Advancement of Systems Designs and Key Engineering Technologies for Materials Based Hydrogen Storage

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United Technologies Research Center

HSECoE

United Technologies
Research Center

DOE Hydrogen Program
Annual Merit Review
Washington, DC
May 15, 2012

Project ID: ST006

Overview

- **Timeline**
  - Start: February 2009
  - End Phase 1: March 2011
  - End Phase 2: June 2013
  - End Phase 3 / Project: June 2014
  - Percent complete: 55% (spending)
- **Budget**
  - $5.91M Total Program
    - Reflects budget reduction with $0.95M
    - $4.58M DOE
    - $1.33M (22.5%) UTRC
  - FY09: $600k DOE
  - FY10: $1,000k DOE
  - FY11: $750k DOE
  - FY12: $750k DOE
- **Barriers***
  - A – J
  - A. System Weight & Volume
  - D. Durability/Operability
  - J. Thermal Management
- **Targets***
  - All
- **Partners**

Pacific Northwest NATIONAL LABORATORY

SRNL SAVANNAH RIVER NATIONAL LABORATORY

Los Alamos NATIONAL LABORATORY

LINCOLN COMPOSITES A Member of Hexagon Composites Group

HRI UQTR Université du Québec à Trois-Rivières

GM

Ford

BASF The Chemical Company

OSU Oregon State UNIVERSITY

JPL

California Institute of Technology 1891

NREL National Renewable Energy Laboratory Innovation for Our Energy Future

HSECoE

IEA HIA Task 22

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* DOE EERE HFCIT Program Multi-year Plan for Storage

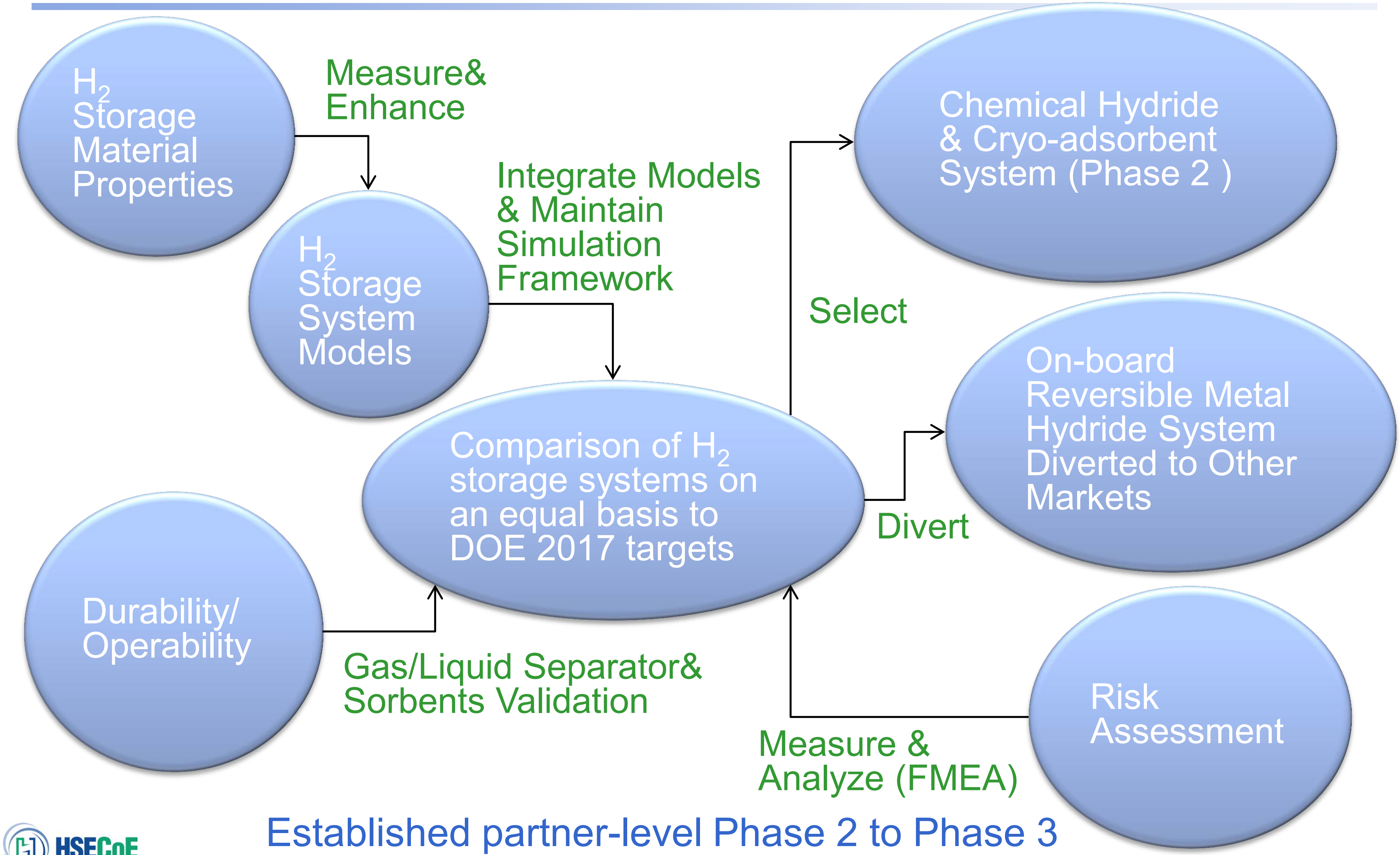
Objectives

- Design of materials based vehicular hydrogen storage systems that will allow for a driving range of greater than 300 miles

Performance Measure	Units	2010	2017	Ultimate
System Gravimetric Capacity	g H_2 /kg system	45	55	75
System Volumetric Capacity	g H_2 /L system	28	40	70
System fill time (for 5 kg H_2)	minutes	4.2	3.3	2.5
Fuel Purity	% H_2	SAE J2719 guideline (99.97% dry basis)		

- Major project impact:
  - Integrated Power Plant Storage System Modeling:
    - Specified on-board reversible metal hydride material requirements. Diverted such a system to different markets.
    - UTRC oversees modeling framework on consistent platform
  - Gas/Liquid separation (GLS) of liquid chemical hydride
  - H_2 quality (NH_3 adsorbent, particulate filter)
  - Compaction/Materials thermal conductivity enhancement
  - Risk Analysis: Failure mode and effect analysis (FMEA)

Approach



H_2 Storage Material Properties

Measure& Enhance

H_2 Storage System Models

Integrate Models & Maintain Simulation Framework

Comparison of H_2 storage systems on an equal basis to DOE 2017 targets

Select

Chemical Hydride & Cryo-adsorbent System (Phase 2)

Divert

On-board Reversible Metal Hydride System Diverted to Other Markets

Durability/ Operability

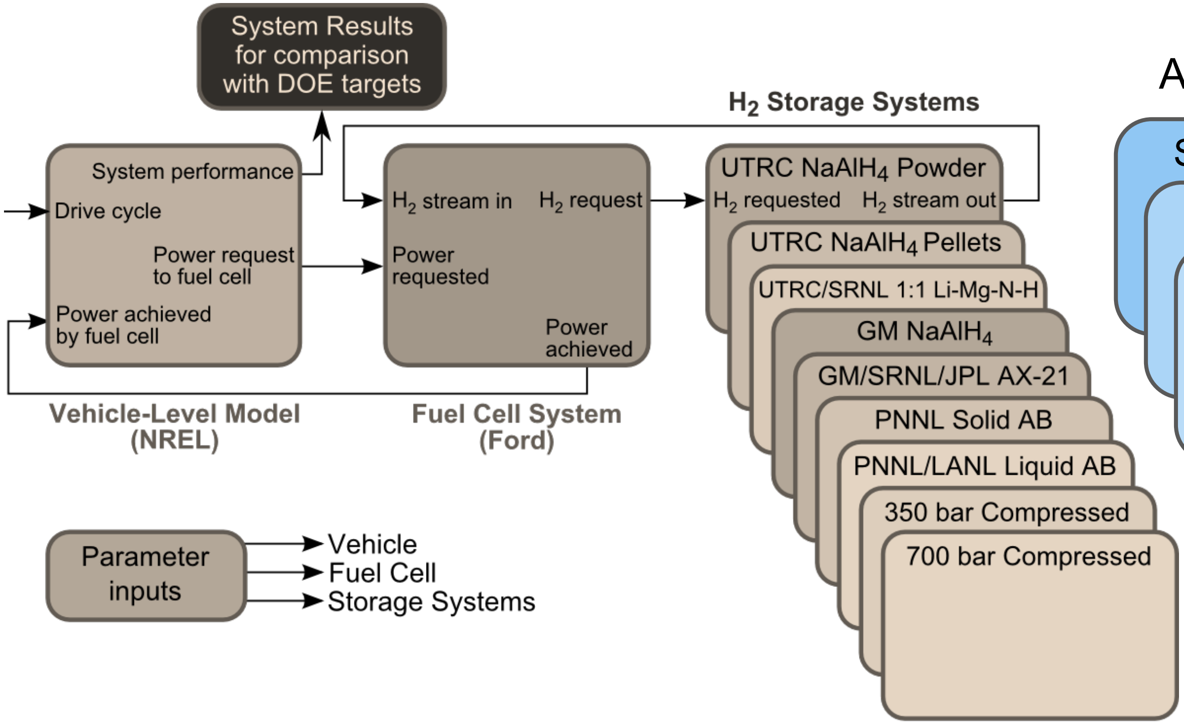
Gas/Liquid Separator& Sorbents Validation

Risk Assessment

Measure & Analyze (FMEA)

Established partner-level Phase 2 to Phase 3 transition criteria in updated SOPO

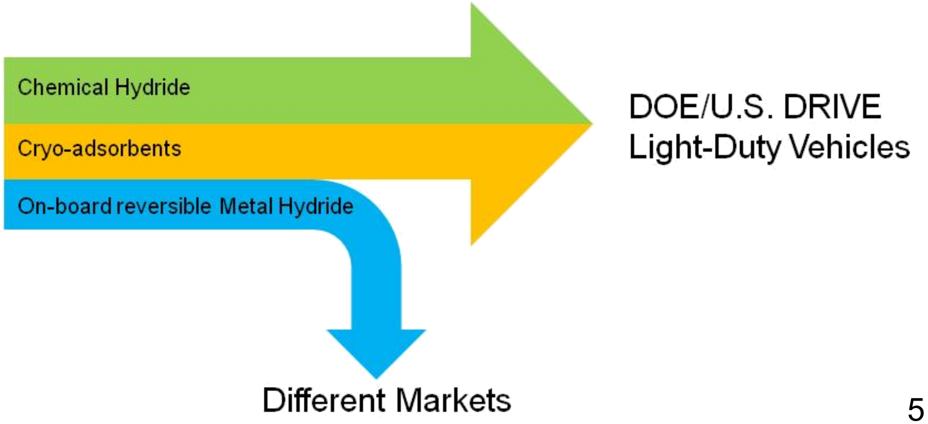
IPPSSM Framework Application



Additional storage models

- SRNL+ MOF-5: 200 bar, 40K
- SRNL+ MOF-5: 200 bar, 80K
- SRNL+ MOF-5: 60 bar, 40K
- SRNL+ MOF-5: 60 bar, 80K
- PNNL/LANL Liquid AB
- PNNL/LANL Alane
- UTRC
  Ideal metal hydride models
  (gap analysis)

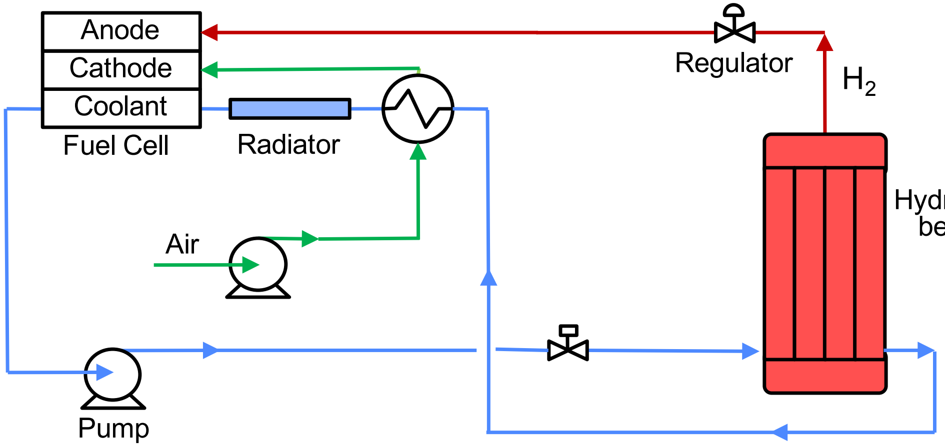
Quantitative comparison of H_2 storage systems on a common basis achieved by team effort



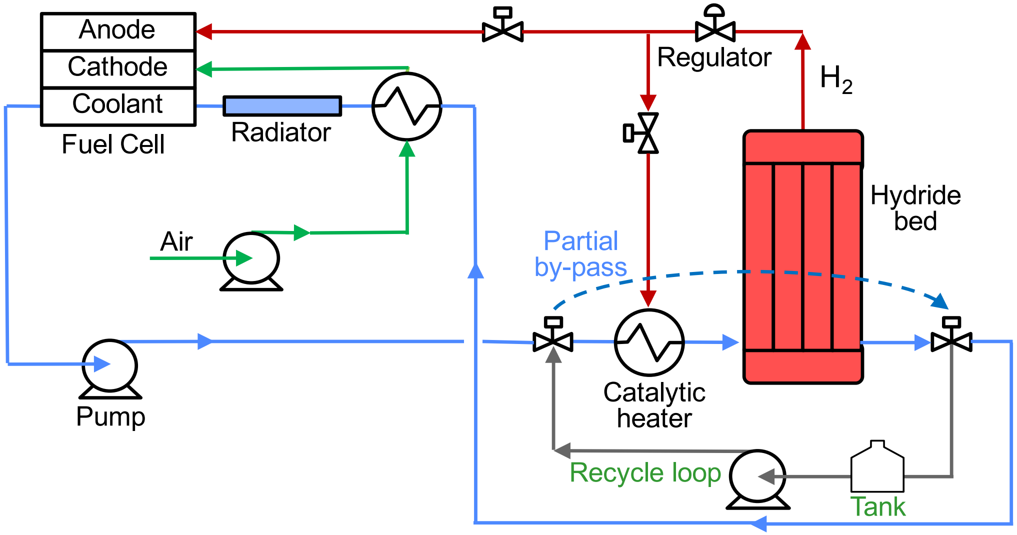
On-Board Reversible Metal Hydrides Diverted to Different Markets

Two qualitatively different systems:

- For higher H_2 pressure materials: use the fuel cell waste heat stream
- Very simple system: selected to determine the minimum material gravimetric capacity needed.
- No separate buffer tank: use H_2 in pores.

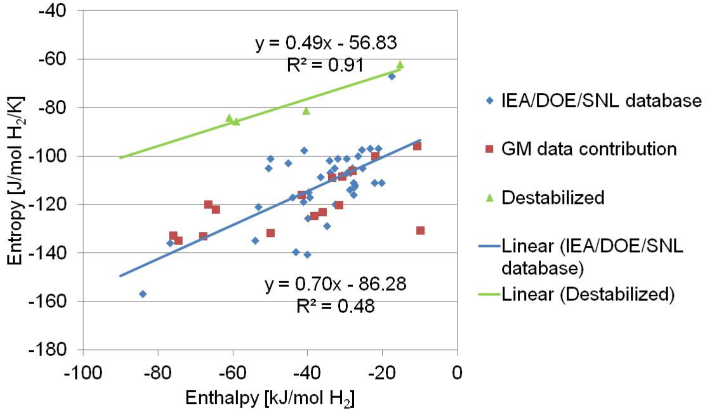


- For lower H_2 pressure materials: Mix of fuel cell coolant, catalytic heater and recycled fluid used for warm-up and to maintain T_{tank}.
- Increased material capacity to compensate for combusted H_2 and heavier BOP.
- No separate buffer tank: use H_2 in pores.



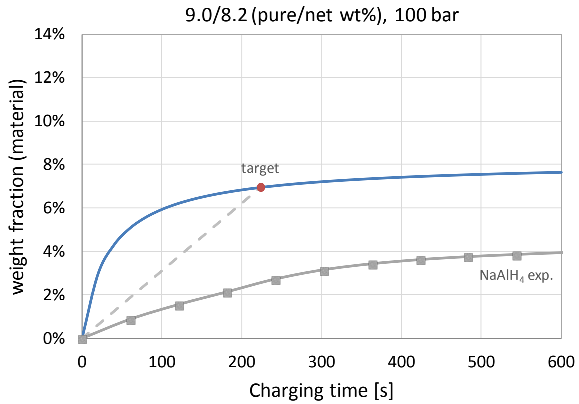
Analysis anchored in metal hydride databases

Thermodynamic Properties



Equilibrium pressure: fit ΔS vs ΔH

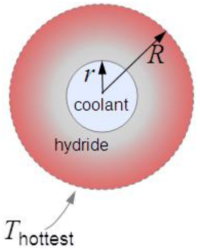
Kinetics



85% of capacity in 3.3 minutes

Heat Transfer (Acceptability Envelope)

$$\overset{\text{heating rate per unit volume}}{\dot{Q}} = \underbrace{\left(\frac{-\Delta H}{w_{H_2}}\right)}_{\text{heat released per mass of } H_2 \text{ absorbed}}\underbrace{\left(\frac{\Delta m_{H_2}}{\Delta t}\right)}_{\text{refueling rate}}\underbrace{\left(\frac{M_{\text{hydride}}}{\rho_{\text{bed}}}\right)^{-1}}_{\text{volume of hydride bed including voids}}$$

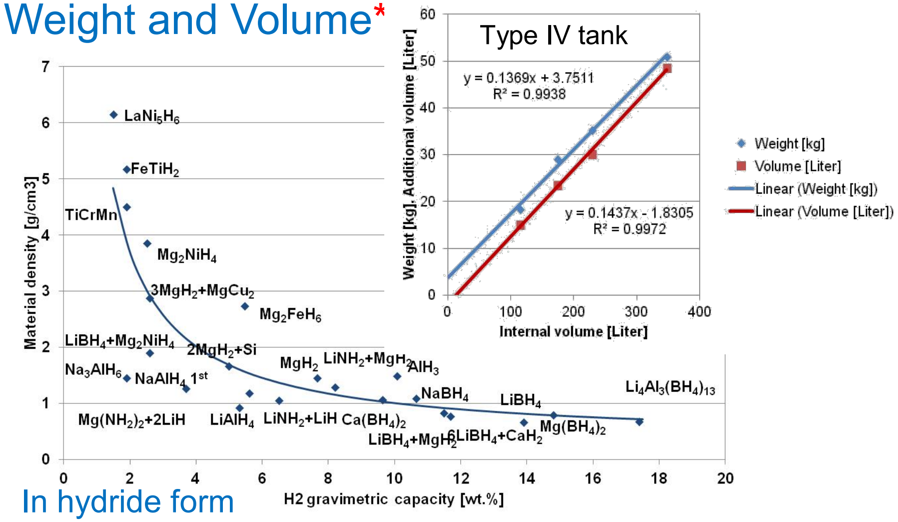


$$\Delta T = \frac{1}{8}\frac{\dot{Q}}{k}\left(R^2 - r^2\right)f(x)$$

10 wt.% ENG “worms”, ΔT=45°C

Short fill time (3.3. minutes)

Weight and Volume*



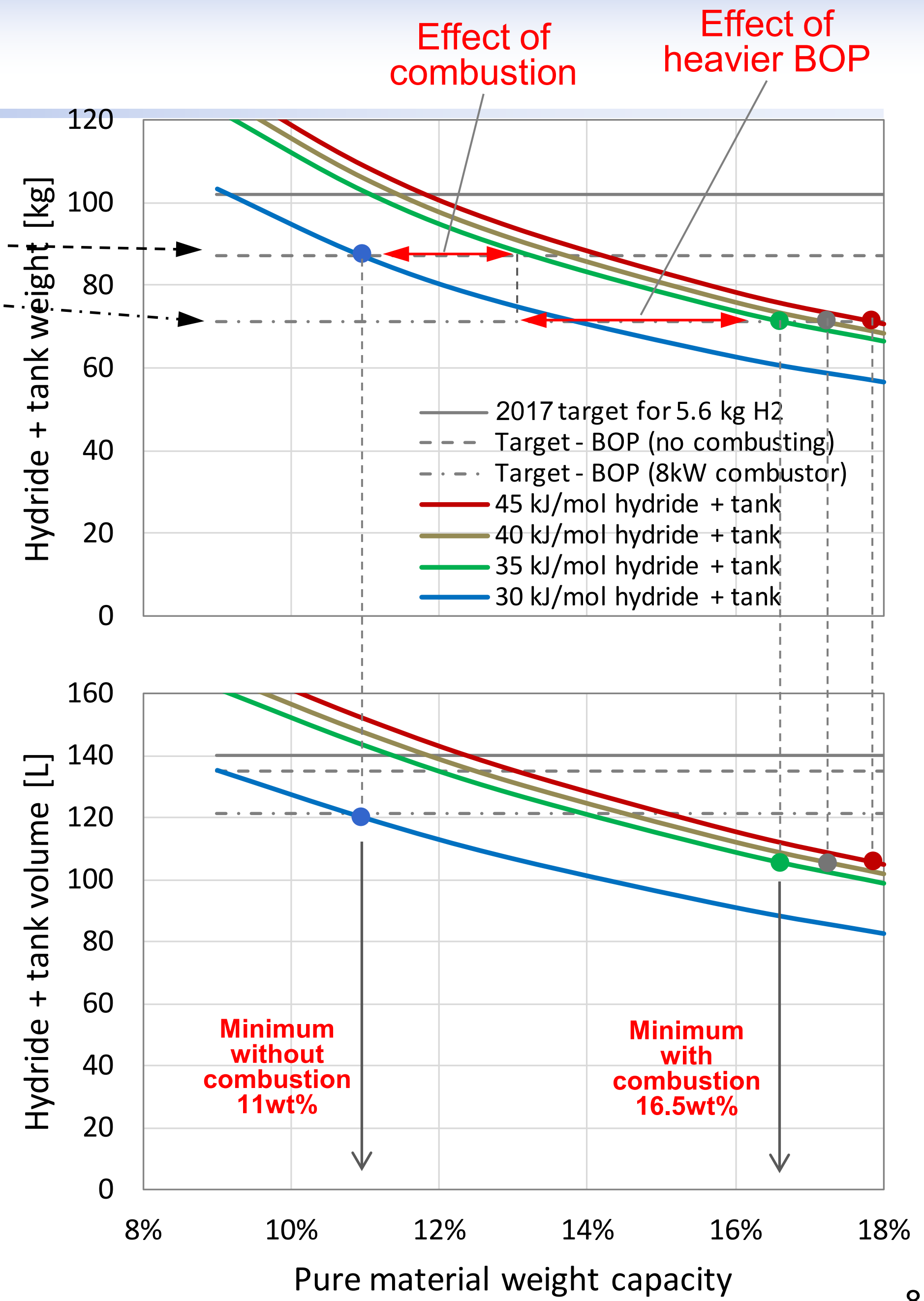
In hydride form

* Other parts from BOP Library (PNNL)

System weight and volume

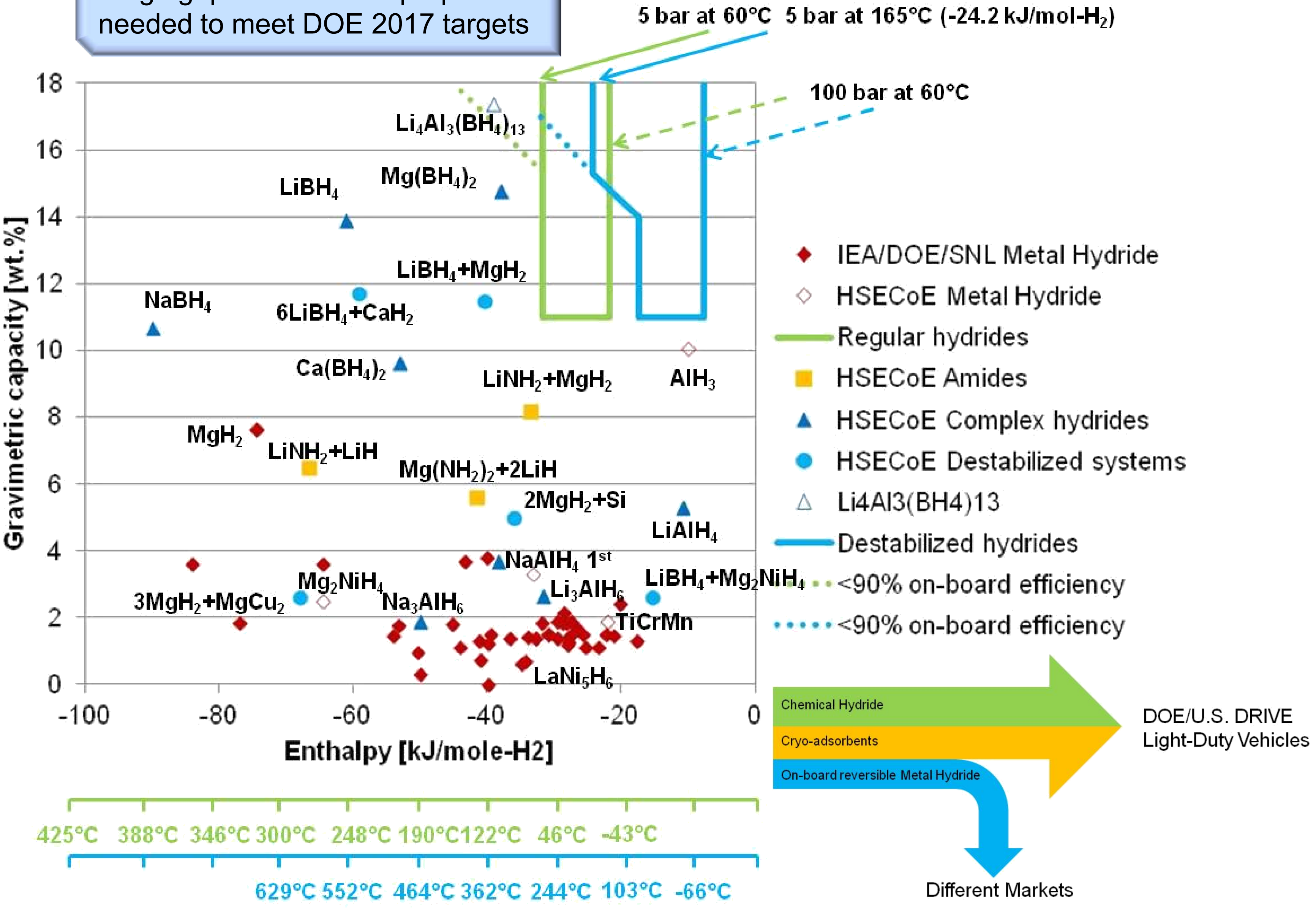
- Allowable (hydride + tank) values
  - Using only waste heat
  - With a 8kW combustor loop

- On-board reversible metal hydride:
  - Systems are limited by weight
  - Waste heat from fuel cell:
    - ΔH < 27 to 32 kJ/mol (depends on drive cycle); >11 wt.%
  - Combustor loop:
    - ΔH > 27 to 32 kJ/mol (depends on drive cycle); >16.5 wt.% due to BOP weight increase and H_2 combustion



Available metal hydride materials vs. requirements

Large gap with material properties needed to meet DOE 2017 targets



Chemical Hydride

Cryo-adsorbents

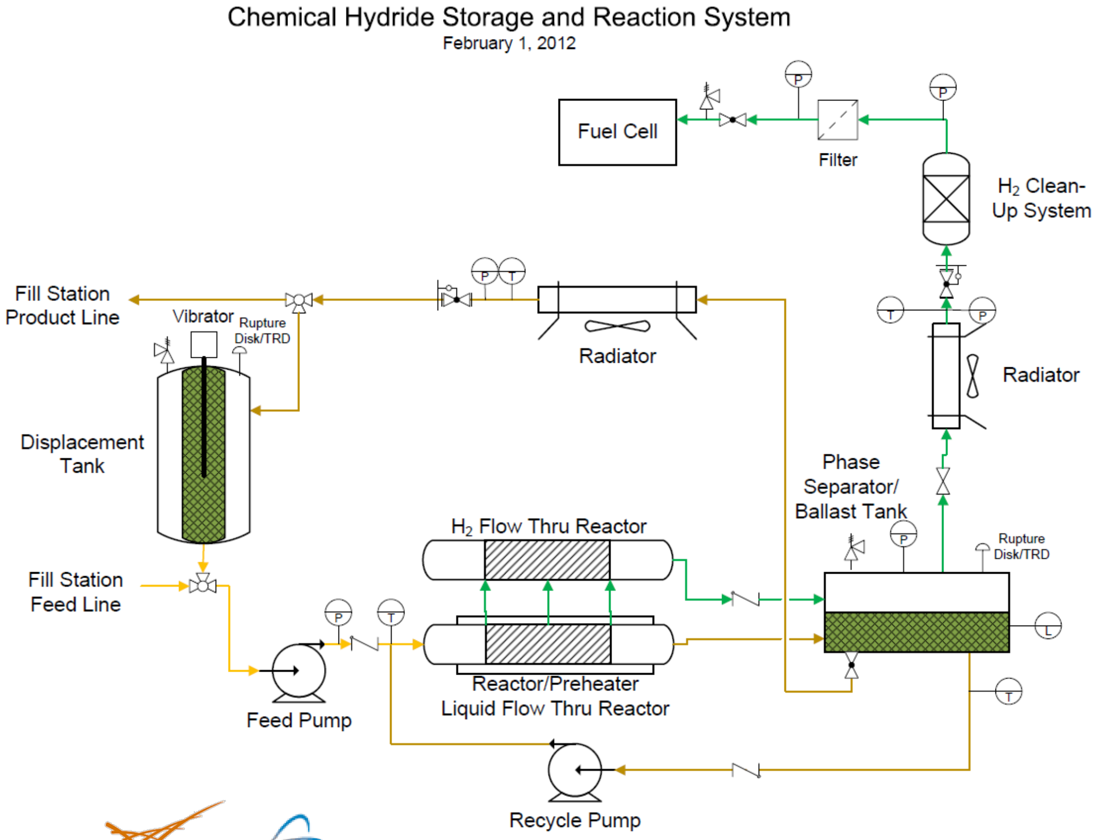
On-board reversible Metal Hydride

DOE/U.S. DRIVE
Light-Duty Vehicles

Different Markets

Liquid Chemical Hydride Operability (GLS Validation)

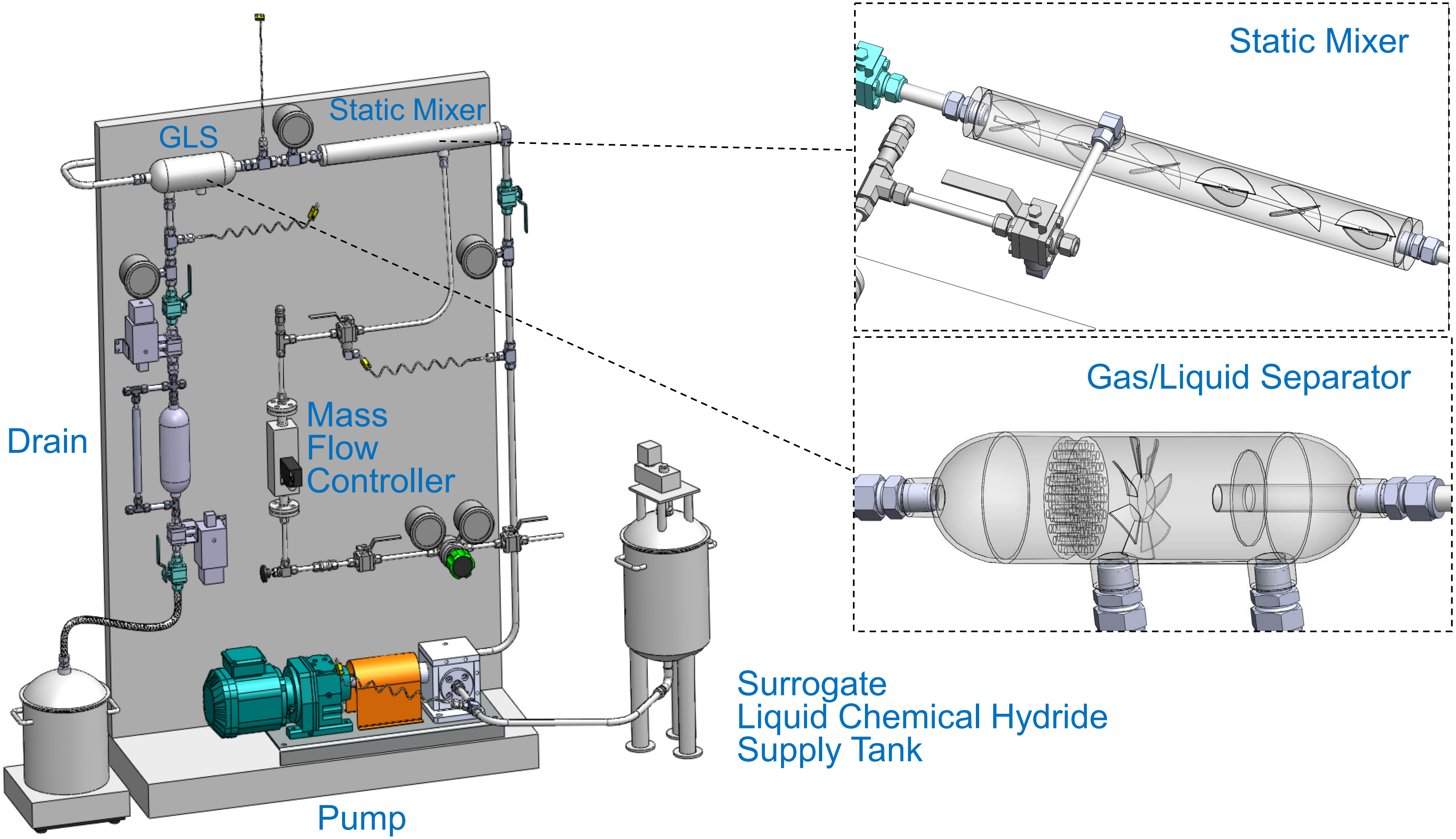
- Hydrogen gas must be separated from the liquid spent fuel following the exothermic thermolysis of ammonia borane.
- Designed gas-liquid separator (GLS) test system.
- UTRC: Surrogate fluid; LANL&PNNL: Engineering fluid form of AB


Chemical Hydride Storage and Reaction System
February 1, 2012

NH_3 filter:
1.2 kg,
1.6 Liter

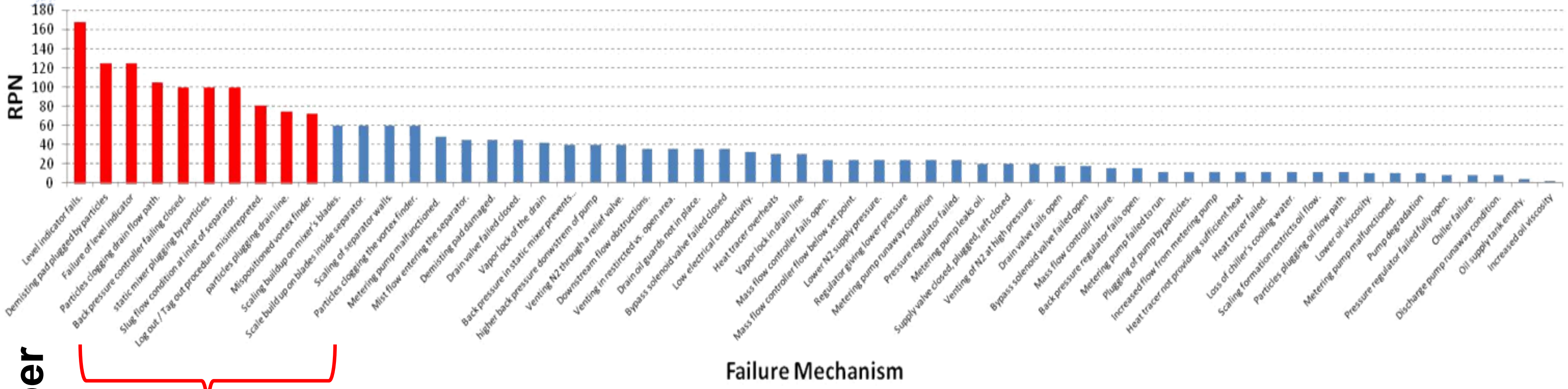
GLS:
5.4 kg
19 Liter

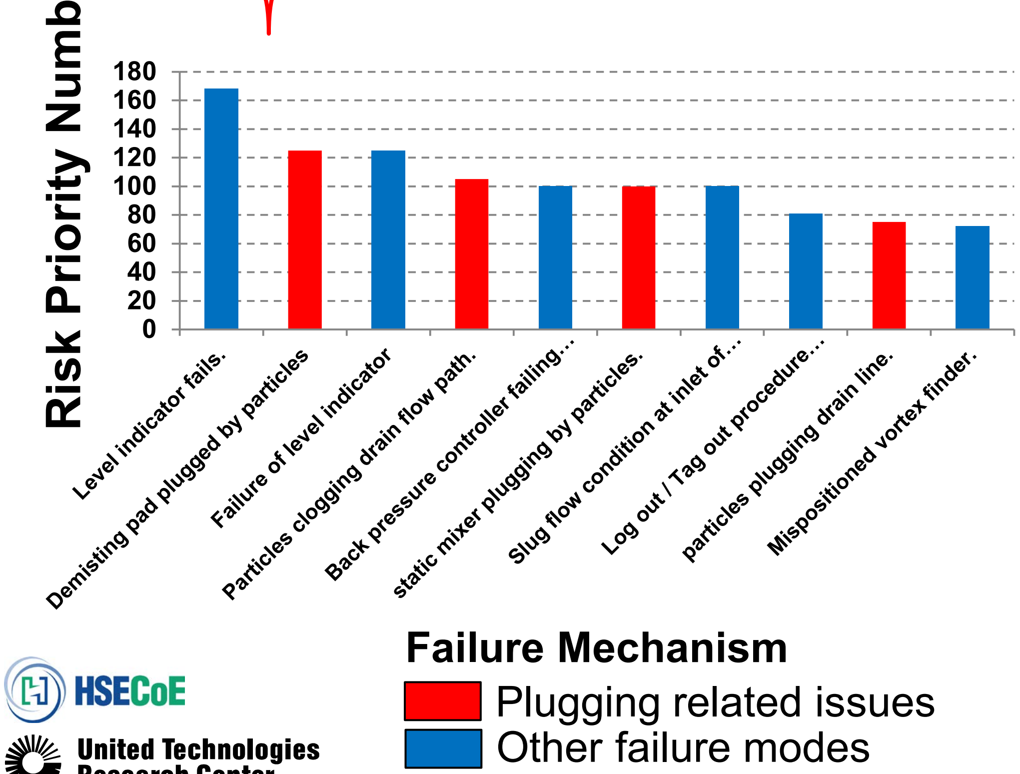
Gas-Liquid Separation Test Facility



Spray nozzle option will also be available

Gas/Liquid Separator (GLS) Test Rig
Failure Mode and Effect Analysis (FMEA)





- Expected learnings from GLS tests:
  - GLS risk factors
  - Mitigation Strategies
  - Efficiency
  - Operability: Slurry pump, Heat exchanger, Gas Liquid Separator(s), Drain, Level Indicator, Plugging issues
- Supported FMEA of Cryo-adsorption and Chemical Hydride Systems in center wide team effort

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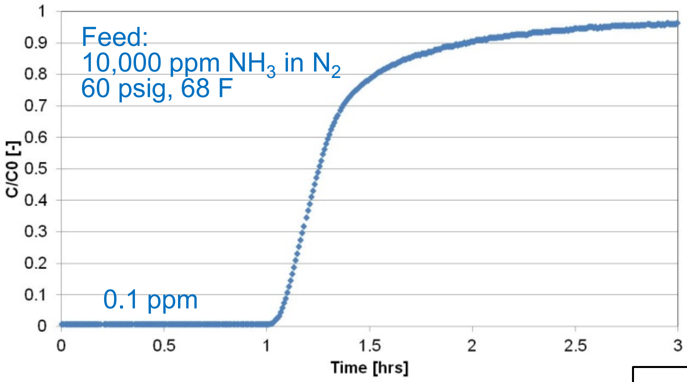
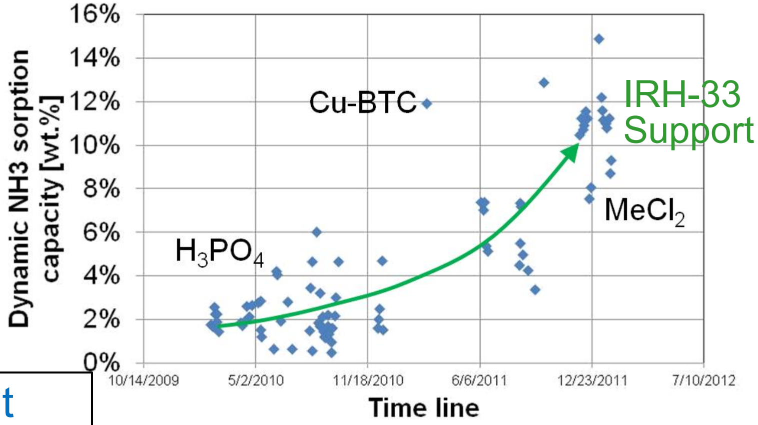
H_2 Quality (NH_3 Mitigation*)

Dynamic Breakthrough

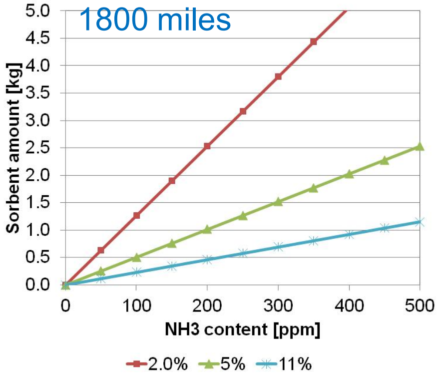


50 wt.% $MnCl_2$ on IRH-33

More Efficient Filter by Capacity Improvement

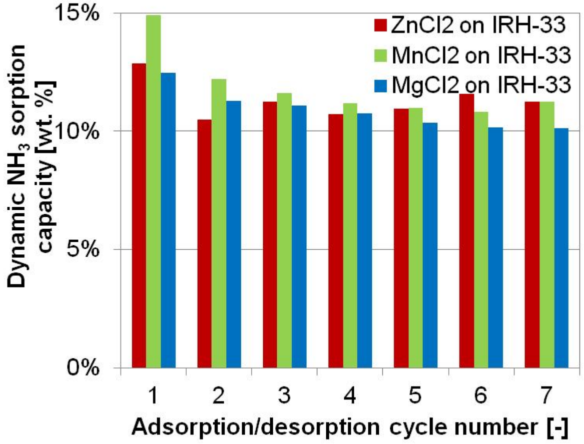


NH_3 Filter Weight

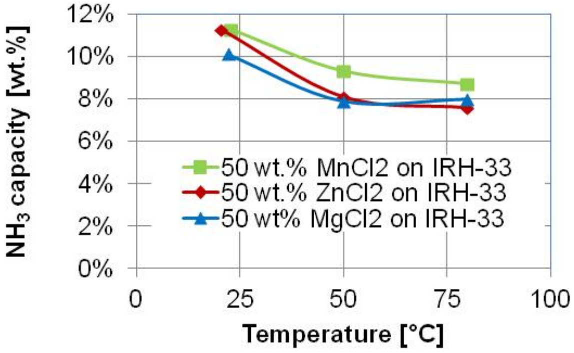


NH_3 filter: 1.2 kg, 1.6 Liter

Regenerable



Capacity over full ambient temperature range



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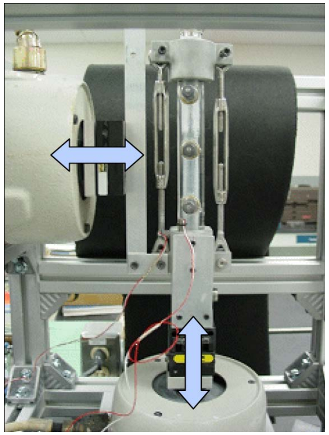
* LANL addresses Boron containing impurities

Cryo-Adsorption System Support: SAC Binderless Compaction

Vibration Packing

- Resulting density is equal to tap density (0.3 g/cm^3):
  - No density enhancement



Compressed Foam Enclosure

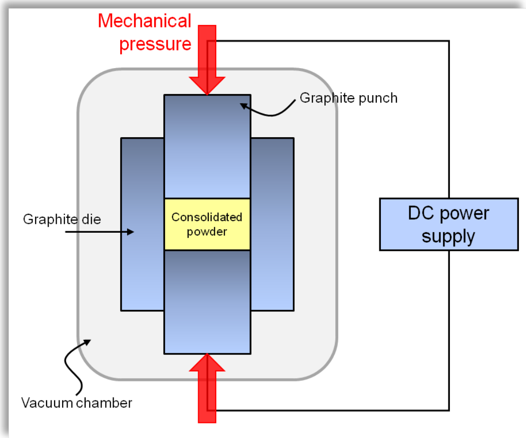
- Springback limits MaxSorb density to 0.3.g/cm^3 with high weight penalty for thermal conductivity enhancement

Filter Press

- Density limited to 0.3 g/cm^3 as only 35 psi pressure in absence of any vibration.

Spark Plasma Sintering (SPS)

- Rapid heat-up and cool down in graphite die to elevated temperatures (1000-1200°C) results in densification to 0.5-0.625 g/cm^3 : Some loss of SA BET but FAST!



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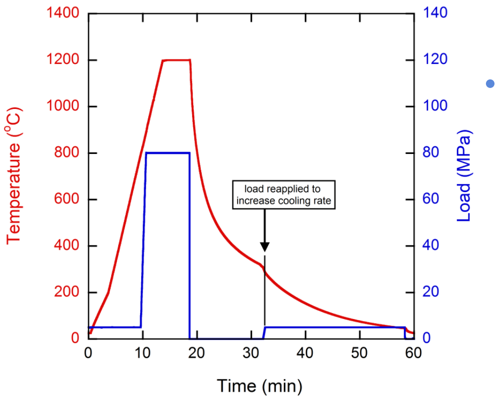
Cryo-Adsorption System Support: Spark Plasma Sintering

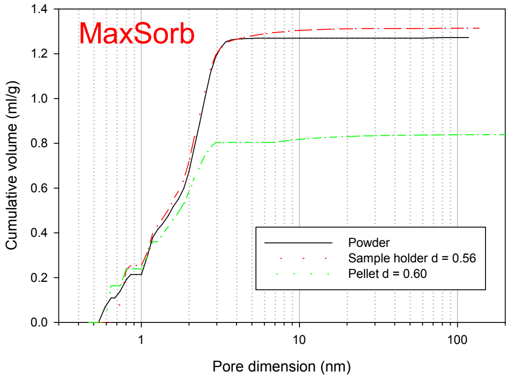
SPS technique



- Impurities reduce required operating temperature
- ρ=0.6 g/cm^3 achieved

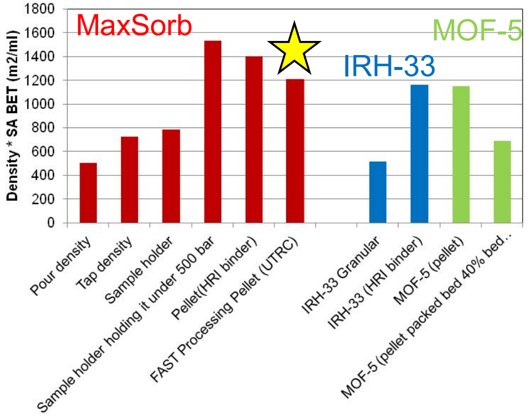
- Sintering reduces pore volume similar as use of binder

Pore volume loss



Volumetric specific surface area

- Comparable to values achieved with binder but faster processing



Scale-up

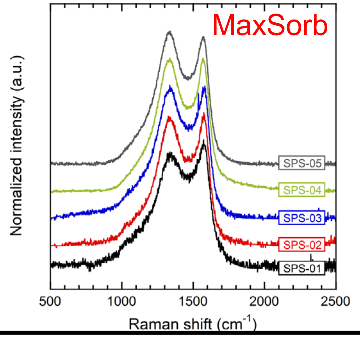
- Applicable to practical size of adsorbents ('hockey puck')

IRH-33

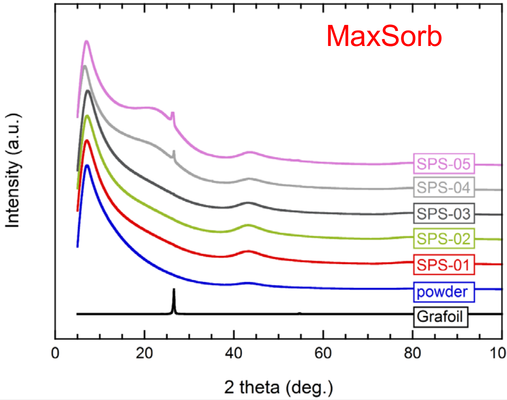
Characterization

SAC material remained highly disordered form of carbon

Raman:



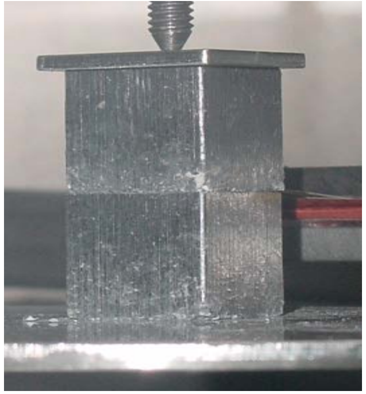
X-ray diffraction:



*** IRH-33 and pore volume measurement kindly provided by UQTR**

Cryo-Adsorption System Support: Conductivity Enhancement

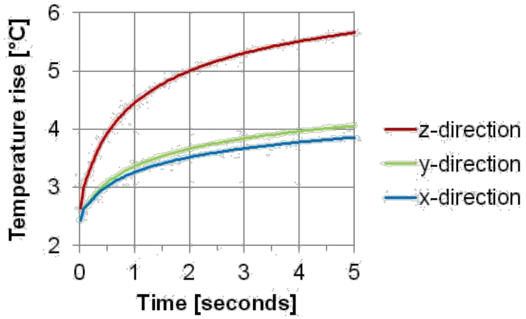
Compacted MOF-5



- MOF-5*+10wt.% ENG “worms”**
- Density: 0.6 g/cm^3
- 25 MPa (3.53 kpsi)

Hot Disk thermal conductivity measurement

- Measurements in each orthogonal direction (x,y, and z(=axis of compaction))
- Parameters: 0.1W, 5s



Thermal Conductivity Anisotropy

- Thermal conductivity in radial direction significant higher than in axial direction (5-10 x)

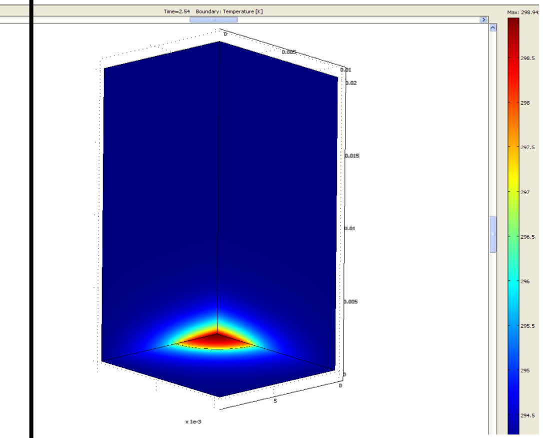
parameter	95% confidence interval	Unit
kX	3.32 < 3.45 < 3.58	W/m/K
kY	1.44 < 1.49 < 1.55	W/m/K
kZ	0.280 < 0.286 < 0.292	W/m/K
C_p	1395 < 1438 < 1484	J/kg/K

- High values of heat transfer coefficients

h_x	638< 645 < 652	W/m^2/K
h_y	706 < 714 < 724	W/m^2/K
h_z	773 < 783 < 795	W/m^2/K

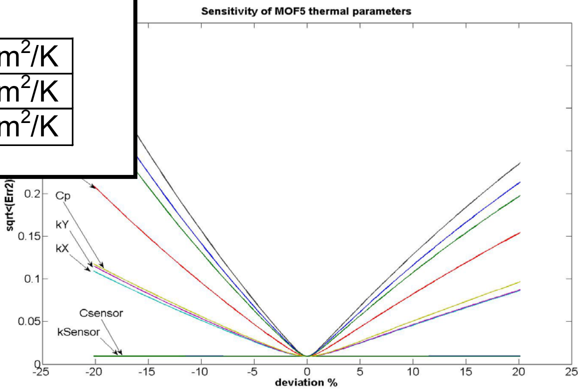
COMSOL Model

- Inverse problem solved with Matlab® optimizer



Dynamic COMSOL Multiphysics model of HotDisk thermal conductivity experiment. The predicted temperature rise with time is fitted to the experimental data with Matlab® optimizer

Error analysis



- High sensitivity of error measure to the sample thermal conductivity parameters results in narrow confidence interval

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* MOF-5 powder produced by BASF and kindly provided by Ford

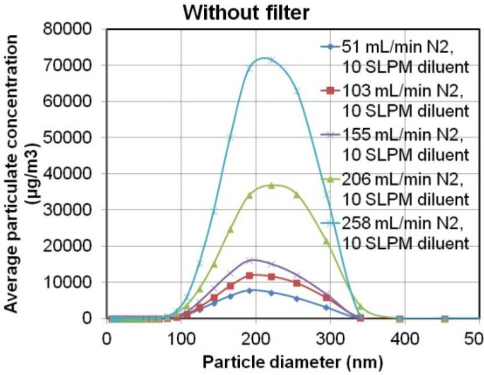
** ENG-worms kindly provided by SGL Carbon

H_2 Quality: Particulate Mitigation

Test Setup

- Particulate analyzer (d_p<0.5 µm)

Concentration & Particulate size without filter



Conclusion:

- Porous SS metal filters are effective (even 10µm),; Need guidance on longevity.:

- Filter needs to be inside bed
- **SAE J2719 April 2008 standard**

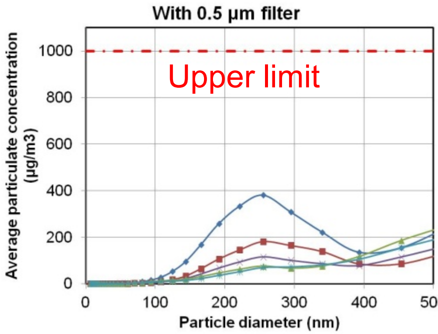
Parameter	Value
Maximum particulate size	< 10 µm
Particulate concentration:	<1000 µg/m^3

Porous SS Metal Filters

- Determine required filter area for longevity of cryo-adsorption system

Concentration & Particulate size with filter

- Particulate concentration well below SAE J2719 guideline even when recorded with 10µm filter



FY12 and FY13 Plan

- Based on revised SOPO resulting from budget reduction

Task	Description	FY12				FY13	
		1Q	2Q	3Q	4Q	1Q	2Q
Project Management	Go/No-Go meeting Phase 2 to Phase 3 transition						☆
	F2F-meetings; Tech Team Review; Annual Merit Review	□		□		□	
	Quarterly Financial and Technical Reports	○	○	○	○	○	○
IPPSSM	Lead IPPSSM Techical Area (TA)	━	━	━	━	━	━
	Support Model Integration	━	━	━	━	━	━
	Maintain Vehicle/Storage System Framework	━	━	━	━	━	━
	Phase 3 lab test scaling guidance			━	━	━	
Material Property Measurement	Thermal Conductivity Enhancement (Cryo-adsorbent)	━	━	━			
	Viscosity for Gas Liquid Separator Surrogate Material			━	━	━	
Chemical Hydride Operability	Gas/Liquid Separator Validation (<5.4 kg, <19 Liters)						☆
	Build Experimental Setup		━	━	━	━	
	Test Gas/Liquid Separator with Liquid AB Surrogate			━	━	━	━
	Mitigate Operability Issues					━	━
H2 Quality (SAE J2719)	NH3 Mitigation Filter (<1.2 kg, <1.6 Liter, 1800 miles) Milestone						☆
	Adsorption Isotherm Measurement			━	━		
	Scale-up to Phase 2 and Phase 3 Requirements			━	━	━	
	Particulate Mitigation		━	━			
Risk Assessment	Flammability Test Liquid AB Formulations (provided by LANL/PNNL)			━	━	━	━
	Dust Explosion Parameters MOF-5			━	━		
	General Support (FMEA/HAZOP)	━	━	━	━	━	━
Heat Exchanger Development	COMSOL Model Comparison with Experimental Data		━	━	━		

Summary

Relevance: Design of materials based vehicular hydrogen storage systems that will allow for a driving range of greater than 300 miles

Approach: Leverage in-house expertise in various engineering disciplines and prior experience with metal hydride system prototyping to advance materials based H_2 storage for automotive applications

Technical Accomplishments and Progress:

- IPPSSM: Completed assessment of on-board reversible metal hydride system and diverted it to different markets.
- Developed on-board reversible metal hydride materials requirements in order for a system to meet the DOE/U.S.Drive 2017 targets.
- Supported FMEA of cryo-adsorption and chemical hydride systems.
- Designed Gas/Liquid Separator (GLS) setup for chemical hydride system.
- Performed FMEA of GLS setup.
- Developed and demonstrated more efficient and regenerable NH_3 filter with high capacity over a wide range of operating temperature.
- Demonstrated binderless compaction of super activated carbon.
- Characterized thermal conductivity anisotropy of MOF-5 + ENG ‘worms’.
- Tested performance of SS particulate filters.

Acknowledgements

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Technical Back-Up Slides

Partner level Phase 2 to Phase 3 Go/No-Go Criteria

Month/Year	Partner-Level Go/No-Go
March/2013	Report on ability to develop a gas liquid separator capable of handling 720 mL/min liquid phase and 600 L/min of H_2 @ STP (40 wt% AB @ 2.35 Eq H_2 and max H_2 flow of 0.8 g/s H_2) fluid having a viscosity less than 1500cp resulting in a gas with less than 100ppm aerosol having a mass less than 5.4 kg and volume less than 19 liters.
	Report on ability to develop an ammonia scrubber with a minimum replacement interval of 1800 miles of driving resulting in a maximum ammonia outlet concentration of 0.1ppm (inlet concentration = 500ppm) having a maximum mass of 1.2 kg and a maximum volume of 1.6 liters.

All targets are equal

Parameter	**Unit**	**2017 Target**	**System +5.6 kg H_2**	**Compressed***	
				350 bar	700 bar
System gravimetric capacity	[wt.%]	5.5	102 kg	117 kg	119 kg
System volumetric capacity	g-H_2/L	40	140 L	329 L	224 L
Refueling time [5 kg-H2]	minutes	3.3			
On-board energy efficiency	%	90			
Purity		SAE J2719			
Operating ambient T	°C	-40 to +60			
Operational cycle life	#	1500			
Minimum delivery pressure (abs.)	bar	5			
Etc.					

*5.6 kg usable H_2: 5 kpsi: 4.8 wt.%, 17 g/L;10 kpsi: 4.7 wt.%, 25 g/L

Minimum balance of plant requirements

- Using waste heat
  Use the TIAX 350 bar system BOP

	Mass	Volume
	kg	L
Check valve	0.2	0.1
Manual valve	0.2	0.1
Solenoid valve	0.6	0.4
Relief valve	0.3	0.1
Pressure transducer	0.1	0.0
Temperature transducer	0.1	0.0
Pressure regulator	2.1	0.7
Pressure relief device	0.5	0.3
Piping	5.0	1.0
Boss	0.4	0.1
Vehicle interface bracket	2.0	0.5
Fill system control module	1.0	1.0
Miscellaneous	2.0	0.5
Total	**14.5**	**4.8**

- Combusting H_2
  Add a combustion loop to the 350 bar BOP
  8 kW microchannel HX/combustor sized by OSU

	Mass	Volume
	kg	L
Coolant valve	1.0	0.4
Coolant fluid	3.0	0.0
Coolant pump	3.0	2.4
Coolant lines	4.0	2.6
System insulation	1.0	5.0
Oil tank	0.7	2.6
Catalytic heater	2.5	0.8
Blower	0.4	0.2
Headers & fittings	0.5	0.1
Sub-Total	**16.0**	**14.1**
	14.5	4.8
Total	**30.5**	**18.9**

T.Q. Hua, R.K. Ahluwalia, J.K. Peng, et al., “Technical assessment of compressed hydrogen storage tank systems for automotive applications,” *Int. J. Hydrogen Energy* **36**, 3037–3049 (2011).

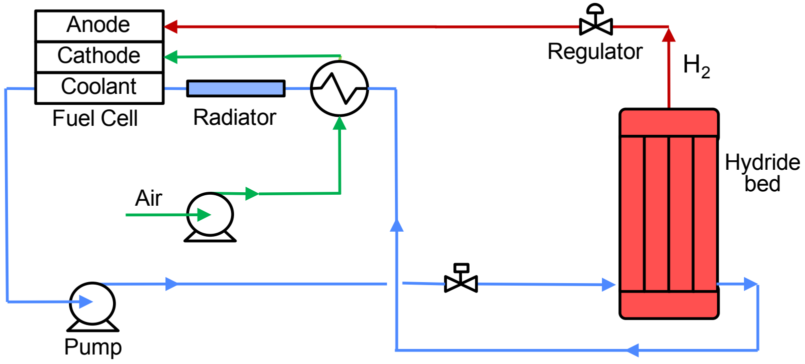
Drive cycles & test conditions for use in the framework

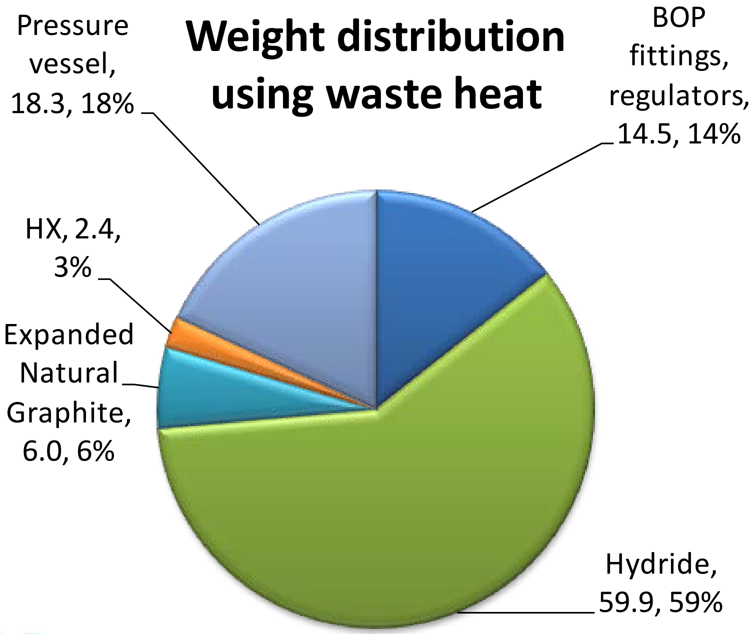
Case	Test Schedule	Cycles	Description	Test Temp (°F)	Distance per cycle (miles)	Duration per cycle (minutes)	Top Speed (mph)	Average Speed (mph)	Max. Acc. (mph /sec)	Stops	Idle	Avg. H2 Flow (g/s)*	Peak H2 Flow (g/s)*	Expected Usage
1	**Ambient Drive Cycle** - Repeat the EPA FE cycles from full to empty and adjust for 5 cycle post-2008	UDDS	Low speeds in stop-and-go urban traffic	75 (24 C)	7.5	22.8	56.7	19.6	3.3	17	19%	0.09	0.69	**1. Establish baseline fuel economy (adjust for the 5 cycle based on the average from the cycles)** **2. Establish vehicle attributes** **3. Utilize for storage sizing**
		HWFET	Free-flow traffic at highway speeds	75 (24 C)	10.26	12.75	60	48.3	3.2	0	0%	0.15	0.56	
2	**Aggressive Drive Cycle** - Repeat from full to empty	US06	Higher speeds; harder acceleration & braking	75 (24 C)	8	9.9	80	48.4	8.46	4	7%	0.20	1.60	**Confirm fast transient response capability – adjust if system does not perform function**
3	**Cold Drive Cycle** - Repeat from full to empty	FTP-75 (cold)	FTP-75 at colder ambient temperature	-4 (-20 C)	11.04	31.2	56	21.1	3.3	23	18%	0.07	0.66	**1. Cold start criteria** **2. Confirm cold ambient capability – adjust if system does not perform function**
4	**Hot Drive Cycle** - Repeat from full to empty	SC03	AC use under hot ambient conditions	95 (35 C)	3.6	9.9	54.8	21.2	5.1	5	19%	0.09	0.97	**Confirm hot ambient capability - adjust if system does not perform function**
5	**Dormancy Test**	n/a	Static test to evaluate the stability of the storage system	95 (35 C)	0	31 days	0	0	0	100%	100%			**Confirm loss of useable H2 target**

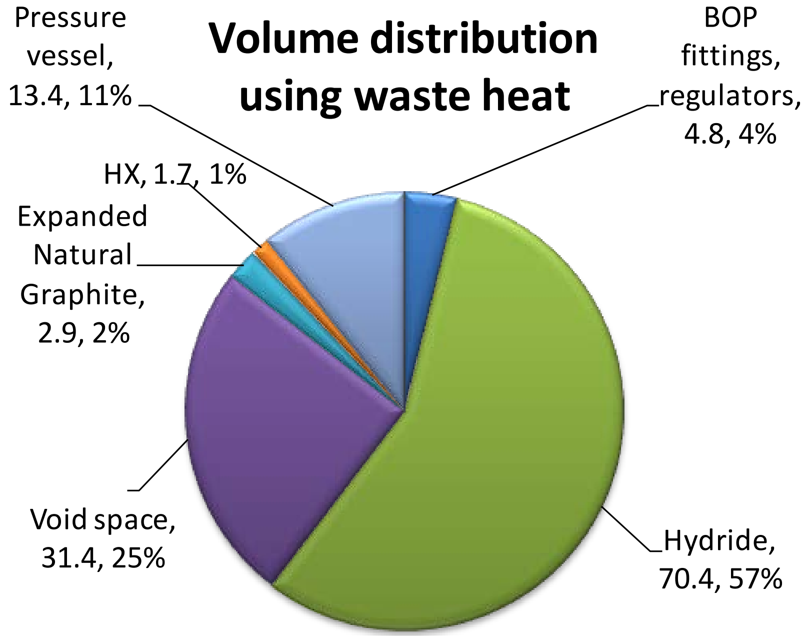
*Based on NREL simulation with compact vehicle, 5.6 kg usable H2, 80 kW fuel cell with a 20 kW battery

Complete system using waste heat only

- Satisfies all targets.
- $\Delta H = -27$ kJ/mol-H_2, $\Delta S = -105$ J/mol-H_2/K
- 11 wt% pure material capacity
- T (5 bar) = 20.7 C
- On-board efficiency: ~100%
- System: 101 kg (5.8 wt%), 124 liters (48 g-H_2/L)
- 66 kg of hydride delivers 5.9 kg-H_2.

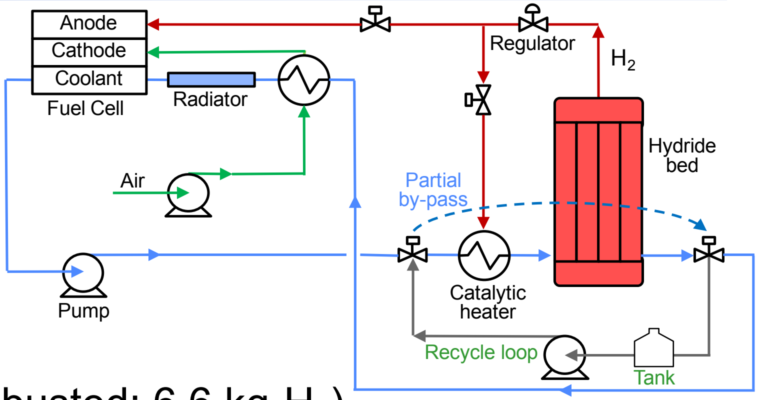


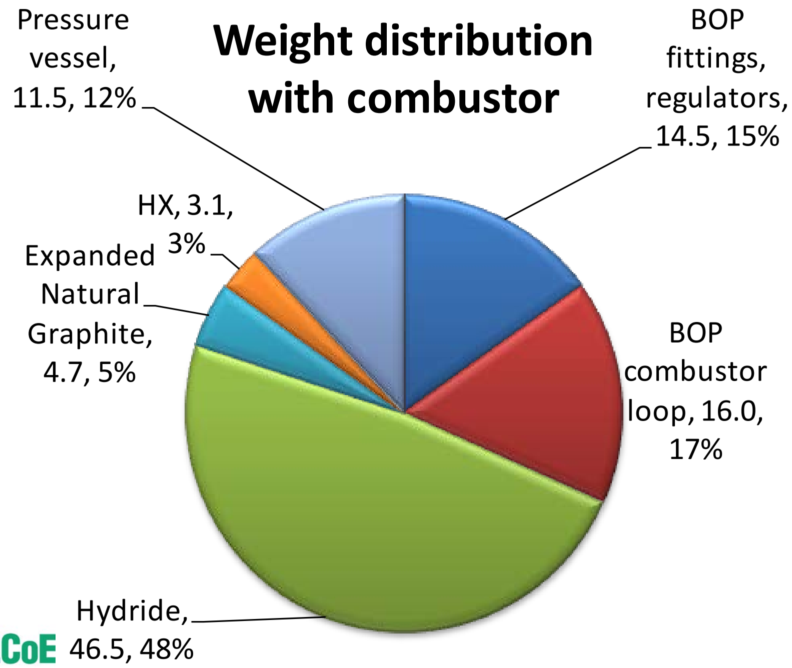


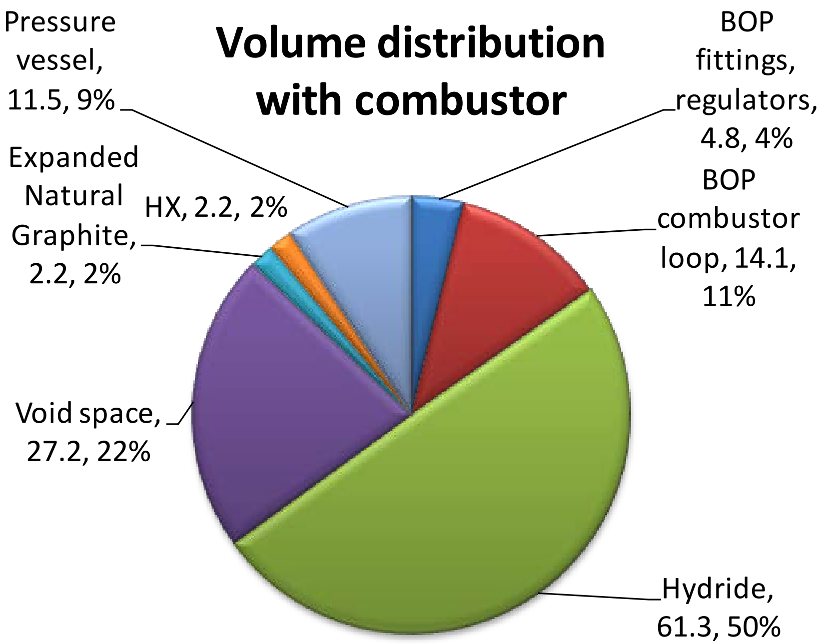


Complete system with combustion

- Satisfies all targets except on-board system efficiency.
- $\Delta H = -40$ kJ/mol-H_2, $\Delta S = -114$ J/mol-H_2/K
- 17 wt% pure material capacity
- T (5 bar) = 122.8 C
- On-board efficiency: ~81%
- System: 103 kg (5.2 wt%), 126 liters (43 g-H_2/L)
- Operating at 130C delivers 5.4 kg-H_2 (delivered + combusted: 6.6 kg-H_2)

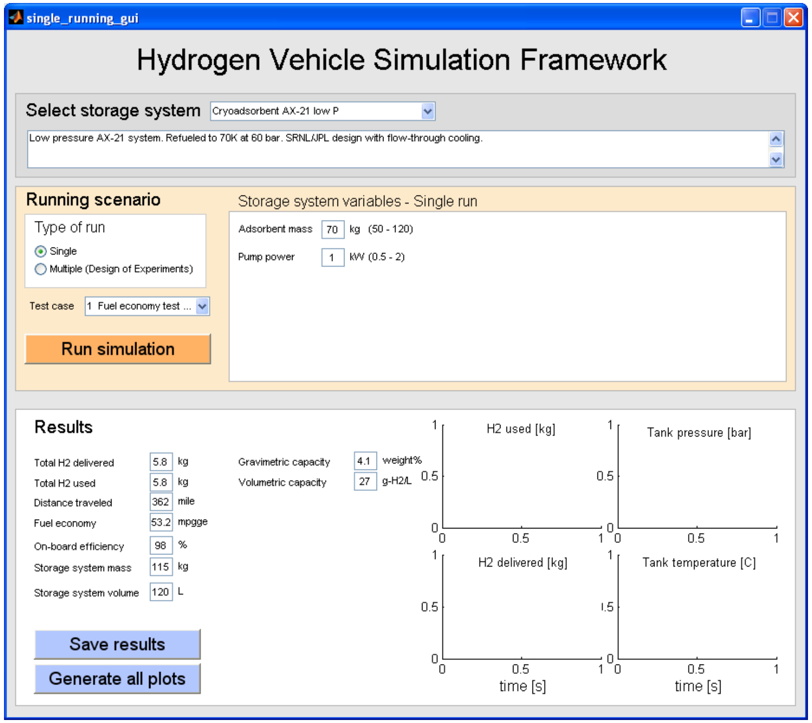






IPPSSM framework development: GUI interface

- Goal: make the framework more user-friendly and expand its capabilities



single_running_gui

Hydrogen Vehicle Simulation Framework

Select storage system: Cryoadsorbent AX-21 low P

Low pressure AX-21 system. Refueled to 70K at 60 bar. SRNL/JPL design with flow-through cooling.

Running scenario

Type of run

● Single
○ Multiple (Design of Experiments)

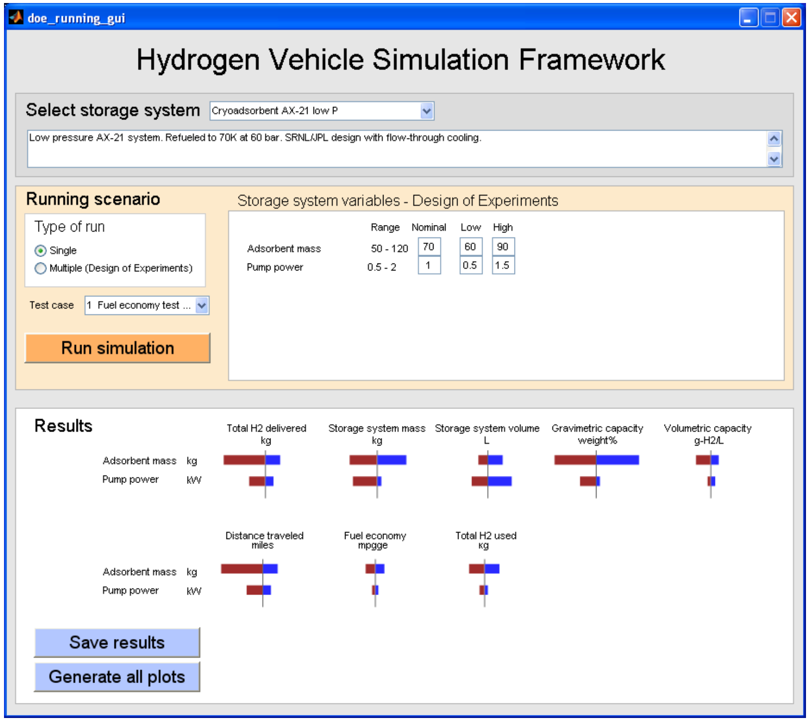
Test case: 1 Fuel economy test ...

Storage system variables - Single run

Adsorbent mass: 70 kg (50 - 120)
Pump power: 1 kW (0.5 - 2)

Results

Total H2 delivered: 5.8 kg
Total H2 used: 5.8 kg
Distance traveled: 362 mile
Fuel economy: 53.2 mpgge
On-board efficiency: 98 %
Storage system mass: 115 kg
Storage system volume: 120 L
Gravimetric capacity: 4.1 weight%
Volumetric capacity: 27 g-H2/L



doe_running_gui

Hydrogen Vehicle Simulation Framework

Select storage system: Cryoadsorbent AX-21 low P

Low pressure AX-21 system. Refueled to 70K at 60 bar. SRNL/JPL design with flow-through cooling.

Running scenario

Type of run

● Single
○ Multiple (Design of Experiments)

Test case: 1 Fuel economy test ...

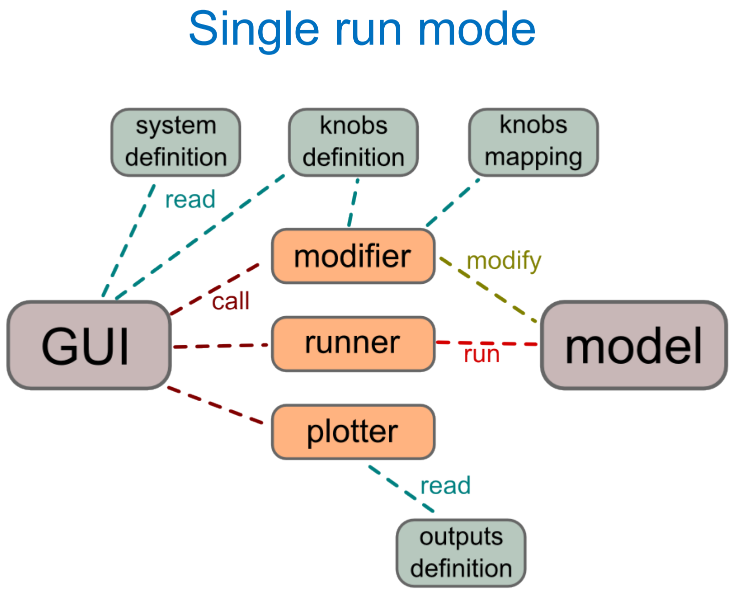
Storage system variables - Design of Experiments

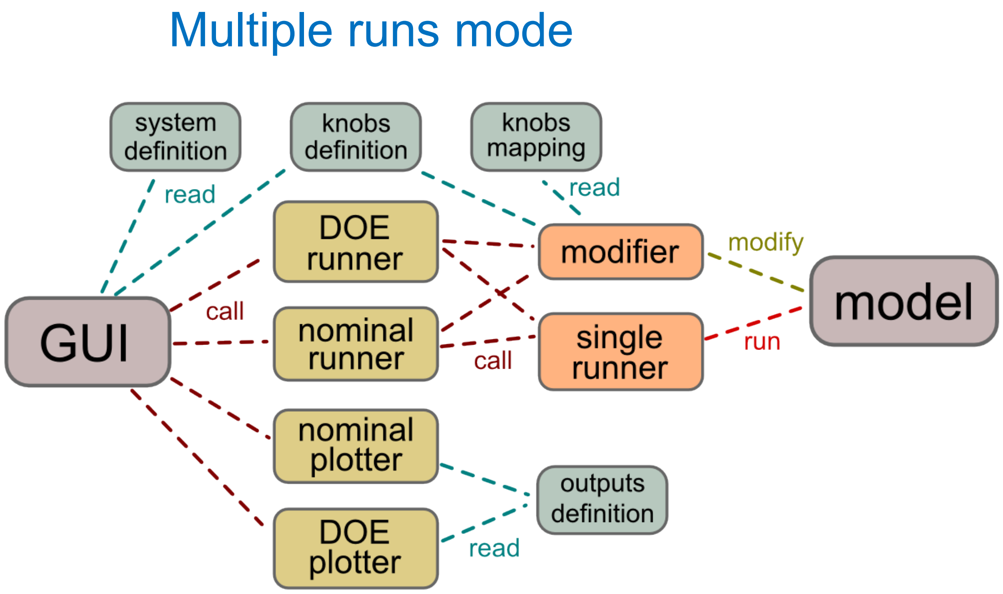
	Range	Nominal	Low	High
Adsorbent mass	50 - 120	70	60	90
Pump power	0.5 - 2	1	0.5	1.5

Results

- Conditions:
  - Single system, single run
  - Single system, parameter sweeps
  - System-to-system comparisons

Framework GUI structure: designed for change





- GUI designed for change: modules with small responsibilities
  - Currently: Matlab®-based
  - Potentially: web-based

HSECoE

United Technologies Research Center

Gas/Liquid Separator Parameters

Parameter	Range
GLS Target Volume	≤ 19 liters
GLS Target Weight	≤ 5.4 kg
GLS Operating Temperature	~ 200-250°C
GLS Operating Pressure	~ 35 bar (508 psi)
Gas Type	Nitrogen
Gas Flow Rate	1,067 slpm (0°C, 1atm))
Liquid Type	Silicone Oil AP 100
Flow Rate	~61 ml/s (1.4 L/min)
Density	0.8-1.4 (g/mL) at 20°C
Viscosity	~20-100 (cP) at 25°C
Surface Tension	~ 0.0375 (N/m)

Flammability Test Apparatus (for Gases or Liquids)

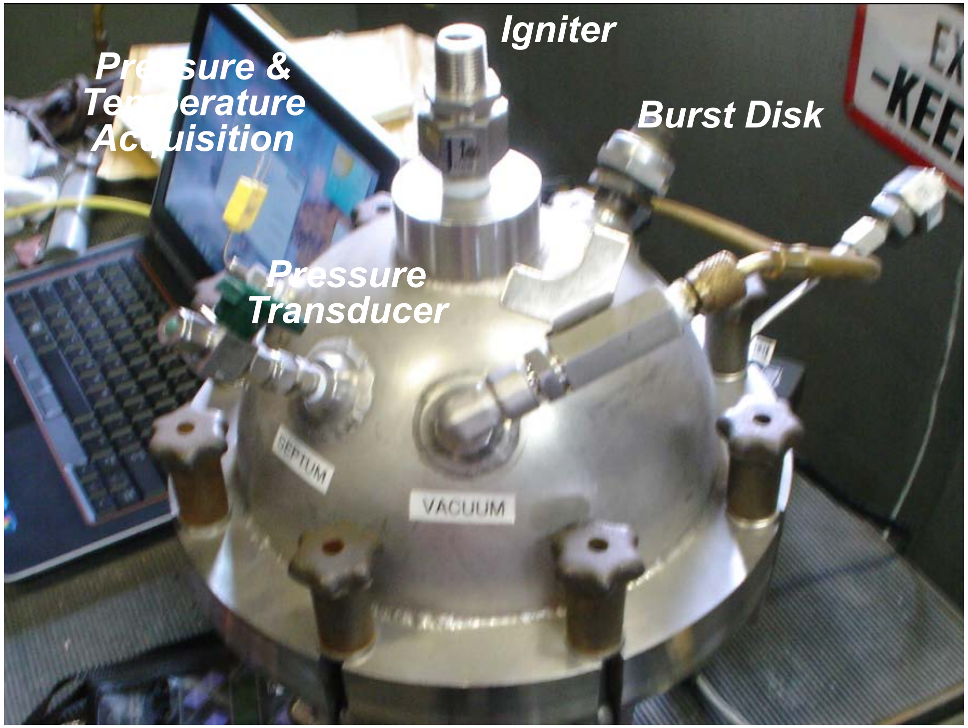


Figure 1: KG Apparatus showing the igniter, burst desk, ports for vacuum, gases, and liquids.

The burst desk is designed for 350 psi.

The data acquisition is set for 1000 samples per second for T & P.

The KG apparatus will be used for measuring the flammability of the slurry (solid AB in silicon oil).

Flammability tests follows *ASTM E-2079*

Figure 2: Two parts of the stainless steel sphere (8.8 liters free volume).

UTRC Contributed to Cryo-adsorption Tank Test Plan

Failure Mechanism	Effects	Validation Test
1) Liner's microcracks initiation and propagation as a result of exposure to LH2 temperature.	Should this failure mechanism occur, H_2 permeation / leakage through the liner could increase over time.	Cryogenic cycling test (for both Type-III and Type-IV liners). Use electron microscopy to compare the liner microstructure before and after the cryogenic cycling test.
2) Delamination and/or blistering of the carbon composite overwrap.	Loss of structural integrity of the tank.	Cryogenic cycling test. Use electron microscopy to compare the composite microstructure before and after the cryogenic cycling test.
3) Debonding of the carbon fiber / epoxy resin bonding matrix material.	Loss of structural integrity of the tank.	Cryogenic cycling test. Use electron microscopy to compare the composite microstructure before and after the cryogenic cycling test.
4) Air leaks into tank due to thermal shock caused by exposure to the cryogenic liquid.	Leaked air condenses at ~ 79^{o}K and, hence, oxygen enrichment is a concern.	Tank leak testing / cryogenic pressure burst test. Pressurize the tank with LN2 (77 ^{o}K or below if possible).

UTRC Contributed to Cryo-adsorption Tank Test Plan (Cont.)

Failure Mechanism	Effects	Validation Test
5) Degradation of mechanical properties (fracture toughness and tensile strength) of the liner and the composite fiber as a results of exposure to LH2.	Loss of structural integrity of the tank. Increased H2 permeation through the liner material.	Mechanical testing of the composite fiber and the liner material. Samples have to be mechanically tested while the submerged in LN2.
6) Type IV liner failure due to thermal fatigue stress concentration.	Liner failure and hydrogen leakage.	Cyclical thermal fatigue test. Cycle the test sample between being submerged in LN2 for several hours and being exposed to ambient air for several hours.

Phase 2: UTRC - Pressure Vessel Safety Tests

Proposed Test	Test Procedure
1. Cryogenic cycling.	• Subject the tank to cryogenic cycles using liquid nitrogen at temperature in the range: 50°K ≤ T ≤ 77°K and at pressure equal to 1 bar. Using temperatures < 77°K is dependent of the existing lab capabilities. • Each cryogenic cycle involves cooling down the tank from room temperature to cryogenic temperature and then warming up to room temperature.
2. Mechanical testing of tank's carbon fiber composite and liner material (Types III and IV).	• Immerse test samples (carbon composite overwrap or the liner) in LN2 at 50 or 77°K for extended period of time. • Test the samples for fracture toughness and tensile strength while the sample is submerged in the LN2.
3. Cryogenic pressure cycling using LN2 (@ T ≤ 77°K).	Subject the tank to pressure cycles between 20 bar (10% of NWP) and 200 bar (100% of NWP) . OR, cycle between 10% NWP and 125% NWP (250 bar). (FMVSS 304)
4. Thermal cycling. (Ambient temperature outside tank).	Subject the tank to temperature cycles between 20°K and 77°K and at 1 bar pressure.
5. Sequential pressure and temperature cycling. (Ambient temperature outside tank).	Subject the pressure cycles between 20 bar and 250 bar followed by one temperature cycle between 20°K and 77°K at 1 bar. Repeat this sequence for a TBD number of cycles.
6. Burst pressure test. (Ambient temperature outside tank).	Subject the new tank (as well as a pressure cycled tank) to a burst test using liquid nitrogen at 77°K.
7. Hydrogen permeation test (Type-IV liner).	Test either the entire tank or a specimen of the liner for H2 permeation. Use LH2 at 20°K and 125% NWP.