# IV.D.6 Achieving Hydrogen Storage Goals through High-Strength Fiber Glass

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Subcontractors:

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- Pacific Northwest National Laboratory, Richland, WA

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Project End Date: August 30, 2017

## Overall Objectives

The objective is to demonstrate a Type IV composite overwrapped pressure vessel (COPV) reinforced exclusively with glass fiber. This will be achieved through the following steps:

- Develop a new glass fiber with strength exceeding Toray T-700 carbon fiber at less than half its cost.
- Demonstrate a novel glass fiber manufacturing process.
- Conduct composite validation laboratory tests to determine the safety factor for the tank made by using new high-strength glass fiber.
- Build cost models to demonstrate the new tank will 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.

## Fiscal Year (FY) 2016 Objectives

- Produce multi-end roving packages of two candidate high-strength glass fibers that offer tensile strength of fiber strands close to 5,000 MPa.
- Build high-strength fiber-reinforced vessels for mechanical evaluations and compare with performance of vessels made from T-700 carbon fibers. (See Table 3b for comparison of mass, burst pressure, and fiber translation efficiency of tanks wound on this project. See Table 5 for modeled cost, gravimetric, and volumetric performance of the DOE 5.6 kg hydrogen tank.)
- Perform stress rupture tests for high-strength fiber strands to provide a basis for determining any changes from the fiber glass safety factor (3.5) currently used for hydrogen tank design to 3.0.
- Demonstrate a new, high-throughput, high-temperature batch melting unit to produce high-strength fiber glass cullet from batch by 4X comparing with the existing melting unit.
- Project the commercial production cost of making high-strength fibers based on the current small-scale fiber-making platform.
- Perform preliminary tank cost calculations and performance projections and compare against the 2020 DOE cost, volumetric, and gravimetric targets (see Table 5).

## Technical Barriers

This project addresses the following technical barriers from the Hydrogen Storage section of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan.

(B) System Cost

## Technical Targets

The project is to demonstrate the technical and commercial feasibility of using high-strength glass fibers to match the tensile strength of Toray T-700 carbon fibers, at about 50% of the cost. At the completion of the project, experimental results and modeling output will enable the team to benchmark with the key parameters shown in Tables 1 and 2. The actual targets for the project are detailed in the Introduction section of this report.

## FY 2016 Accomplishments

During the first phase of the project under FY 2016, the team has successfully completed the following objectives:

- Completed high-strength fiber multi-end roving packages to cover glass fiber chemistry A with two binders and fiber chemistry B with one binder, plus reference E-glass packages with one binder.

**TABLE 1.** Technical System Targets: Onboard Hydrogen Storage for Light-Duty Fuel Cell Vehicles [1]

| Storage Parameter | Units | 2020 | Ultimate | Project Towards Targets (2015) |
|---|---|---|---|---|
| System Gravimetric Capacity | kWh/kg | 1.8 | 2.5 | 0.31 Well Below Target |
| System Volumetric Capacity | kWh/L | 1.3 | 2.3 | 0.43 Well Below Target |
| Storage System Tank Cost | \$/kWh net<br>\$/kg $H_2$ stored | 10<br>333 | 8<br>266 | 34.1 Well Above Target<br>1,136 Well Above Target |

**TABLE 2.** Projected Performance of Hydrogen Storage Systems [1] [a]

| Hydrogen Storage System (Including Balance of Tank Cost) | Gravimetric (kWh/kg sys) | Volumetric (kWh/L sys) | Cost (\$/kWh; Projected to 500,000 units/yr) | Project Towards Targets (2016) |
|---|---|---|---|---|
| 700-bar Compressed Type IV[b] (Estimated Project Performance) | 1.4<br>(0.31) | 0.81<br>(0.43) | 14.8<br>(34.1 + 3.64 = 37.74) | Gravimetric and Volumetric Below Targets. Cost well Above Target. |

[a] Assumes a storage capacity of 5.6 kg of usable $H_2$.
[b] DOE Hydrogen and Fuel Cells Program Record # 15013, “Onboard Type IV Compressed Hydrogen Storage System–Cost and Performance Status 2015.” September 30, 2015. This includes a balance of tank cost of \$3.64/kWh.

- Successfully demonstrated 4X high-throughput, high-temperature melting unit run using high-strength fiber glass batch, making high-strength fiber glass cullet.
- Built 38 all glass fiber COPVs per the STEB02-250 design using reference E-glass and two types of high-strength fibers and confirmed no technical issues for using the existing commercial tank winding process.
- Completed mechanical evaluations for all COPVs, burst pressure, pressure cycle, and stress rupture at 80% burst pressure per Hexagon Lincoln procedures, NGV2-2012 (Hydrostatic Burst Test for Project 4548 REVB 150423 and Ambient Cycle Test for Project 4548 REVB 150423).
- Completed initial performance and translation assessment of high-strength fiber COPV, 81%, against 91% for Toray T-700 COPV. (cf. Table 3b).
- Completed preliminary stress rupture tests on one of the high-strength fiber strands with two types of sizing to compare with reference E-glass fiber and S-glass fiber as a basis for determining potential to change the currently required safety factor for fiber glass pressure vessels. Based on the results, current safety factor of 3.5 should be used unless better quality of high-strength glass fiber can be realized.
- Completed cost modeling for a high-strength glass fiber COPV based on the current high-strength fiber performance in comparison with a Toray T-700 carbon fiber COPV. In terms of composite cost contribution (\$/kWh) and storage system tank cost (\$/kWh net), the current high-strength glass fiber COPV are still too high by 5.2X and 2.8X, respectively (cf. Table 5). However, this result is solely driven by the lower than expected fiber strength which results in the high mass and cost of fiber required in the tank design.

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## INTRODUCTION

This project addresses the Fuel Cell Technologies Office’s intermediate 2017 goals for onboard hydrogen storage for light-duty fuel cell vehicles. Specifically, the team targets a fiber cost less than \$6/lb, a composite contribution to system cost of less than \$6/kWh, a volumetric capacity of 0.86 kWh/L (26 g/L), and a gravimetric capacity of 1.3 kWh/kg (4 wt%), while minimizing increased tank mass compared to T-700 carbon fiber vessels. The project tasks are organized to continually decrease project risk, moving from a technology readiness level of 4 to 6.

## APPROACH

To begin, in Budget Period 1 (BP1), the team develops fibers at the bench and characterizes stress rupture at the fiber level. The team then develops a pilot version of the new glass manufacturing process to produce the high-strength fibers. BP1 ends with test data from prototype tanks built from up to four new fiber samples, i.e., fiber chemistry and sizing chemistry in combination.

In Budget Period 2 (BP2), the team optimizes the best performing fiber and the production process, characterizes stress rupture at the composite level, and investigates alternate tank designs. The project ends with a prototype tank built according to a design tailored to the properties of the new glass that can be tested against a wide range of industry testing standards.

## RESULTS

The project under BP1 has made a total of 1,200 lb of multi-end roving packages (with nominal 450 yield or yd/lb) of high-strength fibers of A-I, A-II, and B-I types. A Type IV composite overwrapped pressure vessel design based upon reference E-glass fiber was completed. Based on the design, 38 all glass fiber COPVs, using A-I, A-II, and B-I packages, were built for mechanical testing. The two selected fiber sizings were compatible with the commercial epoxy resin used for building Toray T-700 carbon tanks; no processing issues were apparent during fabrication of the all glass fiber COPVs. The all glass fiber design is designated as STEB02-250, which is a 250 bar tank designed to a 3.5 factor of safety (875 bar). In comparison, STEB01-250 is an all carbon fiber (T-700) 250 bar design to 2.25 factor safety (563 bar). Figure 1 provides a schematic description of the processes from fiber drawing to vessel winding.

Mechanical properties and density of the high-strength fiber strands are summarized in Table 3a. Also included are properties of reference E-glass strands and T-700 carbon fiber strands for comparison. Due to various limitations of the current small-scale production platform, including fibers with high counts of hollow fibers, large yardage variations, or large fiber diameter variation, thermal inhomogeneity, etc., the final strands of assembled roving showed about 40% translation losses against the pristine fiber strength values reported in 2015 (cf. Table 3). Fiber products from typical commercial scale production furnaces generally exhibit about 15% translation losses as compared with their counterpart of single filament pristine strength. The observed differences point out that the current small scale and discontinuous fiber drawing platform is inadequate in making high quality fiber strand samples. Table 3b compares tank geometry and performance of vessels made from high-strength glass fiber (A-I) and T700 carbon fibers. Deficiency of high-strength glass fibers (cf. Table 3a) translates to poor performance of the vessels against the commercial vessels made from T700 carbon fibers. High-strength fiber reinforced vessels had average translation of 81% as compared with 91% of T700 carbon fiber reinforced vessels.

The vessels were grouped, typically three each, for mechanical testing to determine their burst pressure, pressure cycle, and stress rupture. The results are shown in Figure 2. Relative to the E-glass reference fibers, tanks made from all of the high strength fiber and sizing combinations exhibited improved performance. The A-I fiber tanks performed the best overall, passing both the burst and pressure cycle tests. They also had the longest time to stress rupture when held


**FIGURE 1.** Process flow of high-strength glass fiber production, multi-end roving package assembling, and tank winding processes

**TABLE 3a.** Mechanical Properties and Density of Glass Fiber Strands Compared with T-700 Carbon Fibers*

| Composition Type | A | A | B | B | E (Reference) | T-700 |
|---|---|---|---|---|---|---|
| Sizing Type | I | II | I | II | I | N/A |
| Tensile Strength (MPa) | 3192 ± 79 | 3289 ± 96 | 3372 ± 45 | | 2848 ± 138 | 4900 |
| Tensile Modulus (GPa) | 88.1 ± 1.1 | 89.8 ± 0.7 | 87.7 ± 0.7 | | 82.8 ± 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 |

* Pristine tensile strength of single fiber: Composition A - 5357±71 MPa; Composition B - 5583±58 MPa; N/A - Not applicable

**TABLE 3b.** Vessel Parameters and Vessel Test Results and Comparison Between High-Strength Glass Fiber and T700 Carbon Fiber Reinforcement

| Parameter and Property | STEB01-250 Bar T700 Carbon | STEB02-250 Bar A-I Glass Fiber | Difference relative to T700 (%) |
|---|---|---|---|
| Tank Length (in) | 27.8 | 27.8 | 0.0% |
| Tank OD (in) | 9.95 | 10.65 | 7.0% |
| Nominal Internal Volume (liter) | 24.2 | 24.2 | 0.0% |
| Tank Weight (lbs) | 17.0 | 40.3 | 137.1% |
| Liner Weight (lbs) | 6.3 | 6.3 | 0.0% |
| Fiber Weight (lbs) | 7.1 | 26.3 | 270.4% |
| Resin Weight (lbs) | 3.6 | 7.7 | 113.9% |
| Safety Factor | 2.25 | 3.50 | 55.6% |
| Burst Pressure (avg) (PSI) | 10323 | 13062 | 26.5% |
| Actual Burst Relative to Service Pressure | 2.85 | 3.60 | 26.5% |
| Avg. Translation | 91% | 81% | -11.0% |
| Stress Rupture at 80% Peak Load (min) | indefinite | 661 | - |
| Total Wind Time (min) | 35 | 75 | 114.3% |

OD - outside diameter

at 80% of the average burst pressure. However, significant variations were found in the stress rupture tests.

Stress rupture tests were also performed using fiber strands or rods of the reference E-glass and high-strength glass A-I, and A-II, which were impregnated with the epoxy resin used for T-700 carbon reinforced tanks. These tests were performed to investigate if there is a technical basis to consider revising the current safety factor of 3.5 for glass fiber reinforced tanks to a lower value for the team's high strength glass formulation. The current value of 3.5 is based on the slope of the applied tensile stress vs. time to failure from long-time fiber strand stress rupture tests. Figure 3 summarizes the stress rupture test data along with the S-glass strand data from the literature (used to establish the current 3.5 value) [2] and the reference E-glass stress rupture data from PPG's previous tests [3]. The slopes from the A-I, and the A-II high-strength strand tests are similar to the S-glass strands and the reference E-glass (2026-CR) fibers. The similar slopes suggest that a similar safety factor of 3.5 is warranted for the A-I and A-II fibers that were currently produced.

A model developed by Pacific Northwest National Laboratory (PNNL) was used to assess the cost, volumetric, and gravimetric performance of a DOE standard-sized compressed hydrogen tank (5.8 kg hydrogen, 700 bar, 147.3 L, inside length/diameter = 3.3 in, T-700 carbon fiber) using the achieved glass fiber strengths. As a benchmark, the PNNL model gives tank composite masses that are within 5% of the 2013 and 2015 DOE tank estimates (DOE Records 13010 and 15013). The model was also used to estimate the mass of the standard test evaluation bottles (STEBs) wound by Hexagon Lincoln using the glass fibers. Using the liner dimensions of the Hexagon Lincoln and the A-I average strand strength (3,192 MPa), Table 4 shows that the model predicts composite mass and outside tank dimensions that are very similar to the A-I fiber, 250 bar STEB.

Table 5 presents model results for the DOE standard size compressed hydrogen tank (5.8 kg stored/5.6 kg usable hydrogen, 700 bar, 147.3 L, inside length/diameter = 3.3 in). Seven different design cases are presented along with the 2020 DOE performance targets. Cases 1 through 4 are the reference cases presented in the original proposal. Cases 1 and 2 are tanks with T-700 carbon fiber and E-glass properties. Cases 3 and 4 were the projected BP1 and BP2 performance targets. Note that these numbers are slightly different from the original proposal, due to small adjustments in the fiber stress equations of the model. Case 5 estimates the mass and cost performance of a tank with the properties of 2026-CR E-glass measured during BP1. Cases 2 and 5 with common E-glass strengths estimate very large composite masses. With tank pressure of 700 bar and strand strengths around 3,000 MPa, the tank wall is so thick that the through-thickness composite compression makes it difficult to limit the inner layer stresses by adding more thickness. This is seen in Case 5 for the 2026-CR E-glass (2,848 MPa average strand strength) with estimated composite mass of 653 kg, compared to the Case 2 E-glass (3,000 MPa average strand strength) with estimated composite mass of 543 kg. Case 6 estimates the tank performance for the A-I glass fibers (3,192 MPa average strand strength) produced in BP1. The volumetric capacity is predicted to be 0.48 kWh/L compared to the BP1 goal of 0.81 kWh/L, gravimetric capacity of 0.38 kWh/kg compared to the BP1 goal of 1.1 kWh/kg, and the composite contribution to system cost is predicted to be $27.9/kWh compared to the BP1 goal of $8/kWh. A projected fiber production cost of $5.2/lb (4X standard E-glass at $1.3/lb) is used in the cost estimate. These trends result entirely from the large composite thickness required to support the pressure load with the lower-than-expected fiber strand strengths produced in BP1. The reasons for the low strengths are identified in previous sections of this report. An approach to increase the fiber strand strengths to meet the

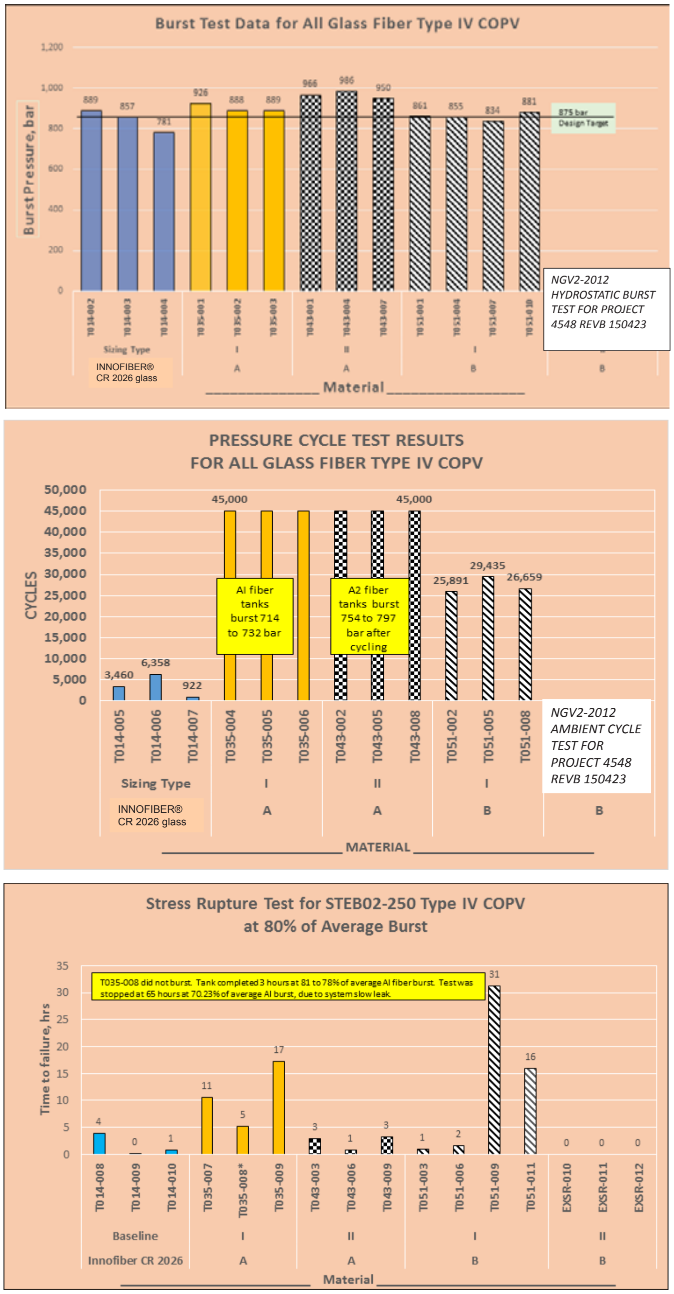


**FIGURE 2.** Tank mechanical evaluations: top – burst pressure, mid – pressure cycle, and bottom – stress rupture

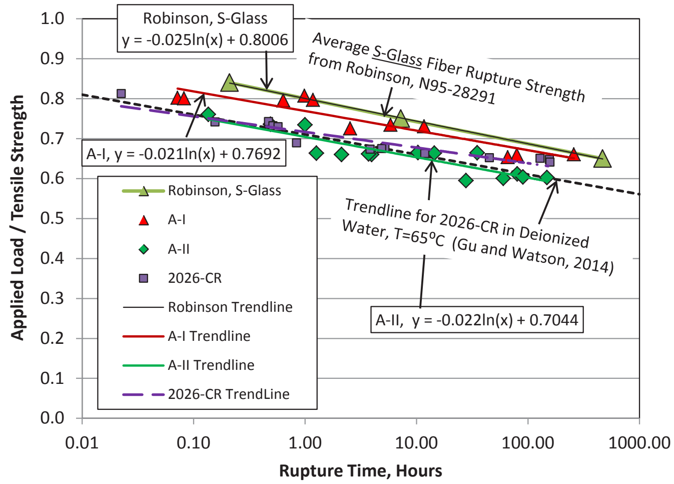


**FIGURE 3.** Stress rupture test data comparing reference E-glass (2026-CR), A-I, and A-II. The literature data for 2026-CR tested in water plus the S-glass stress rupture data reported in literature [2] are included for comparison. Each glass type has similar stress rupture characteristics in terms of the slope of the normalized load vs. time at rupture.

**TABLE 4.** Comparison of As-Wound Tank Mass and Dimensions with PNNL Model Predictions for the A-I Glass Fiber, 250 bar STEB Tank

| Parameter and Property | STEB02-250 Bar A-I Glass Fiber | PNNL Model A-I Glass STEB |
|---|---|---|
| Tank Length (in) | 27.8 With End Bosses | 25.8 Without End Bosses |
| Tank OD (in) | 10.65 | 10.70 |
| Nom. Internal Volume (L) | 24.2 | 24.3 |
| Tank Weight (lb) | 40.3 | 42.4 |
| Liner Weight (lb) | 6.3 With End Bosses | 4.1 Without End Bosses |
| Fiber Weight (lb) | 26.3 | 29.2 |
| Resin Weight (lb) | 7.7 | 9.1 |
| Safety Factor | 3.5 | 3.5 |
| Design Burst Pressure (psi) | 12690 | 12690 |
| Avg. Translation | 81% | 79% |

project goals has been developed and recently discussed with DOE and evaluation of our paths is ongoing

Case 7 estimates the tank performance if an improved A-I glass can be produced with a higher average strand strength of 5,500 MPa. The 10% coefficient of variation results in a design strand strength of 4,950 MPa, 0.76 kWh/L volumetric capacity, 0.88 kWh/kg gravimetric capacity, and a composite contribution to system cost of $11.0/kWh.

Additional cases were simulated with average strand strengths ranging from 3,000 MPa to 7,000 MPa to show the sensitivity of the tank performance trends to strand strength. Figure 4 shows the trends in composite cost, volumetric capacity, and gravimetric capacity. It is estimated that an average strand strength of 6,111 MPa (design strand strength of 5,500 MPa) is required to meet the BP1 goal of 0.81 kWh/L with a gravimetric capacity of 1.0 kWh/kg, and composite cost of $9.6/kWh (based on $5.2/lb fiber cost). At average strand strength of 6,500 MPa (5,850 MPa design strand strength) the estimated volumetric capacity is 0.82 kWh/L with a gravimetric capacity of 1.07 kWh/kg, and a composite cost of $8.8/kWh. At 7,000 MPa (6,300 MPa design strand strength) the estimated volumetric capacity

**TABLE 5.** The estimated performance of glass fiber tanks compared with the BP1 and BP2 goals. Estimated performance of the carbon fiber reference tank is also listed. All calculations are for the DOE standard size pressurized hydrogen tank (5.8 kg stored/5.6 kg usable hydrogen, 700 bar, 147.3 L, inside length/diameter = 3.3 in).

| Case # | 1 | 2 | 3 | 4 | 5 | 6 | 7 | |
|---|---|---|---|---|---|---|---|---|
| | | | BP1 Goal | BP2 Goal | BP1 Actual | BP1 Actual | Conceptual | |
| Summary Metrics | T-700 Carbon Fiber | E-Glass | High-Strength Glass Design-1 | High-Strength Glass Design-2 | 2026-CR E-Glass | Glass A-I | Increased Strength Glass A-I | 2020 DOE Targets |
| Fiber Cost ($/lb) | 13 | 1.3 | 5.2 | 5.2 | 1.3 | 5.2 | 5.2 | 6 |
| Average Fiber Strand Strength, S, MPa | 4,900 | 3,000 | 6,111 | 6,111 | 2,848 | 3,192 | 5,500 | |
| Coefficient of Variation, Cv | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | |
| Design Strand Strength, S*(1-Cv) | 4,410 | 2,700 | 5,500 | 5,500 | 2,563 | 2,873 | 4,950 | |
| Resin Density (g/cm$^3$) | 1.25 | 1.20 | 1.20 | 1.00 | 1.20 | 1.20 | 1.20 | |
| Safety Factor | 2.25 | 3.50 | 3.50 | 3.00 | 3.50 | 3.50 | 3.50 | |
| Storage System Tank Cost ($/kWh net) | 14.2 | 13.0 | 9.7 | 7.8 | 15.6 | 28.1 | 11.3 | 10.0 |
| Composite Cost Contribution ($/kWh) | 12.2 | 12.8 | 9.4 | 7.5 | 15.4 | 27.9 | 11.0 | 6.5 |
| Gravimetric Capacity (kWh/kg) | 1.44 | 0.34 | 1.02 | 1.24 | 0.28 | 0.38 | 0.88 | 1.80 |
| Volumetric Capacity (kWh/L) | 0.85 | 0.45 | 0.79 | 0.85 | 0.40 | 0.48 | 0.76 | 1.30 |
| Tank Mass without $H_2$ (kg) | 123 | 543 | 178 | 145 | 653 | 487 | 205 | |
| Tank Composite Mass (kg) | 103 | 523 | 157 | 124 | 632 | 466 | 184 | |

is 0.84 kWh/L with a gravimetric capacity of 1.16 kWh/kg, and a composite cost of $8.1/kWh. It is important to note that these are only model trends (not actual glass fiber performance) which are useful to project glass composite performance at higher strand strengths.

The trends in Figure 4 suggest that high strength glass fibers must exceed T-700 tensile strength to reach the project goals. Gravimetric capacity is particularly challenging since glass fiber has a higher density than carbon fiber. It is estimated that the best expected performance of the team's current A or B fibers would be 5,500 MPa. In practice, the

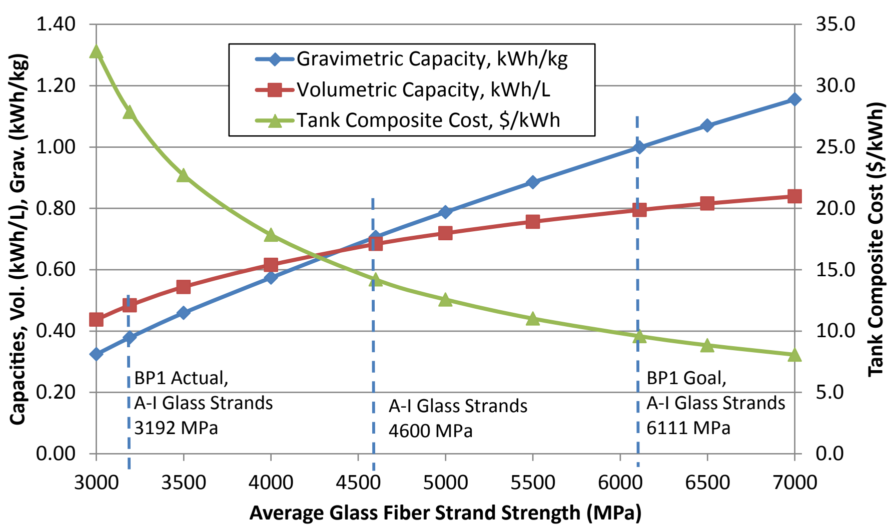


**FIGURE 4.** Sensitivity of cost, volumetric, and gravimetric performance to glass fiber strand strength

best achievable strand tensile strength would then be about 4,600 MPa (based on 15% loss). Therefore, at 4,600 MPa strand strength, Figure 4 would estimate tank performance to be about 0.68 kWh/L volumetric capacity, 0.71 kWh/kg gravimetric capacity, and about $14.2 kWh composite contribution in a 700 bar pressure vessel capable of storing 5.6 L of usable hydrogen at room temperature.

The assessment discussed above was performed using a safety factor of 3.5. BP2 relies on the new fibers exhibiting improved stress rupture characteristics, i.e., the rupture time would need to be less sensitive to the level of applied tensile stress than what was determined for the A-I fibers or S-fibers reported in literature [2]. If achieved, this improved performance could be used to justify the use of a lower safety factor for tank design, making it possible to close the gap reaching the target strand tensile of 5,500 MPa instead of 6,500 MPa by the projection discussed earlier (Figure 4).

## CONCLUSIONS AND FUTURE DIRECTIONS

Progress during BP1 has not achieved the project goal demonstrating high-strength fiber strand with 5,500 MPa tensile strength. High single-fiber strengths were achieved, however 40% translation losses in the strand strength (relative to pristine fiber strength) were caused primarily by processing challenges in the small scale glass-melting and fiber-forming platform, plus the inability to make fiber packages on a continuous basis. The deficiencies can be resolved in BP2 by using a continuous, larger scale fiber production platform that is under consideration. The new platform can enable the team to produce larger, more consistent fiber forming packages for assembling to reduce translation losses as it has been commercially used. In turn, fibers and final assembly roving packages with better quality can translate to greater tank performance improvements.

## FY 2015 PUBLICATIONS/PRESENTATIONS

**1.** H. Li, "Achieving Hydrogen Storage Goals through High-Strength Fiber Glass," at U.S. DRIVE Technical Meetings in Detroit, MI, on May 19, 2016.

**2.** H. Li, "Achieving Hydrogen Storage Goals through High-Strength Fiber Glass," at 2015 U.S. DOE Hydrogen and Fuel Cells Program and Vehicle Technologies Office Annual Merit Review and Peer Evaluation Meeting, Washington D.C., June 9, 2015.

## REFERENCES

**1.** http://energy.gov/eere/fuelcells/downloads/fuel-cell-technologies-office-multi-year-research-development-and-22

**2.** Robinson, E.Y. 1991. "Design Prediction of Long-Term Stress Rupture Service of Composite Pressure Vessels." The Aerospace Corporation. El Segundo, CA.

**3.** Gu, P., Watson J., "Corrosion Resistance of E-Glass Fiber Reinforced Composites: Boron and Interface Factors," CAMX 2014 Conference Proceedings, Orlando, FL, USA, October 13–16, 2014. CAMX–The Composites and Advanced Materials Expo.