IV.D.2 Next Generation Hydrogen Storage Vessels Enabled by Carbon Fiber Infusion with a Low Viscosity, High Toughness Resin System

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Subcontractors:

- Montana State University, Bozeman, MT
- Spencer Composites Corporation, Sacramento, CA

Project Start Date: August 1, 2014 Project End Date: November 30, 2017

Overall Objectives

The project is focused on supporting the key DOE metrics for a 700-bar, Type IV tank by meeting the following objectives:

- Reduce the carbon-fiber (CF) composite volume by 35%.
- Demonstrate cost of composite materials of \$6.5/kWh. This component cost is an important element of the DOE 2020 system cost target of \$10/kWh.
- Demonstrate industry-standard performance (burst strength of 1,575 bar and 45,000 cycle life).

Fiscal Year (FY) 2017 Objectives

- Manufacture large tanks with more efficient usage of CF and an overall reduction in CF.
- Optimize new process technique for larger (>30 L) tanks to demonstrate scalability.
- Conduct key tests related to burst strength and drop testing to confirm performance meets current standards.

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.

- (A) System Weight and Volume
- (B) System Cost
- (D) Durability/Operability
- (G) Materials of Construction

Technical Targets

The project is focused on the technical targets highlighted in Table 1 related to the gravimetric and cost metrics of onboard automotive hydrogen storage systems. Since a significant portion of the cost is directly from the CF composite overwrap, the project aims to reduce the amount of composite necessary to meet the tank specifications. During FY 2016 and 2017, the project has met some key milestones to provide an updated estimates on the progress towards the technical targets in Table 1.

TABLE 1. Progress towards Technical Targets for OnboardAutomotive Hydrogen Storage System

Characteristic	Units	2020 Target	Current Project Estimates
Gravimetric	kWh/kg sys	1.5	1.6 to 1.8 Estimated*
System Cost	\$/kWh at 500,000 units/yr	10	9 to 10.5 Estimated*

*Estimates based on assumptions of 30% and 15% CF reduction

FY 2017 Accomplishments

- Designed and installed processing vessel and associated systems for material transfer and heating.
- Demonstrated improvements in total processing time including preparation, infusion, oven-curing, and clean-up for high-quality 7.5 L prototype vessels.
- Demonstrated ability to prepare 37 L tanks (11-in diameter) and larger tanks.

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INTRODUCTION

DOE Office of Energy Efficiency and Renewable Energy has established aggressive performance targets for Type IV hydrogen storage vessels for Year 2020. Current designs and materials of construction for composite-overwrapped pressure vessels (COPVs) within the industry do not reach the performance targets, as shown by the base-case published by Ahluwalia et al. from Argonne National Laboratory. The specialty chemical producer, Materia, has developed a novel composite resin system, Proxima[®], with ultra-low viscosity (5 cP to 10 cP) that enables vacuum infusion processing for thick CF composite components. The use of this process with Proxima circumvents some challenges inherent with traditional wet filament winding, such as the presence of voids and dry spots. The use of vacuum infusion processing, also known as vacuum-assisted resin transfer molding, for fiberglass composite parts is commonplace in several largescale industries (marine, wind blades), but the feasibility of vacuum-assisted resin transfer molding for compressed gas vessels is not clear. While the concept of infusing dryfilament wound structures has been mentioned in the open literature, the small inter-fiber gaps associated with highperformance CF composites potentially presents significant processing difficulties. Therefore, the commercial application of this approach appears to be limited, which may be related to traditional resins possessing viscosities >200 cP.

In addition to reducing void content, Proxima-based composites also have significantly improved fracture toughness (>3x higher interlaminar fracture toughness) and fatigue performance over currently employed composites for hydrogen storage tanks. The project seeks to leverage this combination of tough resin and new processing to produce CF composite overwrap with better performance, especially in fatigue and damage-tolerance testing. These high performing composites will enable the reduction of the quantity of CF composite overwrap, which alone can account for over 75% of the storage tank system cost. The processingrelated costs for this new approach are expected to be similar to current processing costs with wet winding. By reducing the CF composite content in COPVs by 35%, the project aims to reduce the cost and weight of COPVs and contribute to meeting the DOE 2020 cost target of \$10/kWh.

APPROACH

Since the project requires an expertise in a variety of fields, the project team includes Spencer Composites Corporation and Hypercomp Engineering to provide the specialized filament-winding activities. Montana State University in Bozeman will experimentally characterize composite materials and also use finite element analysis models to anticipate problem areas in tanks designs. Materia is leveraging its experience in infusion process optimization with low-viscosity resin (<10 cP) to demonstrate a series of prototype parts, including tanks and model flat plates of filament wound composites. In order to manage the risks associated with a new resin and a new process for COPVs, the project activities have been divided into stages and the objectives (1) process optimization, (2) COPV design, (3) design optimization, and (4) scale-up of process for vessel testing.

RESULTS

During FY 2017 the team focused on the design and implementation of a simpler, scalable process technique which takes advantage of resin infusion without the use of traditional vacuum bagging film. Also, during this period the emphasis was placed on producing larger tanks for testing. For the new process technique, the team had previously demonstrated the process in proof of concept experiments by making a few high quality dry-wound parts including a 7.5 L vessel, all from dry-wound forms. The new process is similar to a commercially practiced technique called vacuum pressure infusion.

The schematic in Figure 1 outlines the basic elements of the new process equipment which has similarities to a simple autoclave design used for some aerospace composite parts. First, the dry-wound COPV is secured inside a resin confinement vessel which is sized slightly larger than COPV diameter. Next, the resin-confinement vessel is placed into the pressure vessel to make the final assembled pressure vessel with nested COPV/resin confinement vessel. The schematic in Figure 2 outlines the basic steps of the new process. After the resin/curative mixture is prepared, the resin is vacuum transferred into the center vessel for infusion of the COPV. After complete infusion, excess resin is removed and hot air is introduced to cause preliminary curing to achieve ~70% degree of cure. After this is achieved, the tank can be removed easily and placed in the oven for complete curing.

The equipment has been commissioned using a 7.5 L tank and then a 37 L tank. Currently, tanks are being produced for testing of burst strength and drop testing. For the remaining period of the project, the focus will remain on the continued production and testing of larger Type IV tanks (37 L and 133 L) with lower levels of CF in the tanks. In parallel to the process development activities, the use of an alternative CF (Mitsubishi Grafil tow with 800 ksi strength) was studied in 7.5 L tanks but the Grafil fiber appears to require further optimization to avoid damage during processing.

CONCLUSIONS AND UPCOMING ACTIVITIES

From the current results of the project, the team has derived the following conclusions:

 Preparation of small COPV (Type III, 7.5 L) and larger 37 L COPV can achieve complete resin infusion within 30 min.

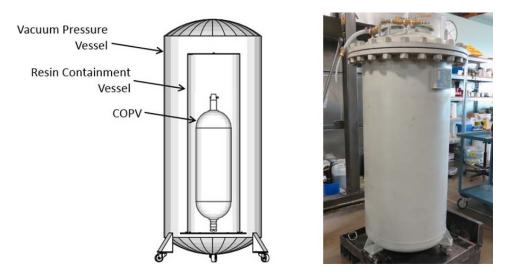
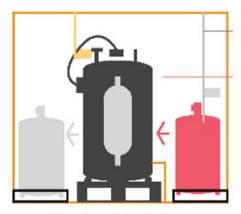
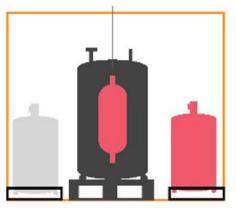


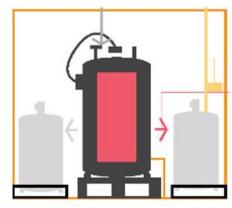
FIGURE 1. Nested arrangement of dry-wound COPV inside a resin containment vessel which is inside a vacuum pressure vessel. Photo of processing pressure vessel positioned under the gantry crane during preliminary test trials.



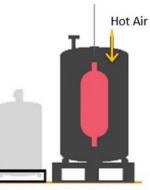
Prep resin/curative Mixture (red)



Remove Excess Resin



Vacuum Transfer Resin to dry-wound COPV



Heat via inline air heater

FIGURE 2. Schematic of the process steps for vacuum pressure infusion processing followed by heat curing using forced hot air.

• Demonstration of new process technique suitable for tank series manufacturing.

The following activities will be the area of focus in the future:

- Produce larger COPVs for testing purposes and as a demonstration of the compatibility for series manufacturing.
- Update current cost model of tanks based on design and processes (Figure 3).
- Generate key performance data including drop-testing larger vessels with lower CF content.

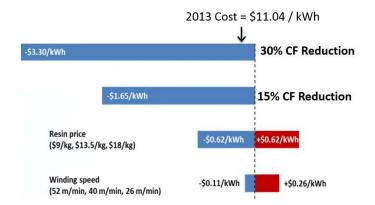


FIGURE 3. Updated sensitivity analysis of COPV cost based on preliminary processing and design estimates. The arrow denotes a 2013 baseline cost of \$11.03/kWh before any CF reductions. (Analysis performed by Strategic Analysis, Inc.).