Hydrogen Storage Engineering CENTER OF EXCELLENCE

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Project ID# ST004

SRNL-STI-2015-00252

Overview

Timeline

- Start: February 1, 2009
- End: June 30, 2015
- 95% Complete (as of 3/1/15)

Budget

- Total Center Funding:
  - DOE Share: $ 35,275,000
  - Cost Share: $ 3,322,000
  - FY ’14 Funding: $3,138,000
  - FY ‘15 Funding: $895,000
- Prog. Mgmt. Funding
  - FY ’14: $ 300,000
  - FY ’15: $ 300,000

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
K. System Life-Cycle Assessment
O. Hydrogen Boil-Off
P. Understanding Physi/Chemi-sorption
S. By-Product/Spent Material Removal

Partners

HSECoE Technical Objectives

Using systems engineering concepts, design innovative material-based hydrogen storage system architectures with the potential to meet DOE performance and cost targets.

- **Develop and validate system, engineering and design models** that lend insight into overall fuel cycle efficiency.
- Compile all relevant materials data for candidate storage media and **define required materials properties to meet the technical targets.**
- **Design, build and evaluate subscale prototype systems** to assess the innovative storage devices and subsystem design concepts, validate models, and improve both component design and predictive capability.

Phased Approach

Phase 2 Go/NoGo Decision:
Go forward with both adsorption and chemical hydrogen systems development.

Phase 3 Go/NoGo Decision:
Go forward with demonstration of two adsorption heat exchanger deigns.

Phase I:
System Requirements & Novel Concepts

Phase II:
Novel Concept Modeling Design & Evaluation

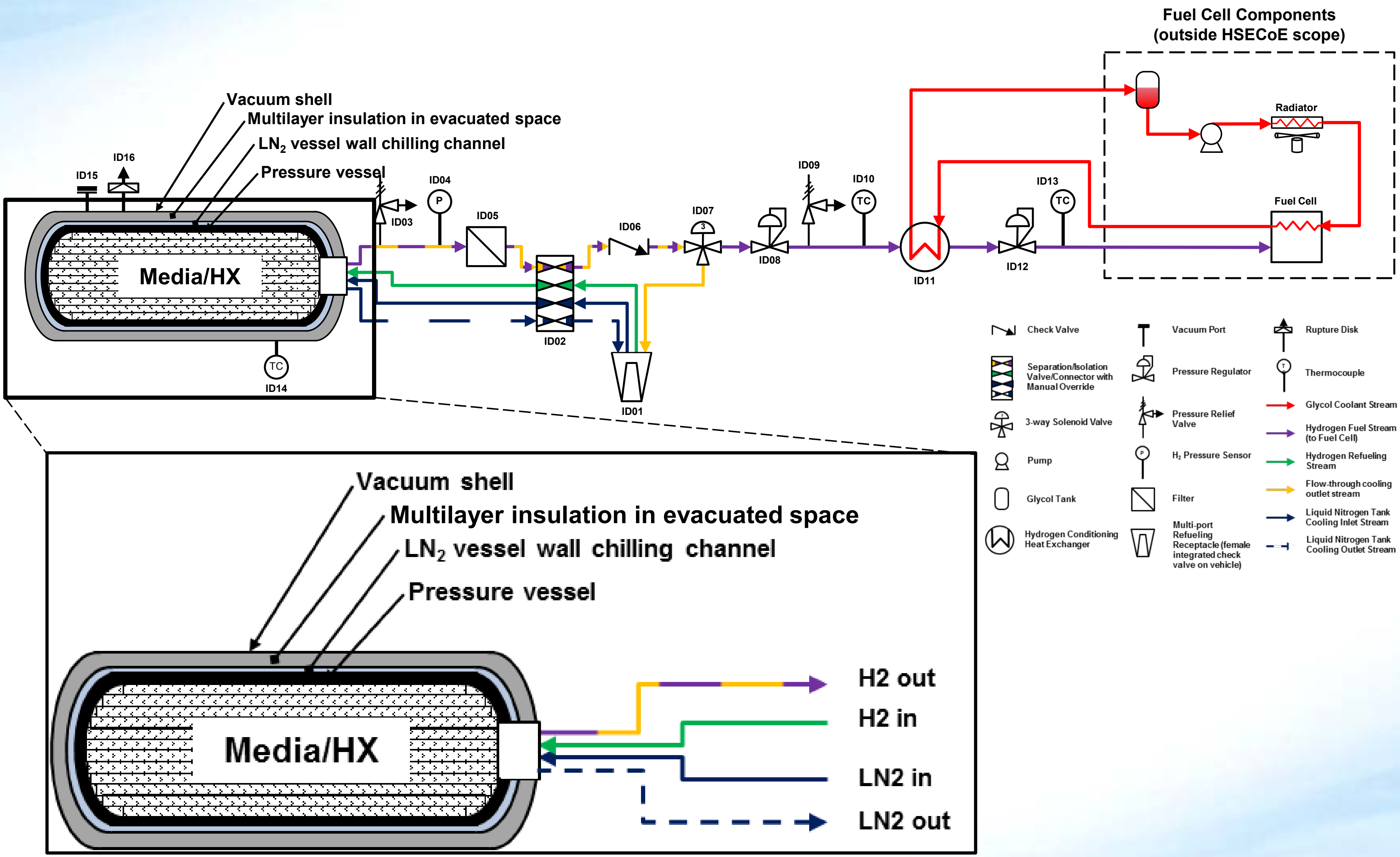
Phase III:
Sub-Scale Prototype Construction, Test and Evaluation

- **Where were we and where can we get to?**
  - Model Development
  - Benchmarking
  - Gap Identification
  - Projecting advances

- **How do we get there (closing the gaps) and how much further can we go?**
  - Novel Concepts
  - Concept Validation
  - Integration Testing
  - System Design

- **Put it all together and confirm claims.**
  - System Integration
  - System Assessments
  - Model Validation
  - Gap Analysis
  - Performance Projections

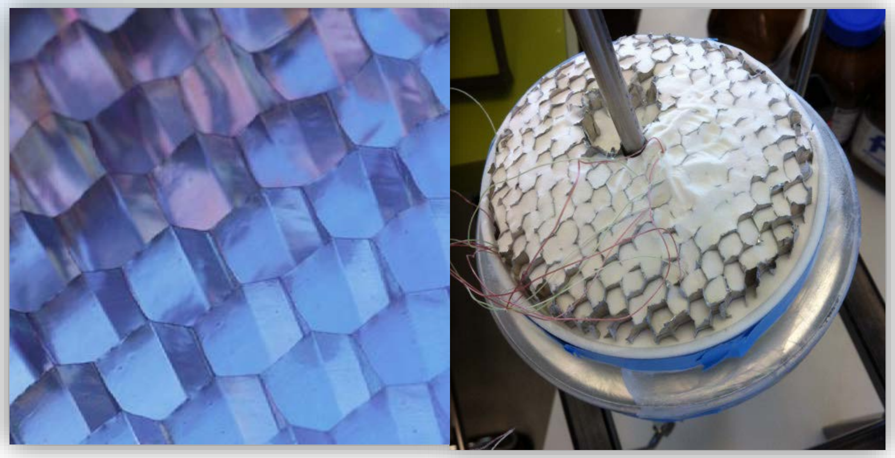
Adsorbent System Overview



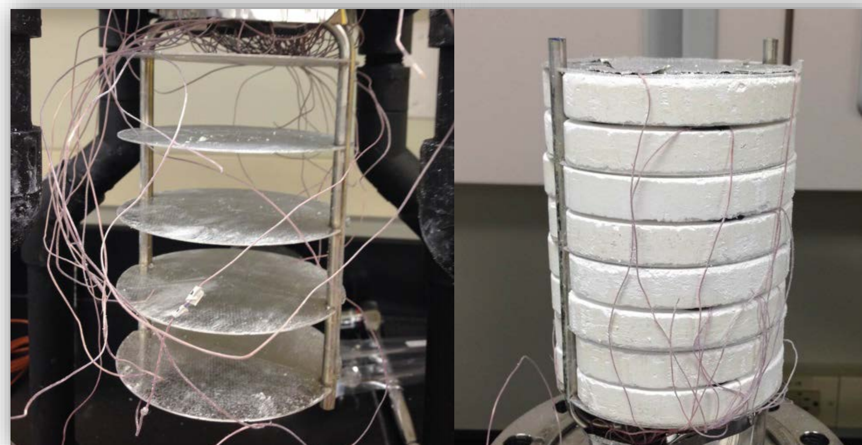
Accomplishment

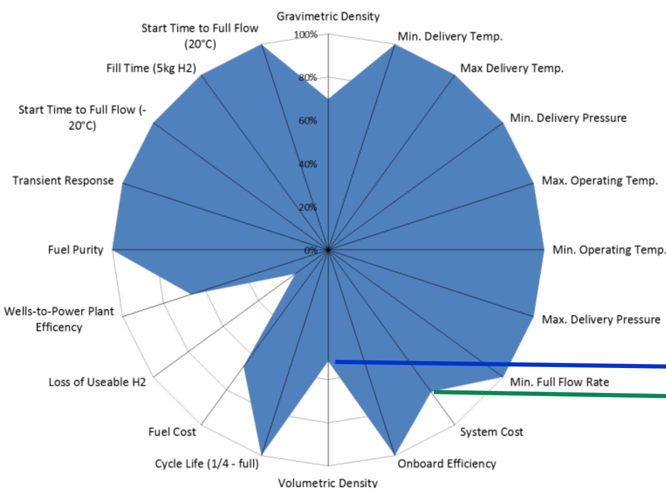
Adsorbent Heat Exchanger Types

HexCell
Flow Through Chilled H_2 Cooling



MATI
Isolated LN2 Flow Cooling

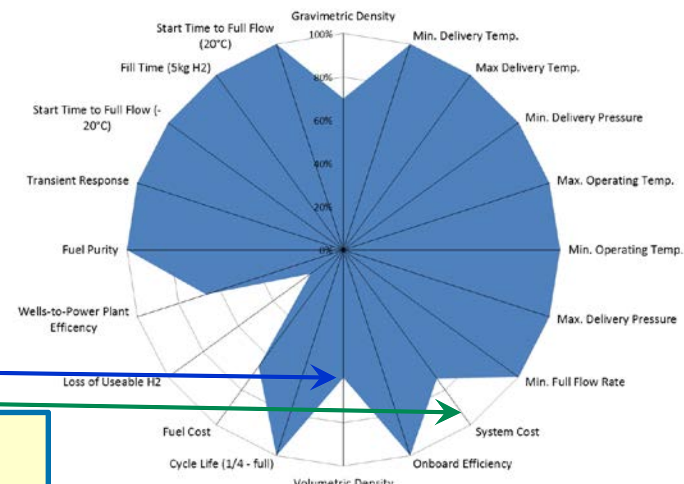




Gain Volumetric Density

in going from loose powder to compacted pucks

at expense of Cost



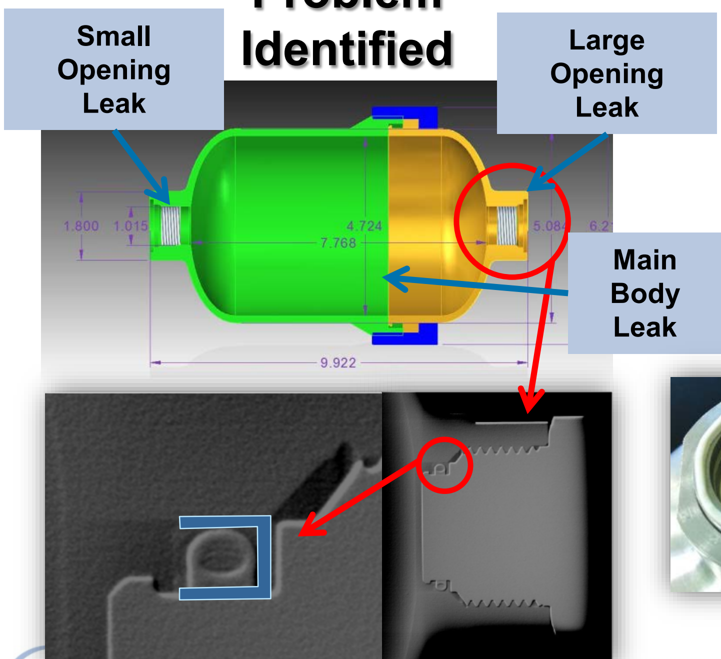
Evaluation of Novel HX Design to Prove Efficacy & Utility

HSECoE

Risk Management: Pressure Vessel Cryogenic Leaks

- Teflon® seals observed to leak at LN2 temps.
- This issue could affect schedule and cost (as of 3/31 3-4 months behind schedule)
- *Tank Seal Tiger Team* formed with weekly telecoms scheduled
- Numerous approaches attempted to solve both waist and large plug leaks
- Waist seal solved with composite Teflon/steel washer allowing testing of HexCell system.
- Large opening seal not solved due to lack of mating surfaces – New stainless steel flange tanks designed, manufactured, tested and delivered allowing MATI system testing.

Problem Identified

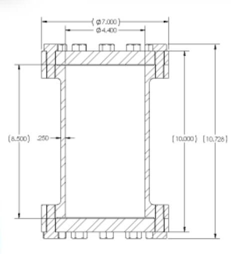


Potential Solutions Investigated

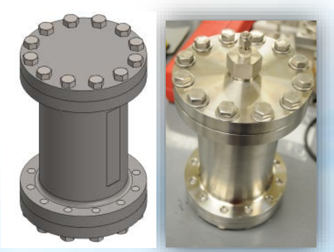
Final Solutions Implemented

SO: Crush Seal
WB: Teflon coated steel washer/w external clamp

HexCell HX



MATI HX



2L Flange Tank

Accomplishment

Adsorbent Media Preparation

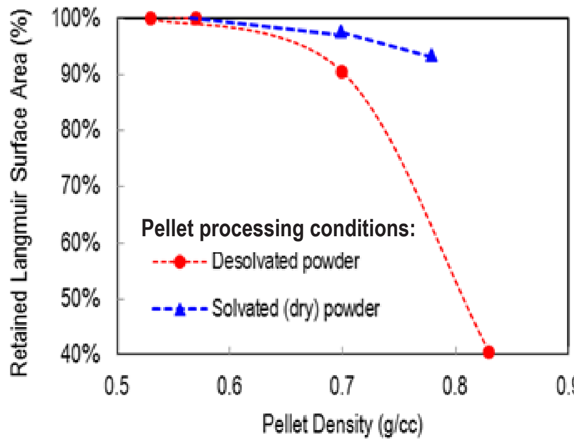
Evaluate **MOF-5 degradation beyond 300 cycles** based on maximum allowable impurity levels as stated in SAE J2719 and report on the ability to mitigate to less than 10%.
Perform a minimum of 10 **heat capacity or thermal conductivity measurements** at temperatures ranging from 70-200K on compacted MOF-5 samples prepared by Ford and to support validating system models and system level designs.

Hydrogen Impurity Cyclic Tests

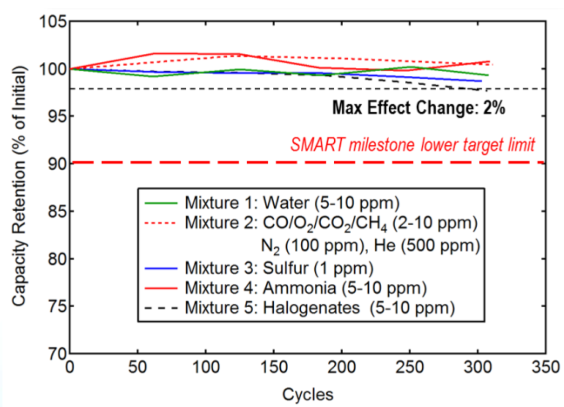
Impurity Test Gas

SAE J2517 Constituent	Chemical Formula	Limits		
Hydrogen fuel index	H_2	> 99.97%		
Total allowable non-hydrogen, non-helium, non-particulate constituents listed below		100		
Acceptable limit of each individual constituent			Test Gas Levels	Mixture Combinations
Water	H_20	5 ppm	5 to 10 ppm	Test Gas Mixture 1
Total hydrocarbons	C_1	2 ppm	2 ppm	Test Gas Mixture 2
Oxygen	O_2	5 ppm	5 ppm	Test Gas Mixture 2
Helium	He	300 ppm	500 ppm	Test Gas Mixture 2
Nitrogen, Argon	N_2, Ar	100 ppm	100 ppm	Test Gas Mixture 2
Carbon dioxide	CO_2	2 ppm	5 ppm	Test Gas Mixture 2
Carbon monoxide	CO	0.2 ppm	2 ppm	Test Gas Mixture 2
Total sulfur	S	0.004 ppm	1 ppm	Test Gas Mixture 3
Formaldehyde	HCHO	0.01 ppm	n/a	Not in Gas Mixture
Formic acid	HCOOH	0.2 ppm	n/a	Not in Gas Mixture
Ammonia	NH_3	0.1 ppm	5 to 10 ppm	Test Gas Mixture 4
Total halogenates		0.05 ppm	5 to 10 ppm	Test Gas Mixture 5
Particulate Concentration		1 mg/kg		

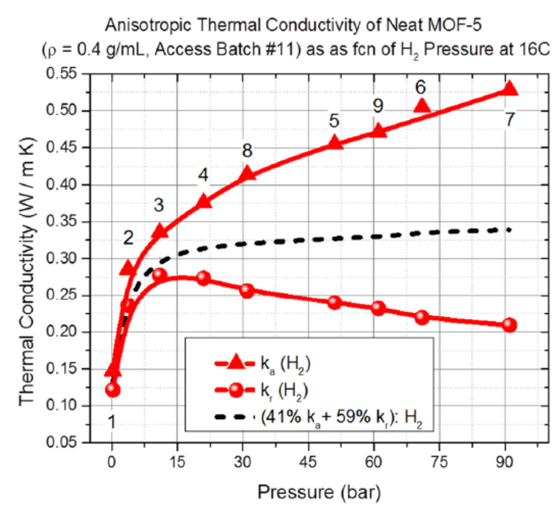
Solvated Compaction



Adsorption Degradation



Anisotropic Thermal Conductivity



HSECoE

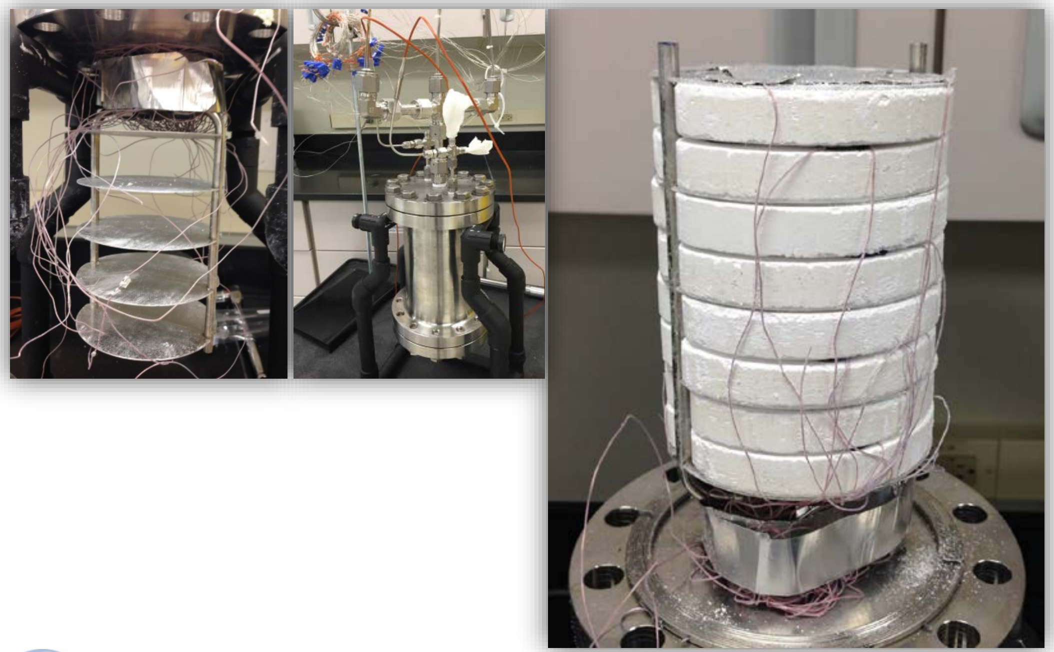
MATI Heat Exchanger & Test Systems

Design and construct a **hydrogen cryo-adsorbent test station** capable of evaluating the performance of a two liter cryo-adsorbent prototype between 80-160K and which would meet all of the performance metrics for the DoE Technical Targets for On-Board Hydrogen Storage Systems.

Demonstrate performance of subscale system evaluations and model validation of a 2L adsorbent system utilizing a MATI thermal management system having 54 g available hydrogen, internal densities of 0.10g/g gravimetric, and 27 g/l volumetric.

Demonstrate a two liter hydrogen adsorption system containing a MATI internal heat exchanger provided by Oregon State University characterizing its performance against each of the sixteen performance DoE Technical Targets for On-Board Hydrogen Storage Systems.

MATI Subscale Prototype Assembled

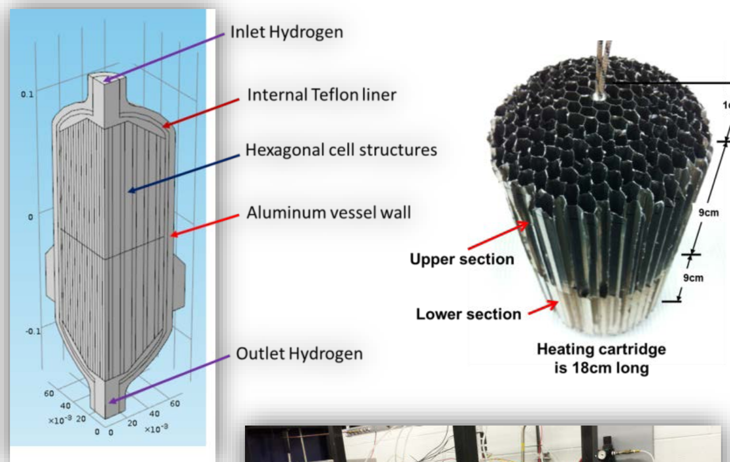


MATI Test Station Completed

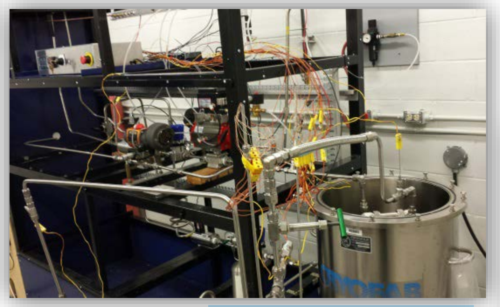
HexCell Heat Exchanger & Test System

Design a 2L adsorbent subscale prototype utilizing a HexCell heat exchanger having 46g avialable hydrogen, internal densities of 0.13g/g gravimetric, and 23.4g/L volumetric.	
Demonstrate performance of subscale system evaluations and model validation of a 2L adsorbent system utilizing a HexCell heat exchanger having 46g avialable hydrogen, internal densities of 0.13g/g gravimetric, and 23.4g/L volumetric.	

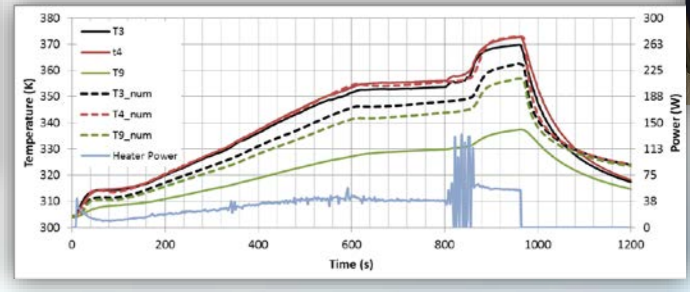
2L HexCell System Design



2L HexCell System Assembly



2L HexCell Test System

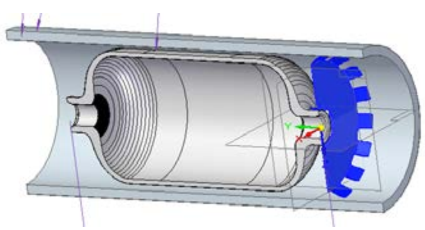


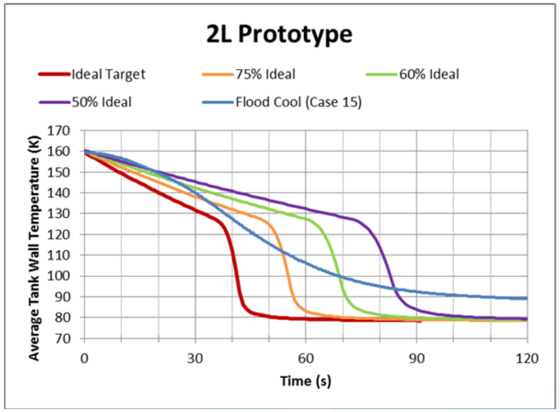
2L HexCell Preliminary Test Results

Pressure Vessel Demonstration

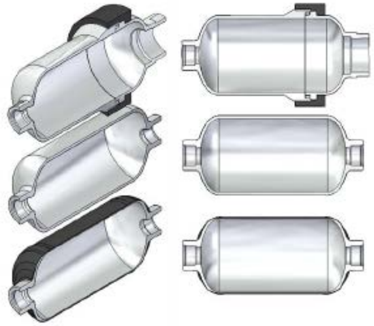
Design and manufacture a baseline, separable Type 1 tank in accordance with size (2L - 6L), pressure (100 bar service pressure), operating temperatures (80K – 160K) and interfaces specified by HSECoE team members, and with a 10% reduction in weight per unit volume compared with the Type 1 tank tested in Phase 2.
Design alternate tank configurations, such as monolithic Type 1, Type 3 with suitable cryogenic liner, and Type 4 with suitable cryogenic liner, that can operate at 100 bar service pressure, at temperatures of 80K – 160K, and offer a further 10% reduction in weight compared with the Phase 3 baseline Type 1 tank, and are consistent with safety requirements established by industry for hydrogen fuel containers.
Hexagon-Lincoln will fabricate and PNNL will demonstrate a minimum one liter scale LN2 jacketed tank. With this device they will measure the transient heat loss for dormancy and demonstrate the LN2 thermos bottle tank cooling concept. This experiment will be scaled to the full size 5.6 kgH2 size and shown experimentally to meet the DOE technical targets for dormancy and refueling time.

Tank Cooling Design and Test Apparatus





Alternate Tank Configurations

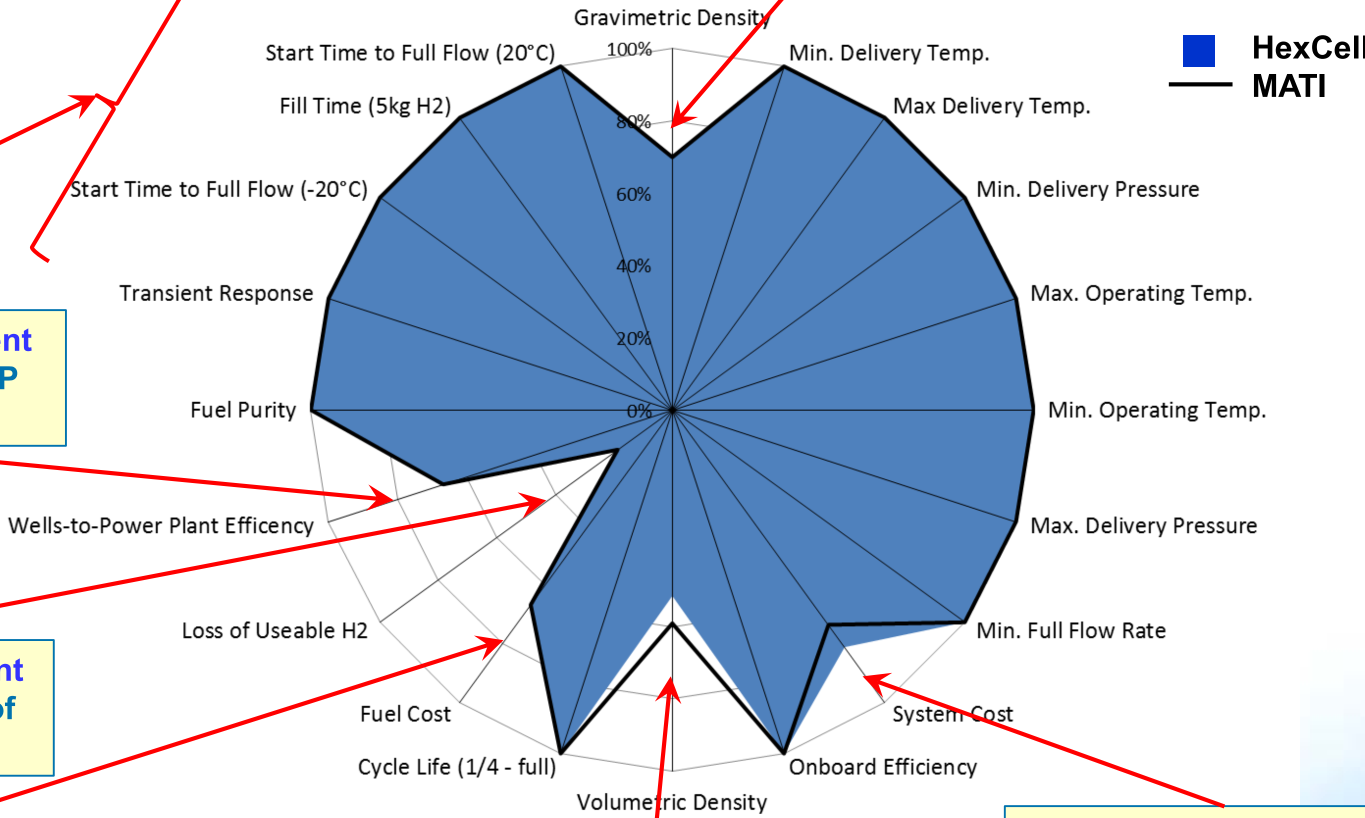


Vessel	Wt. (lb)	% 1	% n-1
1) T1 (1st 3 piece)	5.9	n/a	n/a
2) T1 (2nd 3-piece)	5.0	84	84
3) T1 (1-piece)	3.0	51	60
4) T3	2.23	38	74
5) T4	tbd	tbd	tbd

HSECoE

Adsorbent System *White Space*

Heat Exchanger: HexCell
Media: MOF-5
P: 5-60 bar – Type I Al Pressure Vessel
T: 80-160K - MLVI



New adsorbent needed to meet gravimetric density target.

Fill time and performance targets achieved due to advanced heat exchanger designs.

Higher enthalpy adsorbent needed to achieve WTPP efficiency target.

Higher enthalpy adsorbent needed to achieve loss of useable H_2 target.

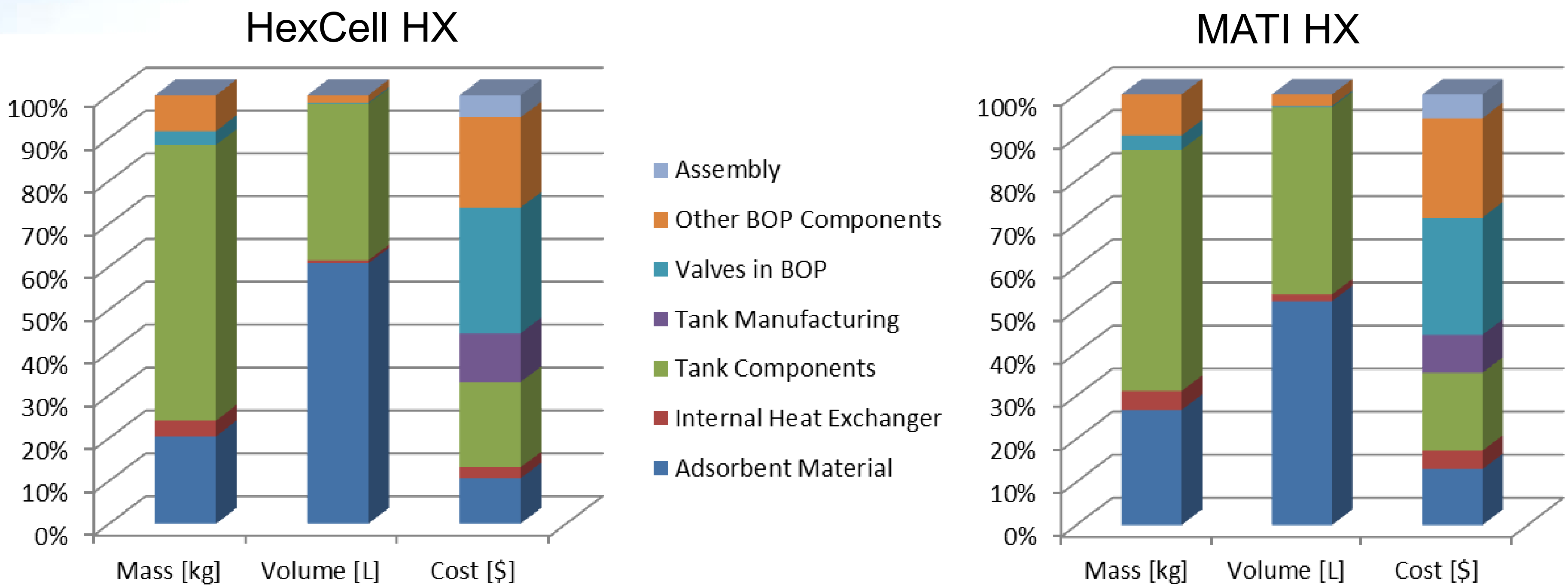
Higher enthalpy adsorbent needed to achieve fuel cost target.

New adsorbent and densification methods needed to achieve volumetric target.

Less expensive tank & BoP needed to achieve cost target.

HSECoE

HexCell & MATI Mass/Volume/Cost Comparison



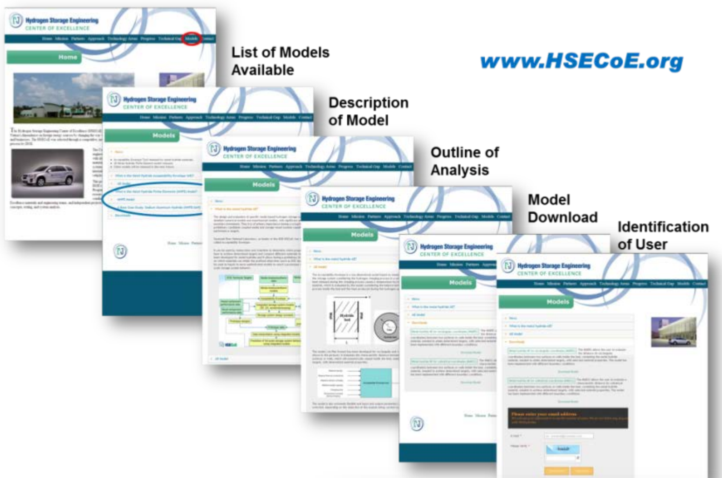
Adsorbent Systems are Primarily:
Mass: ~60% Tank and Insulation
Volume: HexCell 60% Adsorbent
MATI 52% Adsorbent
Cost: ~50% BoP

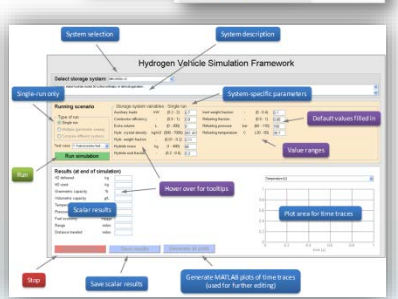
HSECoE

System Modeling

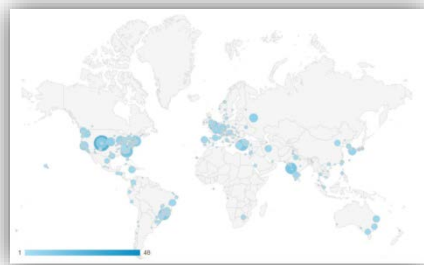
Prepare a report on the impact of system design changes on the tank to wheels efficiency and document progress relative to a 300 mile range for adsorbent systems.
Update the cryo-adsorbent system model with Phase 3 performance data, integrate into the framework; document and release models to the public.
Complete the failure mode and effects analysis (FMEA) associated with real-world operating conditions for a MOF-5-based system, for both HexCell and MATI concepts based on the Phase 3 test results. Report on the ability to reduce the risk priority numbers (RPN) from the phase 2 peak/mean and identify key failure modes.

Models Available on WEB site





Model Usage Tracked



Model Available and Planned

MH Acceptability Envelope	SRNL	complete
MH Finite Element Model	SRNL	complete
Tank Volume/Cost Model	PNNL	complete
MH Framework Model	UTRC/NREL	complete
CH Framework Model	PNNL/UTRC/NREL	complete
AD Framework Model	SRNL/UTRC/NREL	In progress
AD Finite Element Model	SRNL	6/2015

As of Feb. 29, 2015:

- 2162 total sessions, 6034 page views and 1720 users
- Model download figures:
  - *Tankinator* – 39
  - MHAE – 9
  - MHFE – 13
  - Vehicle Framework – 25

FMEA used to Stimulate Thinking

Failure Modes and Effects Analysis

Highest risk items identified from initial FMEA

Corrective actions taken

Example actions during phase 2-3 for reducing the Risk Priority Number (RPN)

- Completed MOF-5 air exposure testing
- Completed MOF-5 contaminated gas cyclic testing
- Completed initial material and heat exchanger testing
- Revised tank construction from composite to aluminum and completed cryogenic testing
- Developed designs with deep-dive technical reviews, controls, and test plans

Phase 1 RPN Values
High: 720
Mean: 188

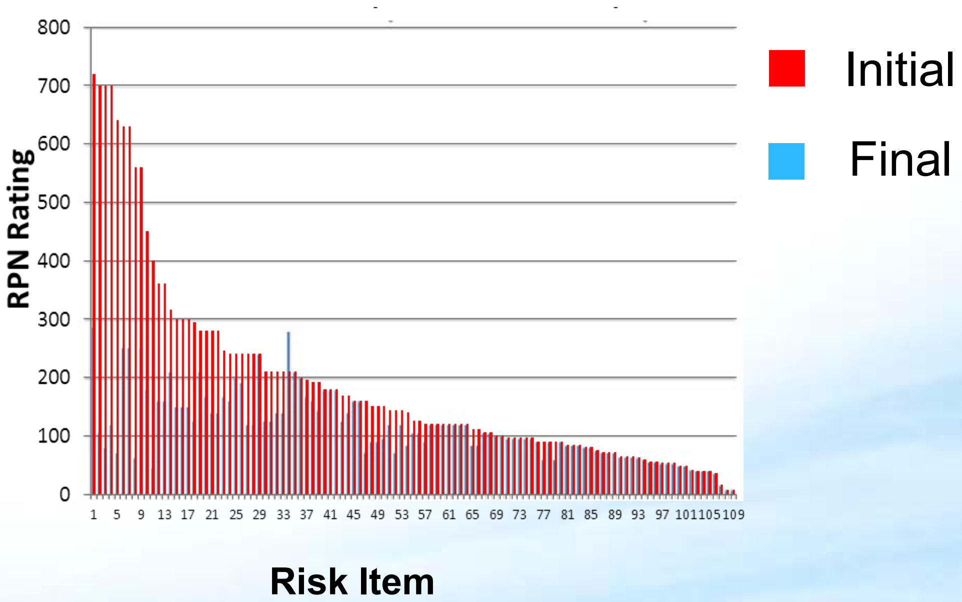
→

Phase 2 RPN Values
High: 512
Mean: 113

→

Phase 3 RPN Values
High: 288
Mean: 114

Use of typical industrial organizational tools, such as FMEA, led to insights into potential system failure modes, alternate research pathways and resultant mitigation methods not thought of previously.



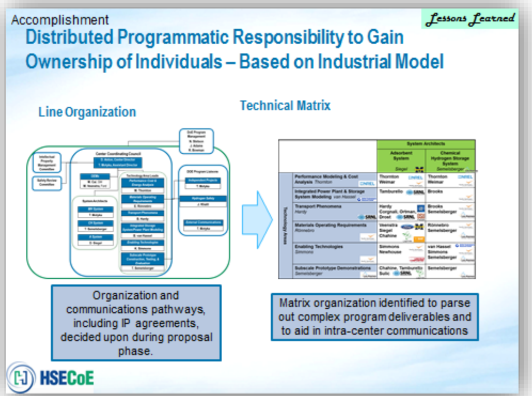
HSECoE

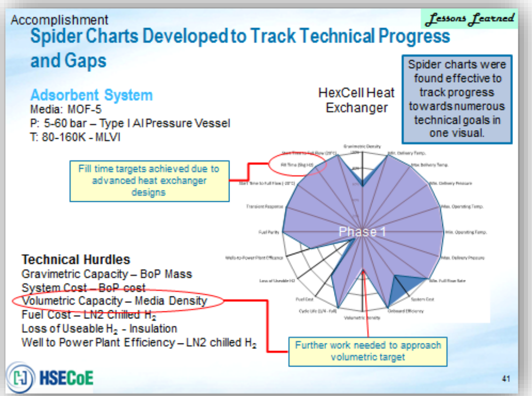
Technology Transfer

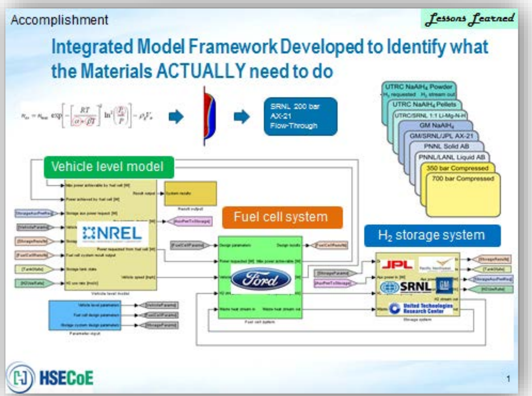
This program has been a technology transfer program with the HSECoE actively partnering with Ford Motor Company and General Motors Co. to develop materials-based hydrogen storage systems for its duration. During this time their active participation has greatly aided the Center in understanding vehicle needs, cost estimation and numerous other areas where only the OEMs have a firm understanding of customer needs and manufacturing capabilities.

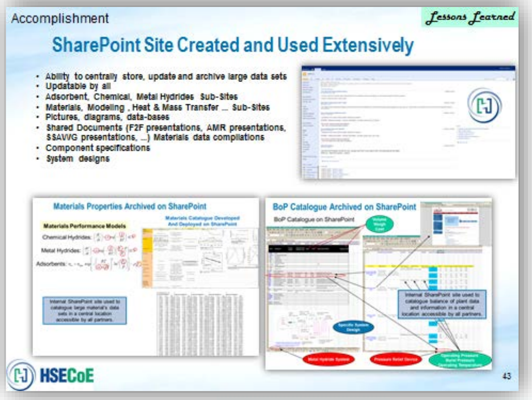
What have we learned about organizing a Center of Excellence?”

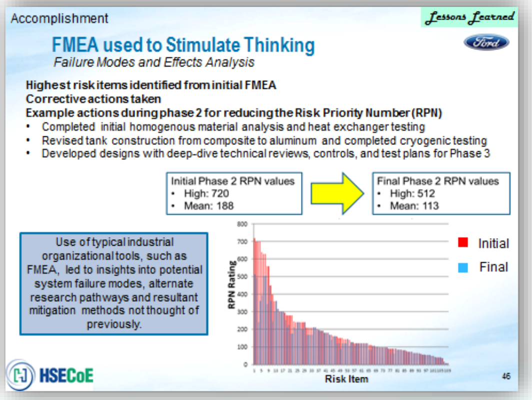
The HSECoE is the Fourth Hydrogen Storage COE
“We are dwarfs perched on the shoulders of giants.”
Bernard of Chartres, 1159-AD

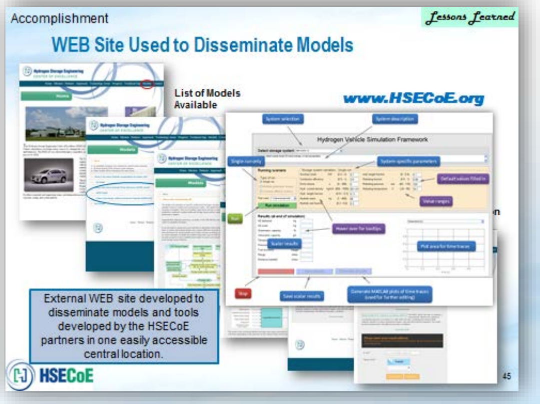






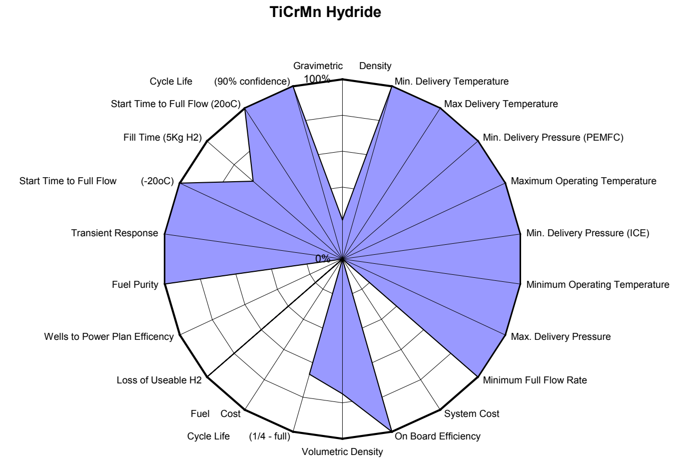


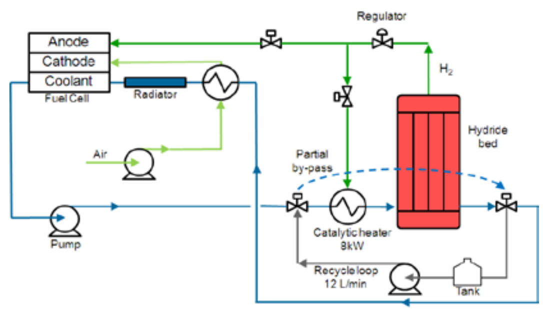


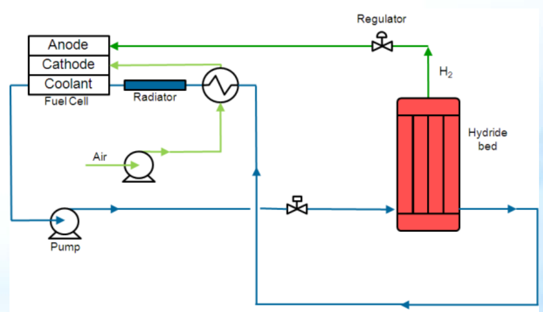


Summary of Challenges and Barriers

- **Metal Hydride System**
  - Low enthalpy materials (i.e. ΔH < 27 kJ/mol-H_2), can use only the waste heat of the fuel cell for discharge, while high enthalpy materials (i.e. ΔH >30 kJ/mol-H_2), require some H_2 combustion and additional BoP.
  - Additional hydrogen capacity (1 to 1.5 wt%) gained by using higher pressure, hybrid tanks would be negated by the additional weight of carbon fiber needed for reinforcement.
  - For most metal hydride densities (>1100 to 1600 kg/m^3) – the volumetric target can be easily met if the gravimetric target is met
  - A material charging kinetics needs to be 3-8X greater than catalyzed $NaAlH_4$, at charging pressures <100 bar.
  - Materials with both high gravimetric capacity **and** low enthalpy of formation need to be developed.







Metal Hydride Materials Requirements

$$\left(\frac{dC}{dt}\right) = A\exp\left(-\frac{E_a}{RT}\right)\left(\frac{P_e - P}{P_e}\right)(C)^{\chi}$$

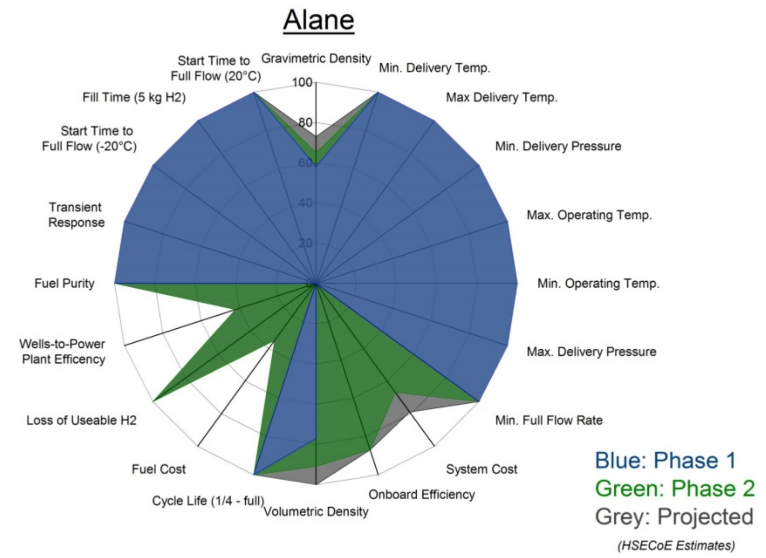
Parameter	Units	Range*
Gravimetric Capacity, $\Delta H<27$ kJ/mol	g_{H2}/g_{media}	11%
Gravimetric Capacity, $\Delta H<40$kJ/mol	g_{H2}/g_{media}	17%
Equilibrium Pressure, P_e	bar	$5<P_e<100$
Exponential, χ		1
Activation Energy, E_a	kJ/mol	3.05
Pre Exponential, A		6.2×10^{8}
Bulk Density	$g_{media}/volume_{media}$	70% Crystal Density
Thermal Conductivity, κ	W/m K	>10

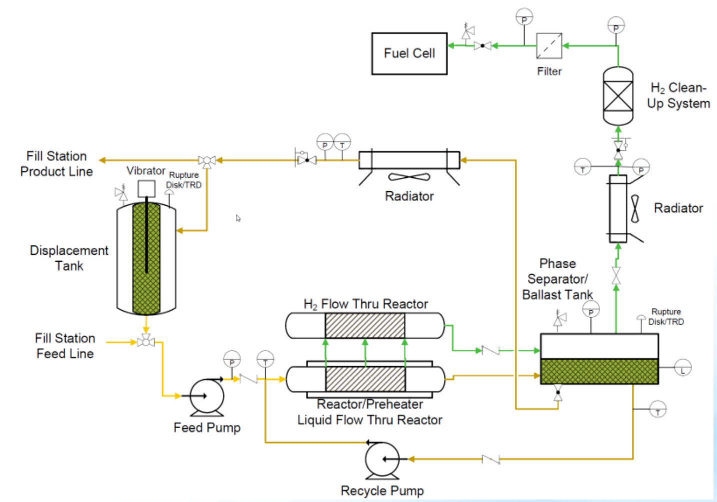
J.M. Pasini, C. Corgnale, B.A. van Hassel, T. Motyka, S. Kumar, K. L. Simmons, *Metal hydride material requirements for automotive hydrogen storage systems*, Intl. J. Hydrogen Energy 2013; 38:9755-9765.

* To meet 2020 targets

Summary of Challenges and Barriers

- **Chemical Systems**
  - H_2 contaminants can be scrubbed.
  - In reactor gas/liquid separation demonstrated.
  - 50 wt.% alane slurry successfully demonstrated in flow through reactor.
  - 50 wt.% ammonia borane slurry not pumpable.
  - Efficient chemical hydride regeneration needs to be developed to address fuel cost and WTPP efficency gap.
  - To mitigate slurry stability and pumping issues, development of a high capacity liquid material both before and after dehydrogenation required.
  - CH which can discard spent fuel environmentally (one-way) optimal business solution.





Chemical Hydride Materials Requirements

$$\left(\frac{dC}{dt}\right) = A\exp\left(-\frac{E_a}{RT}\right)(C)^n$$

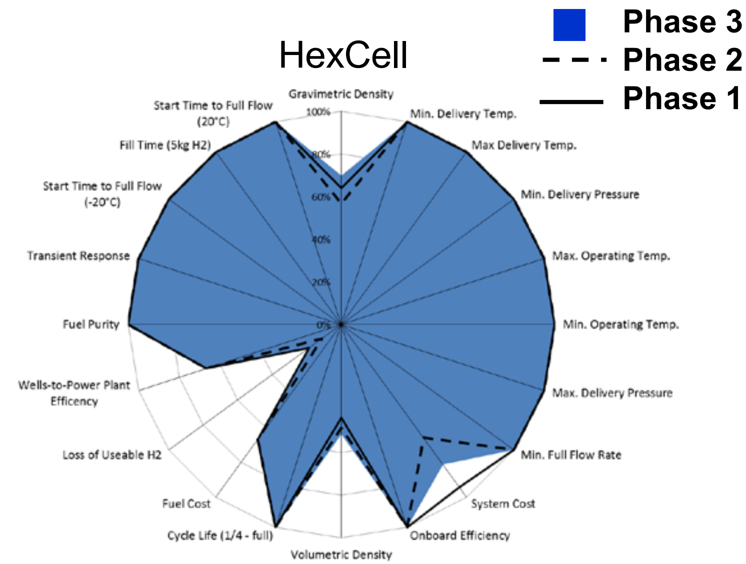
Parameter	Units	Range*
Gravimetric Capacity (liquids)	$g_{H2} / g_{material}$	~ 0.078 (*0.085*)†
Gravimetric Capacity (solutions)	$g_{H2} / g_{material}$	~ 0.098 (*0.106*) †
Gravimetric Capacity (slurries)	$g_{H2} / g_{material}$	~ 0.112 (*0.121*) †
Endothermic Heat of Reaction	kJ / mol H_2	≤ +17 (*15*) †
Exothermic Heat of Reaction	kJ / mol H_2	≤ -27
Kinetics: Activation Energy, E_a	kJ / mol	117-150
Kinetics: Pre-exponential Factor, A		$4 \times 10^9 – 1 \times 10^{16}$
Maximum Reactor Outlet Temperature	°C	250
Media H_2 Density	kg H_2 / L	≥ 0.07
Regeneration Efficiency	%	≥ 66.6%
Viscosity	cP	≤ 1500

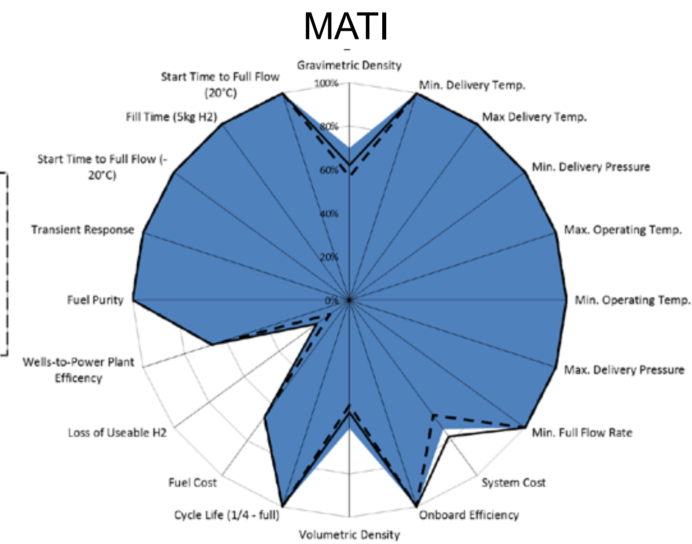
T.A. Semelsberger & K.P. Brooks, *Chemical hydrogen storage material property guidelines for automotive applications*, Journal of Power Sources 279 (2015) 593-609.

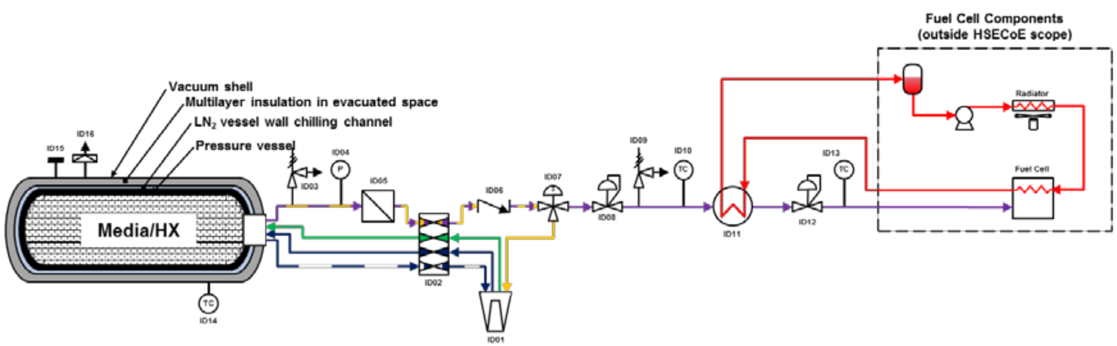
† (if hydrogen gas clean-up needed)
*** To meet 2020 targets**

Summary of Challenges and Barriers

- **Adsorption Systems**
  - Volumetric density improved with microchannel MATI HX design via MOF compaction demonstrated.
  - Charge time addressed with flow through cooling and independent LN2 tank cooling.
  - Low enthalpy adsorbents require low temperatures and eventual loss of hydrogen in dormancy.
  - High density powder compact need to be developed to address volumetric density.







Adsorbent Materials Requirements

$$n_a = \frac{n_{max}RT}{(E_{max} - E_{min})} ln\left(\frac{e^{-\Delta S_0/R} + \frac{P}{P_0}e^{E_{max}/RT}}{e^{-\Delta S_0/R} + \frac{P}{P_0}e^{E_{min}/RT}}\right)$$

$$n_{Total} = n_a + \mathrm{c}(\mathrm{V}_v - \mathrm{V}_p)$$

Parameter	Units	Range*
Maximum Excess Capacity, n_{max}	mol $_{H2}$ / kg material	~ 200
Minimum Binding Energy, E_{min}	kJ/mol	~ 4.49
Maximum Binding Energy, E_{max}	kJ/mol	~E_{min}
Entropy, DS_o	J / mol K	≤ -65
Reference Pressure, P_0	bar	1
Absolute Pressure, P	bar	5<P<100
Bulk Density, r_{bulk}	Kg/m^3	181
Bed Void Volume, V_v-V_p	m^3/kg_{media}	0.00391
Temperature, T	K	77<T<160

Personal Communication B.J. Hardy
*** to meet 2020 DOE targets using MOF-5 as nominal starting material**

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Materials Based Hydrogen Storage Systems Summary

	Mass* (kg)	Volume* (liters)	Cost* ($)	Gravimetric Density (gH_2/ g system)	Volumetric Density (gH_2/ liter system)	Cost ($/kWh)
Metal Hydride System						
$NaAlH_4/Ti$	457	489	8008	1.2%	11.5	42.95
Chemical System						
AB	122	136	3011	4.6%	41.0	16.50
AlH_3	164	151	4133	3.4%	37.0	22.16
Adsorbent System						
HexCell/MOF-5	161	304	2720	3.5%	18.5	14.59
MATI/MOF-5	159	263	2897	3.5%	21.3	15.54
2020 DOE Targets				5.5%	40.0	10.00

** for 5.6 Kg usable hydrogen*

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LANDMARK Innovations

What has the Center done to change the way we look at hydrogen storage?

- **Overall**
  - Technical target prioritization
  - Development of models which integrate the storage system, fuel cell and vehicle drive cycles
- **Metal Hydrides**
  - MH acceptability envelope
  - Microchannel catalytic burner

- **Chemical Hydrogen Storage**
  - CH material requirements
  - Auger reactor for slurries and helical reactor for neat liquids
  - Demonstrated 60wt.% alane slurry reactor
  - Ammonia/diborane scrubber
  - Gas/Liquid separator
- **Adsorbents**
  - Adsorbent materials requirements
  - LN2 tank cooling strategy
  - Low cost flow-through HX design
  - Combined MOF compaction/ augmentation
  - Microchannel HX in compacted media design

Where have we gone?

Materials Based Hydrogen Storage Systems for Automotive Applications

Materials CoEs

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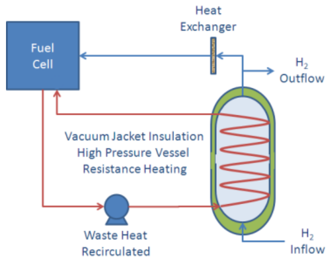
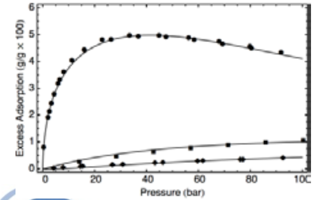
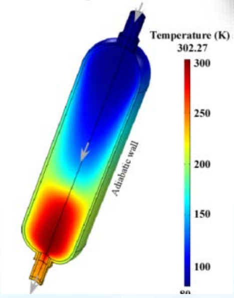
TRL 1	TRL 2	TRL 3	TRL 4	TRL 5	TRL 6	TRL 7	TRL 8	TRL 9
Basic Technology Research		Research to Prove Feasibility	Technology Development		Technology Demonstration	System Commissioning		System Operation
Basic Principals	**Concept Formulation**	**Characteristic Proof of Concept**	**System Validation in Laboratory Environment**	**System Validation in Relevant Environment**	**Pilot Scale System Validation**	**Full Scale System Validation**	**Actual System Qualification**	**Actual System Operation**







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NREL

SRNL

JPL

PNNL

LANL

UTRC

OSU

Hexagon-Lincoln

Technical Back-Up Slides

Reviewers Comments

“How will the models on the web site be maintained once the funding is gone?”

- **DOE will be supporting model updates next year through AOP.**

“A key component of the final report should be statements from the OEMs as to the practical potentials they see for the materials and containment designs developed in this project.”

- **This will be incorporated into the final report.**

“Further attention could be paid to explaining a long term vision for what the on-board system components might look like.”

- **Significant effort was put forth on design and modeling of consolidated BoP components such as valves, pressure transducers and couplings.**

“Greater emphasis should be placed on dealing with the problem areas and technical obstacles identified by "white spaces" in the spider charts.”

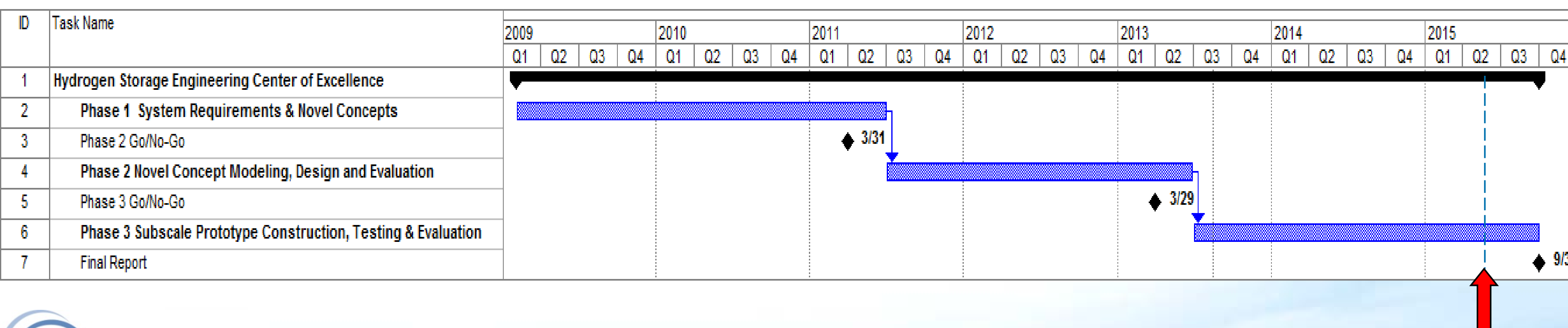
- **This could only be accomplished at the expense of not demonstrating subscale prototypes, a contractual obligation which could not be minimized under the current budgetary constraints.**

“A comprehensive set of material requirements based on system needs should be published in a journal that is widely read by researchers engaged in new material development.”

- **This comprehensive list of materials requirements has been accomplished and presented at the Hydrogen Storage Summit held in January. Articles detailing these results are being prepared for publication.**

Important Dates

- Duration: 6.7 years
  - Phase 1 Start: Feb. 1, 2009
  - Phase 1-2 Transition: March 31, 2011
  - Phase 1 End: June 30, 2011
  - Phase 2 Start: July 1, 2011
  - Phase 3 Go/No-Go Determination: March 31,2013
  - *Phase 2 End:* June 30, 2013
  - Phase 3 Start: July 1, 2013
  - Completion Date: ~~June 30, 2015~~ ⇨ Dec. 31, 2015



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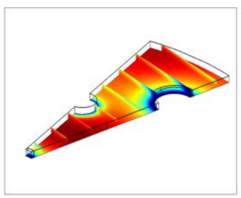
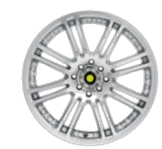
Here we are today.

Why Perform Materials Development and System Engineering in Parallel?

continuous feedback with system design through the integrated model identifying materials requirements





Materials → Thermal Management → H_2 Storage BoP → Fuel Cell → Vehicle → Wheels

- Materials → Engineered Materials Properties
- Thermal Management → Heat Transfer Designs
- H_2 Storage BoP → BoP Component Requirements
- Fuel Cell, Vehicle, Wheels → What is Needed of the Hydrogen Storage Media & System

DOE Materials Based Hydrogen Storage Summit Supported

January 27-28, 2015
Golden, CO

HSECoE partners played a fundamental roll in the DOE H_2 Storage Summit. This DOE sponsored workshop should help guide the materials development community by outlining the major materials characteristics required to meet the DOE technical targets.

Materials requirements for metal hydride, chemical hydrogen and adsorbent materials were reviewed along with Center models on the WEB and a review of *niche* opportunities for hydrogen storage.

DOE Materials-Based Hydrogen Storage Summit:
Defining Pathways for Onboard Automotive Applications

January 27: Day 1

8:00–8:30: Check-in
8:30–8:45: Welcome and meeting logistics – Matt Thornton (NREL)
8:45–9:00: Introduction to workshop objectives – Ned Stetson (DOE)
9:00–9:30: Onboard automotive targets: an OEM perspective – Mike Veenstra* (Ford)
9:30–10:00: Metal hydrides – Ted Motyka* (SRNL)
10:00–10:30: Adsorbents – Don Siegel* (U. Michigan)
10:30–10:45: Break
10:45–11:15: Chemical hydrogen – Troy Semelsberger* (LANL)
11:15–11:45: Off-board regeneration thermodynamics – Rajesh Ahluwalia* (ANL)
11:45–12:30: Lunch
12:30–2:00: Breakout session 1(a)

- Chemical hydrogen
- Metal hydrides
- Adsorbents

2:00–2:15: Break
2:15–4:00: Breakout session 1(b)
4:00–4:15: Break
4:15–5:00: Walk-through of HSECoE web-based system models – Jose Miguel Pasini* (UTRC)

January 28: Day 2

8:30–8:40: Chemical hydrogen breakout session report out
8:40–8:50: Metal hydride breakout session report out
8:50–9:00: Adsorbent breakout session report out
9:00–9:30: Fundamental research directions – TBD
9:30–10:00: Niche application opportunities – Bart van Hassel* (UTRC)
10:00–10:15: Break
10:15–12:15: Breakout session 2

- Bridging fundamental and applied research
- High value added applications

12:15–1:15: Lunch
1:15–2:30: Conclusion / wrap-up

*invited

HSECoE