IV.D.2 Thermomechanical Cycling of Thin Liner High Fiber Fraction Cryogenic Pressure Vessels Rapidly Refueled by LH_2 Pump to 700 bar

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Subcontractors
- Spencer Composites Corporation, Sacramento, CA
- Linde LLC, Hayward, CA

Project Start Date: January 2014
Project End Date: January 2017

Overall Objectives

- Develop ultra-light cryogenic pressure vessels with 12 in diameter and 700 bar rating
- Optimize metallic liner thickness, composite fiber fraction and ultra-thin vacuum jacket
- Quantify liquid hydrogen (LH_2) pump durability over 6,000 refuelings to 700 bar
- Demonstrate full scale system density of 50 g H_2/L_{system} and 9 wt% H_2, and a cycle life of at least 1,500 refills

Fiscal Year (FY) 2015 Objectives

- Submit safety plan for cryogenic H_2 cycling facility rated for 5 kg H_2 prototype vessels and obtain DOE/LLNL approval
- Fabricate 700 bar 163 L system using commercial vessel to test insulation and supports
- Complete civil construction of H_2 test facility
- Design and fabricate initial full scale (63.5 L) prototype 700 bar pressure vessel with a high ratio between inner and outer volume (80+% volumetric efficiency)
- Demonstrate 1,600 bar cryogenic (LN_2) strength of initial prototype (2.28 safety factor)

Technical Barriers

This project addresses the following technical barriers from the Hydrogen Storage section (3.3.5) of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan:

(A) System Weight and Volume

(D) Durability/Operability

(N) Hydrogen Venting

Technical Targets

This project specifically addresses three technical targets for onboard hydrogen storage in light duty fuel cell vehicles: system gravimetric capacity, system volumetric capacity, and operational cycle life. Previous projects on the development of the cryogenic high pressure storage technology achieved peak gravimetric and volumetric capacities of 0.074 kg H_2/kg system and 0.045 kg H_2/L system, respectively, using a 4,000 psi, 151 L (10.7 kg H_2 capacity), 25-in internal diameter, aluminum lined pressure vessel [1]. This current project aims at exploring designs with a 700 bar (10,000 psi) operating pressure, 63.5-L internal volume (5.6 kg H_2 capacity), 12-in internal diameter and a non-Al liner. FY 2015 efforts at designing, fabricating, and cryogenically strength testing pressure vessels produced a 63.5 L, non-Al liner Type III pressure vessel with 81% volume efficiency and 32 kg mass for 700 bar operating pressure, performance that would enable to meet our projected target of 0.090 kg H_2/kg system and 0.050 kg H_2/L system. Those projected performances will be demonstrated with a full size insulated system at the end of this project. Theoretical design calculations have also shown that this pressure vessel would have an operational life of at least 1,500 cycles. This cycle life will be experimentally tested through up to four full size prototype vessels in FY 2016. Table 1 summarizes our projected performance towards the three main technical targets addressed in this project.

FY 2015 Accomplishments

- Sought and obtained LLNL safety approval of 65 bar containment for transient H_2 peak pressure and dynamic vessel wall loading of 2.5–5 kg H_2 over the full pressure and temperature range
- Completed civil site construction and control room installation for cryogenic H_2 cycling facility using 875 bar LH_2 pump

TABLE 1. Progress towards Meeting Technical Targets for Onboard Hydrogen Storage for Light Duty Fuel Cell Vehicles

Storage Parameter	Units	2020	Ultimate	Projected
System Gravimetric Capacity	kg H_2/kg system	0.055	0.075	0.090
System Volumetric Capacity	kg H_2/L system	0.040	0.070	0.050
Durability/Operability Operational Cycle Life	Cycles	1,500	1,500	1,500

- Designed and fabricated a 63.5 L thin lined carbon fiber overwrapped prototype vessel rated to 700 bar with 81% volumetric efficiency
- Demonstrated cryogenic strength of prototype vessel to 1,560 bar

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INTRODUCTION

Storing cryogenic H_2 in a pressurized, insulated system has many benefits in terms of safety, volumetric and gravimetric densities and ownership cost that have been studied and demonstrated by LLNL [2,3] and external parties [4-7]. High utilization (>1,500 kg H_2/d) commercial scale fueling stations will likely require the use of liquid H_2 by means of a fast, energy efficient LH_2 pump. Until now, the development of cryogenic pressure vessels by LLNL has used “off the shelf” pressure vessels with an aluminum liner, a maximum operating pressure limited to 350 bar with large capacity (151 L, equivalent to 10.7 kg H_2) and large diameter (25 in). We believe that system densities (both volumetric and gravimetric), cycle life and manufacturability could be improved by developing pressure vessels specifically tailored towards cryogenic utilization, even at a 5.6 kg H_2 scale, by exploring: thin liner design (especially important for 12 in diameter), non-Al liner materials, high fiber fraction for the composite overwrap, 700 bar operating pressure and ultra-thin vacuum jacket designs.

APPROACH

Within the project, we are designing, manufacturing and cryogenically pressure testing full scale (65 L) 700 bar pressure vessels with a thin (<2 mm), non-Al liner and high fiber fraction. Our primary goal is to assess the *cryogenic* strength of these prototype composite vessels after 1,500 thermomechanical H_2 cycles, while secondary objectives will be accomplished in parallel to (1) measure LH_2 pump performance at 700 bar after 6,000 refuelings (~24 tonnes of LH_2), (2) demonstrate lightweight vacuum jackets for cryogenic H_2 pressure vessels, and (3) design and fabricate an experimental cryogenic H_2 storage system with 5.6 kg H_2 capacity.

In order to achieve the thermomechanical cycling, a LH_2 testing facility will be constructed next to the existing 875 bar LH_2 pump, capable of rapidly cycling full scale (63.5 L) non-certified cryogenic pressure vessels up to 700 bar and performing strength testing of those vessels up to 160 K, 1,300 bar. One to two vessels could be cycled at the same time in this single-manned, remotely operated facility that would also include a vent stack and a 40 kW heat exchanger.

RESULTS

The time period (July 2014 to July 2015) covered by this report focused on five tasks.

Development of Thin-Lined, High Fiber Fraction Vessels

Following the room temperature tensile testing done in FY 2014, ring burst tests, cryogenic tensile test, and pipe welding test completed the small-scale testing for the development of thin-lined high fiber fraction vessels. A design study was then carried out, including liner fatigue and finite element analyses.

The ring burst test procedure was used to evaluate liner buckling after autofrettage, where a liner “ring” (about 1.5 in wide) is overwrapped with a thin carbon fiber ring and then pressurized. This test enables to experimentally determine the minimum thickness the liner can have before it buckles. Thicknesses between 1.3 mm and 1.8 mm were shown to be sufficient for our conditions. Preliminary off-site cryogenic tensile strain testing was then conducted. This mechanical testing was necessary to determine basic variations of the properties of the liner material at cryogenic temperatures, such as Young modulus, elastic strain and tensile (ultimate) strength. It was observed that both the Young modulus and the elastic strain increase with decreasing temperatures. Also, the ultimate strength varied from 80 ksi at room temperature to almost 200 ksi at cryogenic LN_2 temperature. The results from the weld quality testing, tensile tests and ring burst tests showed that the selected material would perform as required.

A design study concerning the construction of the thin lined filament wound composite prototype pressure vessel was then carried out. The pressure vessel was designed based on the American Society of Mechanical Engineers

(ASME) Boiler and Pressure Vessel Code, Section X for Class III vessels with non-load sharing liners for gaseous hydrogen stationary service. A finite element analysis was also performed. The composite design was developed based on netting analysis and a helical-to-hoop fiber stress ratio of 75%. The hoop plies were consolidated into four groups to limit the number of elements required to uniquely model the helical and hoop layers. The dome shape for this pressure vessel was developed using geodesic-isotensoid theory. A winding table was generated.

Safety Analysis and Approval of 2.8 m^3, 65 bar Vessel for Containment of Non-Certified Pressure Vessels during Pressurization and Cycling

Following the safety plan submitted to DOE in June 2014 and a visit from the DOE H_2 Safety Panel in August 2014, it was decided by DOE management that authorization for the design and operation of the containment of non-certified pressure vessels under cycling and strength testing conditions should ultimately be granted by LLNL's pressure safety committee. Internal reviews were performed with pressure and containment experts leading to the ultimate approval of the operation as initially proposed.

Civil Construction of the H_2 Vessel Test Facility

Civil construction of the H_2 vessel test facility started in early FY 2015, and was completed in February 2015. It consisted of the construction of several concrete pads next to the existing LH_2 pump to accommodate a 100 psi air compressor, a control room, support racks for cryogenic high pressure H_2 lines, instrumentation and control air, and a 30 ft by 30 ft pad where the H_2 equipment (containment vessels, vent stacks, heat exchanger) will be installed. Figure 1 is a picture of the completed civil construction together with markers showing the future location of the main H_2 components.

Manufacture and Cryogenic Strength Test of the First Thin-Lined, Full Scale (63.5 L), 700 bar Pressure Vessel Prototype

The first thin-lined full scale 700 bar prototype vessel was fabricated by Spencer Composites then delivered to LLNL for cryogenic strength testing. Based on weight and geometrical measurements, we estimated that the vessel had 81% volumetric efficiency (ratio between inner and outer volume). The vessel was installed in an unmanned pressure test cell and initially filled with liquid N_2 at atmospheric pressure. It was then pressurized with room temperature N_2. Given the difficulty of maintaining a reasonable pressurization rate, the vessel underwent rather large pressure spikes (see Figure 2) before bursting at 1,560 bar and at an estimated temperature of 150 K. Based on the analysis of these results, DOE agreed to validate this go/no-go milestone.

Assembly of the H_2 Vessel Test Facility

Assembly of the test facility started after passing the first go/no-go milestone (previous task) and included instrumentation (pressure transducers, temperature sensors, vacuum transducer, H_2 sensor), air controlled valves (rated for cryogenic H_2 up to 30,000 psi), lines and fittings (rated for cryogenic H_2 up to 20,000 psi), vent stacks, 40 kW heat exchanger, and the containment vessel (Figure 3).

We anticipate completing construction of the H_2 test facility (without the heat exchanger, scheduled to arrive during the fall) by the end of FY 2015 allowing the beginning of prototype vessel cycle testing by that time.

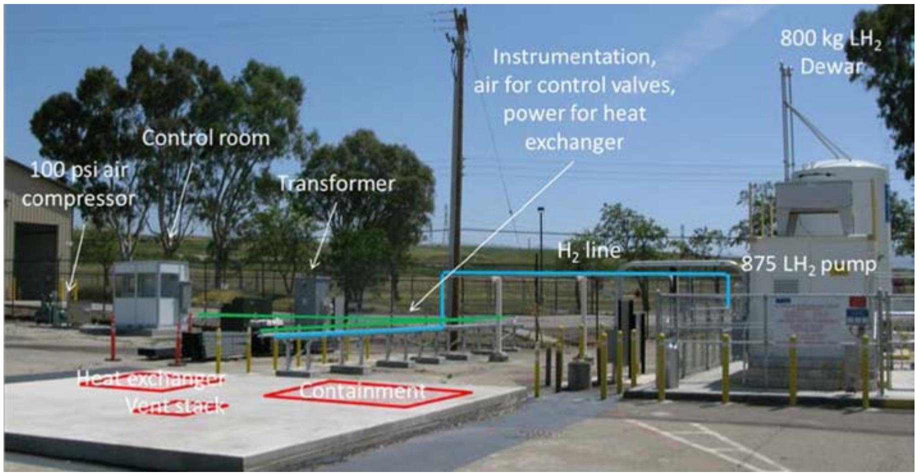

FIGURE 1. Completed site construction of the future H_2 test facility. Future locations of the main components (heat exchanger, vent stack, containment vessel and H_2 line) are shown.

CONCLUSIONS AND FUTURE DIRECTIONS

Small scale tests (welded/non-welded, LN_2/room temperature tension tests, and ring tension tests) were first carried out to determine the behavior of candidate liner metals for the prototype vessels. This was followed by a finite element analysis based on the geometrical and cycling requirements for the thin lined, carbon fiber overwrapped prototype pressure vessel, to help determine winding patterns. The first full scale prototype was then fabricated by Spencer Composites Corporation, and tested at LLNL. We successfully completed the cryogenic strength test of 63.5 L prototype with 81% volumetric efficiency burst at 1,560 bar, which represented the first go/no-go milestone for this project. In parallel, we completed the civil site construction and control room installation for the H_2 test facility. LLNL safety approval for simultaneous testing of multiple vessels within the containment vessel was also received.

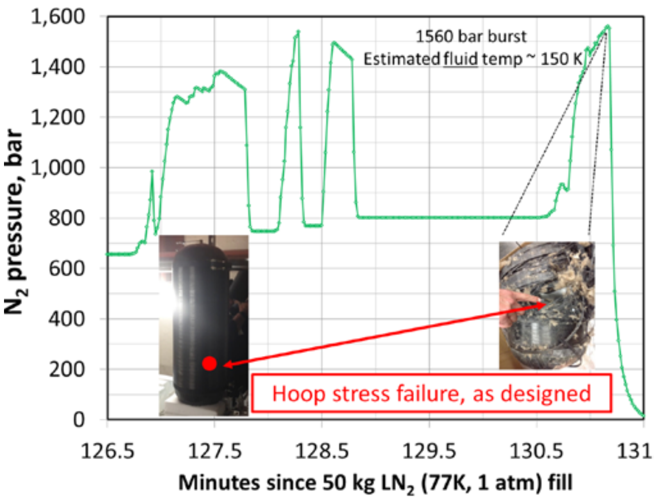

FIGURE 2. Cryogenic strength test of 63.5 L prototype with 81% volumetric efficiency burst at 1,560 bar

Future directions include the following:

- Fabricate two 63.5 L prototype vessels for cycle and subsequent strength testing
- Complete assembly and commissioning of H_2 test facility
- Cycle test two prototype vessels 1,500 times each with cryogenic H_2 followed by a strength test; a successful pressure test of either prototype with 80% volumetric

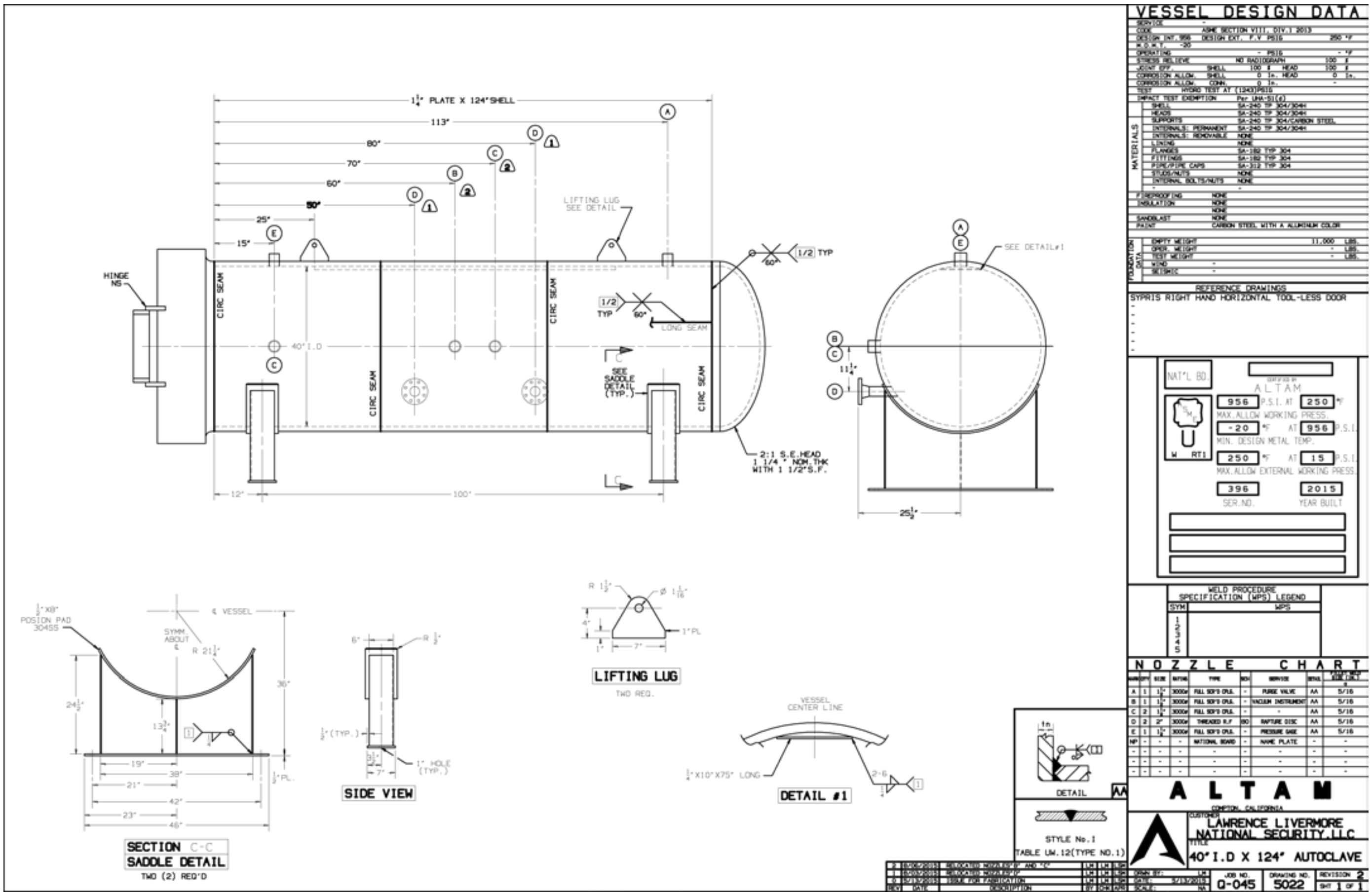

FIGURE 3. ASME certified 65 bar containment vessel design. A hinged-door will be installed on the left hand side.

efficiency when refueled at 72 g H_2/L density from the LH_2 pump will complete the second go-no go milestone

SPECIAL RECOGNITIONS & AWARDS/ PATENTS ISSUED

1. Threaded Insert for Compact Cryogenic-Capable Pressure Vessels, Francisco J. Espinosa-Loza, Timothy O. Ross, Vernon A. Switzer, Salvador M. Aceves, Nicholas J. Killingsworth, Elias Ledesma-Orozco, US Patent 9,057,483 B2, June 16, 2015.

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2. Salvador M. Aceves, Francisco Espinosa-Loza, John W. Elmer, Robert Huber, "Comparison of Cu, Ti and Ta Interlayer Explosively Fabricated Aluminum to Stainless Steel Transition Joints for Cryogenic Pressurized Hydrogen Storage," International Journal of Hydrogen Energy, Volume 40, pp. 1490-1503, 2015.

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7. Ahluwalia R.K., Hua T.Q., Peng J-K., Roh H.S., Bailey J., Kumar R. "System Level Analysis of Hydrogen Storage Options," DOE Annual Merit Review, Washington DC: 2012.