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

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Pacific Northwest National Laboratory

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Project ID # **ST101**

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Overview

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Timeline

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

Budget

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

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

Partners

- Project Lead - PNNL
- Collaborating Team Members
  - Hexagon Lincoln
  - Toray CFA
  - AOC, LLC
  - Ford Motor Company

Relevance

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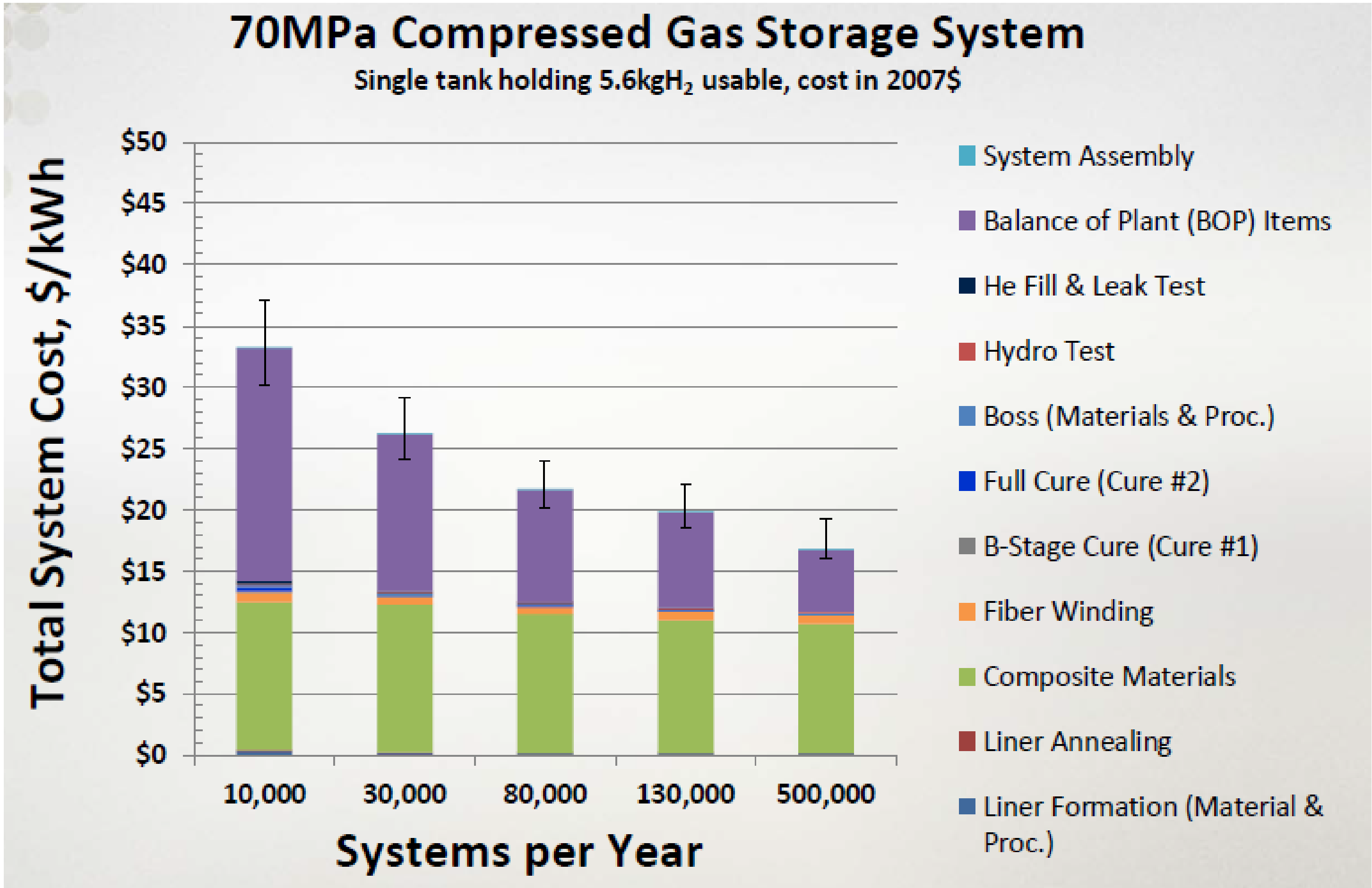
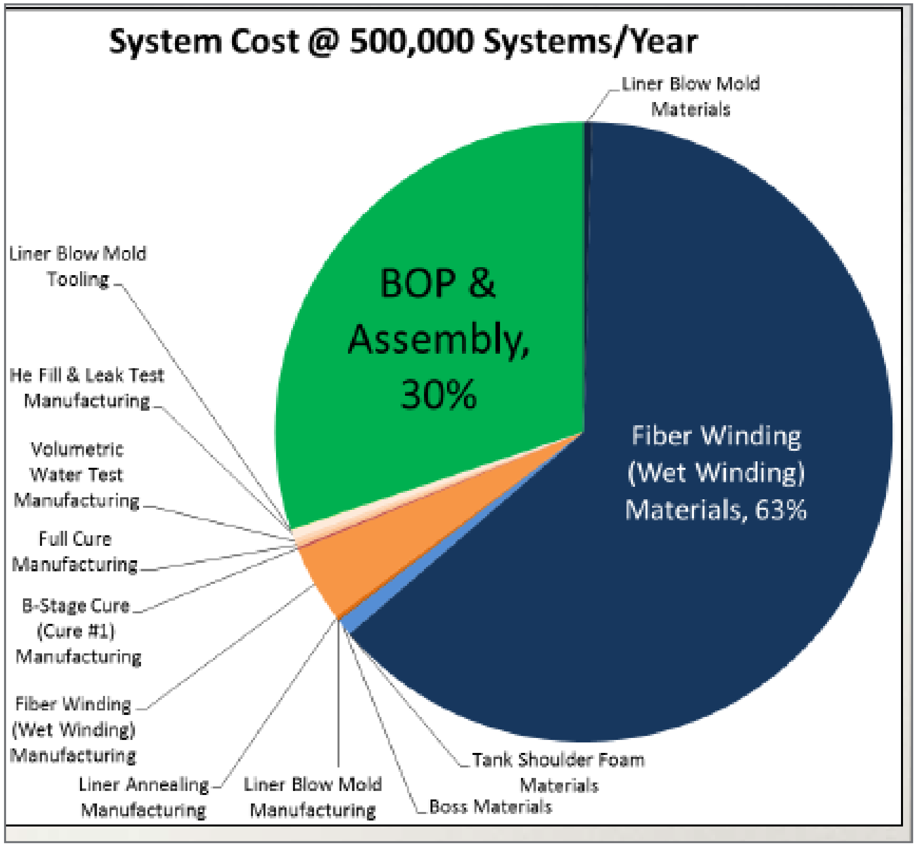
System Cost Analysis Study

2013 AMR Presentation - Strategic Analysis



Relevance

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



500k System per Year
System Cost: $3,134
$600/kgH$_2$
$17/kWh

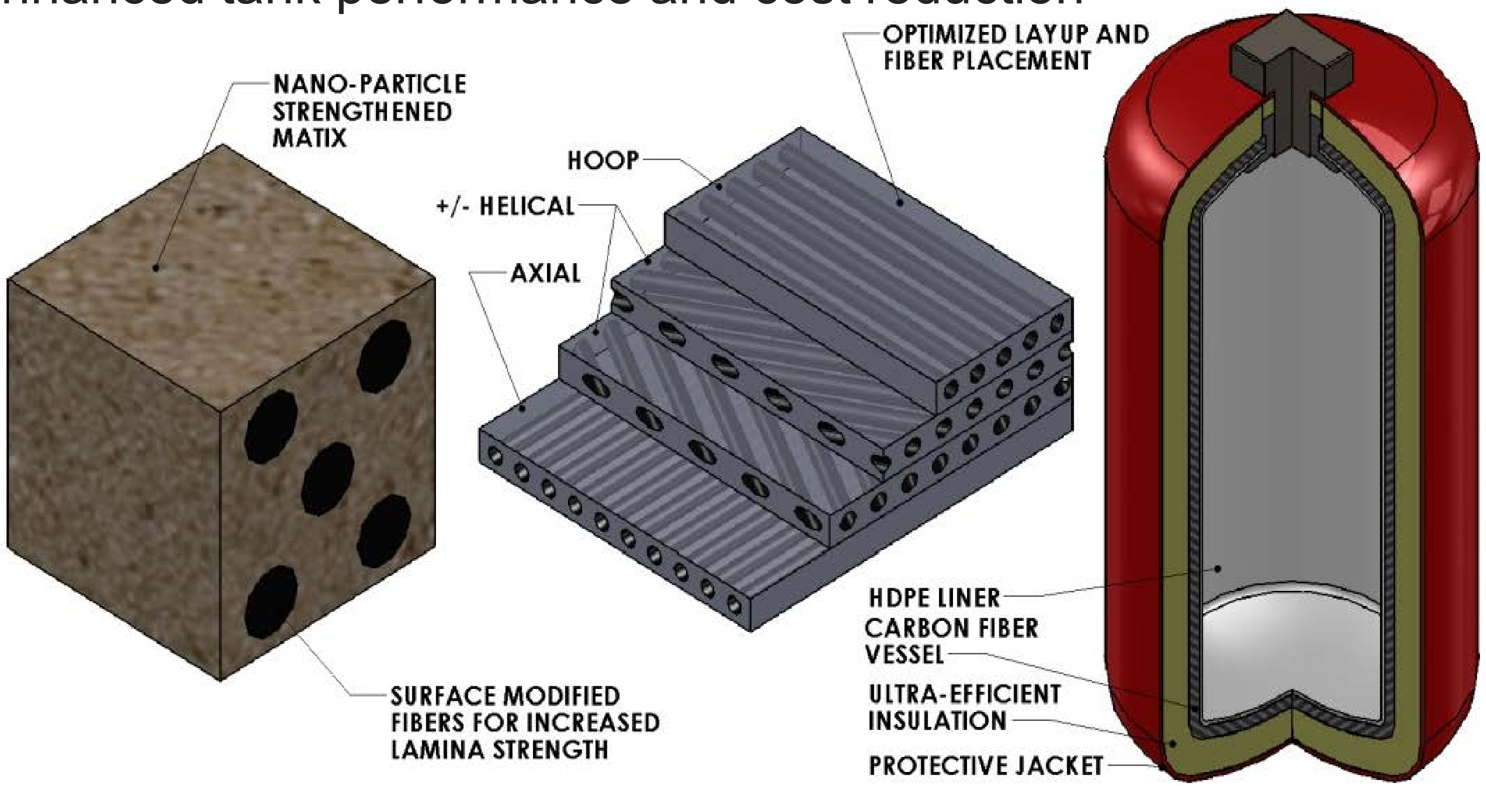
Onboard automotive hydrogen storage system cost targets:

- 2017 - $12/kWh of useable H_2
- Ultimate - $8/kWh of useable H_2

Materials make up 63% of the tank cost.

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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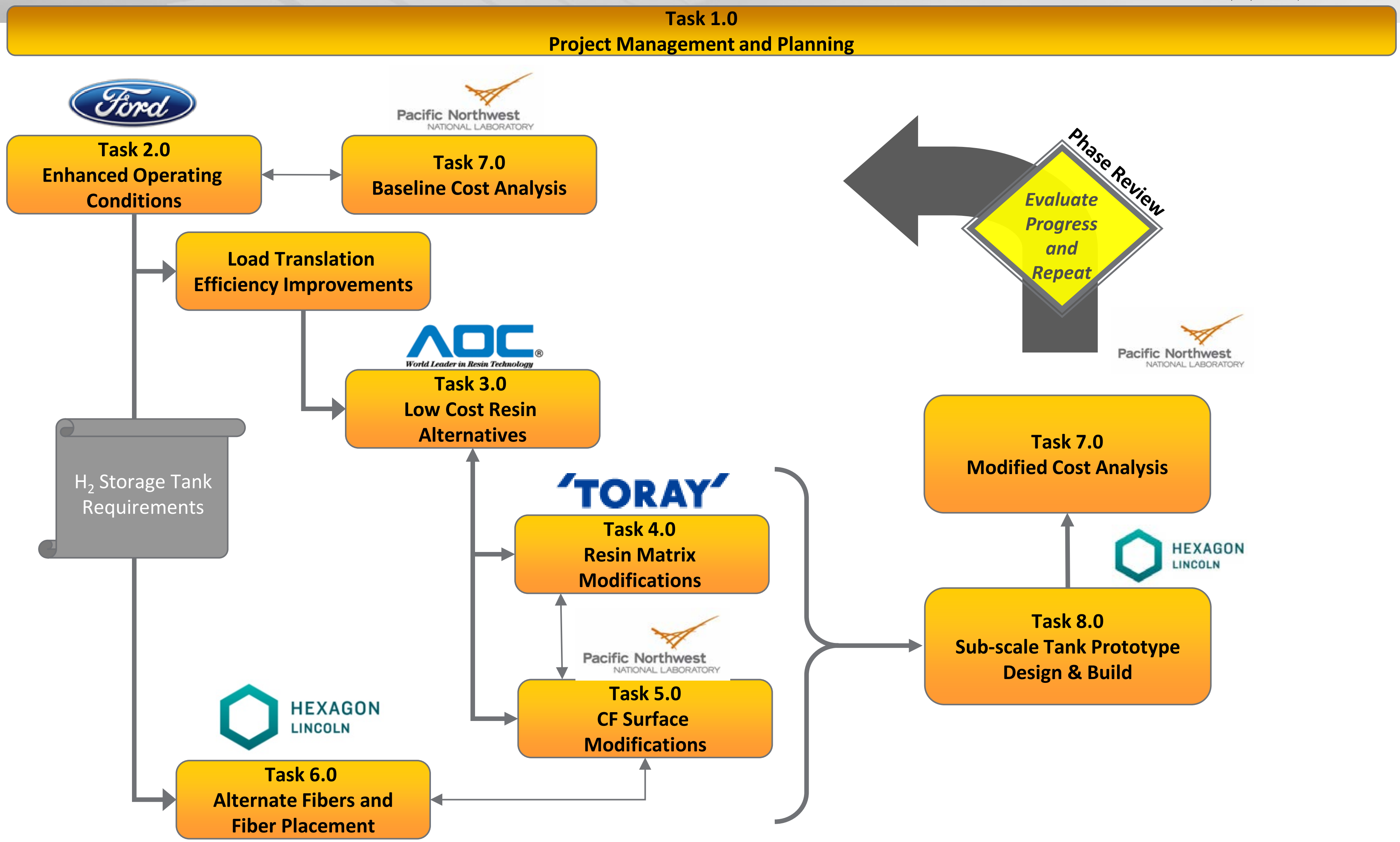
Date	Go/No-Go Decision	Status
3/31/2013	Go/No-Go: "PNNL, with partners Toray Carbon Fibers America, AOC Inc., Lincoln Composites, and Ford Motor Company, will develop a feasible pathway to achieve at least a 10% ($1.5/kWh) cost reduction, compared to a 2010 projected high-volume baseline cost of $15/kWh for compressed H_2 storage tank through detailed cost modeling and specific individual technical approaches."	Completed
6/30/2014	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.	In progress

Project Approach



Flow chart illustrates the approach of the project and inner relationship of each task (task leads are indicated)

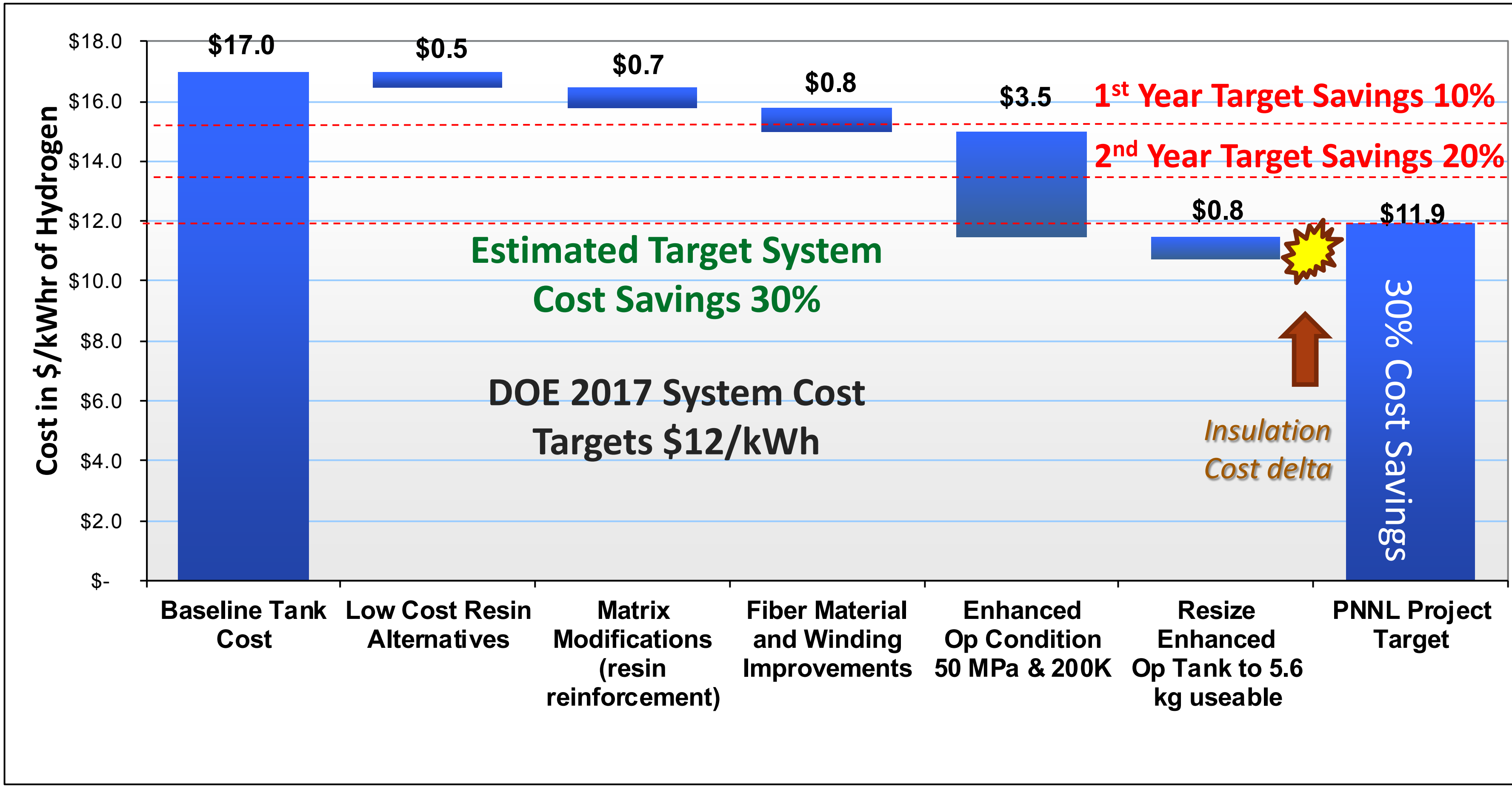
Project Approach
Baseline Cost analysis

- 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.

Technical Accomplishment - Cost Analysis Reduction Opportunities

70 MPa H2 Type 4 Tank Cost Analysis Projections

5.6 kg useable H2 (baseline system cost based on DOE's 2013 700 bar storage system cost record)



Currently identified additional cost reduction opportunities through cold gas storage to achieve a 30% system cost savings and projected path to target

Technical Accomplishment - Spider Chart

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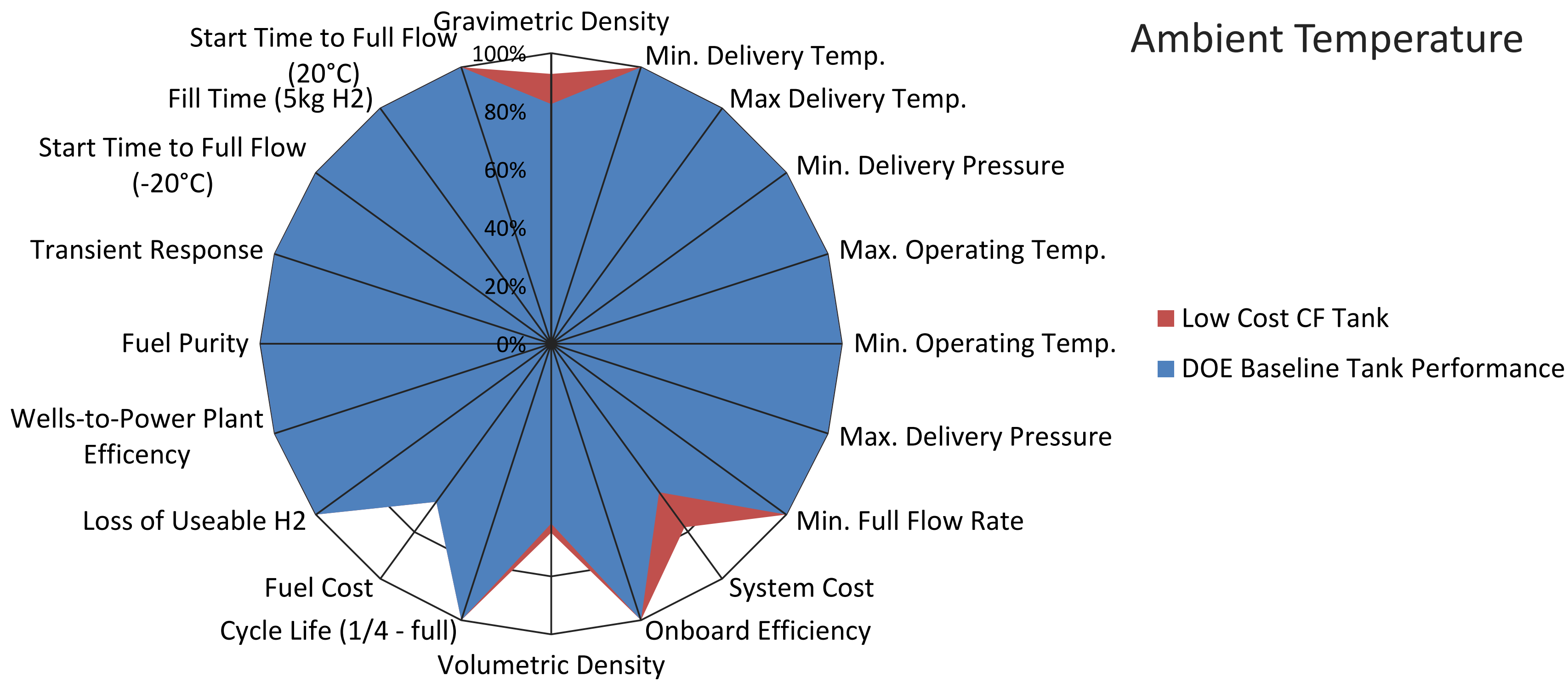
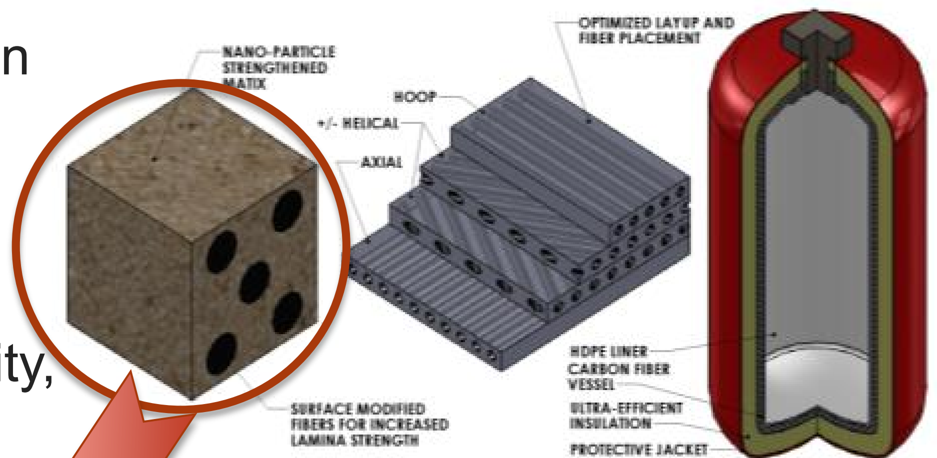
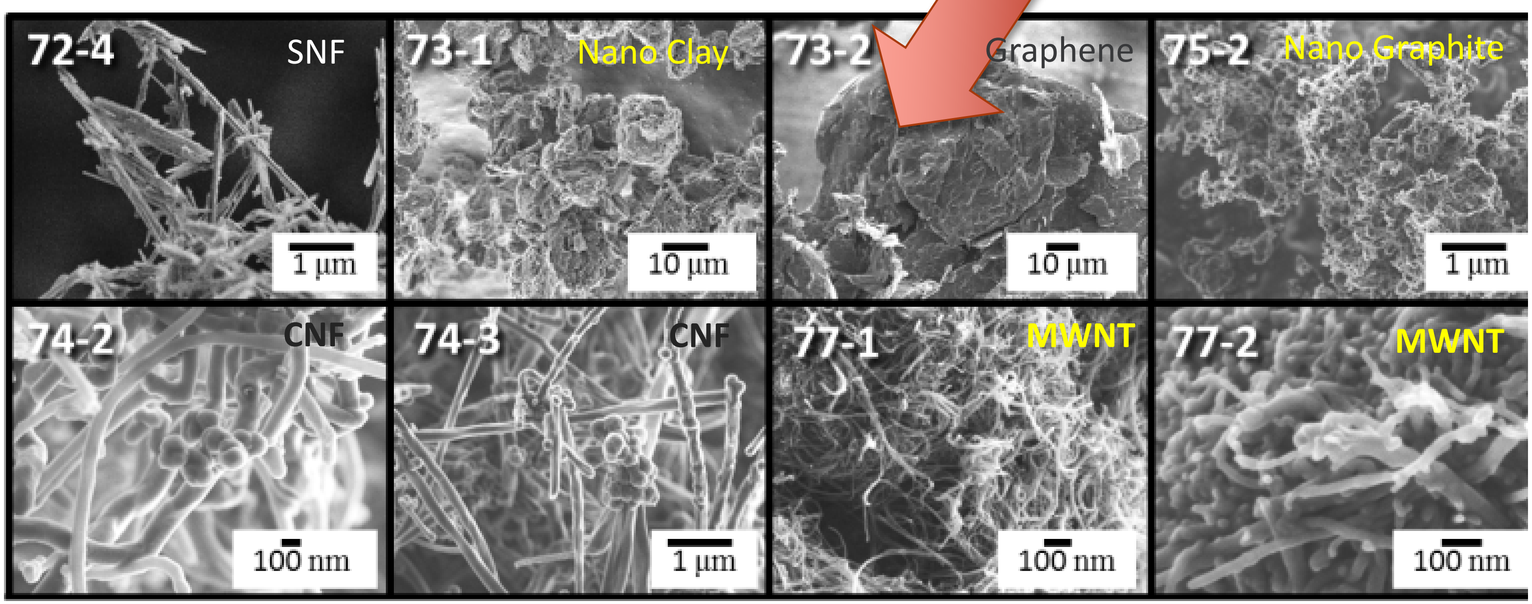
700 Bar Type IV Single Tank System Compared Against 2017 Targets



Technical Accomplishment – Nanoscale Resin Additives

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



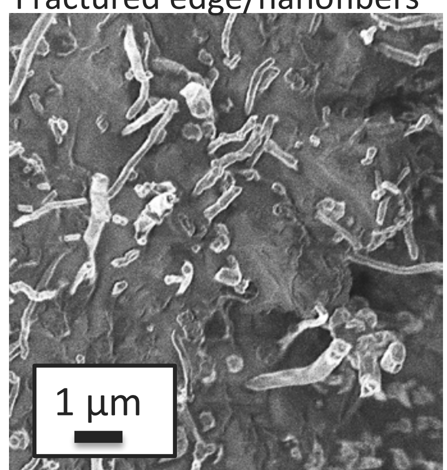


Technical Accomplishment – Matrix Modifications: testing of nanoscale additives in alternate resins

- 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

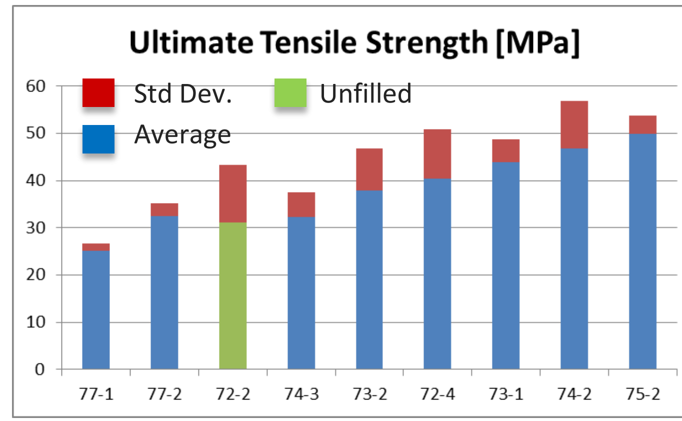
Tensile testing nano-filled resin

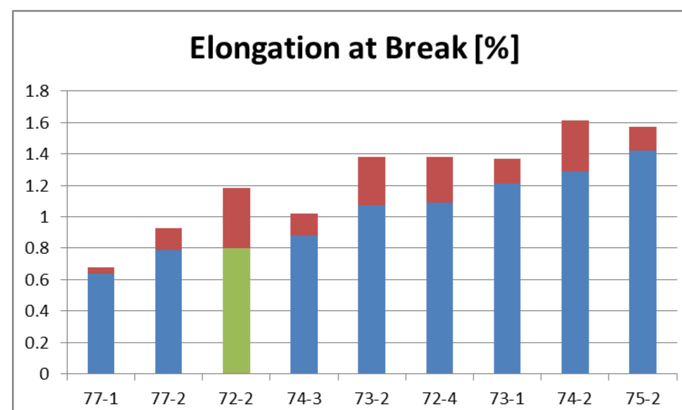
Fractured edge/nanofibers



neat resin

nano-filled resin





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Technical Accomplishment – Matrix Modifications: Rheology of nanoscale additives in alternate resins

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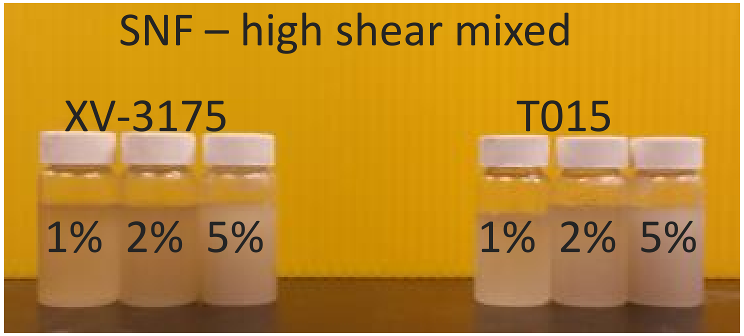
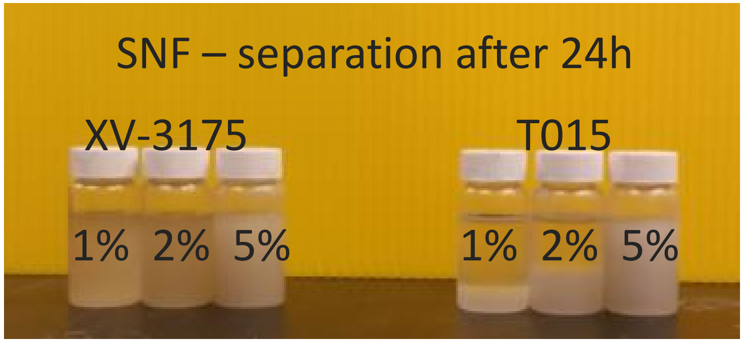
- A rheology study was performed on top performing nano-additives
- High-shear mixing required
- Higher concentrations tried
- Noticed some issues with gelling (after sonication) of CNF in T015 – removed from list
- XV-3175 has higher viscosity – allows for longer dispersion working time than T015
 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





AOC® World Leader in Resin Technology

Technical Accomplishment – Matrix Modifications: Rheology of nanoscale additives in alternate resins (part 2)

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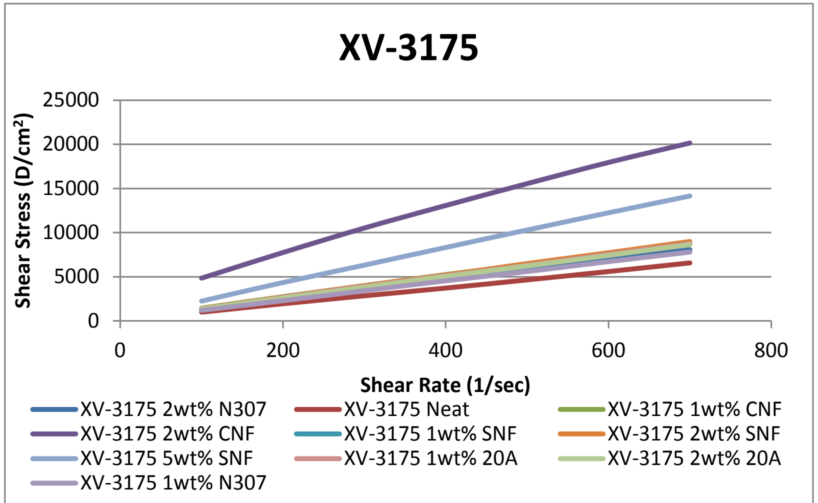
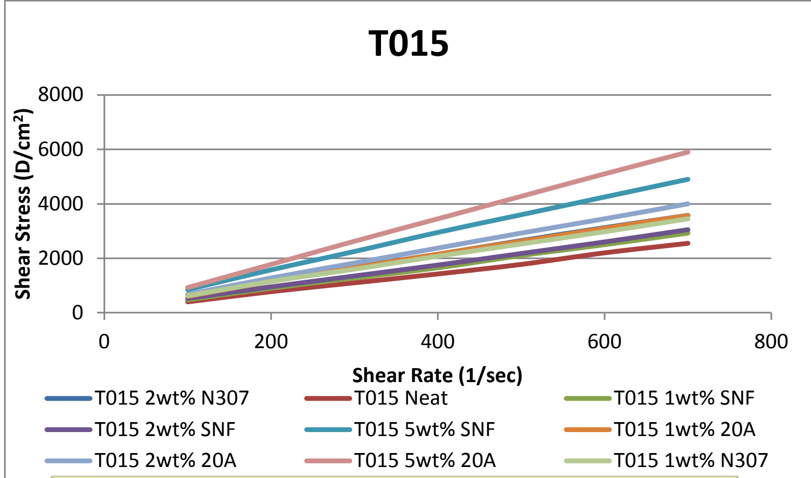
- PNNL prepared new nano additive resins and AOC tested using standard procedures
- Evaluated higher concentrations for larger effects on properties

Resin	Additive	v(cps)
XV-3175	Neat	922
	1wt% CNF	1200
	1wt% SNF	1096
	2wt% SNF	1260
	1wt% 20A	1226
	2wt% 20A	1213
	1wt% N307	1101
	2wt% N307	1129
T015	Neat	356
	1wt% SNF	406
	2wt% SNF	418
	5wt% SNF	673
	1wt% 20A	493
	2wt% 20A	551
	5wt% 20A	829
	1wt% N307	466
	2wt% N307	485





T015 Resin system viscosity in range for filament winding

Technical Accomplishment - Matrix Modifications: Catalyst and Filler Interactions

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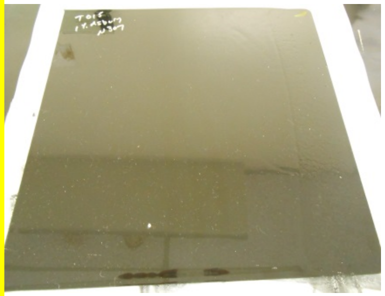
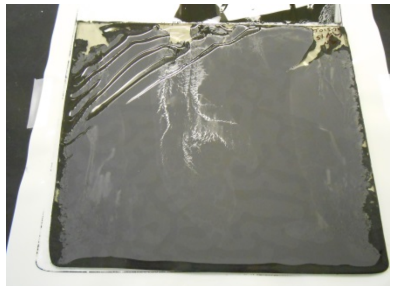
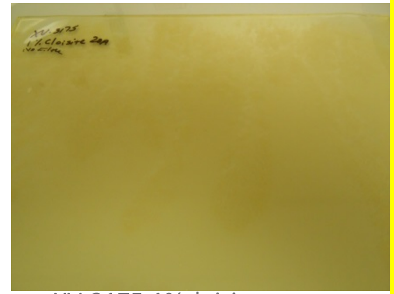


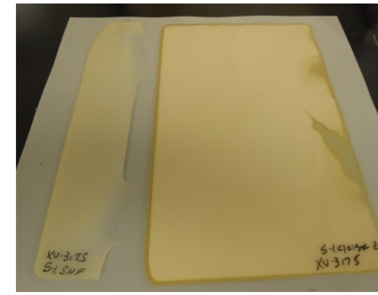
T015 1%Asbury – small white defects



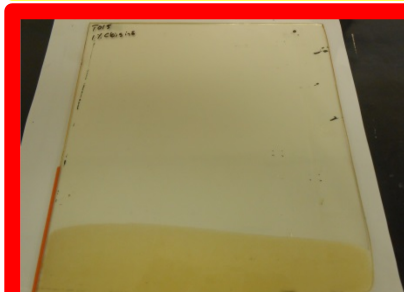
T015 5%Asbury – wrinkling, white defects over large area



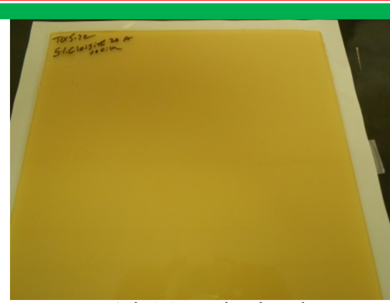
XV-3175 1%cloisite – nonuniform?



XV-3175 5%cloisite – nonuniform edge issue?



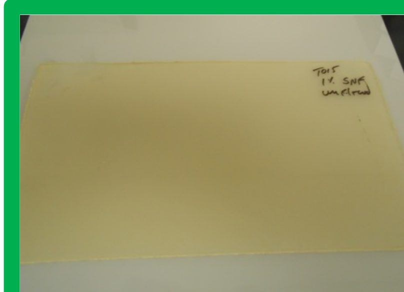
T015 1%cloisite – complete separation



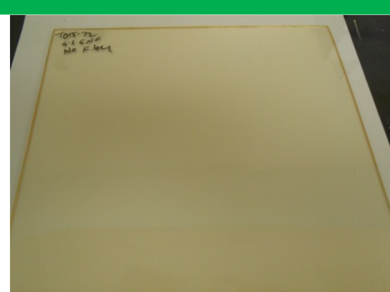
T015 5%cloisite – looks ok

XV-3175 1%CNF – white defects?

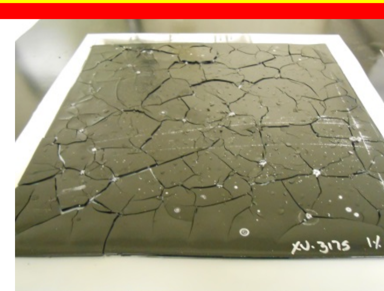
XV-3175 1%SNF – looks ok



T015 1%SNF – looks ok



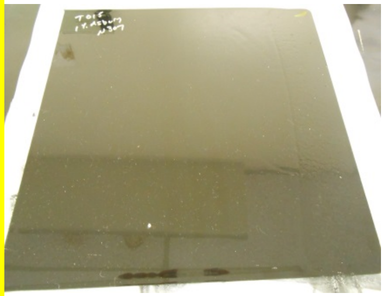
T015 2%SNF – looks ok



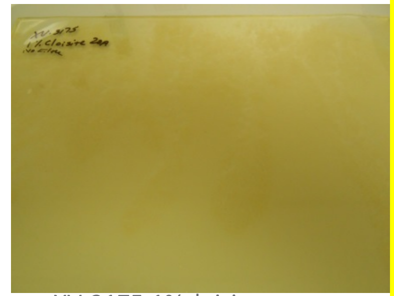
XV-3175 1%Asbury – cracking, white defects

XV-3175 5%Asbury – cracking, white defects

Technical Accomplishment - Matrix Modifications: Catalyst and Filler Interactions


T015 1%Asbury – small white defects


T015 5%Asbury – wrinkling, white defects over large area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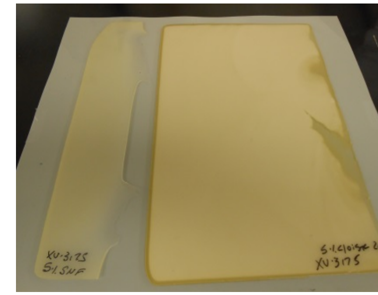
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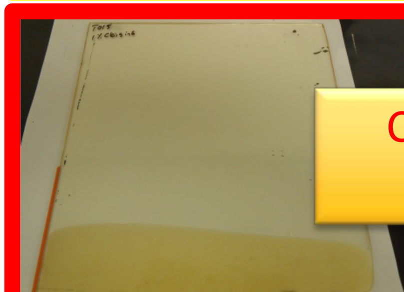
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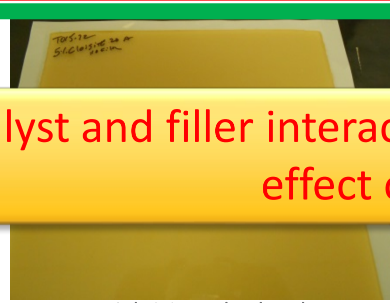
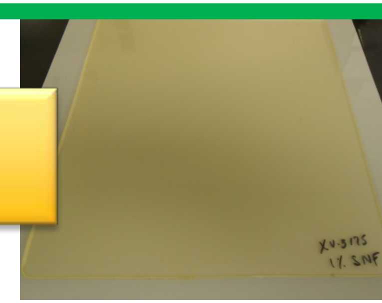
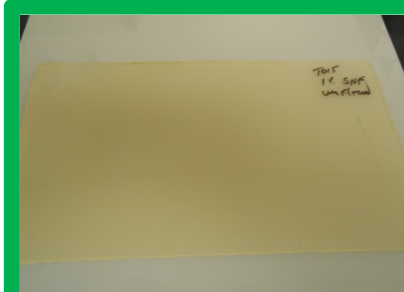

T015 5%cloisite – looks ok


XV-3175 1%CNF – white defects?


XV-3175 1%SNF – looks ok

Catalyst and filler interaction has shown to have an effect on curing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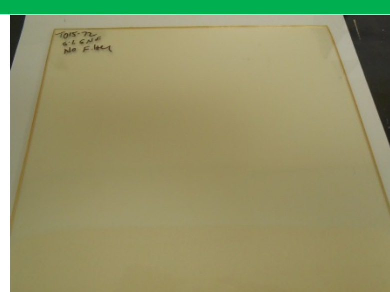
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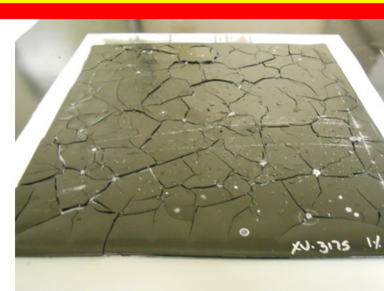
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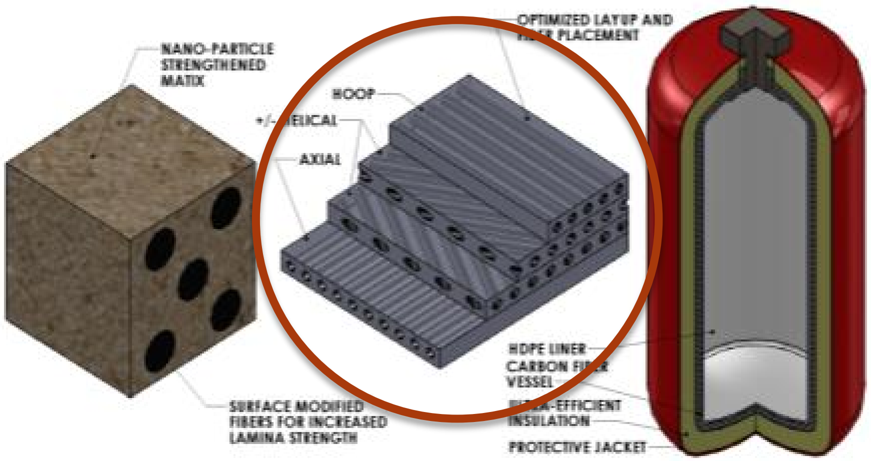
XV-3175 5%Asbury – cracking, white defects

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



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Technical Accomplishment – Alternate Fiber Placement and Multiple Fiber Types

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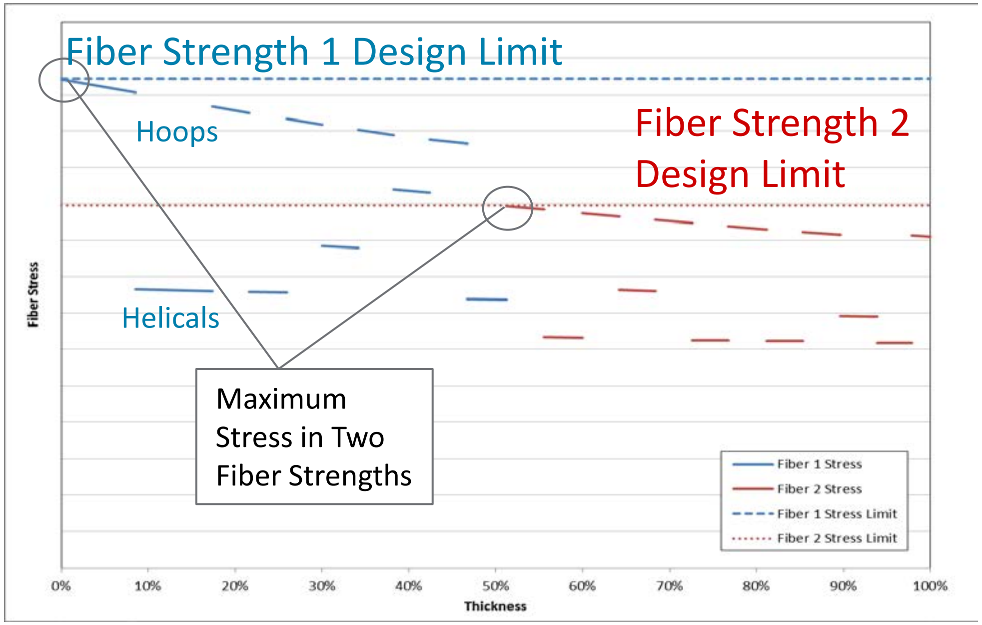

Fiber Properties Used

Material Property	E-Glass	T300	T700	T720	T800
Tensile Strength [ksi]	350	512	711	850	850
Tensile Modulus [Msi]	12.0	33.4	33.4	38.7	42.7
Fiber Count [x1000]	2	12	24	24	24
Yield [ft/lb]	1341	1862	903	1367	1446
Density [lb/in3]	0.093	0.064	0.065	0.065	0.065

Single Fiber Designs Compare to T700 Baseline

Evaluation Criteria	T300	T720	T800
Percent Change in Cost	+19%	+9%	+63%
Percent Change in Mass	+59%	-30%	-30%

Combinations of Modulus and Strength Fiber Designs Compared to T700 Baseline Design

Evaluation Criteria	Hybrid Modulus Design	Hybrid Strength Design
Percent Change in Cost	+38%	-1%
Percent Change in Mass	-34%	-23%

Low and High Angled Helical Combinations Compared to T700 Baseline Design

Evaluation Criteria	Mild Tailoring	Aggressive Tailoring
HAH Percent Change in Cost	-3%	-14%
HAH Percent Change in Mass	-3%	-14%
LAH Percent Change in Cost	-7%	-16%
LAH Percent Change in Mass	-7%	-16%

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Gains in cost and mass savings up to 16% through controlled fiber placement

Technical Accomplishments – Model Validation Matrix for Tank and Material Designs Phase I

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Build	Build Desc.	Build Dep.	Fiber	Resin	Design	Qty. Planned	Qty. Produced	Qty. Tested	Status
1	Baseline	-	T700	HL Epoxy	Baseline	6	6	6	Completed
2	Angle Tailor 1	1	T700	HL Epoxy	Sorted HAH	6	6	0	Built, waiting testing
3	Angle Tailor 2	1	T700	HL Epoxy	All sorted	6	1	0	In-progress
4	Alternative Resin 1	1	T700	TBD	Baseline	6	0	0	Awaiting supply of alternative resin
5	Alternative Resin 2	1	T700	TBD	Baseline	6	0	0	Awaiting supply of alternative resin
6	A/H Ratio Increase 1	1	T700	HL Epoxy	Baseline minus LAHs	6	0	0	At risk, evaluating failure modes
7	A/H Ratio Increase 2	1	T700	HL Epoxy	Baseline minus LAHs	6	0	0	At risk, evaluating failure modes
8	A/H Ratio Increase 3	1	T700	HL Epoxy	Baseline minus LAHs	6	0	0	At risk, evaluating failure modes
9	Fiber Alternative 1	1	T800	HL Epoxy	Baseline	6	0	0	Awaiting production and improved burst equipment
10	Fiber Alternative 2	1	T720	HL Epoxy	Baseline	6	0	0	Awaiting production and improved burst equipment
11	Fiber Alternative 3	1	TBD	HL Epoxy	Baseline	6	0	0	Original choice not available, may substitute or cancel
12	Strength Hybrid	1-4	TBD	HL Epoxy	Baseline	6	0	0	Awaiting alternative fiber testing results to finish designs
13	Modulus Hybrid	1-4	TBD	HL Epoxy	Baseline	6	0	0	Awaiting alternative fiber testing results to finish designs

Technical Accomplishments – Model Validation Matrix for Tank and Material Designs Phase I

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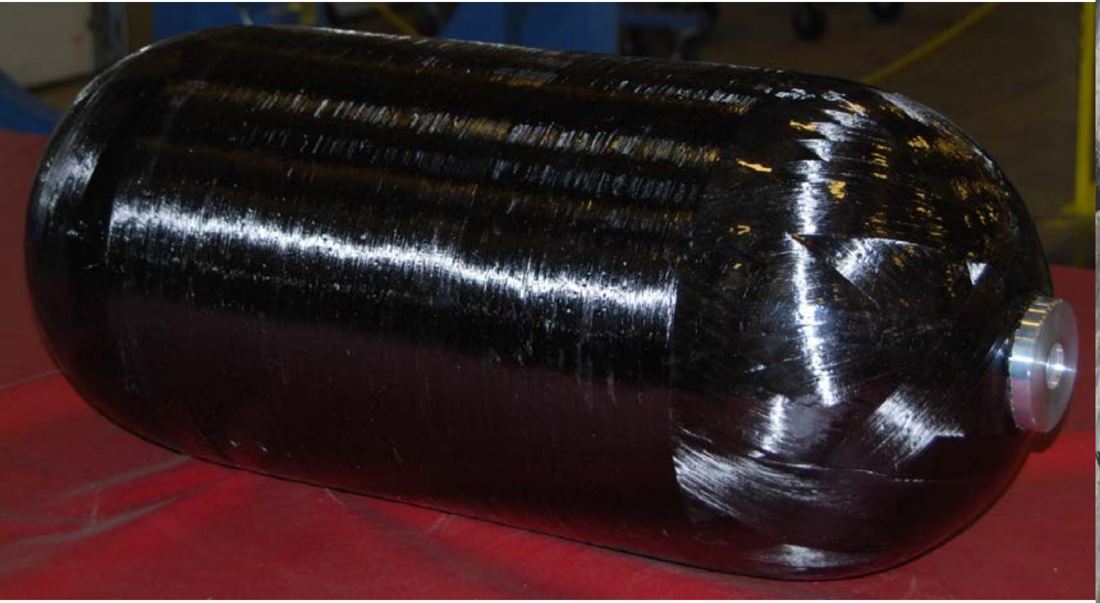
Build	Build Desc.	Build Dep.	Fiber	Resin	Design	Qty. Planned	Qty. Produced	Qty. Tested	Status
1	Baseline	-	T700	HL Epoxy	Baseline	6	6	6	Completed
2	Angle Tailor 1	1	T700	HL Epoxy	Sorted HAH	6	6	0	Built, waiting testing
3	Angle Tailor 2	1	T700	HL Epoxy	All sorted	6	1	0	In-progress
4	Alternative Resin 1	1	T700	TBD	Baseline	6	0	0	Awaiting supply of alternative resin
5	Alternative Resin 2	1	T7						pply of alternative resin
6	A/H Ratio Increase 1	1	T700	HL Epoxy	LAHs	6	0	0	At risk, evaluating failure modes
7	A/H Ratio Increase 2	1	T700	HL Epoxy	Baseline minus LAHs	6	0	0	At risk, evaluating failure modes
8	A/H Ratio Increase 3	1	T700	HL Epoxy	Baseline minus LAHs	6	0	0	At risk, evaluating failure modes
9	Fiber Alternative 1	1	T800	HL Epoxy	Baseline	6	0	0	Awaiting production and improved burst equipment
10	Fiber Alternative 2	1	T720	HL Epoxy	Baseline	6	0	0	Awaiting production and improved burst equipment
11	Fiber Alternative 3	1	TBD	HL Epoxy	Baseline	6	0	0	Original choice not available, may substitute or cancel
12	Strength Hybrid	1-4	TBD	HL Epoxy	Baseline	6	0	0	Awaiting alternative fiber testing results to finish designs
13	Modulus Hybrid	1-4	TBD	HL Epoxy	Baseline	6	0	0	Awaiting alternative fiber testing results to finish designs

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

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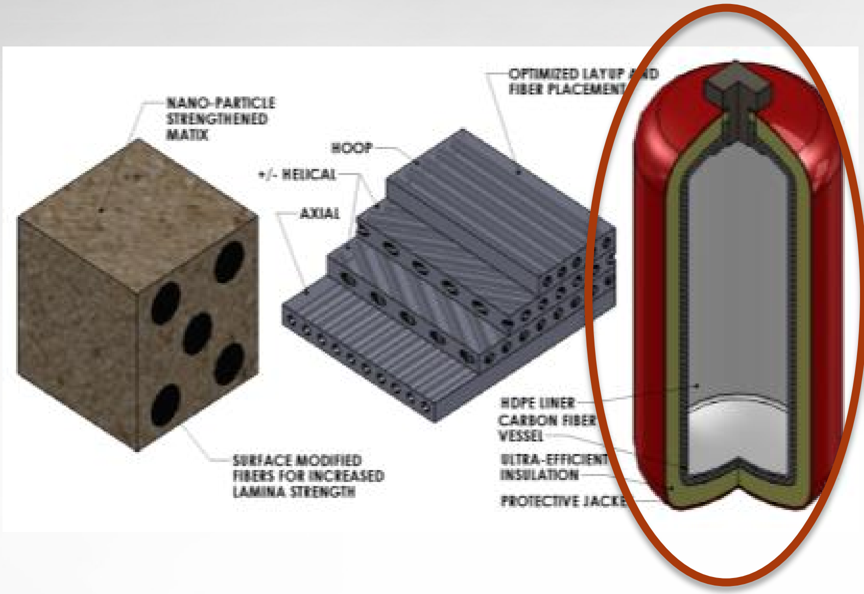
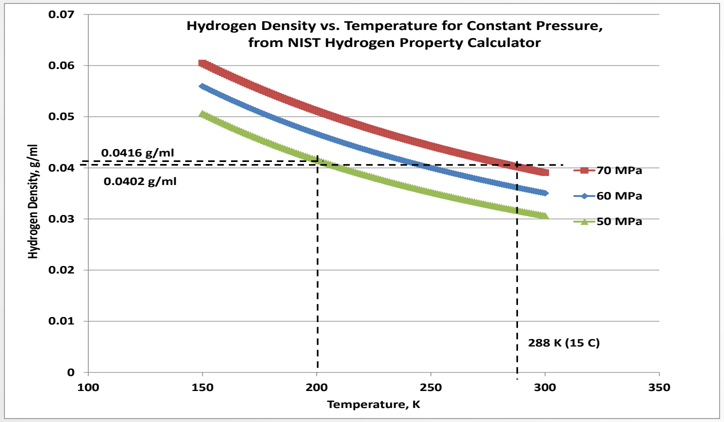
- Assess the operating condition alternatives
- Pros
  - 1. Allows equivalent density at lower pressure which reduces the carbon fiber and cost
  - 2. Lower pressure allows for a thinner, lighter, efficient pressure vessel
- Cons
  - 1. Insulation is required to maintain temperature and extend dormancy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





	Current H_2 Tank	Enhanced H_2 Tank
Operating Conditions	700 bar at 15° C	500 bar at -73° C
Density	40 g/l	42 g/l
Tank Mass	93.6 kg	48.2 kg

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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-Compress vs. Cold Gas Dormancy

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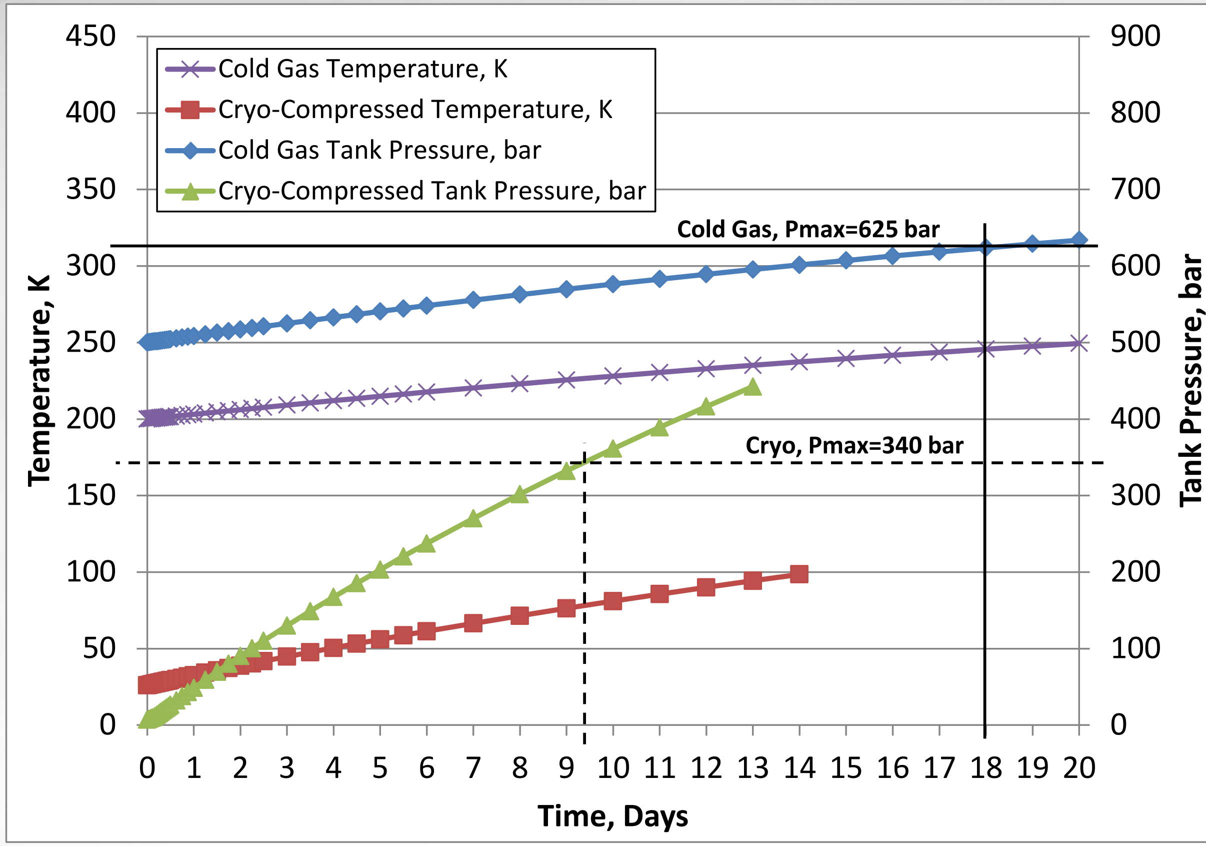
- Cryo-Compressed
  - Initial / final:
    - 26K (-247 C) and 4 bar
    - 77K (-196 C) and 340 bar
  - H2 mass = 9.8 kg
  - Heat and thermal mass:
    - 4.71 W to 4.66 W
    - 59 to 78 kJ/K
  - **9.3 days**
- Cold Gas
  - Initial / final:
    - 200K (-73 C) and 500 bar
    - 248K (-25 C) and 625 bar
  - 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

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Technical Accomplishment - Enhanced Operating Conditions: Insulation Systems

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- 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

Ahluwalia, RK, TQ Hua, JK Peng, S Lasher, K McKenney, J Sinha, and M Gardiner. 2010. *Technical assessment of cryo-compressed hydrogen storage tank systems for automotive applications.* International Journal of Hydrogen Energy. Elsevier, Vol. 35, pp. 4177-4184.

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Technical Accomplishments – Cost Analysis Improvements in Tank Cost Reductions

Case	Useable Hydrogen Mass kg	Composite (fiber + resin) Mass Kg	% Reduction of Composite Mass from Baseline	Est. Tank Cost w/o BOP (without profit) $
1. Baseline, T=288K, P=70 MPa	5.6	93.6	0%	$2,551
2. Lower Cost Resin, T=288K, P=70 Mpa	5.6	93.6	0%	$2,454
3. Nano-Strengthened Resin, T=288K, P=70 Mpa	6.0	87.7	6%	$2,351
4. Fiber Material and Winding Design, T=288K, P=70 Mpa	6.1	83.6	10%	$2,249
5. Cold Gas, Same Outer Volume, T=200K, P=50 MPa	7.0	59.1	36%	$1,637
6. Cold Gas, Resized for 5.8kg H_2, T=200K, P=50 MPa	5.6	48.2	48%	$1,362
PNNL Target 37% Composite Cost Reduction				$1,607
Insulation Margin for 37% total Reduction				$245

37% Tank Cost Savings

Reviewers Comments

- 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

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- 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 (-73ºC) 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

Back Up Slides

Technical Accomplishment - Cost Analysis Reduction Opportunities

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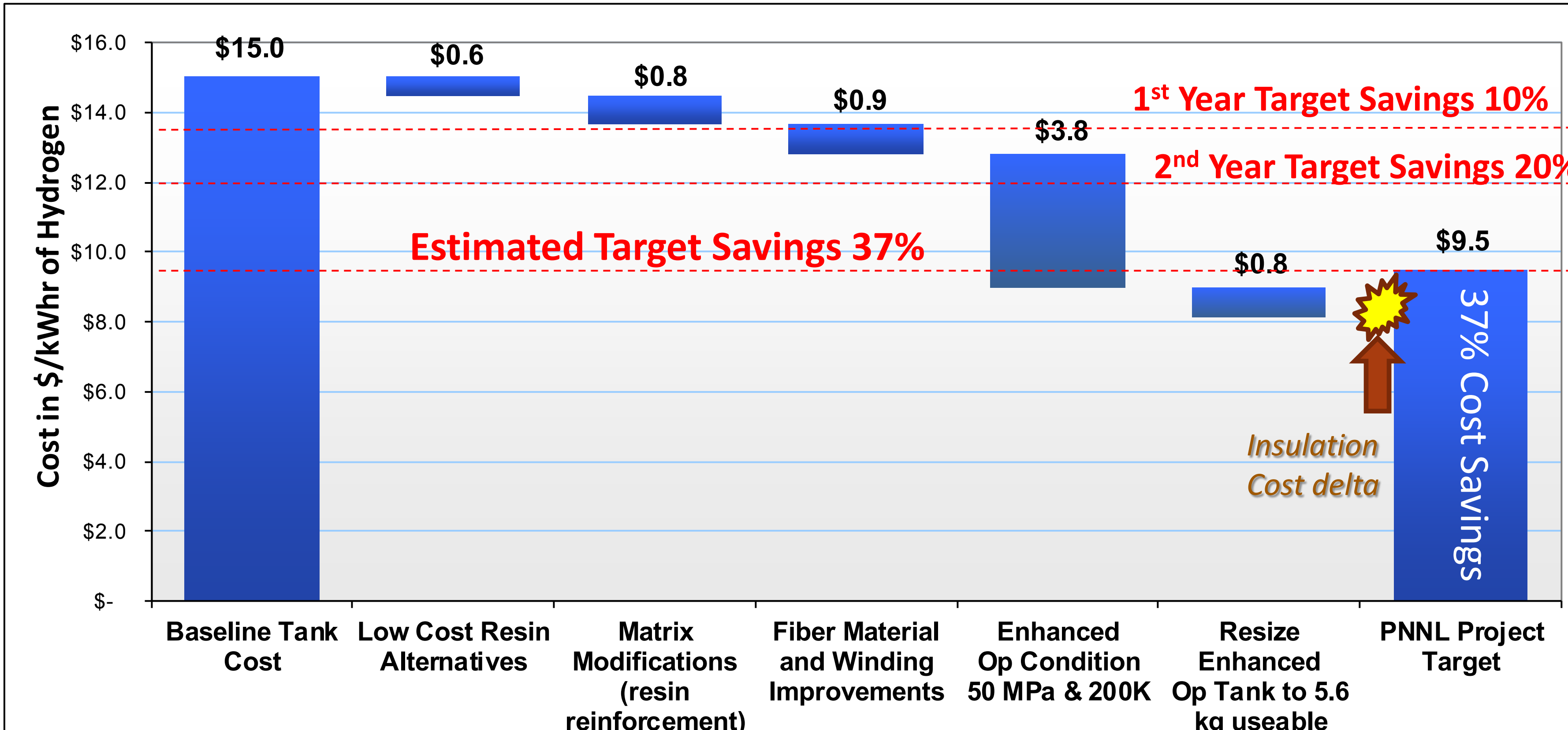
70 MPa H2 Type 4 Tank Cost Analysis Projections

5.6 kg useable H2 (tank only excludes system cost)



Water fall plot from the original baseline from TIAX 2010 results and the project proposal

The AMR slide has been updated to reflect new baseline results from Strategic Analysis AMR 2013

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

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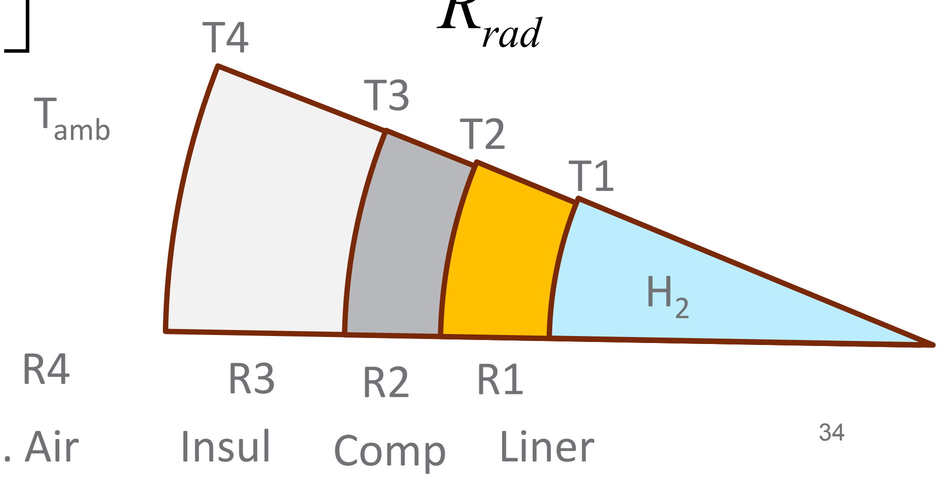
- Radiation heat transfer of Multi-layer Vacuum Insulation (MLVI) with ***n*** layers.
- T_1 = Vacuum jacket temperature
- T_2 = Gas temperature
- Temperature Dependent Thermal Resistance, $\boldsymbol{R_{rad}}$, is updated as the tank temperature increases

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

$$q = \left[\frac{A\varepsilon\sigma(T_4^2 + T_3^2)(T_4 + T_3)}{(1+n)}\right](T_4 - T_3) = \frac{A(T_4 - T_3)}{R_{rad}}$$

- 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

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- 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.