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


United Technologies Research Center

H2

DOE Hydrogen Program

Annual Merit Review
Washington, DC
May 15, 2012

Project ID: ST006

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

* DOE EERE HFCIT Program Multi-year Plan for Storage

IEA HIA Task 22
Objectives

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

<table>
<thead>
<tr>
<th>Performance Measure</th>
<th>Units</th>
<th>2010</th>
<th>2017</th>
<th>Ultimate</th>
</tr>
</thead>
<tbody>
<tr>
<td>System Gravimetric Capacity</td>
<td>g H₂ /kg system</td>
<td>45</td>
<td>55</td>
<td>75</td>
</tr>
<tr>
<td>System Volumetric Capacity</td>
<td>g H₂ /L system</td>
<td>28</td>
<td>40</td>
<td>70</td>
</tr>
<tr>
<td>System fill time (for 5 kg H₂)</td>
<td>minutes</td>
<td>4.2</td>
<td>3.3</td>
<td>2.5</td>
</tr>
<tr>
<td>Fuel Purity</td>
<td>% H₂</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Relevance

- 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
- H₂ quality (NH₃ adsorbent, particulate filter)
- Compaction/Materials thermal conductivity enhancement
- Risk Analysis: Failure mode and effect analysis (FMEA)
Established partner-level Phase 2 to Phase 3 transition criteria in updated SOPO
IPPSSM Framework Application

Collaborations

System Results for comparison with DOE targets

Vehicle-Level Model (NREL)
- System performance
  - Drive cycle
  - Power request to fuel cell
  - Power achieved by fuel cell
- Parameter inputs
  - Vehicle
  - Fuel Cell
  - Storage Systems

Fuel Cell System (Ford)
- H₂ stream in
- H₂ request
- Power requested
- Power achieved

H₂ Storage Systems
- UTRC NaAlH₄ Powder
  - H₂ requested
  - H₂ stream out
- UTRC NaAlH₄ Pellets
- UTRC/SRNL 1:1 Li-Mg-N-H
- GM NaAlH₄
- GM/SRNL/JPL AX-21
- PNNL Solid AB
- PNNL/LANL Liquid AB
- 350 bar Compressed
- 700 bar Compressed

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 H₂ storage systems on a common basis achieved by team effort

Chemical Hydride
Cryo-adsorbents
On-board reversible Metal Hydride

DOE/U.S. DRIVE Light-Duty Vehicles

Different Markets
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.
### Thermodynamic Properties

Equation for equilibrium pressure: fit $\Delta S$ vs $\Delta H$

- $y = 0.49x - 56.83$, $R^2 = 0.91$
- $y = 0.70x - 86.28$, $R^2 = 0.48$

#### Heat Transfer (Acceptability Envelope)

\[
\dot{Q} = \left(\frac{-\Delta H}{w_{H_2}}\right)\left(\frac{\Delta m_{H_2}}{\Delta t}\right)\left(\frac{M_{hydride}}{\rho_{bed}}\right)^{-1}
\]

- $\dot{Q}$: heating rate per unit volume
- $\Delta H$: heat released per mass of $H_2$ absorbed
- $\Delta m_{H_2}$: refueling rate
- $M_{hydride}$: 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", $\Delta T = 45^\circ C$

Short fill time (3.3 minutes)

### Kinetics

- 85% of capacity in 3.3 minutes

### Weight and Volume

**Type IV tank**

\[
y = 0.1368x + 3.7511, \quad R^2 = 0.9938
\]

- Other parts from BOP Library (PNNL)

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**Technical Accomplishments and Progress**

Analysis anchored in metal hydride databases
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:
    - $\Delta H < 27$ to $32 \text{ kJ/mol}$ (depends on drive cycle); >11 wt.%
  - Combustor loop:
    - $\Delta H > 27$ to $32 \text{ kJ/mol}$ (depends on drive cycle); >16.5 wt.% due to BOP weight increase and H$_2$ combustion
Technical Accomplishments and Progress

Available metal hydride materials vs. requirements

Large gap with material properties needed to meet DOE 2017 targets

Gravimetric capacity [wt.%]

Enthalpy [kJ/mole-H2]

- 5 bar at 60°C
- 5 bar at 165°C (-24.2 kJ/mol-H2)
- 100 bar at 60°C

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

NH₃ filter:
1.2 kg, 1.6 Liter

GLS:
5.4 kg, 19 Liter
Technical Accomplishments and Progress

Gas-Liquid Separation Test Facility

- Static Mixer
- Gas/Liquid Separator
- GLS
- Mass Flow Controller
- Pump
- Drain
- Surrogate Liquid Chemical Hydride Supply Tank

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
H$_2$ Quality (NH$_3$ Mitigation*)

Dynamic Breakthrough

Feed:
10,000 ppm NH$_3$ in N$_2$
60 psig, 68 F

0.1 ppm

50 wt.% MnCl$_2$ on IRH-33

More Efficient Filter by Capacity Improvement

Cu-BTC
IRH-33

Support
MeCl$_2$

NH$_3$ Filter Weight

1800 miles

NH$_3$ filter: 1.2 kg, 1.6 Liter

Regenerable

Dynamic NH$_3$ sorption capacity [wt. %]

<table>
<thead>
<tr>
<th>Adsorption/desorption cycle number [-]</th>
<th>ZnCl$_2$ on IRH-33</th>
<th>MnCl$_2$ on IRH-33</th>
<th>MgCl$_2$ on IRH-33</th>
</tr>
</thead>
<tbody>
<tr>
<td>0%</td>
<td>15%</td>
<td>10%</td>
<td>5%</td>
</tr>
<tr>
<td>5%</td>
<td>10%</td>
<td>5%</td>
<td>11%</td>
</tr>
</tbody>
</table>

Capacity over full ambient temperature range

NH$_3$ capacity [wt. %]

* LANL addresses Boron containing impurities
Springback limits MaxSorb density to 0.3 g/cm³ with high weight penalty for thermal conductivity enhancement.

**Vibration Packing**
- Resulting density is equal to tap density (0.3 g/cm³):
  - No density enhancement

**Compressed Foam Enclosure**
- Springback limits MaxSorb density to 0.3 g/cm³ with high weight penalty for thermal conductivity enhancement.

**Filter Press**
- Density limited to 0.3 g/cm³ 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/cm³: Some loss of SA BET but FAST!
Technical Accomplishments and Progress

Cryo-Adsorption System Support: Spark Plasma Sintering

SPS technique

- Impurities reduce required operating temperature
- $\rho=0.6 \text{ g/cm}^3$ achieved

Pore volume loss

- Sintering reduces pore volume similar as use of binder

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

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/cm³
- 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)

<table>
<thead>
<tr>
<th>parameter</th>
<th>95% confidence interval</th>
<th>Unit</th>
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</thead>
<tbody>
<tr>
<td>kX</td>
<td>3.32 &lt; 3.45 &lt; 3.58</td>
<td>W/m/K</td>
</tr>
<tr>
<td>kY</td>
<td>1.44 &lt; 1.49 &lt; 1.55</td>
<td>W/m/K</td>
</tr>
<tr>
<td>kZ</td>
<td>0.280 &lt; 0.286 &lt; 0.292</td>
<td>W/m/K</td>
</tr>
<tr>
<td>C_p</td>
<td>1395 &lt; 1438 &lt; 1484</td>
<td>J/kg/K</td>
</tr>
</tbody>
</table>

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

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

- High values of heat transfer coefficients

| h_x | 638 < 645 < 652 | W/m²/K |
| h_y | 706 < 714 < 724 | W/m²/K |
| h_z | 773 < 783 < 795 | W/m²/K |

* MOF-5 powder produced by BASF and kindly provided by Ford
** ENG-worms kindly provided by SGL Carbon
H₂ 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.

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

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum particulate size</td>
<td>&lt; 10 μm</td>
</tr>
<tr>
<td>Particulate concentration</td>
<td>&lt;1000 μg/m³</td>
</tr>
</tbody>
</table>

Technical Accomplishments and Progress
# FY12 and FY13 Plan

- Based on revised SOPO resulting from budget reduction

## Proposed Future Work

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

<table>
<thead>
<tr>
<th>Month/Year</th>
<th>Partner-Level Go/No-Go</th>
</tr>
</thead>
<tbody>
<tr>
<td>March/2013</td>
<td>Report on ability to develop a gas liquid separator capable of handling 720 mL/min liquid phase and 600 L/min of H₂ @ STP (40 wt% AB @ 2.35 Eq H₂ and max H₂ flow of 0.8 g/s H₂) 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.</td>
</tr>
<tr>
<td></td>
<td>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.</td>
</tr>
</tbody>
</table>
All targets are equal

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

*5.6 kg usable H₂: 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

- Combusting H₂
  Add a combustion loop to the 350 bar BOP
  8 kW microchannel HX/combustor sized by OSU

<table>
<thead>
<tr>
<th>Item</th>
<th>Mass (kg)</th>
<th>Volume (L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Check valve</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>Manual valve</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>Solenoid valve</td>
<td>0.6</td>
<td>0.4</td>
</tr>
<tr>
<td>Relief valve</td>
<td>0.3</td>
<td>0.1</td>
</tr>
<tr>
<td>Pressure transducer</td>
<td>0.1</td>
<td>0.0</td>
</tr>
<tr>
<td>Temperature transducer</td>
<td>0.1</td>
<td>0.0</td>
</tr>
<tr>
<td>Pressure regulator</td>
<td>2.1</td>
<td>0.7</td>
</tr>
<tr>
<td>Pressure relief device</td>
<td>0.5</td>
<td>0.3</td>
</tr>
<tr>
<td>Piping</td>
<td>5.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Boss</td>
<td>0.4</td>
<td>0.1</td>
</tr>
<tr>
<td>Vehicle interface bracket</td>
<td>2.0</td>
<td>0.5</td>
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<tr>
<td>Fill system control module</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Miscellaneous</td>
<td>2.0</td>
<td>0.5</td>
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<tr>
<td><strong>Total</strong></td>
<td><strong>14.5</strong></td>
<td><strong>4.8</strong></td>
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<table>
<thead>
<tr>
<th>Item</th>
<th>Mass (kg)</th>
<th>Volume (L)</th>
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<tbody>
<tr>
<td>Coolant valve</td>
<td>1.0</td>
<td>0.4</td>
</tr>
<tr>
<td>Coolant fluid</td>
<td>3.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Coolant pump</td>
<td>3.0</td>
<td>2.4</td>
</tr>
<tr>
<td>Coolant lines</td>
<td>4.0</td>
<td>2.6</td>
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<tr>
<td>System insulation</td>
<td>1.0</td>
<td>5.0</td>
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<tr>
<td>Oil tank</td>
<td>0.7</td>
<td>2.6</td>
</tr>
<tr>
<td>Catalytic heater</td>
<td>2.5</td>
<td>0.8</td>
</tr>
<tr>
<td>Blower</td>
<td>0.4</td>
<td>0.2</td>
</tr>
<tr>
<td>Headers &amp; fittings</td>
<td>0.5</td>
<td>0.1</td>
</tr>
<tr>
<td><strong>Sub-Total</strong></td>
<td><strong>16.0</strong></td>
<td><strong>14.1</strong></td>
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<table>
<thead>
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<th>Item</th>
<th>Mass (kg)</th>
<th>Volume (L)</th>
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<tbody>
<tr>
<td><strong>Total</strong></td>
<td><strong>14.5</strong></td>
<td><strong>4.8</strong></td>
</tr>
</tbody>
</table>

**Total**                     | **30.5**  | **18.9**   |

## Drive cycles & test conditions for use in the framework

<table>
<thead>
<tr>
<th>Case</th>
<th>Test Schedule</th>
<th>Cycles</th>
<th>Description</th>
<th>Test Temp (°F)</th>
<th>Distance per cycle (miles)</th>
<th>Duration per cycle (minutes)</th>
<th>Top Speed (mph)</th>
<th>Average Speed (mph)</th>
<th>Max. Acc. (mph/sec)</th>
<th>Stops</th>
<th>Idle</th>
<th>Expected Usage</th>
</tr>
</thead>
</table>
| 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 |
| 2    | Aggressive Drive Cycle  
- Repeat from full to empty | US06 | Higher speeds; harder acceleration & braking | 75 (24 C) | 8 | 9.9 | 80 | 48.3 | 3.2 | 0 | 0% | 0.15 | 0.56 | 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  
- Static test to evaluate the stability of the storage system | n/a | 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.
- $\Delta H = -27 \text{ kJ/mol-H}_2$, $\Delta S = -105 \text{ J/mol-H}_2/\text{K}$
- 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$_2$.

Weight distribution using waste heat:
- Hydride: 59.9, 59%
- Expanded Natural Graphite: 6.0, 6%
- HX: 2.4, 3%
- Pressure vessel: 18.3, 18%

Volume distribution using waste heat:
- Hydride: 70.4, 57%
- Void space: 31.4, 25%
- Expanded Natural Graphite: 2.9, 2%
- HX: 1.7, 1%
- Pressure vessel: 13.4, 11%
- BOP fittings, regulators: 14.5, 14%
Complete system with combustion

- Satisfies all targets except on-board system efficiency.
- \( \Delta H = -40 \text{ kJ/mol-H}_2 \), \( \Delta S = -114 \text{ J/mol-H}_2/\text{K} \)
- 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

- Conditions:
  - Single system, single run
  - Single system, parameter sweeps
  - System-to-system comparisons
GUI designed for change: modules with small responsibilities
- Currently: Matlab®-based
- Potentially: web-based
# Gas/Liquid Separator Parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>GLS Target Volume</td>
<td>$\leq 19$ liters</td>
</tr>
<tr>
<td>GLS Target Weight</td>
<td>$\leq 5.4$ kg</td>
</tr>
<tr>
<td>GLS Operating Temperature</td>
<td>$\sim 200$-250°C</td>
</tr>
<tr>
<td>GLS Operating Pressure</td>
<td>$\sim 35$ bar (508 psi)</td>
</tr>
<tr>
<td>Gas Type</td>
<td>Nitrogen</td>
</tr>
<tr>
<td>Gas Flow Rate</td>
<td>1,067 slpm (0°C, 1 atm)</td>
</tr>
<tr>
<td>Liquid Type</td>
<td>Silicone Oil AP 100</td>
</tr>
<tr>
<td>Flow Rate</td>
<td>$\sim 61$ ml/s (1.4 L/min)</td>
</tr>
<tr>
<td>Density</td>
<td>0.8-1.4 (g/mL) at 20°C</td>
</tr>
<tr>
<td>Viscosity</td>
<td>$\sim 20$-100 (cP) at 25°C</td>
</tr>
<tr>
<td>Surface Tension</td>
<td>$\sim 0.0375$ (N/m)</td>
</tr>
</tbody>
</table>
Flammability Test Apparatus (for Gases or Liquids)

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

Flammability tests follows ASTM E-2079

The burst desk is designed for 350 psi.

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

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

Figure 2: Two parts of the stainless steel sphere (8.8 liters free volume).
# UTRC Contributed to Cryo-adsorption Tank Test Plan

<table>
<thead>
<tr>
<th>Failure Mechanism</th>
<th>Effects</th>
<th>Validation Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) Liner’s microcracks initiation and propagation as a result of exposure to LH2 temperature.</td>
<td>Should this failure mechanism occur, H₂ permeation / leakage through the liner could increase over time.</td>
<td><strong>Cryogenic cycling test</strong> (for both Type-III and Type-IV liners). Use electron microscopy to compare the liner microstructure before and after the cryogenic cycling test.</td>
</tr>
<tr>
<td>2) Delamination and/or blistering of the carbon composite overwrap.</td>
<td>Loss of structural integrity of the tank.</td>
<td><strong>Cryogenic cycling test</strong>. Use electron microscopy to compare the composite microstructure before and after the cryogenic cycling test.</td>
</tr>
<tr>
<td>3) Debonding of the carbon fiber / epoxy resin bonding matrix material.</td>
<td>Loss of structural integrity of the tank.</td>
<td><strong>Cryogenic cycling test</strong>. Use electron microscopy to compare the composite microstructure before and after the cryogenic cycling test.</td>
</tr>
<tr>
<td>4) Air leaks into tank due to thermal shock caused by exposure to the cryogenic liquid.</td>
<td>Leaked air condenses at ~ 79°K and, hence, oxygen enrichment is a concern.</td>
<td><strong>Tank leak testing / cryogenic pressure burst test</strong>. Pressurize the tank with LN2 (77 °K or below if possible).</td>
</tr>
</tbody>
</table>

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33
<table>
<thead>
<tr>
<th>Failure Mechanism</th>
<th>Effects</th>
<th>Validation Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>5) Degradation of mechanical properties (fracture toughness and tensile strength) of the liner and the composite fiber as a result of exposure to LH2.</td>
<td>Loss of structural integrity of the tank.</td>
<td>Mechanical testing of the composite fiber and the liner material.</td>
</tr>
<tr>
<td></td>
<td>Increased H2 permeation through the liner material.</td>
<td>Samples have to be mechanically tested while the submerged in LN2.</td>
</tr>
<tr>
<td>6) Type IV liner failure due to thermal fatigue stress concentration.</td>
<td>Liner failure and hydrogen leakage.</td>
<td>Cyclical thermal fatigue test.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Cycle the test sample between being submerged in LN2 for several hours and being exposed to ambient air for several hours.</td>
</tr>
</tbody>
</table>
## Phase 2: UTRC - Pressure Vessel Safety Tests

<table>
<thead>
<tr>
<th>Proposed Test</th>
<th>Test Procedure</th>
</tr>
</thead>
</table>
| 1. Cryogenic cycling.                                                         | - Subject the tank to cryogenic cycles using liquid nitrogen at temperature in the range: $50^\circ K \leq T \leq 77^\circ K$ and at pressure equal to 1 bar. Using temperatures $< 77^\circ K$ 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 $77^\circ K$ 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 (@ $T \leq 77^\circ K$).                | 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 $20^\circ K$ and $77^\circ K$ 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 $20^\circ K$ and $77^\circ K$ 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 $77^\circ K$.                              |
| 7. Hydrogen permeation test (Type-IV liner).                                  | Test either the entire tank or a specimen of the liner for H2 permeation. Use LH2 at $20^\circ K$ and 125% NWP.                           |