

IV.D.1k Development of Improved Composite Pressure Vessels for Hydrogen Storage

Jon Knudsen (Primary Contact), Norm Newhouse, John Makinson
Lincoln Composites, Inc.
5117 NW 40th Street
Lincoln, NE 68524
Phone: (402) 470-5039
E-mail: jknudsen@lincolncomposites.com

DOE Technology Development Manager:
Monterey Gardiner
Phone: (202) 586-1758
E-mail: Monterey.Gardiner@ee.doe.gov

DOE Project Officer: Jesse Adams
Phone: (303) 275-4954
E-mail: Jesse.Adams@go.doe.gov

Contract Number: DE-FC36-09GO19004

Project Start Date: January 1, 2009
Project End Date: December 31, 2013

Objectives

- To improve the performance characteristics, including weight, volumetric efficiency, and cost, of composite pressure vessels used to contain hydrogen in media such as metal hydrides, chemical hydrides, or adsorbants.
- To evaluate design, materials, or manufacturing process improvements necessary for containing metal hydrides, chemical hydrides, or adsorbants.
- To demonstrate these improvements in prototype systems through fabrication, testing, and evaluation.

Technical Barriers

This project addresses the following technical barriers from the Hydrogen Storage section of the Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan:

- (A) System Weight and Volume
- (B) System Cost
- (G) Materials of Construction

Technical Targets

This project is conducting fundamental studies for the development of improved composite pressure vessels

for hydrogen storage. Insights gained from these studies will be applied toward the design and manufacturing of hydrogen storage vessels that meet the following DOE 2010 hydrogen storage targets:

	2010	2015
Gravimetric capacity:	>4.5%	>6%
Volumetric capacity:	>0.045 kg H ₂ /L	>0.081 kg H ₂ /L
Storage system cost:	To be determined	To be determined



Introduction

Lincoln Composites is conducting research to meet DOE 2010 and 2015 Hydrogen Storage Goals for a storage system by identifying appropriate materials and design approaches for the composite container. At the same time, continue to maintain durability, operability and safety characteristics that already meet DOE guidelines for 2010 and 2015. There is a continuation of work with Hydrogen Storage Engineering Center of Excellence partners to identify pressure vessel characteristics and opportunities for performance improvement. Lincoln Composites is working to develop high-pressure vessels as are required to enable hybrid tank approaches to meet weight and volume goals and to allow metal hydrides with slow charging kinetics to meet charging goals.

Approach

Lincoln Composites is establishing and documenting a baseline design as a means to compare and evaluate potential improvements in design, materials and process to achieve cylinder performance improvements for weight, volume and cost. Lincoln Composites will then down-select the most promising engineering concepts which will then be evaluated to meet Go/No-Go requirements for moving forward.

The following areas will be researched and documented:

- Evaluation of alternate fiber reinforcement
- Evaluation of boss materials and designs
- Evaluation of resin toughening agents
- Evaluation of alternate liner materials
- Evaluation of damage vs. impact
- Evaluation of stress rupture characteristics
- Evaluation of in situ non-destructive examination (NDE) methods to detect damage

Results

Lincoln Composites has completed the documentation of a baseline design as a means to compare and evaluate potential improvements in design, materials and process to achieve cylinder performance improvements for weight, volume and cost. Baseline characteristics are shown in Table 1.

Lincoln Composites is in the process of conducting testing on alternate fibers relative to fiber strength and impact tolerance. Baseline fiber was selected as Toray T700. Five alternative fibers were selected as part of the study. Vessels were constructed with each of the five fibers using the same parameters on each: mandrel, wind patterns, tooling and processing. Tow count was adjusted, per fiber, to maintain consistent band cross-sectional area. One vessel constructed of each fiber was hydrostatically burst. Stress in the fiber at failure is calculated based on fiber certifications and normalized to Toray T700. Further testing of alternative fibers is being completed. Vessels, that were manufactured along with the burst vessels, are in the process of being impacted (drop testing), cycled and then burst tested. This testing is in process and following the completion of this testing, strength versus cost will be evaluated.

Lincoln Composites is looking into alternative boss materials as part of this project. Specifically, investigation methods are underway to create bosses constructed with Aluminum 7075-T73. Properties, of which, are difficult to acquire through entire thickness. High strength would allow reduction in boss size and allow aluminum use at high temperatures. To date, near net shaped bosses have been machined from 7075-T6 aluminum with the following surface finishes: smooth machining, rough machining, sand blasted and chemical etching. These bosses were then heat treated to a T73 condition. Bosses have been sectioned for review and locations mapped for hardness testing. Results

of hardness testing have been received and are in the process of being evaluated. Results to date indicate that heat treating will be sufficient if the parts are rough machined first. Plans are being made for testing of the boss material with respect to stress corrosion cracking.

Investigations into alternate resin compilation are underway to determine effects on the toughening properties of a full-scale vessel. First phase was to research and perform testing on alternate hardeners that could be used with our current baseline resin. Several experiments were run with alternate hardeners with an end result that our current hardener performs best. Next step is to use this hardener to begin looking at different resin formulations. First step is to down-select based on screening of viscosity and T_g results. Further testing is planned to determine mechanical and environmental/chemical properties. Upon completion, a down-select activity will determine what resin formulations will be used to produce coupons for further testing. The last activity will then be to build full-scale vessels with the alternate resin formulations and to perform further testing such as impact.

Studies are ongoing with respect to alternate materials to minimize the permeability of gas through the high density polyethylene (HDPE) liners that Lincoln Composites currently uses. Evaluation of coatings and surface treatments has shown blistering following a hydrogen soak and blow down. Treatments have not been shown to be effective. The first investigation into Nanoclay gave unsuccessful results. The molecular properties of HDPE did not promote dispersion. However, new material found from an alternate vendor has shown some improvements. HDPE with titanium dioxide has resulted in a 25% reduction in permeation. Lincoln Composites has also worked with the addition of ethylene vinyl alcohol (EVOH). We encountered problems with layered materials including the ability to weld. We looked at adding an outside

TABLE 1. Service Conditions and Nominal Cylinder Properties

Service Pressure	5,000 psi (344.7 bar)
Gas Settling Temperature	59 °F (15 °C)
Maximum Fill Pressure	6,500 psi (448 bar)
Service Life	20 years
Gas Fill Temperature Limits	-40 to 149 °F (-40 to 65 °C)
Operating Temperature Limits	-40 to 180 °F (-40 to 82 °C)
Proof Test Pressure	7500 psi (517 bar)
Minimum Rupture Pressure	11,700 psi (807 bar)
Cylinder Diameter	21.4 inches (543.4 mm)
Cylinder Length (unpressurized)	63.0 inches (1600 mm)
Cylinder Length at Maximum Fill Pressure	63.34 inches (1609 mm)
Cylinder Empty Weight (excluding hardware)	231 lbs (105 kg)
Cylinder Volume	15,865 in ³ (260 L)
Cylinder Volume at Service Pressure	16,132 in ³ (264.4 L)
Cylinder interior diameter	19.2 inches (488 mm)

layer to keep the material away from the weld joint, however, issues with adhesion of the EVOH to the HDPE were experienced. Lincoln Composites is in the process of looking at EVOH that has been modified to increase ductility. The evaluation of nylon as a filler has also been targeted. The cost of nylon, when compared with HDPE, would generate a large cost increase. Liners have been built with the following conditions: HDPE (baseline), HDPE/standard nanoclay, HDPE/development nanoclay, and HDPE/titanium dioxide. These will be wound into full vessels and testing will then move forward on full-scale models. A permeation rate versus cost relative to HDPE is shown in Figure 1. HDPE is the baseline at 1:1. HDPE fillers show 40% reduction with limited cost increase. Alternate materials show promise of significant permeation reduction while others are prohibitively expensive.

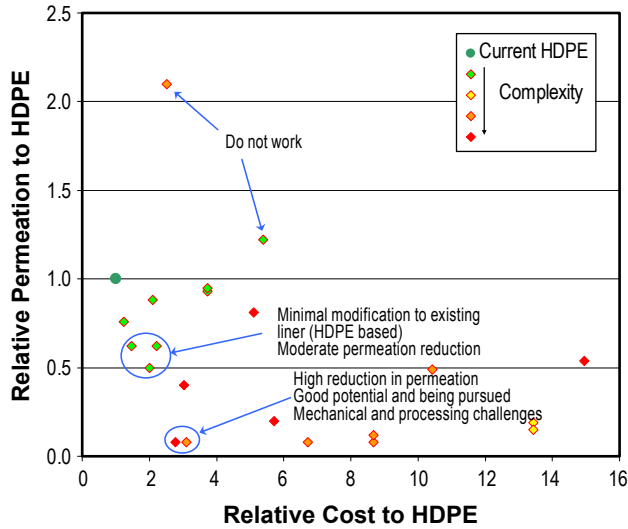


FIGURE 1. Permeation Rate versus Cost Relative to HDPE Liner Material

Lincoln Composites is looking into an improved database for stress rupture of carbon fiber that may allow for reduced safety factors. This will in turn maintain projected reliability and reduce cost, weight and increase volumetric efficiency with thinner walls. A stress rupture project presented at an industry workshop to gain feedback and support was conducted. This project is currently being refined with some collaborators and funding identified. Additional collaboration and funding is being sought. Stress rupture, fatigue and damage tolerance are all being considered in the study. The evaluation of damage vs. impact is being considered to characterize safety and ability to remain in service after damage. NDE as a means of monitoring the structural integrity is being considered which will allow for thinner laminates and removal from service before rupture.

Conclusions and Future Directions

Future work for this project will be to continue progress on evaluating potential improvements. This is work that is being completed in Phase 1 of the project. After completion of Phase 1, Lincoln Composites will down-select most promising engineering concepts and evaluate against DOE 2010 and 2015 Hydrogen Storage Go/No-Go criteria. Phase 2 is continuation of container development in support of system requirements and finally, Phase 3 will be the fabrication of subscale vessels to support assembly of prototype systems for evaluation.

FY 2010 Publications/Presentations

1. 2010 DOE Hydrogen Program Annual Merit Review, June 9, 2010, Washington, D.C.