Hydrogen Storage Engineering
CENTER OF EXCELLENCE

Systems Engineering of Chemical Hydrogen, Pressure Vessel, and Balance of Plant for On-Board Hydrogen Storage

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DOE Fuel Cell Technology Program
Annual Merit Review

Washington, DC
May 15, 2012
Technology Development Managers: Ned Stetson and Jesse Adams

U.S. Department of Energy
Energy Efficiency and Renewable Energy
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Project ID: ST005

Overview

- Timeline
  - Start: Feb. 2009
  - Project End: Jan. 2014
    - End Phase 1: 2011
    - End Phase 2: 2013
    - End Phase 3: 2014
  - Percent complete: 55%
- Budget
  - $5.8M Total (PNNL) Program
    - DOE direct funded
    - No cost-share required for National Lab
  - FY12: $895k
  - FY11: $960k
- Barriers
  - A. System Weight and Volume
  - B. System Cost
  - C. Efficiency
  - D. Durability
  - E. Charging / Discharging Rates
  - G. Materials of Construction
  - H. Balance of Plant (BOP) Components
  - J. Thermal Management
  - O. Hydrogen Boil-Off
  - S. By-Product/Spent Material Removal
- Partners

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Lincoln Composites A Member of Hexagon Composites Group
SRNL Savannah River National Laboratory
OSU Oregon State University
HRI UQTR
BASF The Chemical Company
HSM Systems
Ford
United Technologies Research Center
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Relevance: Hydrogen Storage

- Impact to FCT Program
  - Demonstrate hydrogen storage system that meets DOE 2015 targets for light duty vehicles using chemical hydrogen storage
  - Apply materials discoveries from the Materials Centers of Excellence
  - Discover/develop engineering solutions to overcome material's deficiencies
  - Identify minimal performance for materials to be applicable in engineered H_2 storage systems for light duty vehicles.
- Hydrogen Storage Community at Large
  - Develop and/or advance modeling and simulation tools for the optimum design and engineering of on-board storage systems
  - Provide functional prototype systems available to OEMs
  - Provide engineering methodologies, analysis tools, and designs applicable to stationary storage and portable power applications
  - Demonstrate on-board storage to advance state of the art.
  - Identify, develop and validate critical components either for performance, mass, volume, or cost.

Approach:

- PNNL's Roles Supporting Engineering Center Structure
  - Lead Technology Area (TAL) for Materials Operating Requirements
  - Coordinate activities as the Technology Team Lead (TTL)
    - Bulk Materials Handling (Transport Phenomena)
    - Pressure Vessels (Enabling Technologies)
    - Manufacturing and Cost Analysis (Performance Analysis)
  - Liaison to VT Program projects and resources
- Technical Objectives of PNNL Scope:

Chemical Hydrogen

- Design chemical hydrogen H_2 storage system & BOP components
- Develop system models to predict mass, volume, performance
- Reduce system volume and mass while optimizing storage capability, fueling and H_2 supply performance

All Systems

- Mitigate materials incompatibility issues associated with H_2 embrittlement, corrosion and permeability
- Demonstrate the performance of economical, compact lightweight vessels for hybridized storage
- Guide design and technology down selection via cost modeling and manufacturing analysis
- Perform value engineering of BOP to minimize cost, volume and mass

- Phased/ gated progressions aligning with HSECoE go/no-go decisions

FY12 Objectives

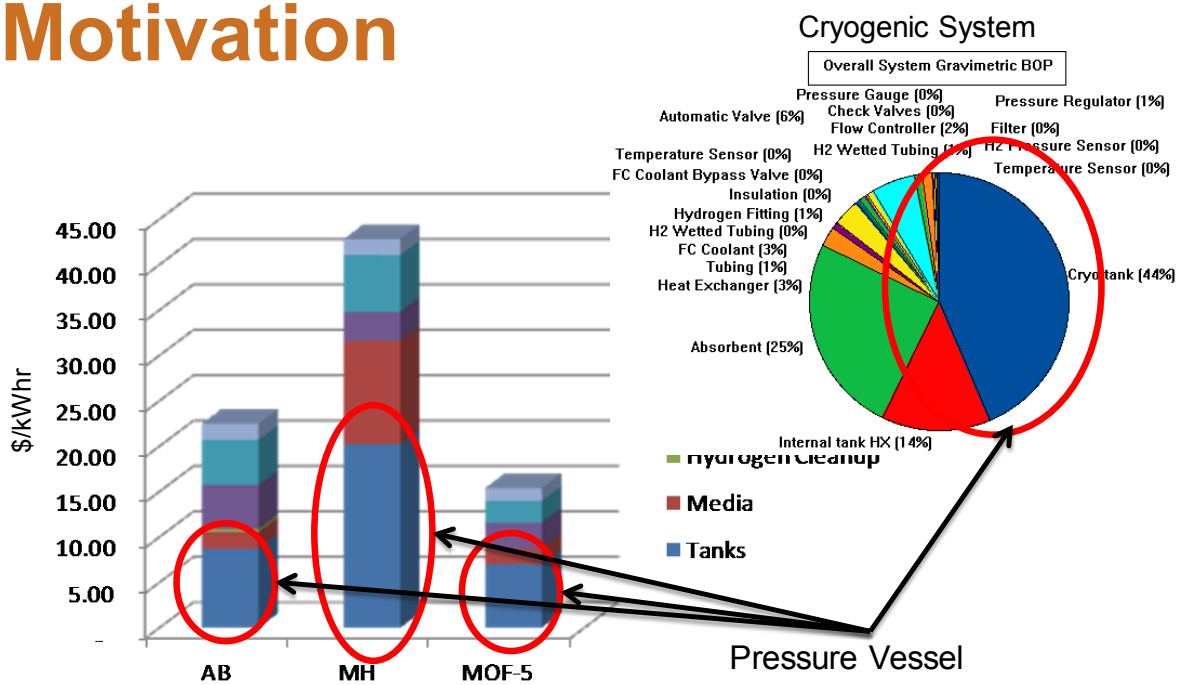
- Pressure Vessel
  - Exercise model to assess materials and design options
  - Optimize vessel design in terms of cost
  - Assess chemical compatibility of polymer liners
  - Assess vessel cost as function of pressure
- Chemical Hydrogen Storage Design
  - Validate models and concepts via experiments
  - Assess feasibility of liquid-slurry chemical hydrogen storage
  - Assess feasibility of heat exchanger
  - Assess feasibility of slurry use with heat exchanger, pump, valves.
- Balance of Plant
  - Maintain BOP library
  - Size components (heat exchangers, valves, pumps,…)
  - Determine material compatibility
  - Identify where improvements can be made
- Cost Modeling

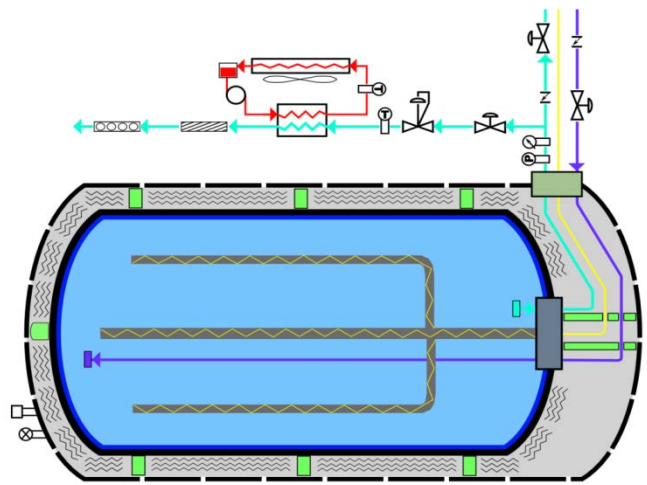
Accomplishments: Milestones FY12

Q3	Task 1	Complete Development of Simulink® AB Slurry Storage Model
Q4	Task 1	Report on sensitivity study to determine operating envelops for viscosity, heat of reaction, flocculation/setting.
Q4	Task 1	Report on feasibility to identify/develop a radiator/heat exchanger capable of cooling the effluent from ~525K to ~360K having a mass less than or equal to 1.15kg and a volume less than 10.9 liters
Q2	Task 2	Report on feasibility to achieve a 40 wt% AB slurry with viscosity <1500cp pre and post dehydrogenation with comparable kinetics.
Q3	Task 4	Report on feasibility to identify BoP materials suitable for the Chemical Hydrogen system to have a system mass no more than 41kg and a system volume no more than 57 liters
Q4	Task 4	Report on ability to identify Type IV tank liner materials suitable for 40K operation having a mass less than or equal to 8kg and a volume less than 3 liters (2.55mm thickness)
Q4	Task 4	Report on ability to identify BoP materials (excluding internal HX, external HX, and combustor) suitable for 60 bar cryogenic adsorbent system having mass less than 17 kg and a volume less than 18.5 liters.
Q1	Task 5	Update Cost Analysis for F2F8 Meeting.
Q2		**Go/No-go assessment of endothermic vs exothermic materials (for the Engineering Center, not DOE)**
Q3		**Go/No-go assessment of liquid vs slurry**

Pressure Vessel Motivation

- Vessel is high cost, high mass
- Initial work (FY10-11) based on 200-250 bar pressure vessel
  - Limited to Type III
- Partners found lower pressure options
- New work (FY12) examine trade-off in pressure and volume for optimized gravimetrics
  - Expand tank types to include Type I and Type IV





Pressure vessel improvement needed to Achieve Mass and Cost Targets

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Different Pressure Vessel Types

	Pro	Challenges
Type I- all metal pressure vessel	• Low outgassing (Limited to hydrogen) • Lower cost	• Higher mass compared to Type IV • Tank fabrication limitations with aluminum
Type II - metal with overwrap cylinder section	• Reduced liner thickness at low pressures	• Large mass penalties at high pressure
Type III - metal lined, composite overwrap	• Previously demonstrated	• Fatigue life challenges
Type IV – polymer lined composite overwrap	• Lightweight	• Not demonstrated • Higher Cost

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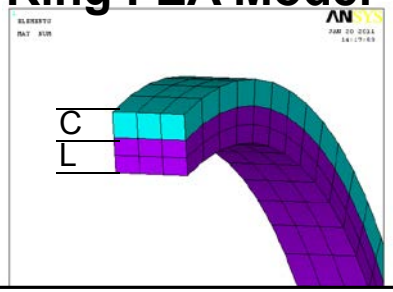
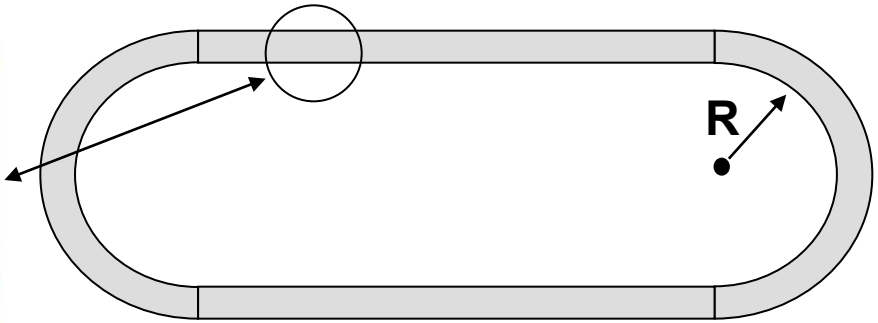
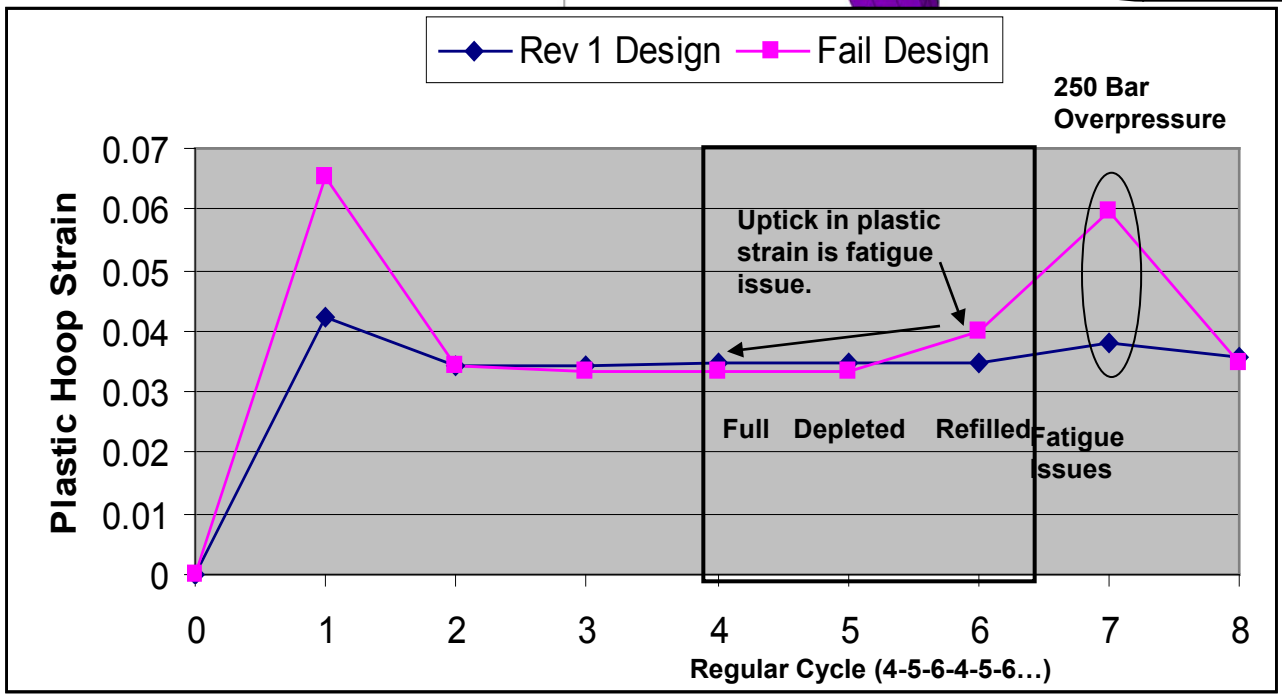
Accomplishments: Type III Wall Cylinder FEA for Tank Thickness

C = composite wall thickness
L = liner wall thickness.
When C/R and L/R ratios are fixed, stress states are constant with R.

Ring FEA Model







Rev 1 = acceptable design, plastic hoop strain remains <u>constant</u> during normal operation.

Fail design = not acceptable from fatigue limits, plastic hoop strain goes from 3.5% to 4.0% back down to 3.5% every cycle.

If the 250 Bar overpressure state becomes part of the normal operation cycle even Rev1 could have fatigue problems.

200L, 200 Bar Cryo (77-120K)
Fail design 7.5mm liner, 7.2 mm shell
Rev 1 design 9.0mm liner, 7.2mm shell

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Accomplishments: Type III Wall Cylinder FEA for Tank Thickness

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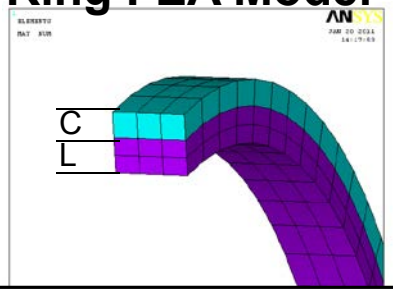

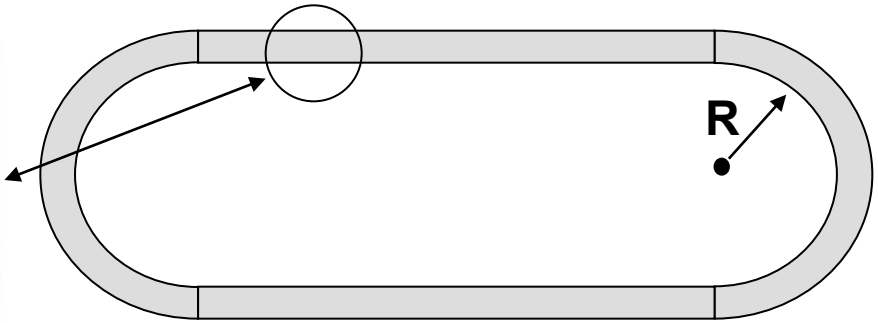

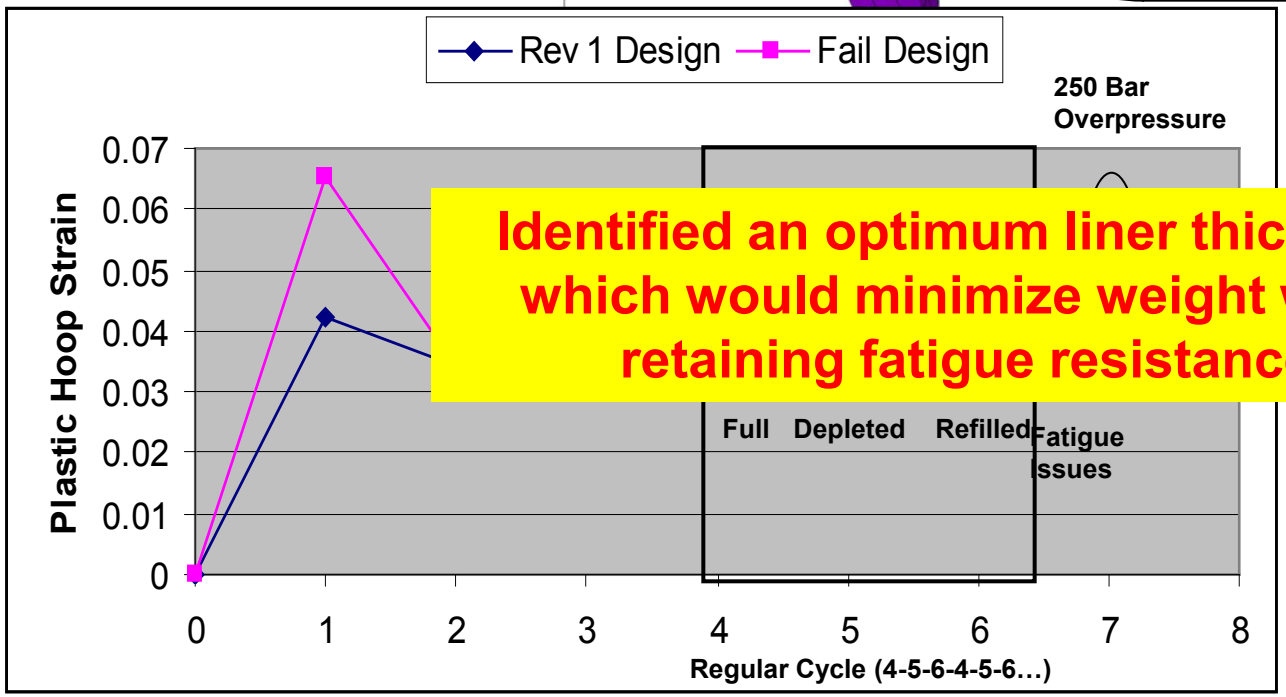

Identified an optimum liner thickness which would minimize weight while retaining fatigue resistance

Rev 1 = acceptable design, plastic hoop strain remains ring normal operation.

= not acceptable from s, plastic hoop strain .5% to 4.0% back down to 3.5% every cycle.

If the 250 Bar overpressure state becomes part of the normal operation cycle even Rev1 could have fatigue problems.

200L, 200 Bar Cryo (77-120K)
Fail design 7.5mm liner, 7.2 mm shell
Rev 1 design 9.0mm liner, 7.2mm shell

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Accomplishments: Cryogenic Test Plan for Polymer Liner Material (Type IV Vessel)

Can we use a type IV tank in cryogenic conditions?

- Cryogenic dewar setup on large load frame
  - Grip design changes
- H_2 Permeation (Lincoln)
- Tensile
- Fatigue
- Weld strength (Lincoln to weld)
- Impact (Lincoln)
- Coefficient of Thermal Expansion
- Dynamic Mechanical Analysis
- Materials
  - HDPE – Completed
  - Kynar® – Completed
    - Homopolymer
    - Copolymer
  - Halar® – Completed,
  - Kel F®
  - Polytetrafluoroethylene
  - Nylon
  - Ethylene Vinyl alcohol
  - Others
  - Wound composite

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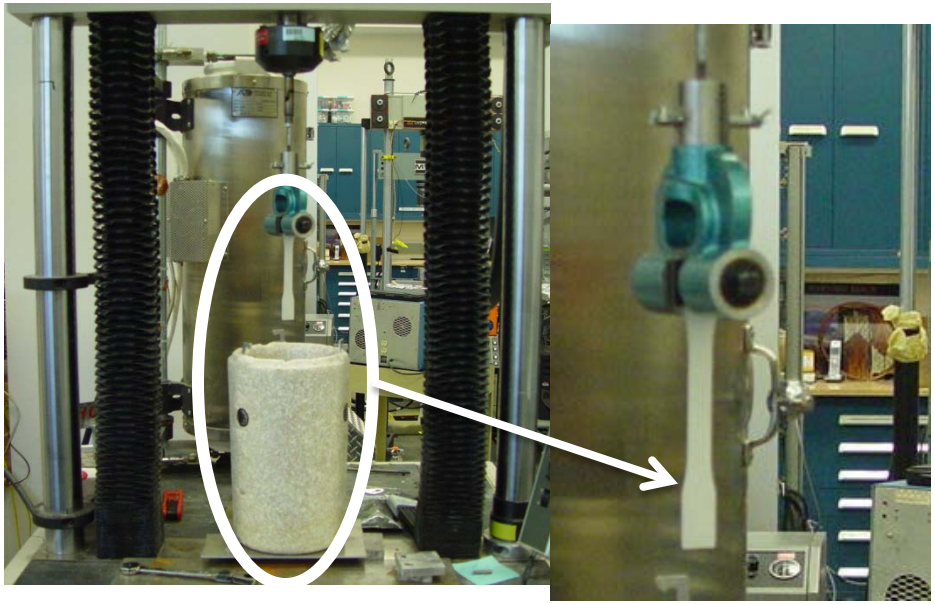
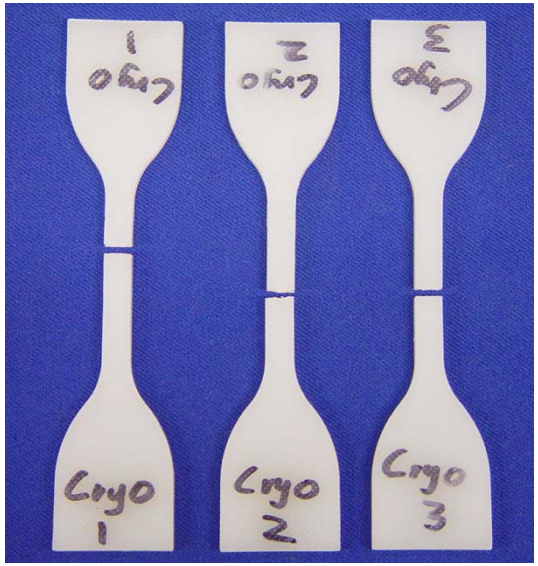
Accomplishments: Cryogenic Test of Liner Materials

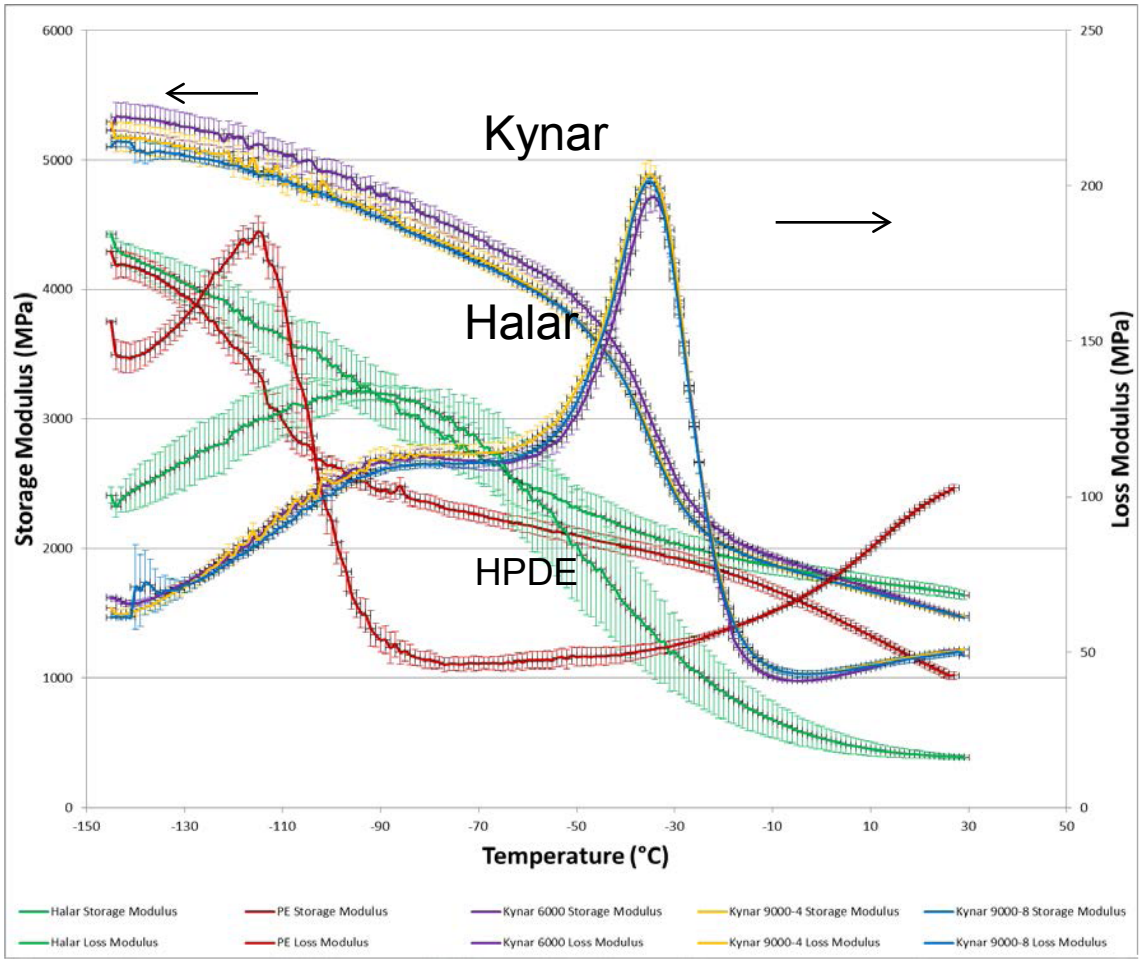


Halar Room Temp.
TS: 42 MPa
Modulus: 1.6 GPa
Elongation: 20%

Halar in Liquid N_2
TS: 143 MPa
Modulus: 4.4 GPa
Elongation: 4.5%



Accomplishments: Dynamic Mechanical Analysis for Type IV Liner Materials



Sample	Storage Modulus - 145°C (GPa)	Glass Transition T_g(°C)
HDPE	**4.29**	**-112.50**
Halar	**4.43**	**-77.30**
Kynar 6000	5.23	-33.63
Kynar 9000 44ccm	5.29	-35.28
Kynar 9000 88ccm	5.10	-34.93

Halar and HDPE are the best performers to date at cryogenic temperatures

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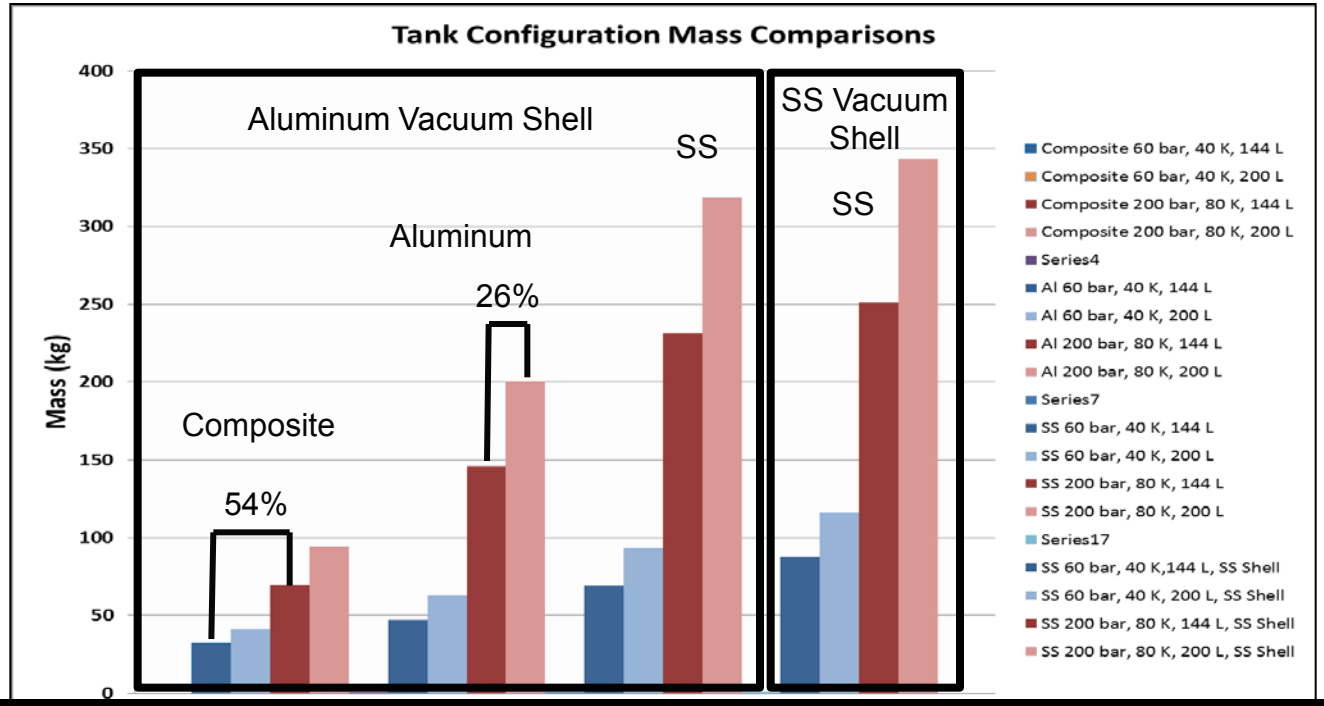
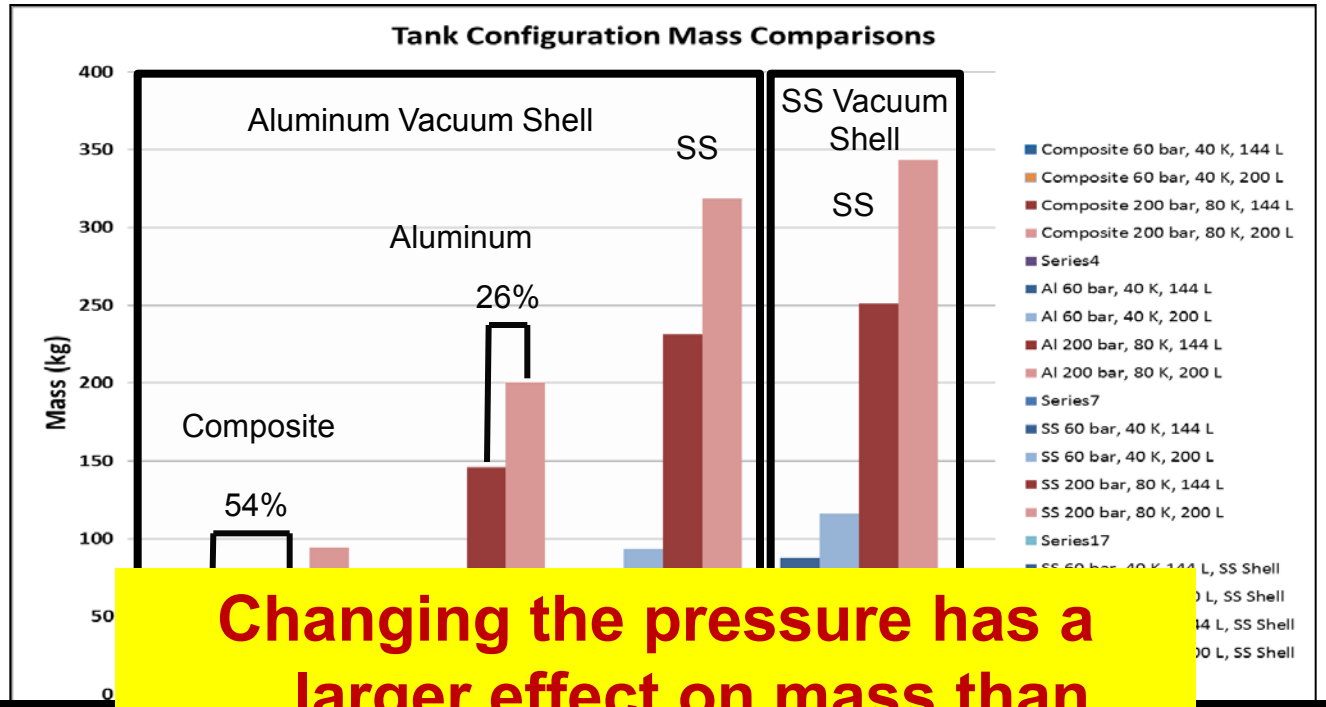
Accomplishments: Sensitivity of Tank Type Mass Relative to Pressure and Volume



Tank Configuration Shell/Inner tank	144 L		200 L		60 bar		200 bar	
	60 bar	200 bar	60 bar	200 bar	144 L	200 L	144 L	200 L
	Increase Pressure		Increase Pressure		Increase Volume		Increase Volume	
Al/Composite/Al (III)	+54% wt		+56% wt		+22% wt		+26% wt	
Al/Al (I)	+68% wt		+69% wt		+25% wt		+27% wt	
SS/Al (I)	+70% wt		+71% wt		+26% wt		+27% wt	
SS/SS (I)	+65% wt		+67% wt		+24% wt		+27% wt	

Accomplishments: Sensitivity of Tank Type Mass Relative to Pressure and Volume



Changing the pressure has a larger effect on mass than changing the volume

Tank Configurat Shell/Inner tank				200 bar
			L	144 L 200 L
	Increase Pressure	Increase Pressure	Increase Volume	Increase Volume
Al/Composite/Al (III)	+54% wt	+56% wt	+22% wt	+26% wt
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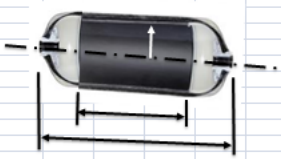
SS/SS (I)	+65% wt	+67% wt	+24% wt	+27% wt

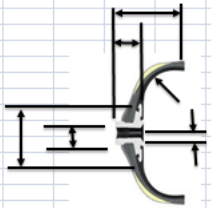
Pressure Vessel Next Step- Combine predictive models with cost models

Home Insert Page Layout Formulas Data Review View Add-Ins Acrobat

I105

1		Input to Tank Model					
2		Output from Tank Model					
3							
4	Lookup row			Modeled Version	Case		
5							
6	1	Selected Column	3		1	2	3
7	2	Modeled		Current	Thiokol	Exp. 1	Current
8	3		Tank Parameters				
9	4	Operating Pressure	bar	100	100	100	100
10	5	Operating Temperature	C	120	120	120	120
11	6	Overall Tank Length	cm	95	95	95	95
12	7	Tank Radius	cm	25	25	25	25
13	8	Tank Volume (Inside Liner Volume)	ccm	151000	151000	151000	151000
14	9	Boss Opening	cm	10	10	10	10
15	10	Tank Type (I, II, III, IV)		IV	IV	IV	IV
16	11						
17	12						
18	13		Boss Production				
19	14	Boss ID	cm	4	4	4	4
20	15	Boss OD	cm	0.05	0.05	0.05	0.05
21	16	Boss base ID	cm				
22	17	Boss base thickness	cm				
23	18	Boss finished volume	ccm				
24	19	Boss bar stock OD	cm				
25	20	Boss bar stock length	cm				
26	21	Boss Bar stock starting volume	ccm				
27	22	Material	Type	AL 6061-T-6		AL 6061-	AL 6061-T-6
28	23	Material density	kg/ccm	0.098			
29	24	Mass of aluminum bar stock	kg				
30	25	Mass of aluminum boss finished	kg				
31	26	Wastage factor	%	0.02	0.02	0.02	0.02
32	27	Boss Material cost/kg	$/kg	9.6	9.6	9.6	9.6
33	28	Program time	hrs	0.25	0.25	0.25	0.25
34	29	CNC machine time	hrs	0.25	0.25	0.25	0.25
35	30	Tooling	$				
36	31	Machinist I Labor for machining	hrs	0.5	0.5	0.5	0.5
37	32	Machinist II Labor for machining	hrs	0.5	0.5	0.5	0.5
38	33	Machinist I Cost/hour	$/hour	24	24	24	24
39	34	Machinist II Cost/hour	$/hour	28	28	28	28
40	35	Production rate for boss	units/hr	4	4	4	4
41	36	packaging	$				
42	37	Anodizing	$/boss				
43	38	Material Cost per boss	$/boss				
44	39	labor cost per boss	$/boss				
45	40	Manufacturing cost per boss	$/boss				
46	41	Manufacturing overhead per boss	$/boss				





Model | Manufacturing Costs | Winding Time Equation | Insulation | Specific Density | Named Ranges

Ready 40%

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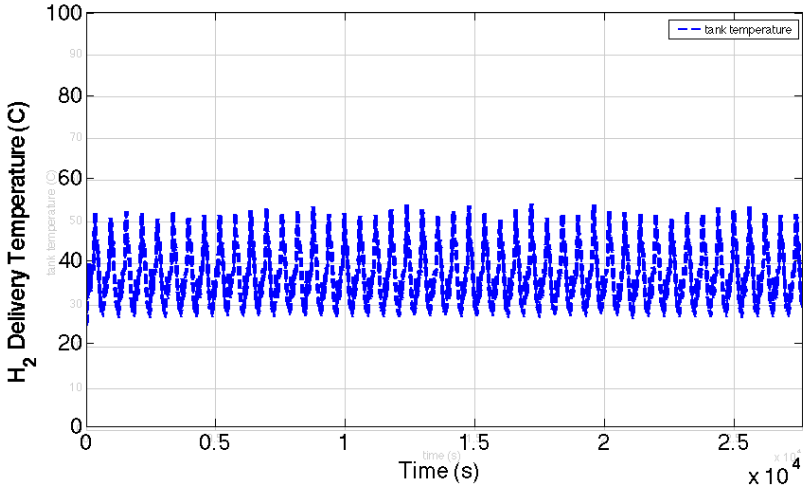
Chemical Hydrogen Storage Development

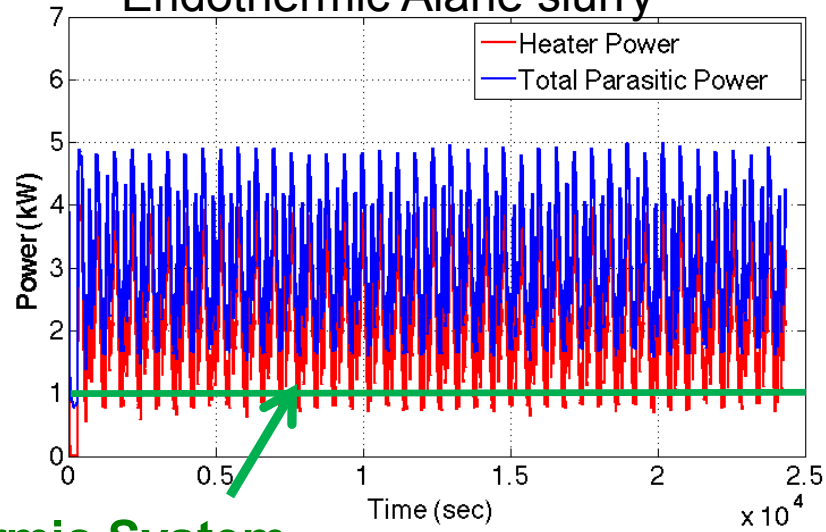
- Modeling and Validation
  - Operational envelope
  - Validation (Reviewer section)
    - Radiator/Heat Exchanger
      - 50% reduction in mass/volume from baseline
      - Validation underway
    - Pump
      - 44% reduction in mass from baseline
      - Validation underway
    - Displacement volume tank – FY13
- Liquid-Slurry development
  - Endothermic liquid-slurry: Alane surrogate leverage BNL's work
  - Exothermic liquid-slurry: Ammonia borane surrogate

Accomplishments: Chemical Hydrogen Storage Results of US06 Drive Cycle in Framework

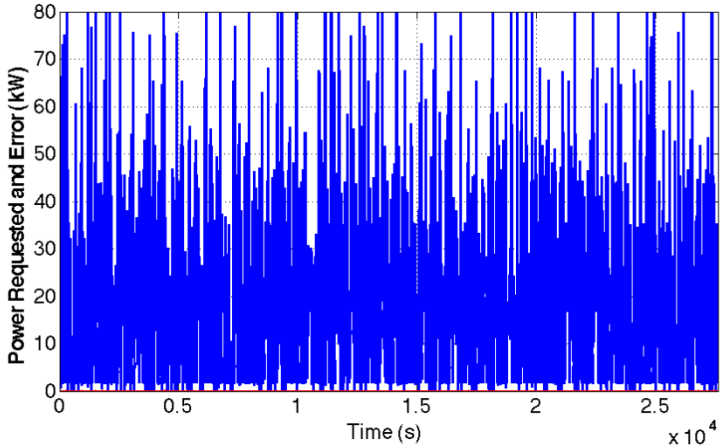


H_2 Delivery Temperature (C)

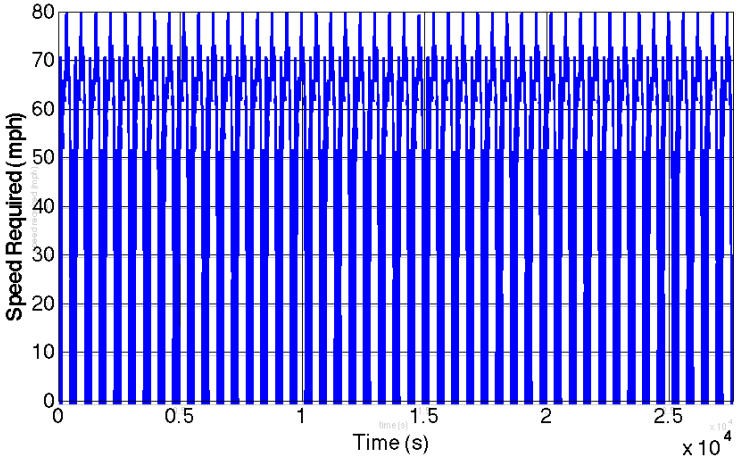
Endothermic Alane slurry



Exothermic System Parasitic Power



Power Requested by Vehicle (kW)



Vehicle Speed (mph)

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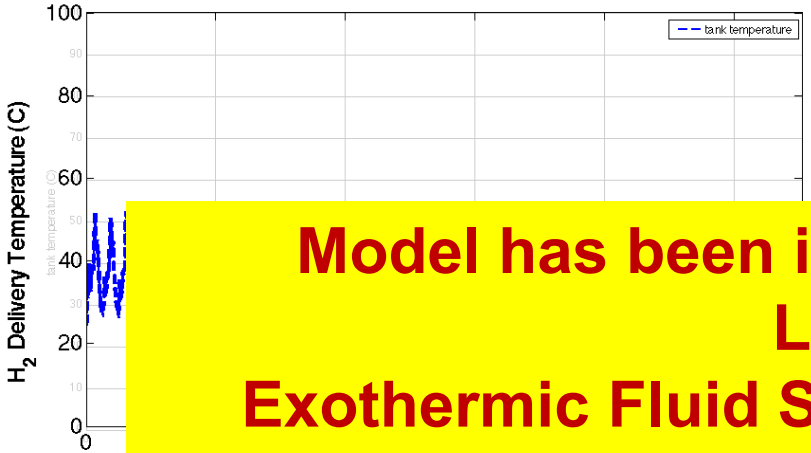
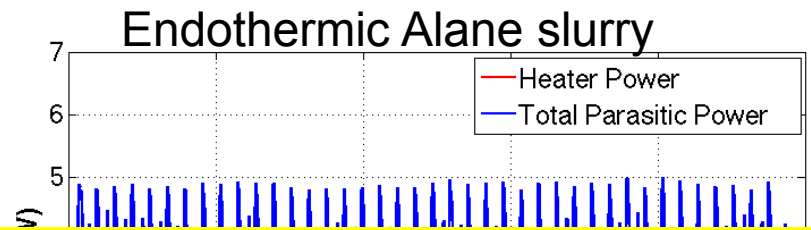
Accomplishments: Chemical Hydrogen Storage Results of US06 Drive Cycle in Framework





Model has been implemented into the Vehicle-Level Framework

Exothermic Fluid System:

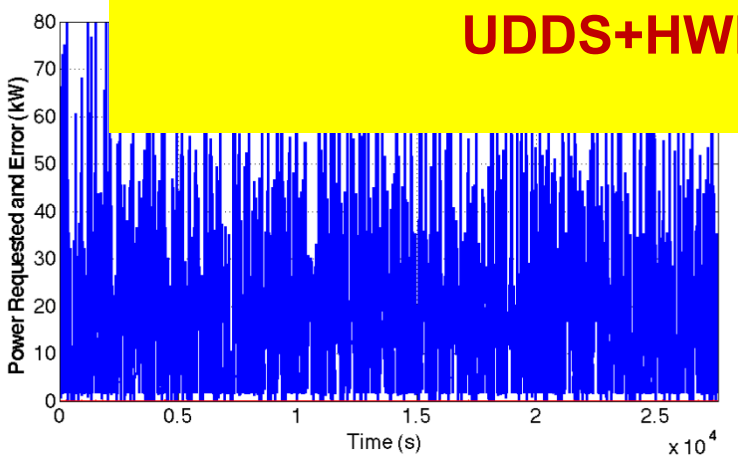
UDDS+HWFET fuel economy 51.2 mpgge

Efficiency > 90%

Endothermic Fluid System:

UDDS+HWFET fuel economy 43.3 mpgge

Efficiency < 90%



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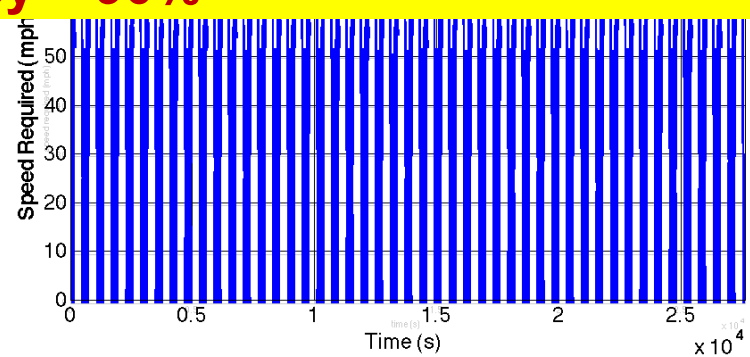

Vehicle Speed (mph)

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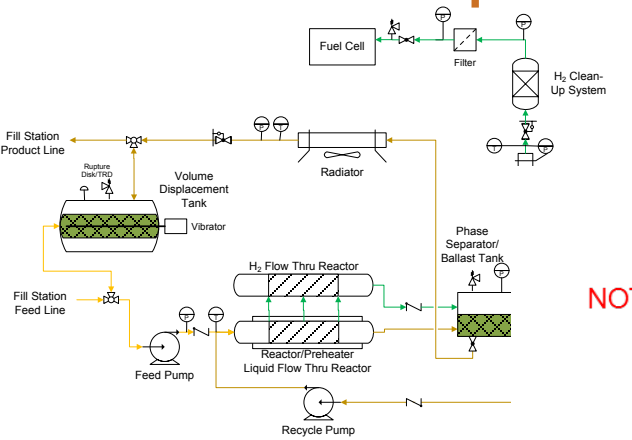
Accomplishments: Exothermic Chemical Hydrogen System

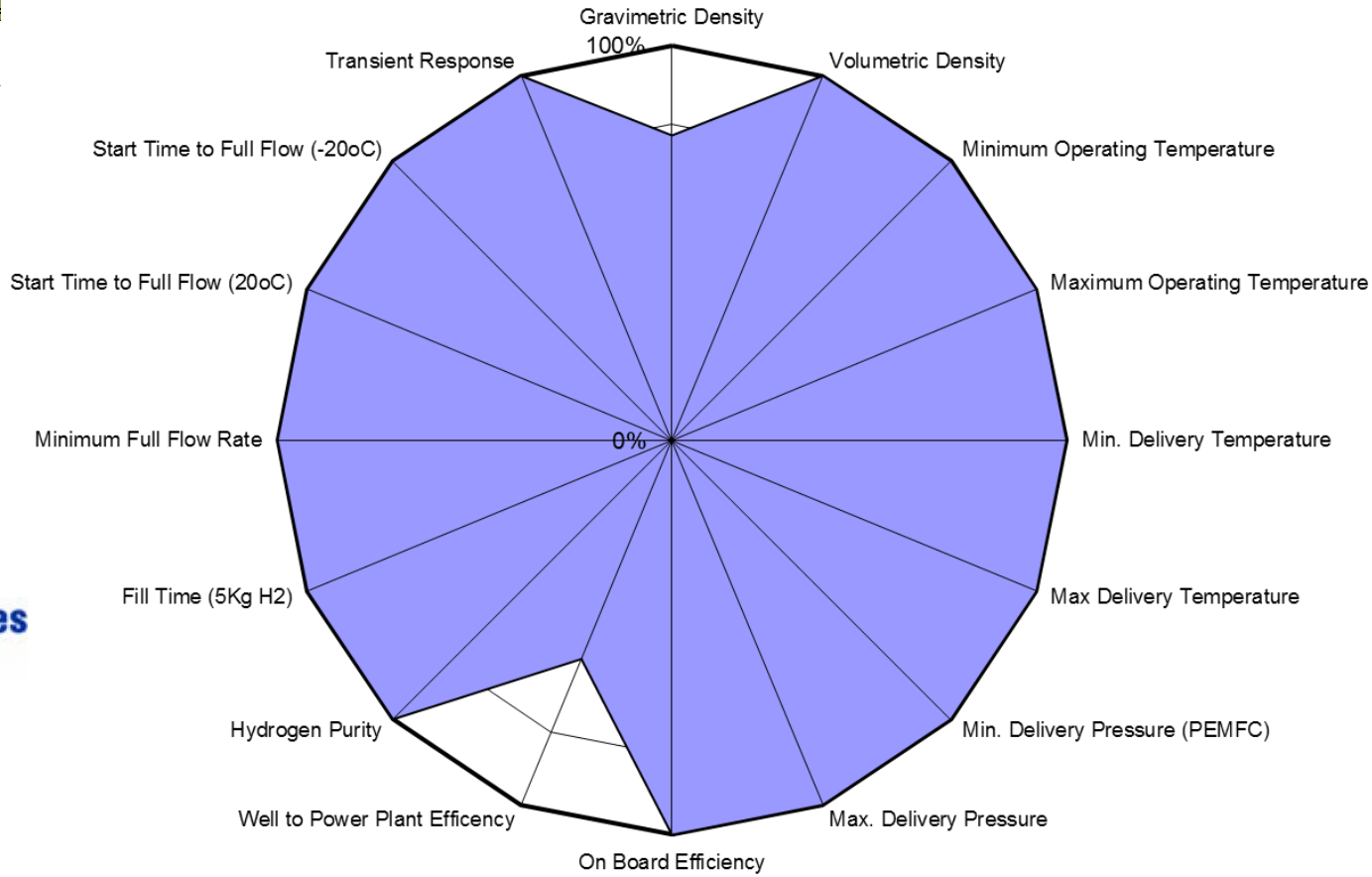
Liquid Slurry Ammonia-Borane: 2017 Targets



14 Targets Met at 100%
All 16 Targets Met > 60%

DOE 2017 Targets for Liquid Ammonia Borane Flow Through System

NOTE: All metrics that exceed DOE targets are plotted at 100% to keep the diagram scaling intact



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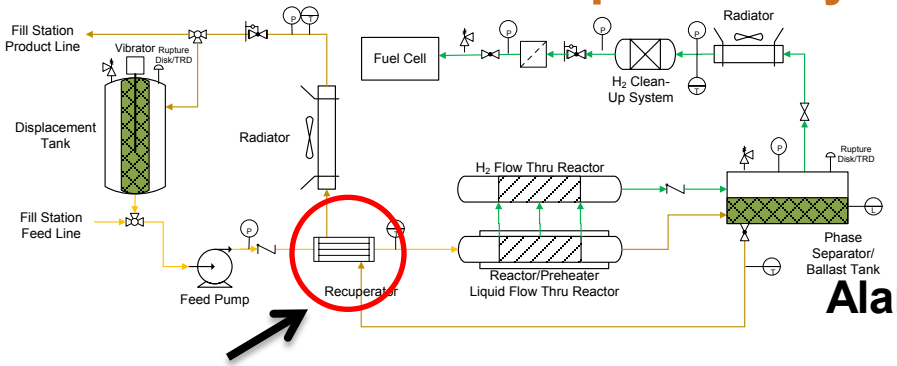
Accomplishments: Endothermic Chemical Hydrogen System (Alane)

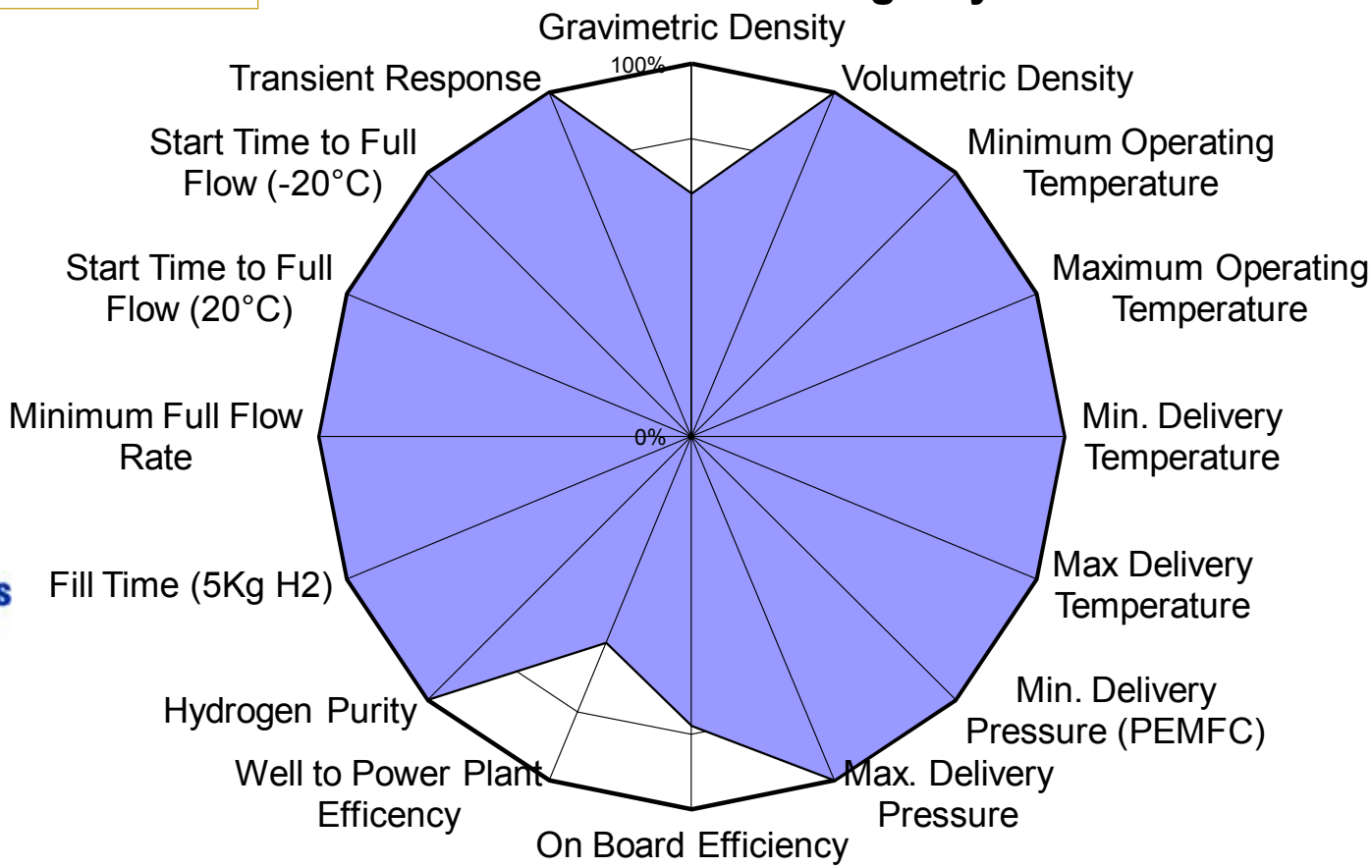
Liquid Slurry Alane: 2017 Targets



Recuperator

13 Targets Met at 100%
All 16 Targets > 60%

DOE 2017 Targets for Liquid Alane 50 wt% Flow Through System



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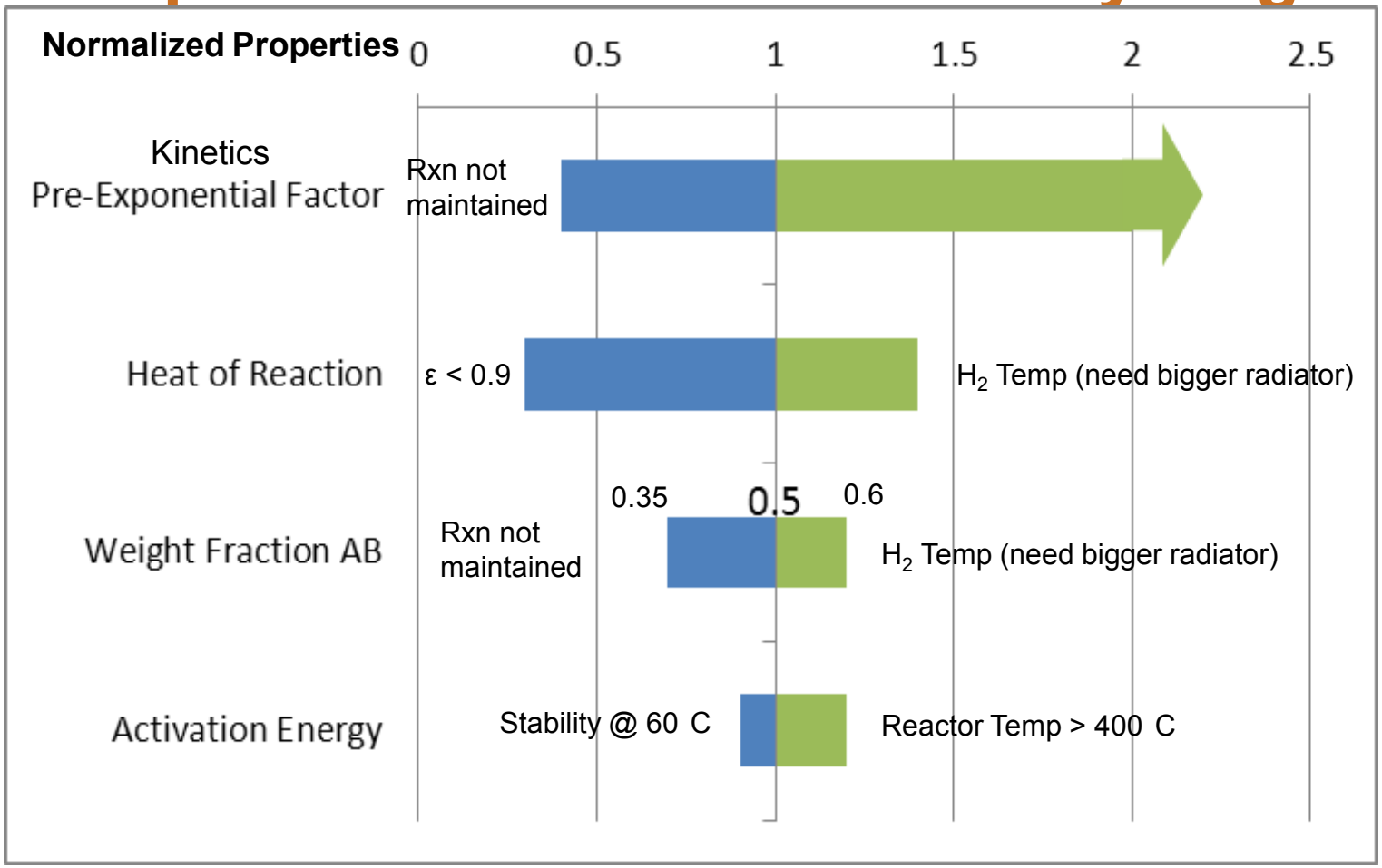
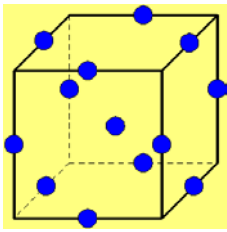
Accomplishment: Sensitivity Analysis Tornado Chart "Operability Envelope"- Example Exothermic Chemical Hydrogen



- **Assumes no changes to the design**

Accomplishments: Box-Behnken Type Sensitivity Analysis Example Endothermic CH (Alane)



Normalized to Alane Properities					
Kinetics	Heat of Reaction	Alane Wt%	Reactor Length (m)	US06 On-Board Efficiency	Fraction of DOE Mass Target
1	0.5	70	1.83	91%	78%
1	0.5	50	1.83	91%	63%
1	0.75	70	1.83	89%	78%
1	0.75	50	1.83	89%	63%
1	1	50	1.83	87%	63%
1	1	70	1.83	87%	78%
10	0.5	70	1.83	92%	78%
10	0.5	50	1.83	92%	63%
10	0.75	70	1.83	89%	78%
10	0.75	50	1.83	89%	63%
10	1	50	1.83	87%	63%
10	1	70	1.83	87%	78%

↑Alane Wt% → ↓ System Mass

↓ ΔHrxn → ↑ Efficiency

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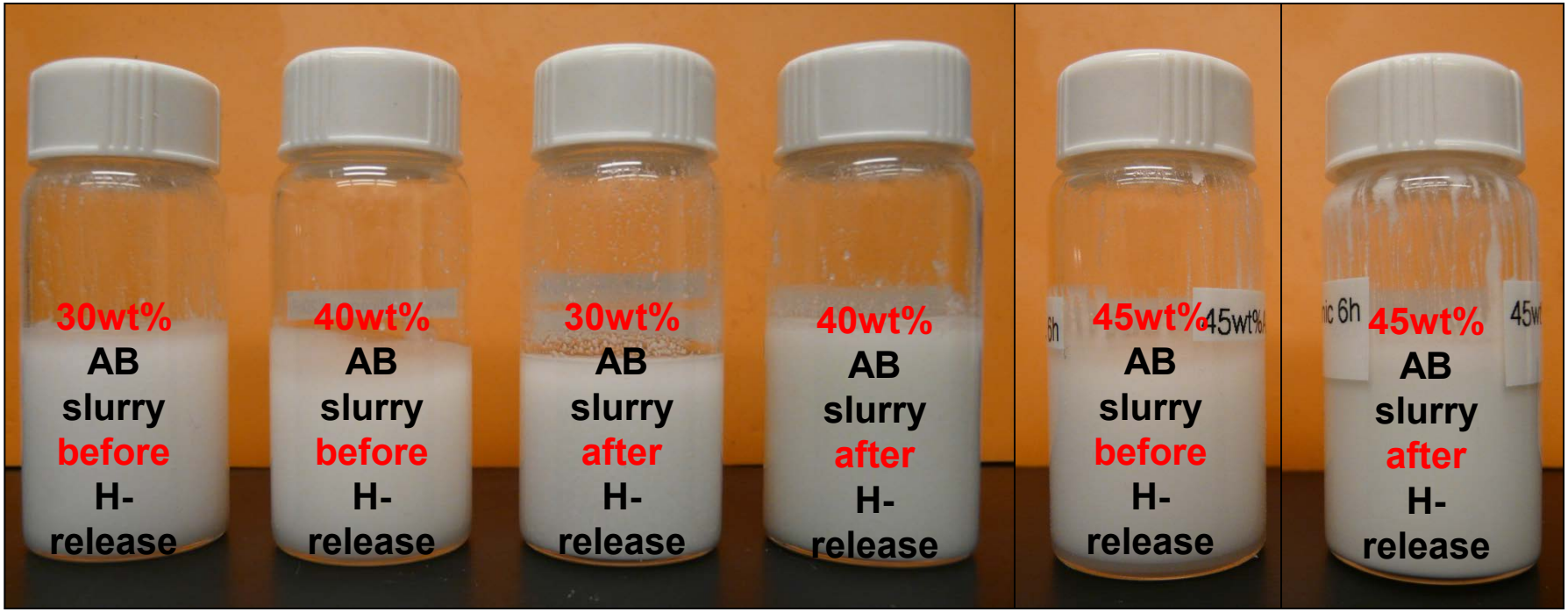
Accomplishments: Exothermic Liquid-Slurry Development (AB Slurry)

- Evaluated
  - 4 candidate carrier liquids
  - 6 additives
  - 7 synthesis techniques
- Results
  - Achieved 45 wt% AB in silicon oil[1] (>7 wt% H_2)
    - Synthesis – Sieve followed by sonication
    - Settling/flocculation evaluated
    - Viscosity measurements completed
    - Kinetics verified
  - Discontinued
    - 3 carrier liquids
    - 3 additives
    - 6 synthesis technique

* We recognize that silicon oil is not an ideal fluid.

Accomplishments: Liquid Slurry



Material remains a liquid-slurry before and after release

Fresh slurry no settling/flocculation for 3+ months

Spent slurry settling within several hours

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~600cP Viscosity of AB Slurry (45wt%) Before & After H-release

Anton Paar MCR301 rheometer with a bob/cup set-up

	AB slurry before H-release	AB slurry after H-release	Measured Temp. (°C)
Plastic viscosity (cP)	~ 617	~ 442	25
Yield stress (Pa)	~ 48	~ 3.7	25

Experimental conditions & Key results:

- *Measuring the shear stress (τ) in the range of 0-1000 1/sec shear rate ($\dot{\gamma}$)*
- *Basic rheological model*

  $\tau = \tau_y + \eta_p \dot{\gamma}$ *(Bingham model with a yield stress τ_y and plastic viscosity η_p)*
- *AB slurry (40wt%) before H-release & decomposed AB slurry (settled down → re-stirring) used for viscometer*
- ***Decomposed AB less viscous (~442 cP) than fresh AB slurry (~617 cP)***

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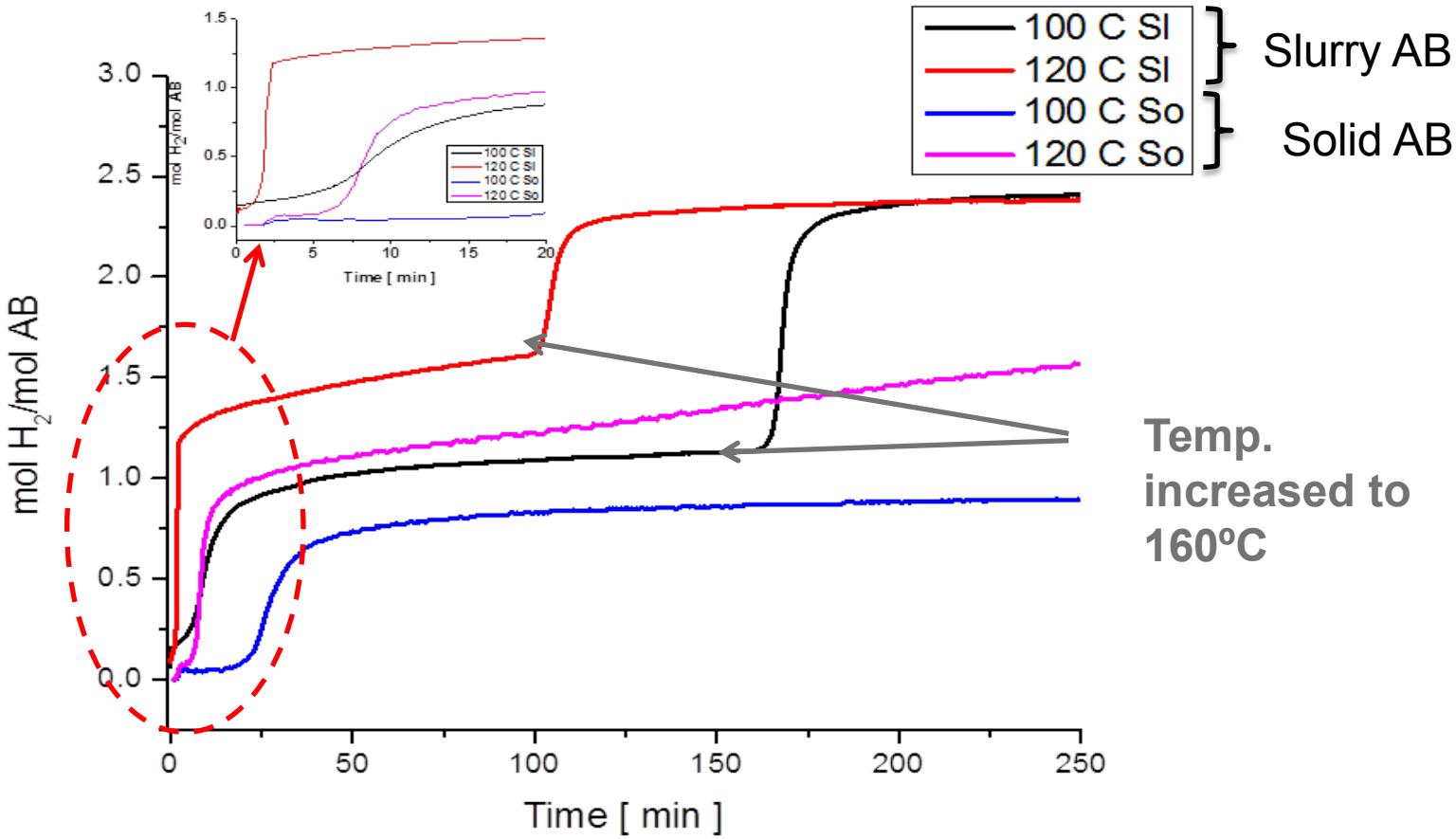
Accomplishments: Dehydrogenation Kinetics of Slurry AB Comparable to Solid AB



Preliminary result shows slurry AB kinetics similar to solid AB with 2.5 equivalent of hydrogen release, but, no induction period

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Collaborative Activities

Hydrogen Storage Engineering Center of Excellence	• Lincoln Composites - study of CF cost and pressure vessel design modeling • GM - design of structured media bed for MH • Ford – characterization of absorbent materials • UQTR - design and materials characterization of carbon absorbent • OSU - microarchetecture device concept development and thermodynamic analysis • UTRC - develop solutions for H_2 impurities filtering • LANL - AB system design and measure H_2 impurities • NREL - input for tank to wheels analysis and system cost models • SRNL - study AB reactivity and kinetics model development
SSAWG	• Participate in group discussions and analysis
Materials 'Reactivity' Program	• Khalil (UTRC) and Anton (SRNL) - understand reactivity properties of AB • Van Hassel (UTRC) - study impurities in H_2
Independent Analysis	• TIAX - provide design details for AB refueling cost and feasibility assessment, plus share cost parameters for system cost modeling

Future Work: FY13

Chemical Hydrogen System

- Detailed Design, Engineering and Analysis
  - Complete sensitivity analysis
    - Viscosity
    - Settling/flocculation
    - Vapor pressure
    - Thermal stability…
- Validate Critical Components
- Complete Solid-Liquid Slurry Development
  - Composition
  - Additives

BOP, Pressure Vessel and Cost Analysis

- Value Engineering
  - Examine BOP volume/mass trade-offs
- Pressure Vessel Engineering
  - Reduce cost, mass
  - Maintain safety
- Materials Compatibility/ Reactivity
  - Finalize H_2 wetted material compatibility in components
  - Determine BOP and pressure vessel materials compatibility
- Cost Analysis
  - Work with partners, vendors on reducing cost
  - Update analysis with detailed design

Summary

- Pressure Vessels
  - Completed the HSECoE tank needs survey for bench top tank production
  - Modeled various cases of type I, III, and IV tanks of pressure and temperature
  - Tested of type IV liner materials at cryogenic temperatures
  - Evaluated mass comparisons between type I, III, and IV
- Cost Analysis
  - Updated MOF – 5
  - Cost analysis being combined with vessel design models
- Chemical Hydrogen System – BOP
  - Identified key components to reduce mass / volume for pump, radiator (Heat exchanger)
  - Validation of performance initiated
- Chemical Hydrogen System – Modeling and Validation Exothermic Slurry (Ammonia Borane as surrogate)
  - Modeled fraction AB critical to meeting DOE mass target
  - 45 wt% AB slurry demonstrated- Slurry pre and post H_2 release

Summary

- Chemical Hydrogen System – Modeling and Validation Exothermic Slurry (Ammonia Borane as surrogate – cont.)
  - Increased ΔH_{rxn} or fraction AB results in T_{H2} outlet excesses (with current design)
    - 150% of ΔH_{rxn} and 70 wt% AB system $T_{H2} > 85°C$
  - 50 wt% AB and a range of kinetics met all targets
  - On-Board Efficiency target can be met with > 8 cold-starts/day
- Chemical Hydrogen System – Modeling and Validation Endothermic Slurry (Alane as surrogate)
  - Alane cannot meet DOE targets for mass for the system specified and conditions evaluated
  - Performed regression analysis on results
    - System Mass is dominated by weight fraction alane
      - Can meet DOE target with current BOP at 0.82 weight fraction alane
- System Efficiency improved with reduced heat of reaction
  - System On-Board Efficiency of 90% possible with US06 if $\Delta H_{rxn}/2$
  - System On-Board Efficiency of 90% possible with Cold FTP if $\Delta H_{rxn} = 0$
  - Kinetics has little effect on the system efficiency or mass (reactor small fraction of total system mass—most is alane itself)
- With multiple start-ups the DOE on-board efficiency target difficult to achieve

Hydrogen Storage Engineering
CENTER OF EXCELLENCE

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Jesse Adams – DOE EERE, Technology Development Manager

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Technical Back-up Slides

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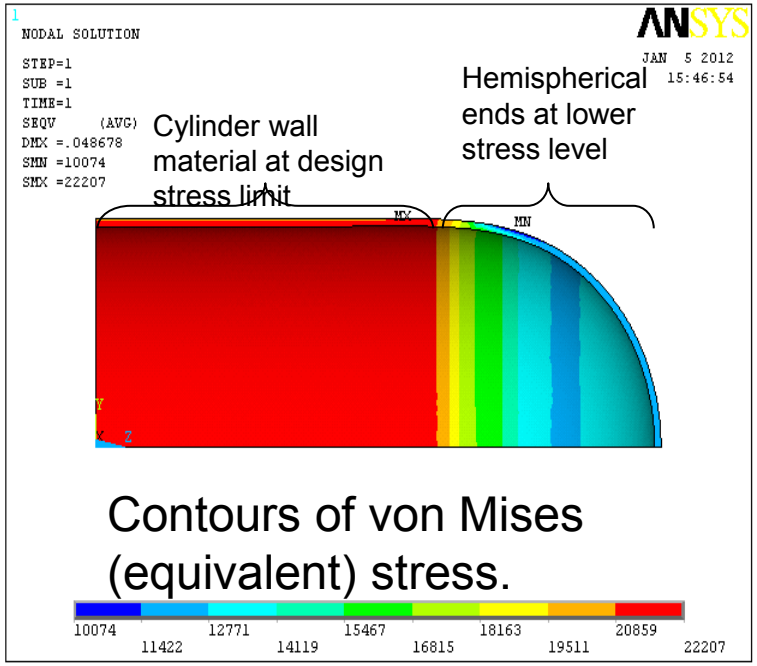
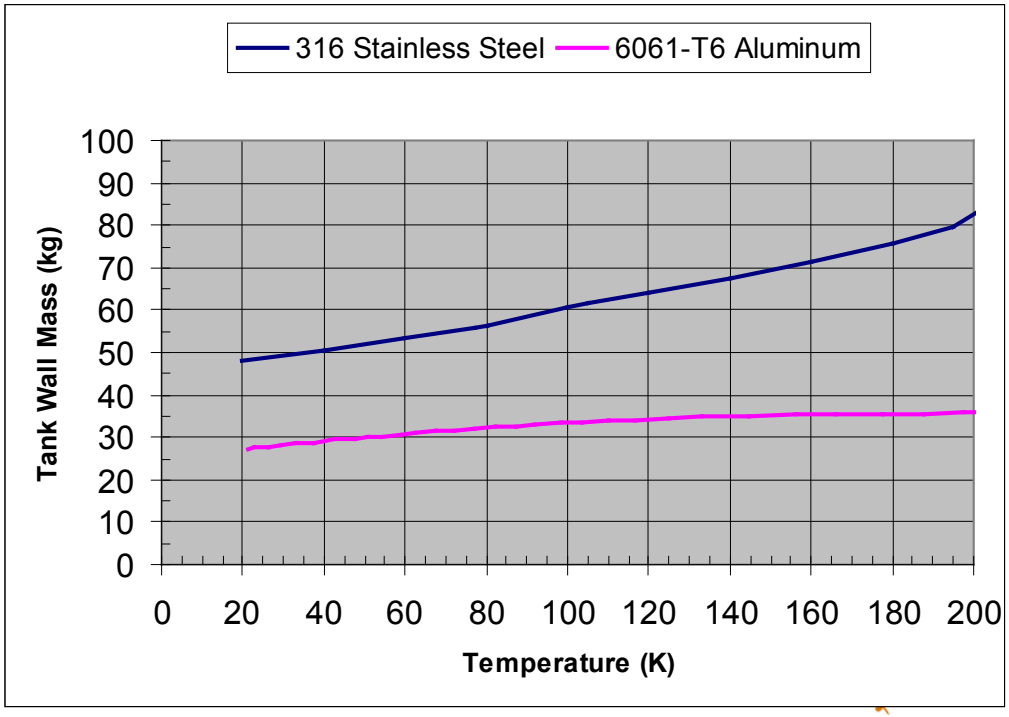
Accomplishments: Modeling of Type I Pressure Vessel



Pressure 60 bar
Temperature = 40K – 120K

R=202.3mm (internal)
L/D=3 (external)
Storage Capacity = ~144L
(varies slightly, 142.6-144.8L)
Wall Thickness = 0.15-0.24” (steel),
0.25-0.34” (aluminum)

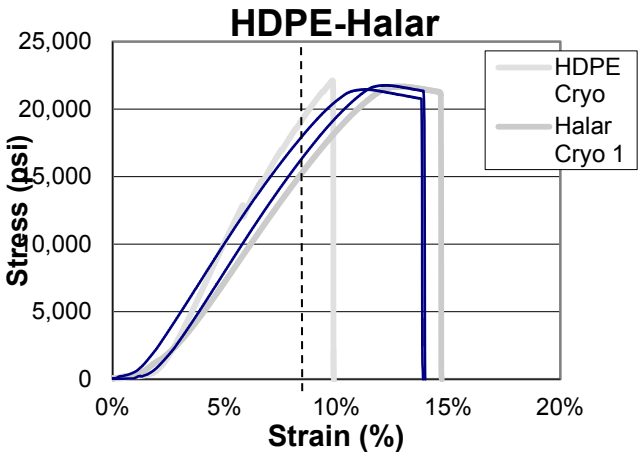


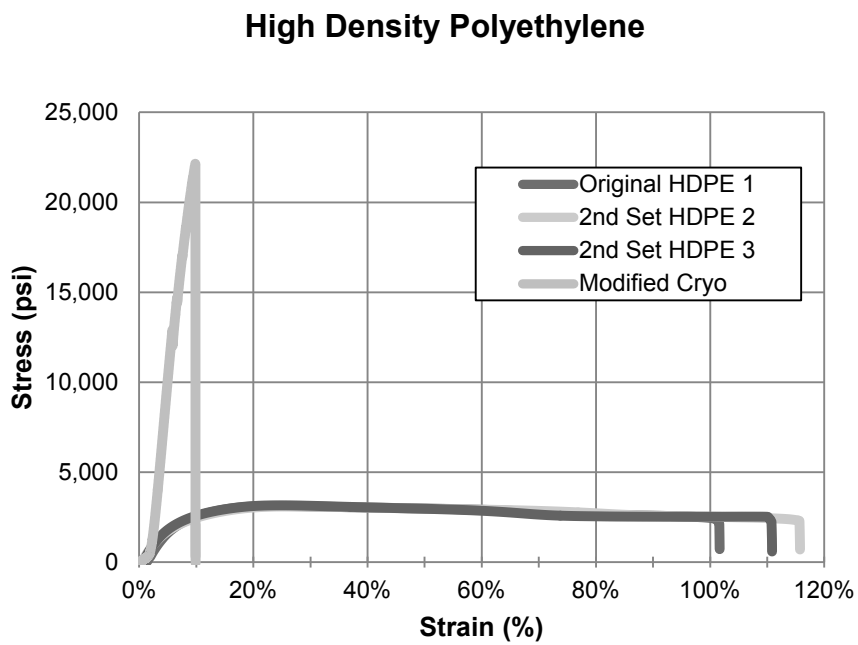
Accomplishments: Type IV Pressure Vessel Data

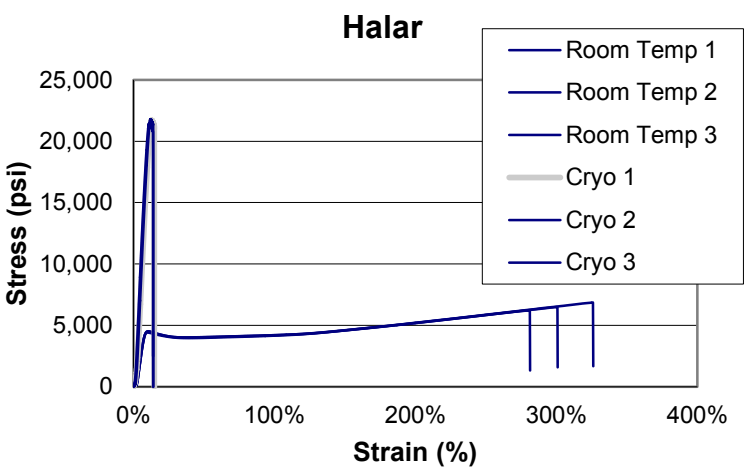
- 200L Capacity, 200 Bar 80K-160K
  - Comparable to Type III designs
  - 180K, 250 Bar overpressure is peak stress/strain state
- 144L Capacity, 60 Bar, 40-120K Temperature Range
  - All cases hit the minimum 3 layer composite limit
    - Larger diameter would be a more effective use of material
  - HDPE liner (assume no load carry)

	60 Bar, 144 L		60 Bar, 144 L		200 Bar, 200 L	
Diameter	300		400		500	
Liner Thickness	3 mm	6 mm	3 mm	6 mm	3 mm	6 mm
Total (kg)	**14.9**	**21.1**	**11.8**	**16.6**	**26.5**	**32.2**
Liner (kg)	5.7	11.5	4.5	9.1	5.1	10.3
Composite (kg)	9.2	9.6	7.3	7.5	21.5	21.9

Accomplishments: Cryogenic Material Testing for Type IV Pressure Vessels







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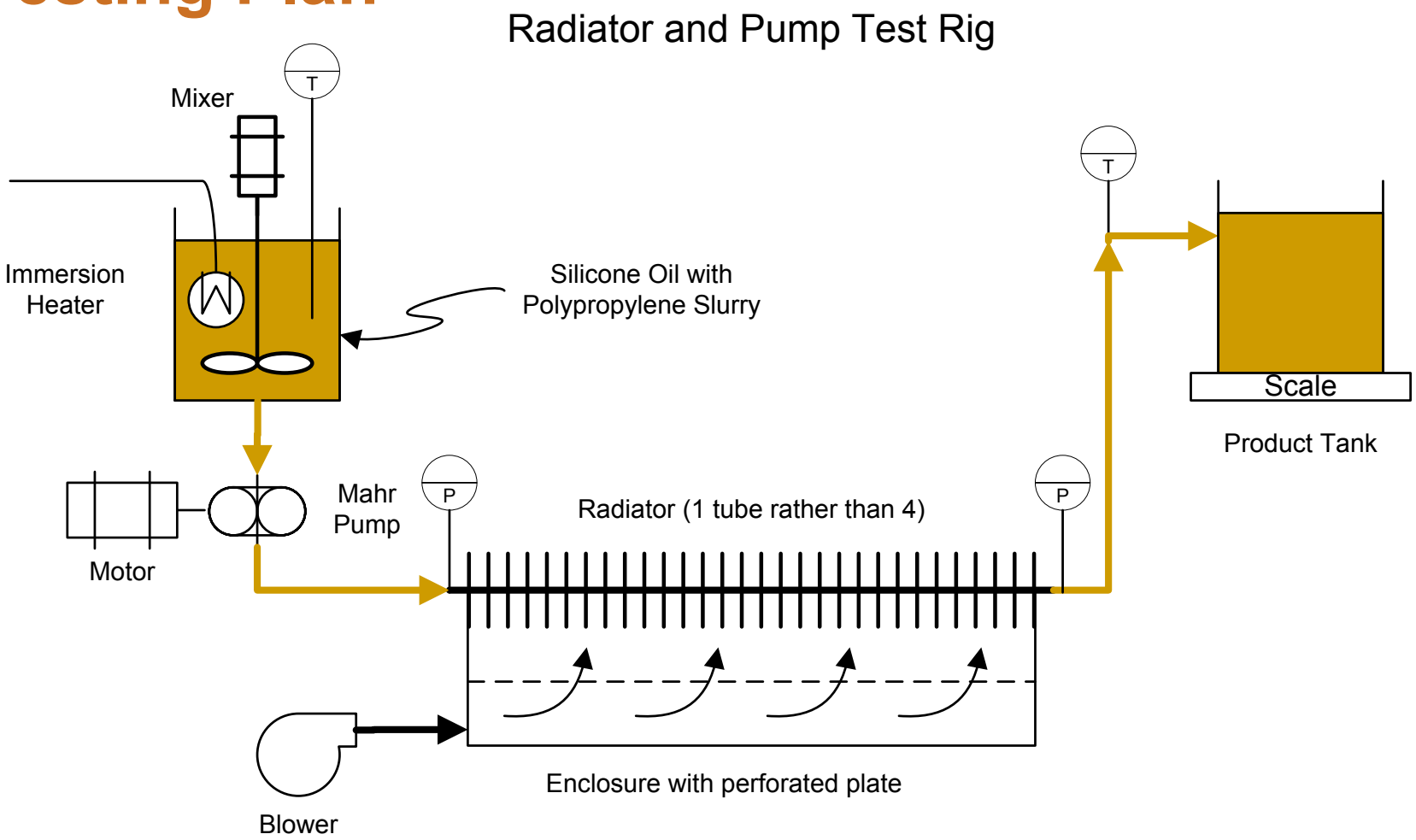
Chemical Hydrogen Component Validation Testing Plan



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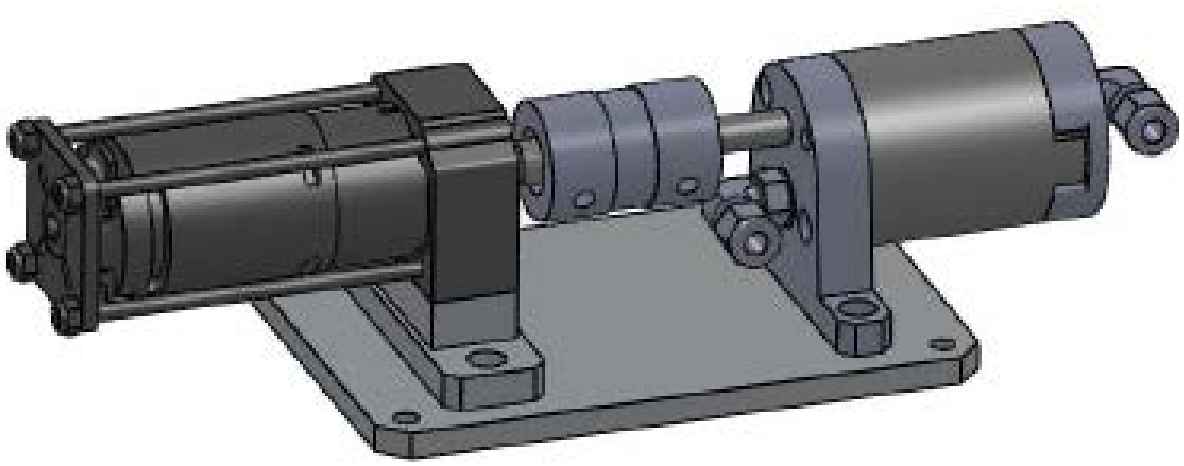
Accomplishments: Chemical Hydrogen System Identified Components (Pump)

Feed Pump	Requirements	Actual Mahr TC-1251 Round Pump
Viscosity	≤1500 cp	≤ 50,000 cp
Pressure	20 bar	≤ 65 bar
Flow rate	1 Liter/minute	1.2 Liter/minute
Weight	≤ 3 kg	2.5 kg (includes pump, motor, couplings etc).
Volume	≤ 0.77 Liter	1.5 Liter



This is a ~44% reduction in mass from baseline

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Accomplishments: Chemical Hydrogen System Identified Components (HX)

HX	Requirements	Actual Energy Transfer MDE
Outlet temperature For US06 and SC03 Hot Cycle	< 60°C	
Fluid: AB/IL 50/50 or AB slurry in silicone oil		
Weight	≤ 1.15 kg	1.32 kg + 1.0 kg for fan
Volume	≤ 10.9 Liter	1.3 Liter+ 5.9 Liter for fan

This is a ~60% reduction in mass and ~50% reduction in volume from baseline

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