Achieving Hydrogen Storage Goals through High-Strength Fiber Glass

2016 U.S. DOE HYDROGEN and FUEL CELLS PROGRAM and VEHICLE TECHNOLOGIES OFFICE ANNUAL MERIT REVIEW and PEER EVALUATION MEETING

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Project ID: ST115

Project Overview

Total Funding: $1,400,006

- Federal: $800,000
- FFRDC: $400,000
- Cost Share: $200,006
- BP1: 781,425
- BP2: 618,581

Cost to Date

- All BP1 funds have been consumed

Timeline

- Start: 09/01/2014
- BP1 End: 08/81/2016 *
- BP2 End: 08/01/2017

* One-year no-cost extension for BP1

Barriers

- Reduced pressure vessel cost while maintaining volumetric capacity

Partners

- Hexagon Lincoln
- Pacific Northwest National Lab

Relevance

Reduce composite contribution to system cost by nearly 50%

- Target a fiber cost <$6/lb.
- Composite contribution to system cost of <$6/kWh

Minimal impact on tank weight and capacity compared to tanks made with T-700 carbon fiber

- Volumetric capacity of 0.86 kWh/L (26 g/L)
- Gravimetric capacity of 1.3 kWh/kg (4wt%)
- Minimizing increases in tank mass compared to T-700 carbon fiber vessels

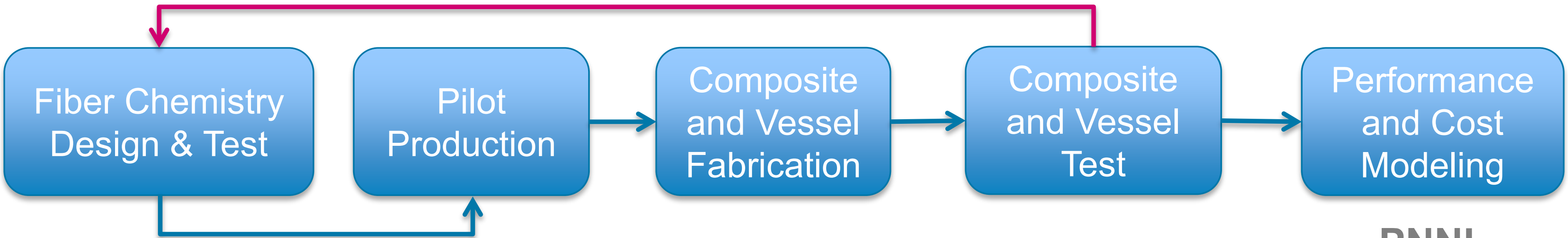
Approach

Demonstrate a Type IV composite overwrapped pressure vessel reinforced exclusively with glass fiber

- New glass fiber with strength exceeding T-700 at less than half its cost
- Novel glass fiber manufacturing process
- Study of the stress rupture behavior of composites made from the new fiber

- **Budget Period 1**
  - Produce fiber glass, prototype new manufacturing process
  - Build and test tanks
  - Model performance and cost improvements using tank data
- **Budget Period 2**
  - Improve fiber glass
  - Build large tanks and test at higher pressure
  - Investigate safety factor reduction

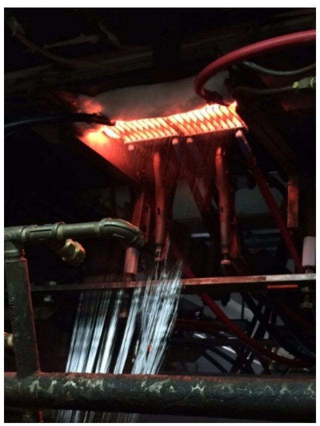
Approach



PNNL

Glass selection (A, B, plus reference E-glass)
Binder selection (I, II)

Produce Pilot Batches of New Fibers


Batch melting | Fiber forming | Package drying | Assembling | Vessel winding

PPG (1,200 lb. of packages produced)

Hexagon Lincoln

Schedule

Task	Partner	Start	Baseline Finish	Planned/ Actual Finish	Comments
1: Novel Fiber Development	PPG	9/1/2014	7/3/2015	4/15/2016	Completed
2: Tank Modeling and Validation	HL	10/1/2014	7/3/2015	6/13/2016	Finishing
3: Stress Rupture Characterization, Target Modeling	PNNL	9/1/2014	7/31/2015	6/10/2016	On Hold, Will Resume
4: Closing Fiber Performance Gaps	PPG	9/1/2015	2/15/2016	11/28/2016	Delayed
5: Tank Modeling and Validation	HL	9/1/2015	9/6/2016	5/15/2017	Delayed
6: Stress Rupture Determination, Cost/perf. Modeling	PNNL	9/1/2015	8/26/2016	6/12/2017	Delayed

Accomplishments: Fiber Glass Development

Produced packages of three high strength fibers and a reference E-glass fiber for tank builds

Completed evaluations of three sets of tanks

- High strength fiber tanks outperformed the reference fiber tanks on burst pressure and cyclic pressure
- One high strength fiber with sizing A significantly outperformed on stress rupture

Glass Type	Reference E (CR)	A (HP2)	B (HPX)
Binder Type I	2026	2026	2026
Binder Type II	N/A	HSPK1.1	HSPK1.1
Fiber Packages Made (lb)	400	400 x 2	400
Packages Delivered (lb)	> 14 x 20 lb/ea	14 x 2 x 20 lb/ea	14 x 20 lb/ea
Vessels Produced (unit)	9 + 3	9 x 2	9

Accomplishments: Fiber Glass Development

Properties of High Strength Glass Fiber Strand

Composition Type	A	A	B	B	E (Reference)	T-700
Sizing Type	I	II	I	II	I	N/A
Tensile Strength (MPa)	3192 $\pm$ 79	3289 $\pm$ 96	3372 $\pm$ 45		2848 $\pm$ 138	4900
Tensile Modulus (GPa)	88.1 $\pm$ 1.1	89.8 $\pm$ 0.7	87.7 $\pm$ 0.7		82.8 $\pm$ 1.1	230
Elongation at Break (%)	5.5	5.6	5.8		5.5	2.1
Density (g/cm^3)	2.58	2.58	2.58	2.58	2.64	1.8

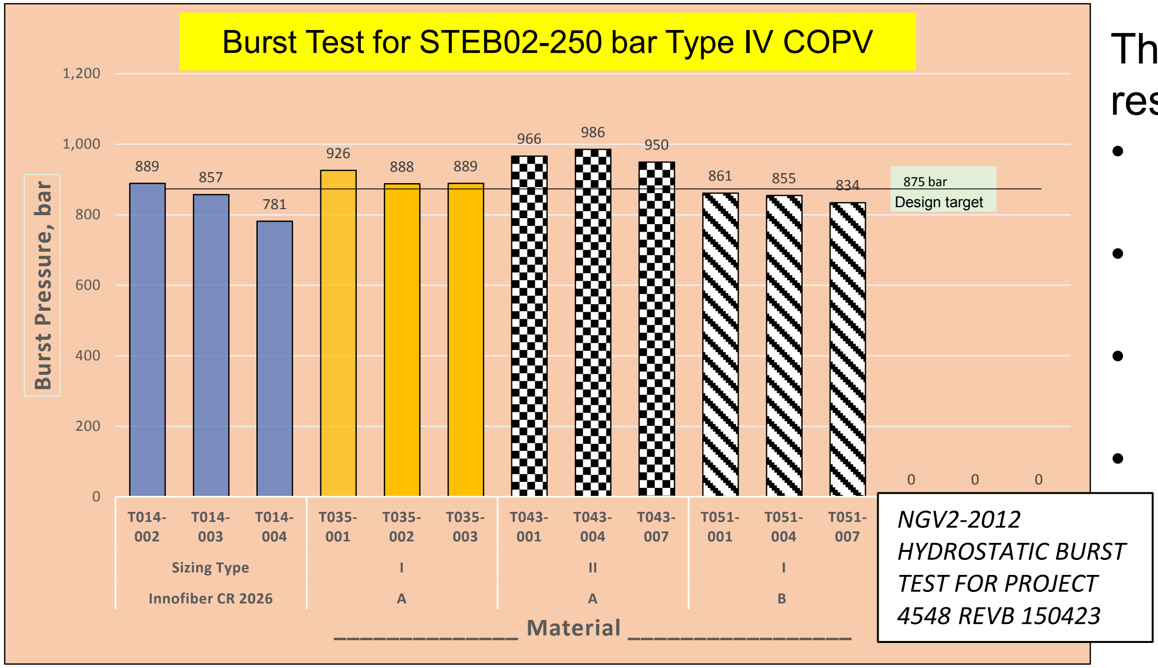
Notes: 1) High strength fiber compositions protected by US 9,278,883 B2, PPG Industries Ohio, Inc. (March 8, 2016)
2) Forming temperature for A glass cullet is lower than B glass cullet

- In small-scale pilot production, we measured a 40% translation loss of strand tensile for high strength fibers against their pristine ***single fiber*** strength (A: 5357 MPa, B: 5583 MPa)
- Standard commercial fiber glass production typically has a fiber strand tensile translation loss within 15% of pristine value
- Options for reducing transition loss will be investigated during BP2, including alternate small-scale pilot production parameters, modest PPG pilot system investments, and a possible production furnace campaign

Accomplishments: Fiber Evaluation

Hexagon Lincoln

- Tanks made with high strength fiber one (A-I, A-II) passed burst testing; tanks made with high strength fiber two (B-I) did not
  - The ability to successfully produce higher temperature B glass is under review
- Sizing affects tank performance under 80% burst pressure, suggesting the importance of optimizing the sizing
- 250 bar tests were used in BP1 to reduce cost



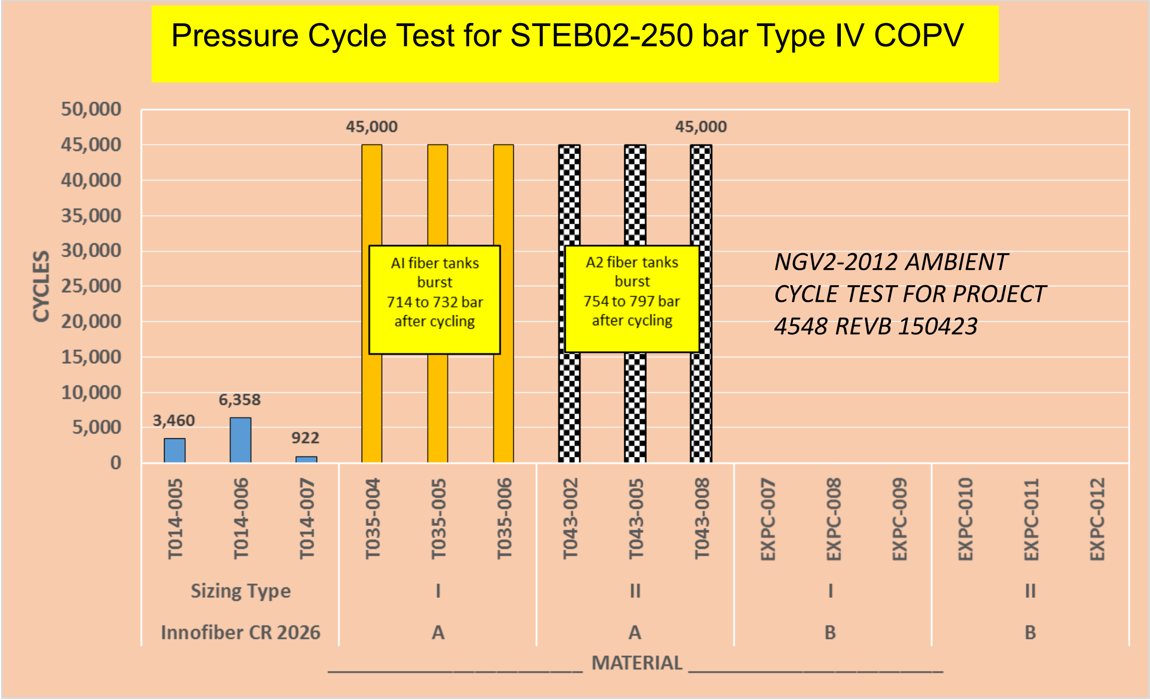
The graph shows the burst test results for the BP1 fiber glass

- Blue bars are the control glass with commercial sizing (binder)
- Yellow bars are the high strength glass one with commercial sizing
- Checkered bars are high strength glass, with a new sizing
- Diagonal lines are high strength glass two with commercial sizing

Accomplishments: Fiber Evaluation

Hexagon Lincoln

- High strength fiber one tanks (A-I, A-II) passed pressure cycle test, and substantially outperformed reference E-glass tanks; high strength fiber two testing underway
- Time to failure curve may be shifted for high strength fiber tanks compared reference E-glass tanks



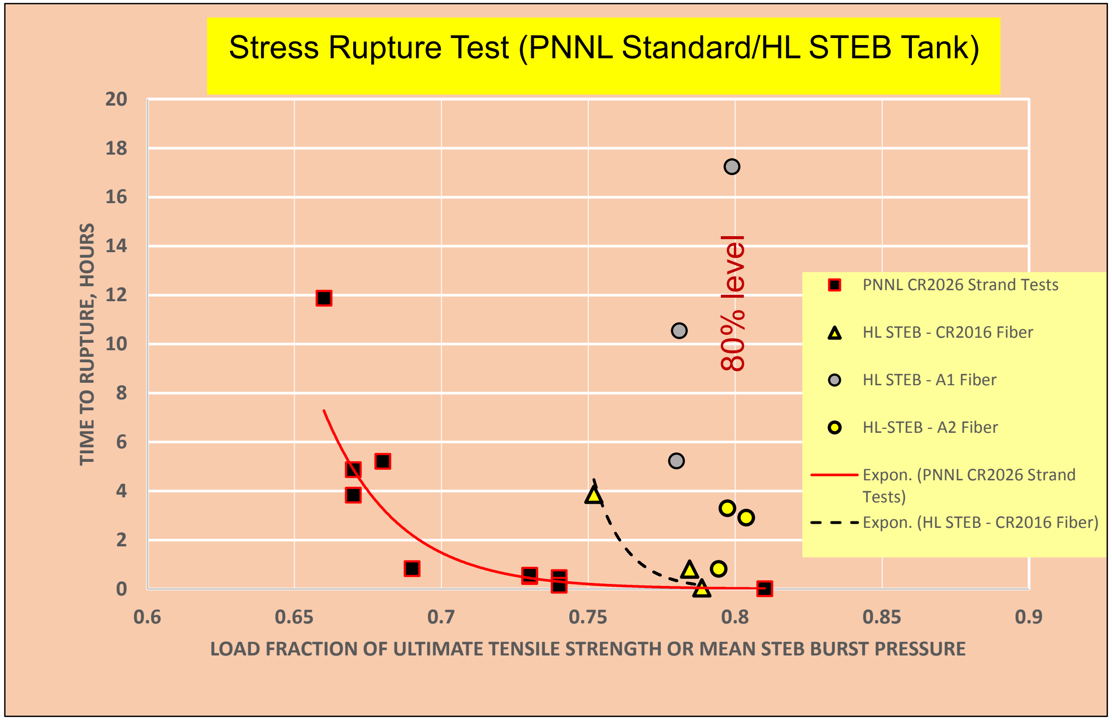
The graph shows the burst test results for the BP1 fiber glass

- Blue bars are the control glass with commercial sizing (binder)
- Yellow bars are the high strength glass one with commercial sizing
- Checkered bars are high strength glass, with a new sizing

Accomplishments: Fiber Evaluation

Hexagon Lincoln
PNNL

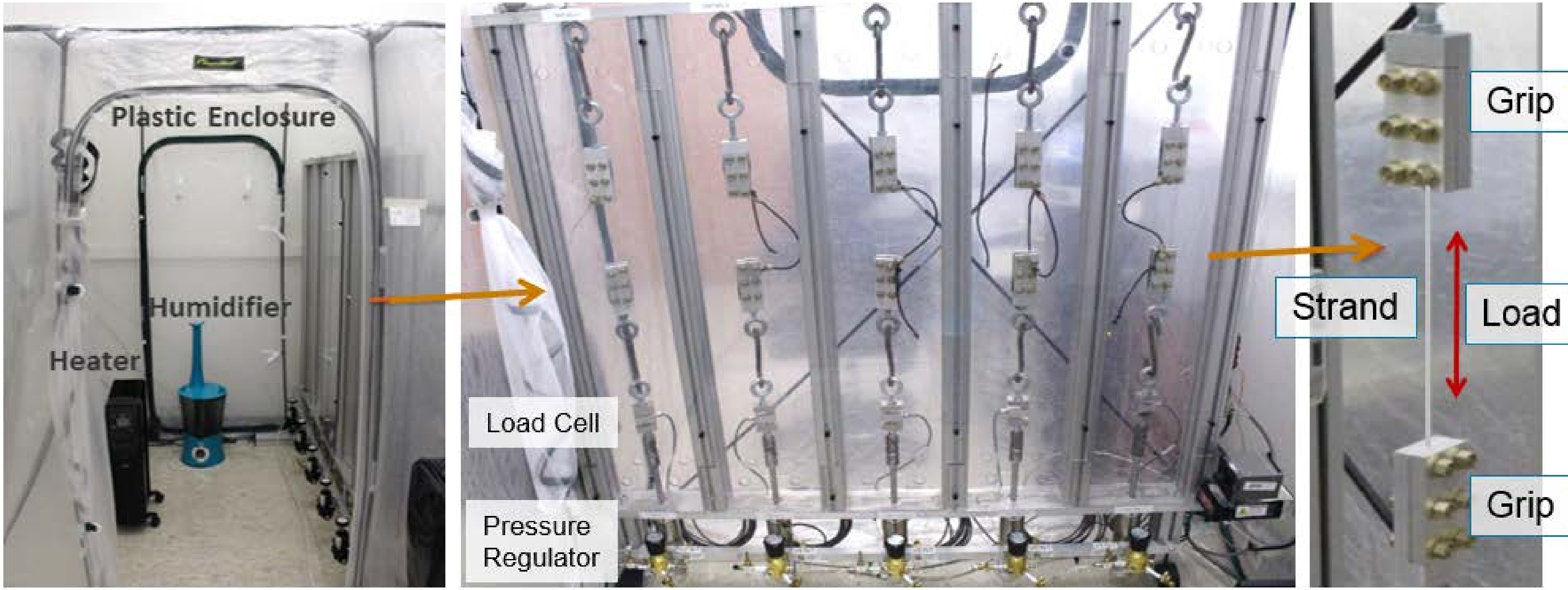
- Time to failure curve may be shifted for high strength fiber tanks compared to reference E-glass tank
  - Uniaxial tests are very different loading conditions compared to the filament wound pressure vessels
    - Initial failure of some highly loaded fibers (or fibers more prone to stress rupture) can shift load to other lower stressed fibers
    - Failure load in a laminated composite often occurs after a certain amount of fiber breakage when the total composite is more equally loaded



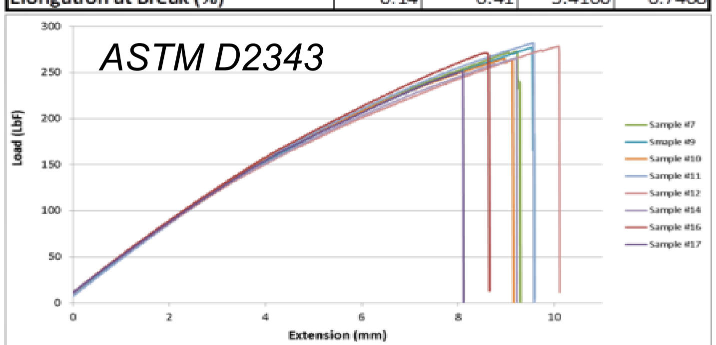
The graph shows the stress rupture test results for the BP1 fiber glass

- Squares are the PNNL control glass
- Triangles are the PPG control glass
- Blue circles are the first high strength fiber glass
- Yellow circles are the same high strength fiber glass, with a new sizing

Accomplishments: Stress Rupture Performance PNNL



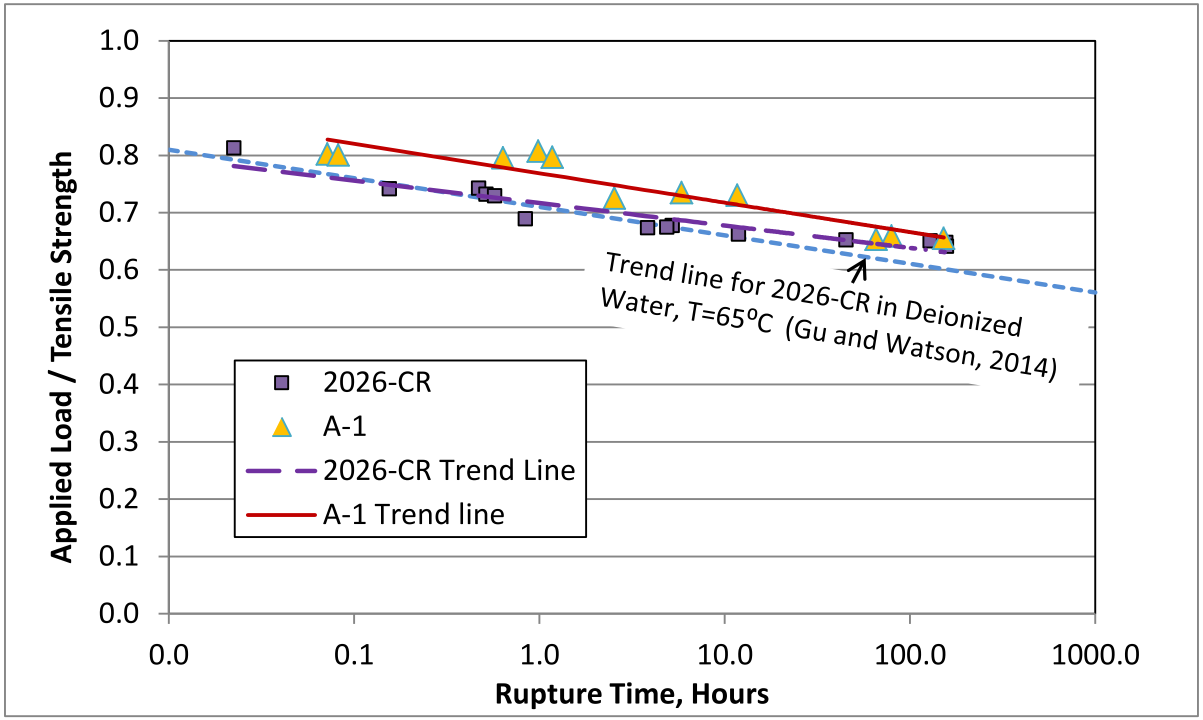
BareFiber PNNL Custom Grip	Average	Stdev	Min	Max
Peak Load (LbF)	270.64	9.87	251.0476	282.2207
Extension (mm)	8.30	2.80	8.1159	10.1112
Elongation at Break (%)	6.14	0.41	5.4106	6.7408



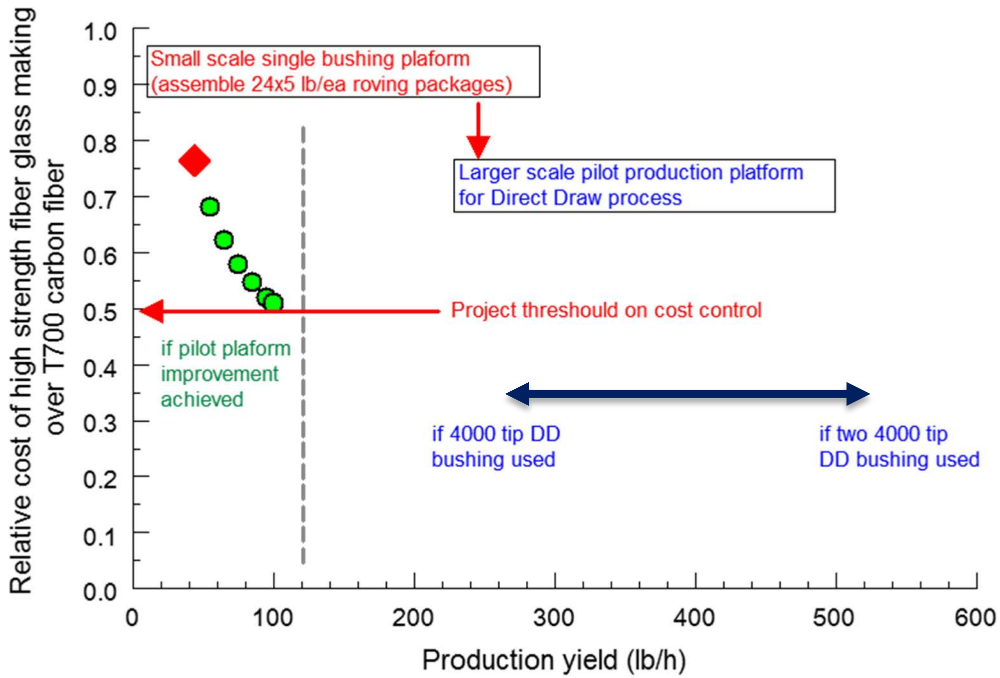
- ASTM D2343 method
- 12 tests of Reference E-glass strands (1100 TEX)
- 8 of 12 ruptured in the gauge
- Average tensile load of 270.6 lb.
- PPG tested 272.0 lb.

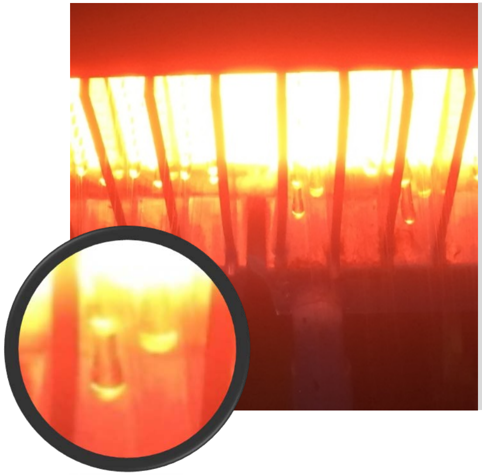
Accomplishments: Stress Rupture Performance

- Reliable test platform has been established to evaluate stress rupture characteristics of high strength fibers
- The slope of the line represents sensitivity of the fiber to moisture attack under applied tensile stress
  - This is the basis from which changing Safety Factor in the future can be determined

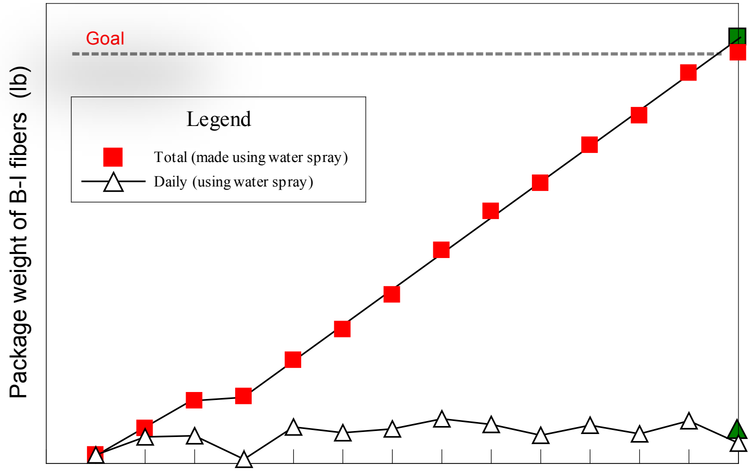


Accomplishments: Cost Modeling and Analysis





Beading from the insulating refractory gasket material is an issue that leads to low efficiency and higher cost.



Daily production rate on small-scale melter

- Beading comes from thermal breakdown of the gasket material due to the stress associated with non-continuous production. Hollow fibers come from trapped air when re-melting cullet to produce fibers. Nether are an issue for fibers made in the large-scale manufacturing platform.
- We believe the high strength glass fiber can be made using a large-scale, traditional manufacturing platform, which will enable PPG to achieve our cost object and DOE cost targets.

Reviewer Comments (summary)

Programmatic

- Schedule delays: Project was granted a one-year, no-cost extension
- PNNL acting as a test lab: PNNL is performing in a modeling and analysis role, that requires test data to support the modeling
- Against cost and design performance modeling: Safety factor reduction is an important part of enabling this technology to be commercialized

Technical

- Consider vol./grav. inefficiencies of glass vs. carbon: Our project used calculations for these parameters in the proposal and will produce actual values during the BP2 of the project
- Lack of hybrid design: This is not within project scope
- T700 tank should be baseline: Comparisons can be made using HL data from commercial products

Collaborations

Partner	Project-Related Information
PPG	PPG has been making glass since 1883 and our Fiber Glass Research Center has been in operation since 1959. We are the second largest domestic supplier of fiber glass into the reinforcement market. New glass compositions will be developed at our Glass Business and Discovery Center near Pittsburgh, PA while sizing and process development takes place at our Fiber Glass Research Center in Shelby, NC.
Hexagon Lincoln	Hexagon Lincoln is the largest manufacturer of Type 4 natural gas and hydrogen vehicle fuel containers in the world with a 50-year history of designing and manufacturing high performance, filament-wound composite structures.
PNNL	PNNL has supported the ongoing DOE hydrogen energy mission since the 1990s. PNNL is a partner in the DOE Hydrogen Storage Engineering Center of Excellence, has created strong partnerships with the domestic automakers through the United States Council for Automotive Research (USCAR), and routinely partners with representatives from academia and industry to develop and transfer new technologies to emerging markets in hydrogen.

Remaining Challenges and Barriers

Short-Term

- 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
- Small bushing
  - Down stream processes
    - Integrity of package after drying
    - Degree of contact damage in roving assembly process

Long-Term

- Maximum temperature capability of large-scale glass production furnace

Proposed Future Work

Project Work

- Complete BP1 Tank Evaluations
- Complete Performance and Cost Modeling and Analysis
- BP1 Go/No-Go Process

Other Work

- Market Development for new low-cost, high strength fiber glass
  - Automotive
  - Aerospace
  - Military
- Investments in advance, large scale, high-temperature fiber glass production technology

Technology Transfer Activities

No hydrogen storage technology transfer activities to date

High strength fiber glass technical evaluations underway

Summary

Objective

- Demonstrate a Type IV composite overwrapped pressure vessel (COPV) reinforced exclusively with glass fiber to reduce 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

- Target a fiber cost <$6/lb.
- Composite contribution to system cost of <$6/kWh
- Volumetric capacity of 0.86 kWh/L (26 g/L)
- Gravimetric capacity of 1.3 kWh/kg (4wt%)

Approach

- New glass fiber with strength exceeding T-700 at less than half its cost
- Novel glass fiber manufacturing process
- Study of the stress rupture behavior of composites made from the new fiber

Accomplishments

- 3 glass/sizing compositions made
- 3 set of tanks made and testing
- Groundwork completed for safety factor evaluation

BACK UP

2015 AMR Review Comments

Project Strength

- High impact if successful
- Relevance and value of a high-strength, lower cost glass fiber for H2 tank fabrication
- Capabilities of technology vertically integrated teams
- Project organization
- Commercial scale up capability in future

Project Weakness

- Schedule slip – fiber production, testing, tank modeling and fabrication, lack of composite data in BP 1
- Fiber rupture testing methods development – just use well established standard methods, National lab should already have this capability, PNNL acting as a testing lab
- Consider volumetric and gravimetric inefficiencies of glass vs. carbon, lack of hybrid design work
- Baseline tank should be T700 Carbon Fiber
- Against cost and design performance modeling

2015 AMR Review Comments

“So far, PPG reported that two fiber compositions have been produced with properties similar to baseline carbon fibers. …..the project tasks appear to have slipped by 3 – 6 months according the dates on Slide #25. Substantial recovery seems unlikely and significant de-scope of objectives is more likely.”

“With the little over a year left for this project, I strongly recommend against any further cost or design modeling. Instead, more effort should be made to develop and test glass fibers and resins with greater potential for high strength along with fabrication and testing of prototype tanks.”

“Drop the low viscosity resin idea and focus on the hybrid fiber design.”

Response

- Equipment issues prevented us from producing fibers according to the original schedule, and we are still struggling with the revised schedule. We worked with DOE, to secure a no-cost project extension to complete our work without removing any objectives. Removal of low viscosity resin would be a DOE decision if the project is approved for BP2

2015 AMR Review Comments

“The project is a good approach to looking at the possibility of developing a high strength glass….The low density resin and hybrid composite portions of the work seem to have been ignored ”

Response

- In BP-2, we will evaluate the potential of low density resin in combination with high strength fibers. The current scope calls for evaluation at a coupon level by PNNL. The current tank build approach does not support low density resin
- The hybrid composite approach, combining T700 carbon fiber with high strength glass fiber, was been discussed among team members during the project proposal. It was not included in the proposal due to cost and schedule limitations.

2015 AMR Review Comments

"The presentation suggests that chemistry has been developed with proper temperatures and binders to produce fibers that exceed T700 strengths. However, the box plots of the data show something completely different with the new glass fiber chemistries showing S-glass type properties. The actual accomplishment is hard to rate because of this discrepancy. The value of the efforts expended on tabbing methods and test strand process is not clearly shown."

"I would favor using a reference tank based on T700 carbon fiber rather than an E-glass control……"

Response

- At the 2015 AMR meeting, we had not produced high strength fibers on this project. We used composite data from other PPG tests to show that the high strength fibers are better than reference E-glass comparable to commercial S-glass fibers.
- E-glass tanks provide the required reference to determine whether the safety factor reduction is technically possible for high strength fibers. There is also significant data already available for T-700 tanks from outside the project.
- We have added references of our test methods for 2016 AMR meeting.

2015 AMR Review Comments

"The companies involved are excellent with very good expertise and reputation…….However, with each participant's efforts so well defined, it is not obvious how much sharing of information or collaboration is actually taking place."

Response

- The team has held regular project meetings discuss progress and issues related to the schedule. We have kept DOE closely informed of the progress and schedule delays.
- PPG and PNNL worked together on grip options, and performed round robin tests using the same samples to build confidence. PNNL visited PPG's testing lab. With the improved grip design, PNNL performed final stress rupture tests using the reference E-glass strands, and the results closely match with PPG results.
- PPG has visited Hexagon Lincoln twice to understand the filament winding and tank production processes.
- DOE and Hexagon Lincoln visited the PPG Shelby plant to see the standard fiber glass commercial process and the project's induction batch melting process.

2015 AMR Review Comments

"This project almost exclusively looks at reducing the cost of Type-IV hydrogen storage vessel through substitution of fiberglass for T700 carbon fibers. However, slide #27 shows circa a 40% mass and ~10% volume penalties for about only a 30% cost benefit through use of just fiberglass alone….However, still unresolved are possible degradation during pressure-temperature cycling of such tanks on vehicles."

"The program has the potential to significantly decrease composite costs. The impact on weight an capacity in switching to fiberglass should quantified."

Response

- The project is structured to provide sufficient data to establish if the actual weight and mass penalty from fiber glass tanks are justified for the cost savings achieved from actual fiber glass production. The project proposal used projects values for mass, volume, and cost. The project will establish the actual values.

2015 AMR Review Comments

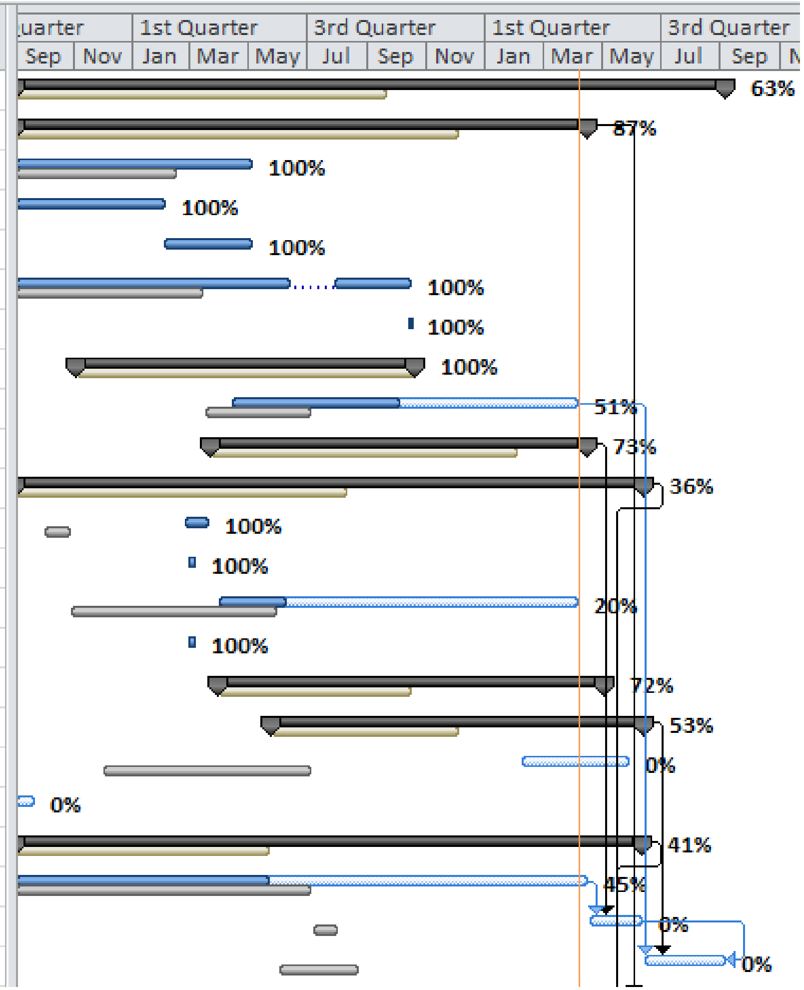
"This effort is very relevant to the overall goals of the hydrogen storage program. The optimization, including hydride development, of the fiber reinforcement is key to minimizing cost and creating value. However, this effort is not new has others have attempted to create high strength fiberglass in the past. The novel chemistry is not clear at this early stage of the project. …. Again, the unique technology is not clear has this well be different than that already in place."

Response

- Existing commercially available high strength glass fibers (S-glass or S2-glass) are single-source, limited production products. The overall production cost for these commercial fibers is very high, and limits use.
- The project's high strength fibers developed can be produced at temperature lower than the existing S-glass fibers. Large commercial-scale furnace operation becomes possible for making high strength fibers. The production scale can lower costs significantly. In addition, the new high strength fibers should have higher tensile strength than existing commercial S-glass fibers.

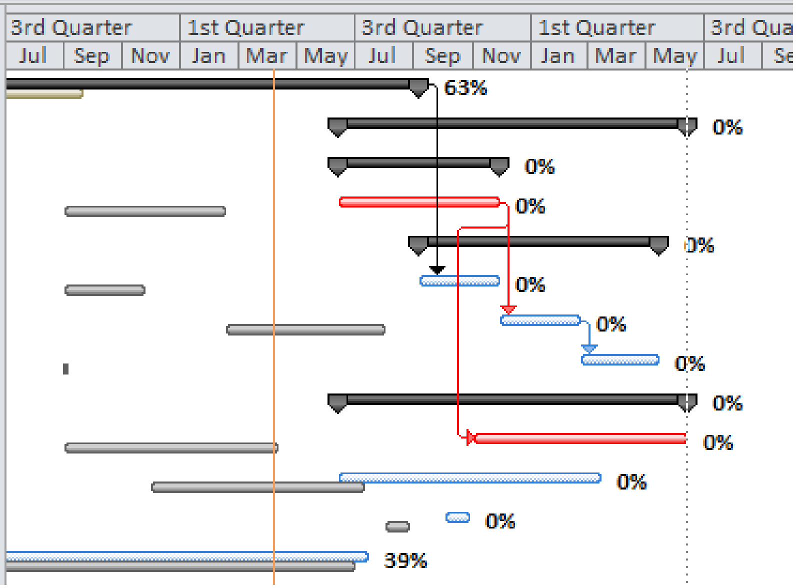
BP1 – Gantt Chart

WBS	Task Name	Duration	Start	Finish
1	**Phase 1**	**517 days?**	**9/1/14**	**9/5/16**
1.1	**Task 1**	**416 days?**	**9/1/14**	**4/15/16**
1.1.1	Task 1.1	8.8 mons	9/1/14	5/4/15
1.1.2	HP2 #1	111 days	9/1/14	2/2/15
1.1.3	HP2 #2	66 days	2/2/15	5/4/15
1.1.4	Task 1.2	13.25 mons	9/1/14	10/16/15
1.1.5	DOE Visit	1 day	10/15/15	10/15/15
1.1.6	**Task 1.2 Activities**	**251 days**	**11/3/14**	**10/19/15**
1.1.7	Task 1.3	248 days	4/13/15	4/5/16
1.1.8	**Task 1.3 Activities**	**271 days?**	**3/23/15**	**4/15/16**
1.2	**Task 2**	**457 days**	**9/1/14**	**6/13/16**
1.2.1	Task 2.1	1 mon	2/23/15	3/20/15
1.2.2	HL Task Design	7 days	2/26/15	3/6/15
1.2.3	Task 2.2	12.9 mons	3/30/15	4/5/16
1.2.4	HL Tank Build and Test Plan	7 days	2/26/15	3/6/15
1.2.5	**HL Tank Builds (E, HP2)**	**277 days**	**3/30/15**	**5/2/16**
1.2.6	**HL Tank Testing**	**267 days**	**5/25/15**	**6/13/16**
1.2.7	Task 2.3	4 mons	2/8/16	5/27/16
1.2.8	HL 700B Tank Design	15 days	9/1/14	9/19/14
1.3	**Task 3**	**456 days**	**9/1/14**	**6/10/16**
1.3.1	Task 3.1	20.8 mons	9/1/14	4/15/16
1.3.2	Task 3.2	2 mons	4/18/16	6/10/16
1.4	Phase 1 Go/No-Go	3 mons	6/14/16	9/5/16



BP2 – Gantt Chart

WBS	Task Name	Duration	Start	Finish
1	**Phase 1**	**517 days?**	**9/1/14**	**9/5/16**
2	**Phase 2**	**260 days**	**6/14/16**	**6/12/17**
2.4	**Task 4**	**120 days**	**6/14/16**	**11/28/16**
2.4.1	Task 4.1	6 mons	6/14/16	11/28/16
2.5	**Task 5**	**180 days**	**9/6/16**	**5/15/17**
2.5.1	Task 5.1	3 mons	9/6/16	11/28/16
2.5.2	Task 5.2	3 mons	11/29/16	2/20/17
2.5.3	Task 5.3	3 mons	2/21/17	5/15/17
2.6	**Task 6**	**260 days**	**6/14/16**	**6/12/17**
2.6.1	Task 6.1	8 mons	11/1/16	6/12/17
2.6.2	Task 6.2	9.8 mons	6/14/16	3/14/17
2.6.3	Task 6.3	1 mon	10/3/16	10/28/16
3	Task 7	24 mons	9/1/14	7/14/16



Goal and Project Metrics

Metrics	T-700 Carbon Fiber	E-Glass	High-Strength Glass Design 1	High-Strength Glass Design 2	2017 DOE Targets
Fiber Cost ($/lb)	13	1.3	Target < 6.0		6.0
Composite Contribution to System Cost ($/kWh)	11	10.7	8.0	5.6	6.5
Gravimetric Capacity (kWh/kg)	1.5	0.33	1.1	1.3	1.8
Volumetric Capacity (kWh/L)	0.87	0.45	0.81	0.86	1.3
Vessel Mass (kg)	120	560	172	141	
Fiber Strand Tensile (MPa)	4900	3000	≥ 4900	≥ 4900	
Resin Density (g/cm^3)	1.2	1.2	1.2	1.0	
Safety Factor	2.25	3.5	3.5	3.0	

Overall Goal: new high strength glass fiber will reduce the *composite contribution* to system cost by nearly 50% (projected for commercial processes) compared to tanks made with T-700S carbon fiber

We protect and beautify the world™