Enhanced Materials and Design Parameters for Reducing the Cost of Hydrogen Storage Tanks

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Pacific Northwest National Laboratory
June 17, 2014

Project ID # ST101

This presentation does not contain any proprietary, confidential, or otherwise restricted information.
Overview

Timeline

- Start date: Jan 2012
- End date: Sept 2015
- Percent complete: 50%

Barriers

- Barriers addressed
  - Reduce the cost of manufacturing high-pressure hydrogen storage tanks
  - Improved material properties to reduce carbon fiber use
  - Alternative tank operating parameters provides wider operating envelope of pressure and volume
  - Strategic alternative fiber types and fiber placement for cost reduction

Budget

- FY13 DOE Funding: $382K
- Planned FY14 DOE Funding: $600K
- Total project funding
  - DOE share: $2,100K
  - Contractor share: $525K (20%)

Partners

- Project Lead - PNNL
- Collaborating Team Members
  - Hexagon Lincoln
  - Toray CFA
  - AOC, LLC
  - Ford Motor Company
System Cost Analysis Study
2013 AMR Presentation - Strategic Analysis

70MPa Compressed Gas Storage System
Single tank holding 5.6kgH₂ usable, cost in 2007$

- System Assembly
- Balance of Plant (BOP) Items
- He Fill & Leak Test
- Hydro Test
- Boss (Materials & Proc.)
- Full Cure (Cure #2)
- B-Stage Cure (Cure #1)
- Fiber Winding
- Composite Materials
- Liner Annealing
- Liner Formation (Material & Proc.)
Relevance

Strategic Analysis Cost Study – High Volume - based on the 2013 AMR reference projections

Materials make up 63% of the tank cost.

Onboard automotive hydrogen storage system cost targets:

- 2017 - $12/kWh of useable H₂
- Ultimate - $8/kWh of useable H₂

System Cost @ 500,000 Systems/Year

BOP & Assembly, 30%
Fiber Winding (Wet Winding) Materials, 63%
Liner Blow Mold Materials
Liner Blow Mold Tooling
He Fill & Leak Test Manufacturing
Volumetric Water Test Manufacturing
Full Cure Manufacturing
B-Stage Cure (Cure #1) Manufacturing
Fiber Winding (Wet Winding) Manufacturing
Liner Annealing Manufacturing Liner Blow Mold Manufacturing
Tank Shoulder Foam Materials Boss Materials

500k System per Year
System Cost: $3,134
$600/kgH₂
$17/kWh
Project Approach

- Improvement of the individual constituents for synergistically enhanced tank performance and cost reduction

Reduced tank costs and mass through engineered material properties for efficient use of carbon fiber
### Updated Milestones

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<th>Date</th>
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<td>3/31/2013</td>
<td>Go/No-Go: &quot;PNNL, with partners Toray Carbon Fibers America, AOC Inc., Lincoln Composites, and Ford Motor Company, will develop a feasible pathway to achieve at least a 10% ($1.5/kWh) cost reduction, compared to a 2010 projected high-volume baseline cost of $15/kWh for compressed H₂ storage tank through detailed cost modeling and specific individual technical approaches.”</td>
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<td>6/30/2014</td>
<td>PNNL, with partners Toray Carbon Fibers America, AOC Inc., Hexagon Lincoln, and Ford Motor Company, will develop a feasible pathway through cold gas enhanced operating conditions to achieve at least an additional 20% ($3.4/Kwh) cost (mass reduction of 18.7 kg composite or 13.3 kg carbon fiber) reduction for compressed hydrogen storage tank above the 15% (13.5 kg composite, 9.6 kg carbon fiber) accomplished in FY13 through resin modification and fiber placement. This will be demonstrated through detailed cost modeling of specific low cost thermal insulating approaches. Percent improvements are based on a 2013 projected high-volume baseline (composite mass 93.6 kg, carbon fiber mass 66.3 kg) cost of $17/kWh for 70MPa compressed H2 storage tanks.</td>
<td>In progress</td>
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</table>
Flow chart illustrates the approach of the project and inner relationship of each task (task leads are indicated)
Baseline cost model for an on-board vehicle tank was considered a critical element for the project in order to evaluate the starting point and progress.

Cost factors:
- Carbon Fiber Options: material and usage
- Insulation Concepts: vacuum, ultra-insulations
- Design Alternatives: resin, fibers, liner, processing

Compare with prior DOE cost studies by TIAX and Strategic Analysis (SA).

Cost model will allow for trade-off studies to be performed in order for the team to focus on the most promising concepts.

Desire to use a simplified estimator tool for predicting storage system parameters and cost without extensive CAE modeling.
Currently identified additional cost reduction opportunities through cold gas storage to achieve a 30% system cost savings and projected path to target.
700 Bar Type IV Single Tank System Compared Against 2017 Targets

- Gravimetric Density
- Min. Delivery Temp.
- Max. Delivery Temp.
- Min. Delivery Pressure
- Max. Operating Temp.
- Min. Operating Temp.
- Max. Delivery Pressure
- Min. Full Flow Rate
- System Cost
- Onboard Efficiency
- Ambient Temperature

- Low Cost CF Tank
- DOE Baseline Tank Performance
Technical Accomplishment – Nanoscale Resin Additives

- Nanoscale additives strengthen resin
- PNNL validating multiple types
- Mechanical testing
- Viscosity measurements
- Initial down selection – UTS, viscosity, cost

Images of various nanomaterials:
- SNF
- Nano Clay
- Graphene
- Nano Graphite
- CNF
- MWNT
**Technical Accomplishment – Matrix Modifications:**

- Testing of nanoscale additives in alternate resins

- Tensile samples fabricated from vinyl ester resins with nanoscale additives

- Testing shows significantly enhanced UTS and Elongation at break with nano-additives

- Additional testing with different cure recipes is needed and at cryogenic temperatures

- Based on cost and performance, nanoclays and nanoplatelets are top candidates at $3-10/lb

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![Tensile testing nano-filled resin](image)

<table>
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<tr>
<th>Ultimate Tensile Strength [MPa]</th>
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<td>Std Dev.</td>
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<td>77-3</td>
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<td>77-5</td>
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<table>
<thead>
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<th>Elongation at Break [%]</th>
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<td>77-4</td>
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<td>77-5</td>
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</table>
Technical Accomplishment – Matrix Modifications: Rheology of nanoscale additives in alternate resins

- A rheology study was performed on top performing nano-additives
- High-shear mixing required
- Higher concentrations tried
- Noticed some issues with gelling (after sonication) of CNF in T015 – removed from list
- XV-3175 has higher viscosity – allows for longer dispersion working time than T015
  - Indicates daily mixing may be required
Technical Accomplishment – Matrix Modifications: Rheology of nanoscale additives in alternate resins (part 2)

- PNNL prepared new nano additive resins and AOC tested using standard procedures
- Evaluated higher concentrations for larger effects on properties

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<tr>
<th>Resin</th>
<th>Additive</th>
<th>ν(cps)</th>
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<tr>
<td>XV-3175</td>
<td>Neat</td>
<td>922</td>
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<tr>
<td></td>
<td>1wt% CNF</td>
<td>1200</td>
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<tr>
<td></td>
<td>1wt% SNF</td>
<td>1096</td>
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<tr>
<td></td>
<td>2wt% SNF</td>
<td>1260</td>
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<tr>
<td></td>
<td>1wt% 20A</td>
<td>1226</td>
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<tr>
<td></td>
<td>2wt% 20A</td>
<td>1213</td>
</tr>
<tr>
<td></td>
<td>1wt% N307</td>
<td>1101</td>
</tr>
<tr>
<td></td>
<td>2wt% N307</td>
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<tr>
<td>T015</td>
<td>Neat</td>
<td>356</td>
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<tr>
<td></td>
<td>1wt% SNF</td>
<td>406</td>
</tr>
<tr>
<td></td>
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<td>466</td>
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<tr>
<td></td>
<td>2wt% N307</td>
<td>485</td>
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</table>

T015 Resin system viscosity in range for filament winding
Technical Accomplishment - Matrix Modifications: Catalyst and Filler Interactions

T015 1%Asbury – small white defects
T015 5%Asbury – wrinkling, white defects over large area
T015 1%cloisite – complete separation
T015 5%cloisite – looks ok
T015 5%SNF – looks ok
T015 2%SNF – looks ok
XV-3175 1%cloisite – nonuniform?
XV-3175 5%cloisite – nonuniform edge issue?
XV-3175 1%cloisite – nonuniform?
XV-3175 1%CNF – white defects?
XV-3175 1%SNF – looks ok
XV-3175 1%SNF – looks ok
T015 1%Asbury – cracking, white defects
XV-3175 1%Asbury – cracking, white defects
XV-3175 5%Asbury – cracking, white defects
XV-3175 5%Asbury – cracking, white defects
Technical Accomplishment - Matrix Modifications: Catalyst and Filler Interactions

Catalyst and filler interaction has shown to have an effect on curing.
Technical Accomplishment - Alternate Fiber Placement and Multiple Fiber Types

- Investigating alternate carbon fibers
  - Evaluate performance/price
  - Consider heavy tow fibers

- Investigating alternate low-cost fibers
  - Evaluate performance/price
  - Consider strength and other performance issues
  - Consider manufacturability

- Evaluating hybrid fiber reinforcement
  - Some materials give strength
  - Some materials address durability

- Evaluating layering options
  - Higher modulus materials on outside to improve load share with inner layers
  - One material for helical layers, one for hoop layers
Technical Accomplishment – Alternate Fiber Placement and Multiple Fiber Types

Fiber Properties Used

<table>
<thead>
<tr>
<th>Material Property</th>
<th>E-Glass</th>
<th>T300</th>
<th>T700</th>
<th>T720</th>
<th>T800</th>
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<tr>
<td>Tensile Strength [ksi]</td>
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<td>512</td>
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<td>24</td>
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<td>Yield [ft/lb]</td>
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<td>1862</td>
<td>903</td>
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<td>Density [lb/in3]</td>
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<td>0.064</td>
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Gains in cost and mass savings up to 16% through controlled fiber placement
<table>
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<tr>
<th>Build</th>
<th>Build Desc.</th>
<th>Build Dep.</th>
<th>Fiber</th>
<th>Resin</th>
<th>Design</th>
<th>Qty. Planned</th>
<th>Qty. Produced</th>
<th>Qty. Tested</th>
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### Technical Accomplishments – Model Validation Matrix for Tank and Material Designs Phase I

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<th>Fiber</th>
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</tr>
</tbody>
</table>

**Detailed modeling completed and experimental validation in progress**
Technical Accomplishments - Alternate Fiber Placement and Multiple Fiber Types

- Baseline tank design is within 1-2% of design burst pressure
- Prioritized burst testing matrix to test the effects of fiber placement and multiple fiber types
- Tank burst test on filled and unfilled low cost matrix for evaluation of nano filler enhancements
Technical Accomplishment - Enhanced Operating Conditions

- Assess the operating condition alternatives
  - **Pros**
    - 1. Allows equivalent density at lower pressure which reduces the carbon fiber and cost
    - 2. Lower pressure allows for a thinner, lighter, efficient pressure vessel
  - **Cons**
    - 1. Insulation is required to maintain temperature and extend dormancy
    - 2. Insulation reduces the cost and volume benefits of the lower pressure

<table>
<thead>
<tr>
<th>Current H₂ Tank</th>
<th>Enhanced H₂ Tank</th>
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<tbody>
<tr>
<td>Operating Conditions</td>
<td>700 bar at 15° C</td>
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<tr>
<td>Density</td>
<td>40 g/l</td>
</tr>
<tr>
<td>Tank Mass</td>
<td>93.6 kg</td>
</tr>
</tbody>
</table>

![Graph of Hydrogen Density vs. Temperature for Constant Pressure](image)
Technical Accomplishment - Enhanced Operating Conditions

- Transient heat transfer model calculates tank temperature and pressure rise based on thermal properties and mass of tank components and hydrogen gas.
- Model easily links to Ford and PNNL tank cost estimators.
- For cold gas operation, estimate:
  - Dormancy for a given insulation system
  - Insulation cost and volume that offset composite savings and package size

**Insulation Dormancy Study**
- Benchmark thermal model against measured performance of LLNL cryo-compressed vacuum insulated jacket.
- Show dormancy improvement of cold gas operation compared to cryo-compressed temperature and pressure.
- Does the vacuum insulation jacket provide enough dormancy for the cost ($290, Tiax cost model)?
Cryo-Compressed vs. Cold Gas Dormancy

Cryo-Compressed

- Initial / final:
  - 26K (-247 C) and 4 bar
  - 77K (-196 C) and 340 bar
- H2 mass = 9.8 kg
- Heat and thermal mass:
  - 4.71 W to 4.66 W
  - 59 to 78 kJ/K
- 9.3 days

Cold Gas

- Initial / final:
  - 200K (-73 C) and 500 bar
  - 248K (-25 C) and 625 bar
- H2 mass = 6.3 kg
- Heat and thermal mass:
  - 3.78 W to 2.59 W
  - 63 to 66 kJ/K
- 18 days

The cold gas storage with similar insulation to cryo-compressed can double the dormancy period.
Technical Accomplishment - Enhanced Operating Conditions: Insulation Systems

- Tiax estimated $290 for the manufactured cost of the vacuum insulation system based on the 151 liter capacity of the Gen-3 tank.
- Estimated Cost Margin $245 satisfies our project goal of 30% overall system cost savings.
- Further cost reduction potential:
  - Smaller 141 L capacity is required for 5.8 kg H2.
  - Reduced dormancy to 7 days could allow a lower cost insulation system.
- Next Steps: Evaluate high performance physical insulation materials for cost and volume tradeoffs with vacuum jacket technology.

Current cost estimates under our cost margin to meet our goal of 30% system cost savings

## Technical Accomplishments – Cost Analysis

### Improvements in Tank Cost Reductions

<table>
<thead>
<tr>
<th>Case</th>
<th>Useable Hydrogen Mass Kg</th>
<th>Composite (fiber + resin) Mass Kg</th>
<th>% Reduction of Composite Mass from Baseline</th>
<th>Est. Tank Cost w/o BOP (without profit) $</th>
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</thead>
<tbody>
<tr>
<td>1. Baseline, T=288K, P=70 MPa</td>
<td>5.6</td>
<td>93.6</td>
<td>0%</td>
<td>$2,551</td>
</tr>
<tr>
<td>2. Lower Cost Resin, T=288K, P=70 Mpa</td>
<td>5.6</td>
<td>93.6</td>
<td>0%</td>
<td>$2,454</td>
</tr>
<tr>
<td>3. Nano-Strengthened Resin, T=288K, P=70 Mpa</td>
<td>6.0</td>
<td>87.7</td>
<td>6%</td>
<td>$2,351</td>
</tr>
<tr>
<td>4. Fiber Material and Winding Design, T=288K, P=70 Mpa</td>
<td>6.1</td>
<td>83.6</td>
<td>10%</td>
<td>$2,249</td>
</tr>
<tr>
<td>5. Cold Gas, Same Outer Volume, T=200K, P=50 MPa</td>
<td>7.0</td>
<td>59.1</td>
<td>36%</td>
<td>$1,637</td>
</tr>
<tr>
<td>6. Cold Gas, Resized for 5.8kg H₂, T=200K, P=50 MPa</td>
<td>5.6</td>
<td>48.2</td>
<td>48%</td>
<td>$1,362</td>
</tr>
</tbody>
</table>

PNNL Target 37% Composite Cost Reduction: $1,607

Insulation Margin for 37% total Reduction: $245

**37% Tank Cost Savings**
FY13 Reviewer Comment: Future work looks to be a weakness as the efforts do not appear to further address the remaining 40% cost reduction goal. The effort to optimize the use of different fiber types is the right approach. However, the future work does not appear to leverage the success of the modeling effort with an optimized pressure vessel geometry and ultimately the efficient use of different fiber types.

FY14 Response: The project is currently validating the models with a baseline tank geometry for varied fiber types that would determine the optimum use of the various fiber types.

FY13 Reviewer Comment: So far focus on simulations. Experimental verification is missing but planned for the future.

FY14 Response: Correct, the project is currently experimentally validating the assumptions made in the modeling through testing the various resins, nano additives, and tank layup designs. The tanks are ultimately the final target for improvements in burst testing with lower weights or material costs.
Proposed Future Work

FY14

► Complete testing of material modification enhancements with higher concentrations
► Fabricate tanks with baseline geometry with alternate fiber placement and multiple fiber types
► Fabricate baseline tank geometry with material property enhancements
► Complete test matrix burst testing

FY15

► Integration of individual material constituents into full scale tank builds
► Burst testing of full scale tank designs based on performance data from FY14 small scale tank builds
► Correlate full scale tank build material masses into cost savings
► Complete testing on insulating materials cost and performance
Collaborations

- Pacific Northwest National Laboratory: Kevin Simmons (PI), Ken Johnson, Kyle Alvine
  - Project management, material and cost models, resin modifications
- Hexagon Lincoln: Norm Newhouse, Brian Yeggy
  - Tank modeling, tank fabrication, tank and materials testing
- Ford Motor Company: Mike Veenstra, Dan Houston
  - Enhanced operating conditions, cost modeling, materials testing
- Toray Carbon America: Anand Rau
  - Carbon fiber surface modification and testing
- AOC Resins: Thomas Steinhausler, Mike Dettre
  - Resin system design and materials testing
Project Summary

- Down selected specific matrix modifiers and currently focusing on higher concentrations and the impact on viscosity
- Completed extensive thermal performance model on insulating quality and cost
- Thermal insulating performance models indicates with cold gas temperatures (-73°C) dormancy could extend out to 18 days or a reduction in tank insulation could lower the insulating costs
- Identified reduction opportunities to achieve up to a 48% composite tank cost savings before insulating costs
- Identified an insulating cost margin of $245 per tank allowed for a 37% composite tank cost savings
Project Summary

Relevance: Reducing pressure vessel cost, mass, and volume

Approach: Establish baseline cost and reduce tank costs and mass through engineered material properties through efficient use of carbon fiber

Technical Accomplishments: Developed a feasible pathway to achieve at least a 30% ($5.1/kWh) system cost reduction, compared to a 2013 projected high-volume baseline system cost of $17/kWh for 700 bar Type IV pressure vessel through detailed cost modeling, cold gas operation, and specific individual technical approaches

Technology Collaborations: Active collaborations with Hexagon Lincoln, Ford Motor Company, Toray CFA, and AOC, LLC

Proposed Future Research: Validate predictive models with experimental data
Currently identified additional cost reduction opportunities through cold gas storage to achieve a 37% tank cost savings and projected path to target.
Technical Accomplishment - Enhanced Operating Conditions: Temperature Dependent Thermal Performance of Vacuum Insulation

- Radiation heat transfer of Multi-layer Vacuum Insulation (MLVI) with \( n \) layers.
- \( T_1 \) = Vacuum jacket temperature
- \( T_2 \) = Gas temperature
- Temperature Dependent Thermal Resistance, \( R_{rad} \), is updated as the tank temperature increases

\[
q = \frac{A \varepsilon \sigma (T_4^4 - T_3^4)}{(1 + n)}
\]

- Incremental heat transfer modelled in Excel
- Vacuum insulation and H2 properties update each time step
- 1D solution x tank surface area
Technical Accomplishment -
Enhanced Operating Conditions:
Vacuum Insulation Model Progression

- Benchmark the MLVI model against the LLNL Gen-2 Dormancy Tests (match reported 5W heat gain and 16K/hr temperature rise).
- Confirm Model performance of Gen-3 tank performance reported by ANL. Gen-3 has thicker aluminum liner and less composite. (Supercritical H2 at 350 bar and 63K had 2 days dormancy to final pressure of 425 bar. PNNL model also predicted 2 days.)
- Model ANL dormancy cases for cryo-compressed initial conditions of 26K and 40 bar to final 340 bar. (ANL predicted 5 to 11.7 days for 85% and 60% full tank. PNNL model predicts 9.3 days assuming supercritical H2 properties).
- Increase initial conditions to 200K and 500 bar. Calculate dormancy to 625 bar. PNNL model predicts 18 day dormancy, double the 9 day dormancy at cryo-compressed conditions.