# Ford/BASF-SE/UM Activities in Support of the Hydrogen Storage Engineering Center of Excellence

Mike Veenstra (PI), Jun Yang, and Chunchuan Xu



#### Manuela Gaab and Ulrich Müller



#### Don Siegel and Yang Ming



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## Overview



## Timeline

- Project Start: February 2009
- Project End: June 2014
- Percent Complete: 70%

## Budget

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- Total Project Funding:
  - DOE Share: \$2,140K
  - Contractor Share: \$643K
- Funding for FY12: \$400K
- Funding for FY13: \$350K

## **Barriers**

All DOE System Targets\*

\*http://www1.eere.energy.gov/hydrogenandfuelcells/storage/pdf s/targets\_onboard\_hydro\_storage.pdf

## **Partners**

- Project Lead: Ford
- Subcontractors: BASF and U. Michigan
- Center Partners:





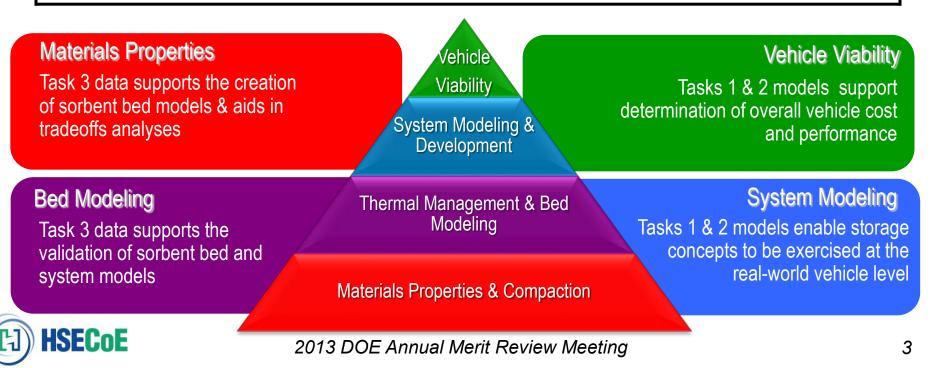
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Three Technical Tasks Contribute to the Overall HSECoE Mission

 Task 1: Develop dynamic vehicle parameter model that interfaces with diverse storage system concepts

Task 2: Development of robust cost projections for storage system concepts

 Task 3: Devise and develop system-focused strategies for processing and packing framework-based sorbent hydrogen storage media
 Image: Comparison of the system storage media





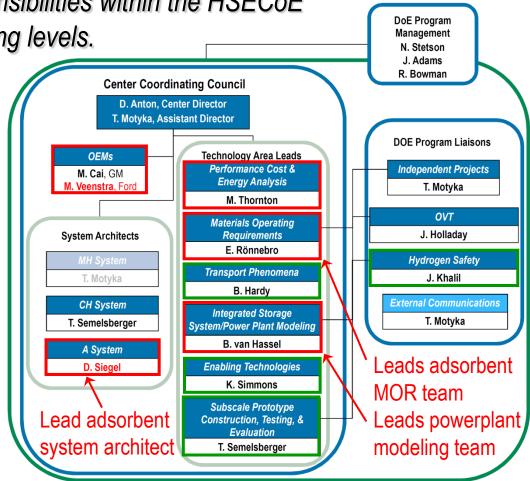
# **Relevance:** Organizational

Ford has many roles and responsibilities within the HSECoE at both the executive and working levels.

Key organizational functions:

- As technical contributors, disseminate data & models across the HSECoE
- As team leads, foster interpartner communication & streamline & align research
- Act as liaisons between the HSECoE and the C&S and Storage Tech. Teams
- Provide an automotive perspective & context

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- Core contribution areas of project outcomes [red]
- Ancillary contribution areas of project outcomes [green]



# **Approach:** System Architect and OEM perspective

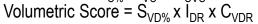
## System Architect Role (D. Siegel)

- Coordinated design/performance trade-offs
- Identified strategic decisions (i.e. pressure, temp, tank)
- Developed criteria for media selection and milestones
- Completed Phase 3 test plan and target matrix
- Organized analysis for Phase 3 Go/No-go Review

## **OEM** Perspective Role (M. Veenstra)

- Developed fuel cell model and vehicle use cases
- Supported cost studies with high volume analysis
- Provided FMEA guidance to avoid failure modes
- Quantified objective function for system rankings

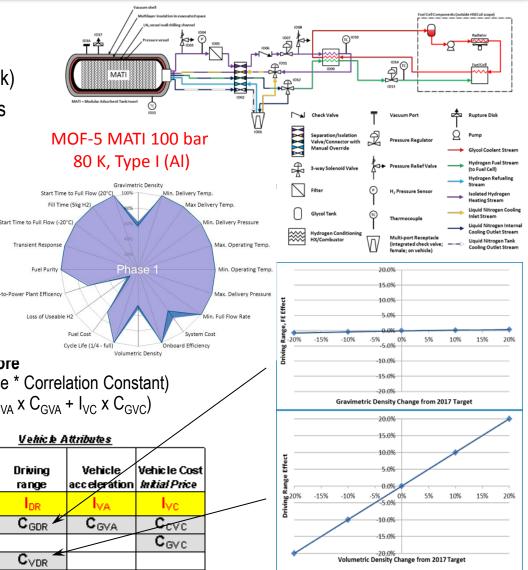
System Score = Grav. Score + Cost Score + Vol. Score **Target Score** = (% of Target Obtained)\* $\Sigma$ (Importance \* Correlation Constant) Gravimetric Score =  $S_{GD\%}$  ( $I_{FE} \times C_{GFE} + I_{DR} \times C_{GDR} + I_{VA} \times C_{GVA} + I_{VC} \times C_{GVC}$ ) Cost Score = S VI VC



Rating value based o important to custome - Used HSTT OEM A Hierarchy Process (A with sales and survey

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netric Score = $S_{VD\%} \times I_{VC} \times C_{CVC}$			Vehick Attributes				
on how			Fuel economy	Driving	Vehicle	Vehic le Cost	
er?			(weight)	range	acc eleration	Initial Price	
Analytic AHP) ey data	<u>System Targets</u>	Importance:	FE	I <sub>DR</sub>	I <sub>VA</sub>	l <sub>VC</sub>	
	Gravimetric Density		C <sub>GFE</sub>	C <sub>GDR</sub> 🖊	C <sub>GVA</sub>	CCVC	
	System Cost					C <sub>GVC</sub>	
	Volumetric Density						



# **Progress:** Reevaluated System Design FMEA

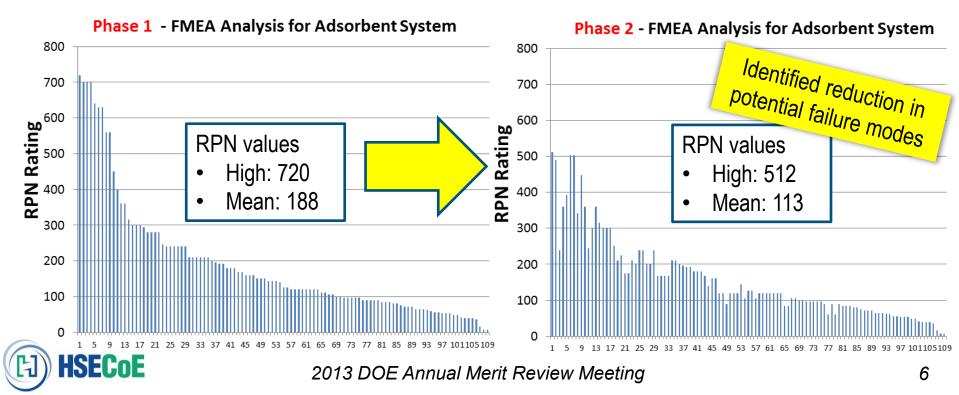


## FMEA = Failure Mode and Effects Analysis (industry tool per SAE J1739)

- o Identifies and evaluates the potential failure of a product and its effects
- Documents the risk and helps prioritize the key actions to reduce failures

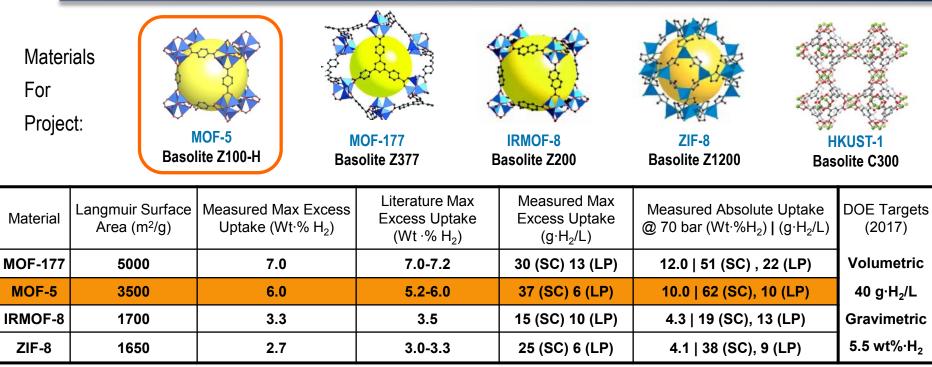
## Example actions during phase 2 for reducing the Risk Priority Number (RPN)

- 1. Completed initial homogenous material analysis and heat exchanger testing
- 2. Revised tank construction from composite to aluminum and completed cryogenic testing
- 3. Developed designs with deep-dive technical reviews, controls, and test plans for Phase 3



## Progress: Adsorbent Material Down-Select





'SC' and 'LP' indicate whether the volumetric capacities are based on single crystal (SC) or loose powder (LP) density, These values help by providing upper and lower bounds to volumetric uptake.

#### Adsorbent Material Down-selection was based on:

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- Performance: MOF-5 outperforms MAXSORB in gravimetric density and in volumetric density (along with other MOFs).
- Availability: MOF-5 has been provided supplied in high quantities to the center by BASF
- Future Prospects: MOF-5 is a member of the larger class of Framework Materials, which has a large potential.
- Safety: MOF-5 in not believed to present any known safety hazards

MOF-5 was selected as the primary adsorbent material for the HSECoE



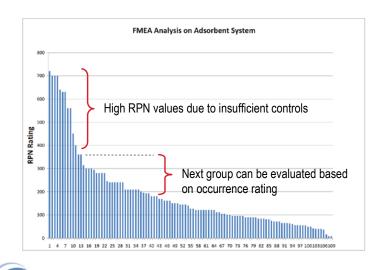
# Approach: Phase 2 SMART Milestones and Tasks



Adsorbent System					
Component	Partner	S*M*A*R*T Milestone			
Materials Development	Ford/UM/ BASF	Report on ability to develop compacted MOF-5 adsorbent media having a total hydrogen material density of greater than or equal to 0.3 g/cc, H2 density of 11 wt. % and 33 g/liter and thermal conductivity of 0.5 W/m-K at P = 60-5 bar and T = $80-160$ K.			
Materials Development	Ford/UM/ BASF	Report on ability to demonstrate a composite MOF-5 adsorbent monoliths having H2 effective kinetics equivalent to 5.6 kg usable H2 over 3 minutes and permeation in packed and powder particle beds with flow rate of 1 m/s superficial velocity and pressure drop of 5 bar.			

#### MOF-5 Material Development Tasks

- Density of  $\geq$  0.3 g/cc with **total capacity**:  $\geq$  11% and  $\geq$  33 g/l
- Thermal conductivity of  $\geq$  0.5 W/m-K at 5-60 bar and 80-160 K
- o Demonstrate effective kinetics for 3 minute fill of 5.6 kg
- o Demonstrate permeation with flow rate of 1 m/s and pressure drop of 5 bar



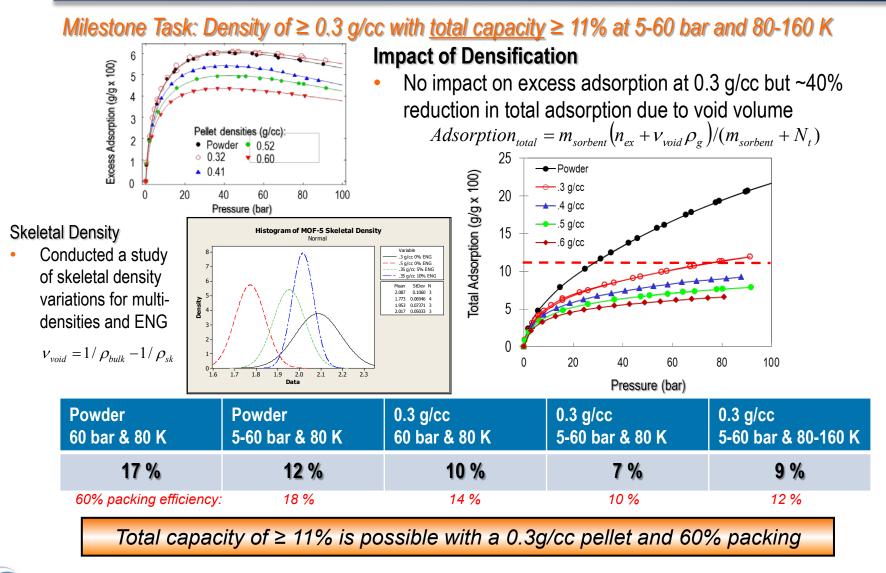
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#### MOF-5 Material FMEA Tasks

- **Non-homogenous bed:** Evaluate material variation (i.e. surface area, density, thermal conductivity, scale-up, etc.)
- **Air exposure & in-service activation**: Need to quantify the level of allowable air exposure and in-service activation.
- Cycling over lifetime: Pressure cycling test of pellets
- **Impurity effects:** Evaluate effects with hydrogen purity at or beyond the limits of SAE J2719
- Safety Assessment: Determine the ignition energy levels for handling of the dust and internal pressure effects

## Progress: MOF-5 Gravimetric Density Results



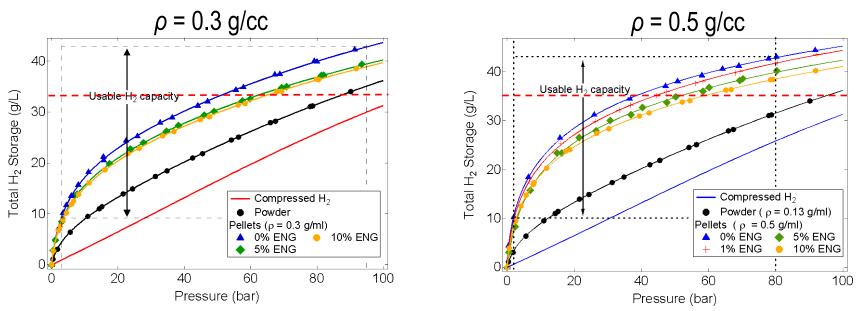


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Milestone Task: Density of  $\geq$  0.3 g/cc with total capacity  $\geq$  33 g/l at 5-60 bar and 80-160 K



Note: All curves currently assume skeletal densities of 2 g/cc and 100% packing efficiency.

Powder 5-60 bar & 80 K	0.3 g/cc + 5% ENG 5-60 bar & 80 K	0.3 g/cc + 5% ENG 5-60 bar & 80-160 K	0.5 g/cc + 5% ENG 5-60 bar & 80 K	0.5 g/cc + 5% ENG 5-60 bar & 80-160 K	
20 g/l	22 g/l	31 g/l	22 g/l	34 g/l	
60% packing efficiency:	20 g/l	26 g/l	21 g/l	27 g/l	
Total capacity of $\geq$ 33 g/l at 5-60 bar is theoretically achievable with 80-160 K					



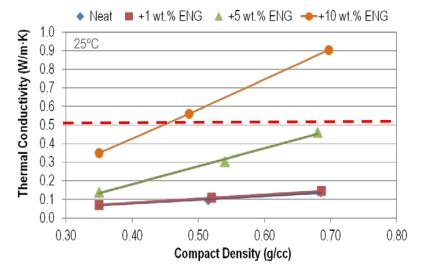
# **Progress:** Thermal Conductivity of MOF-5



New MOF-5

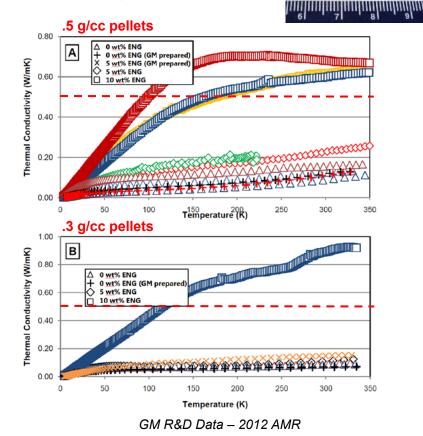


#### Thermal Conductivity Data of MOF-5 and MOF-5/ENG Composites



- MOF-5 has an extremely low thermal conductivity and needs further advancement to optimize the heat exchanger concepts and system design.
- Enhanced Natural Graphite (ENG) at 10 wt% has been shown to significantly improve (~4× to 6x depending on temperature) the thermal conductivity.

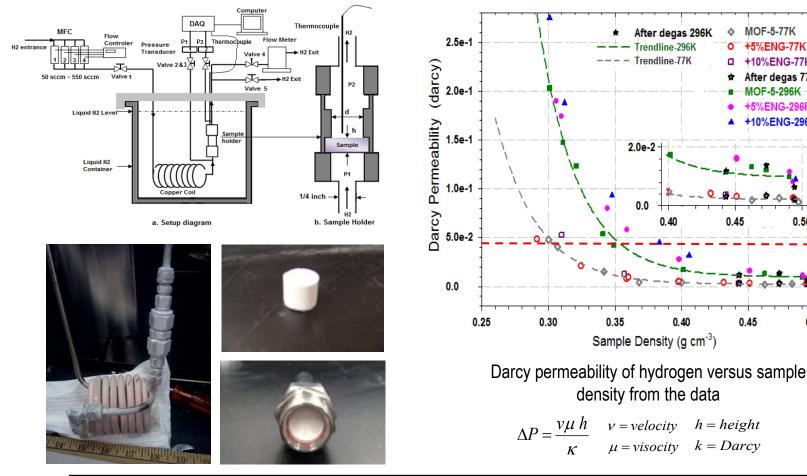
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Thermal conductivity of  $\geq$  0.5 W/m-K requires 10 wt% ENG at ~100 to 150 K

#### **Progress:** Permeation Testing of Densified MOF-5 BASF

#### Milestone Task: Demonstrate permeation with flow rate of 1 m/s and pressure drop of 5 bar



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Pellets of .3 g/cc (77 K) have a projected pressure drop of 3.6 bar at 1 m/s

Projection based on an extrapolation based on test data at .12 m/s with a Darcy of .0486 (or .0465 compressible gas equation)

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MOF-5-77K

+5%ENG-77K

+10%ENG-77K

After degas 77K

10%ENG-296K

0.50

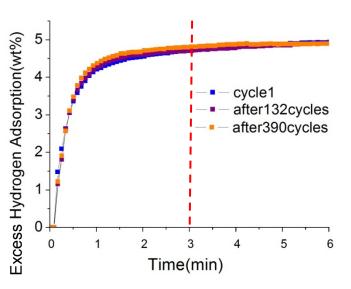
0.50

0.45

OF-5-296K %ENG-296K



#### Milestone Task: Demonstrate effective kinetics for 3 minute fill of 5.6 kg

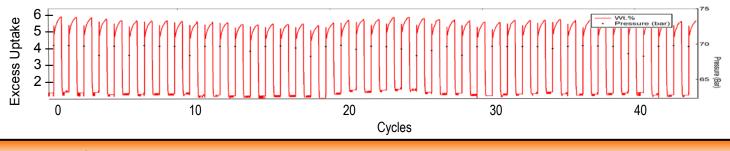


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MOF-5 Pellet, Density=0.39g/cc

MOF-5 pellets have high rate of kinetics and is maintained over multiple cycles.

Initial cycle testing of powder (below) over 240 cycles and pellets over 390 cycles provide stable results for both kinetics and uptake adsorption %.



MOF-5 pellet testing demonstrates consistent kinetics and uptake

## **Progress:** Homogenous Evaluation of MOF-5

FMEA Task: Non-homogenous Bed Failure Mode - Evaluate pellet variation GW0118 – pellets 6x6 mm,1 +/- 0.01% graphite, .377 g/cc with  $\sigma$  = .012 g/cc

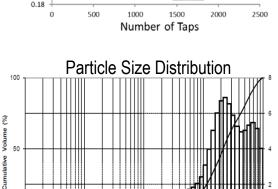
GW0117 – pellets 6x6 mm, 5 +/- 0.1% ENG, .391 g/cc with  $\sigma$  = .013 g/cc



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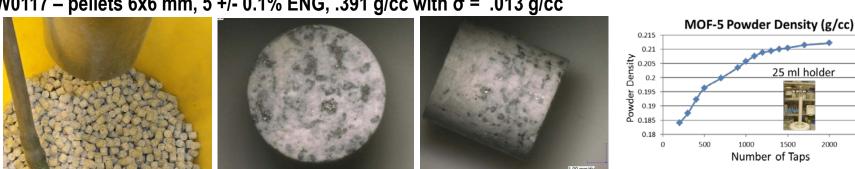
Туре	Particle Size (mm) or Pellet Dia. (mm)	Density [BASF] (g/cc)	BET SA [B-F] (m²/g)	Pore Volume [B-F] (cm <sup>3</sup> /g)
Powder	99% conf: < .86	.19 (tap density)	2680-2763	1.27
Pellets neat	99% conf: 5.9 - 6.0	99% conf: .3441	2477-2489	1.18 - 1.21
Pellets+5%ENG	99% conf: 5.9 - 6.0	99% conf: .3543	2387-2702	1.14 - 1.18

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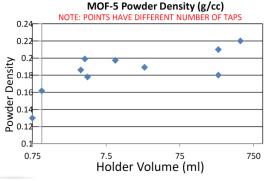


Particle Diameter (µm)

0.1







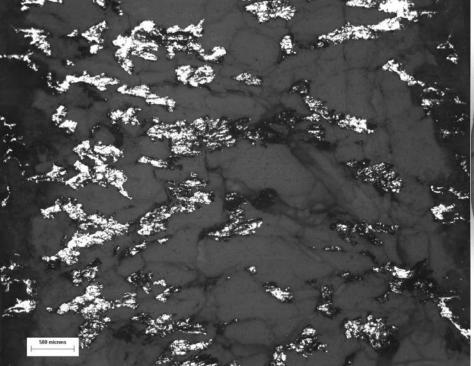


25 ml holder

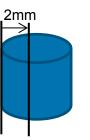
# Progress: Microscopy Analysis of MOF-5 & ENG



#### FMEA Task: Non-homogenous Bed Failure Mode - Evaluate pellet variation

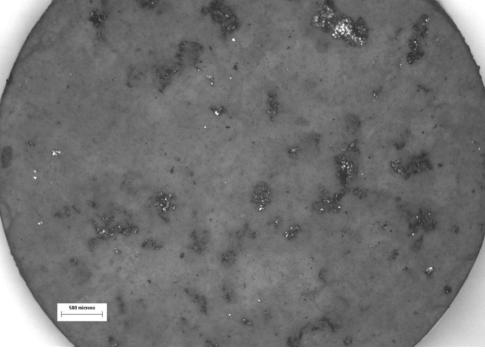


MOF-5 + 5wt% ENG, D=0.39 g/cc Magnification 25x



MOF-5 + 5wt% ENG, D=0.39 g/cc 6mm x 6mm pellet Magnification 25x

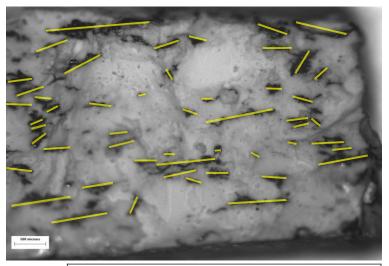




# Progress: Microscopy Analysis of MOF-5 & ENG

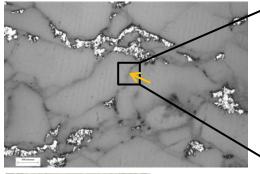


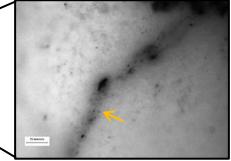
#### FMEA Task: Non-homogenous Bed Failure Mode - Evaluate pellet variation

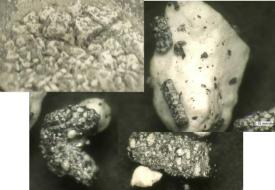


ENG orientation angle within pellets				
	Avg. Angle(degrees)	Avg. Length(mm)		
1	24.30	0.364		
2	24.95	0.310		
3	20.70	0.315		
4	23.44	0.254		
5	19.12	0.322		
Avg:	22.50	0.31		
Std Dev:	2.49	0.04		

MOF-5 + 5wt% ENG, D=0.39 g/cc Boundary lines between ENG and MOF-5, magnifications of 50x and 400x







ENG particles form around MOF-5 conglomerates during processing

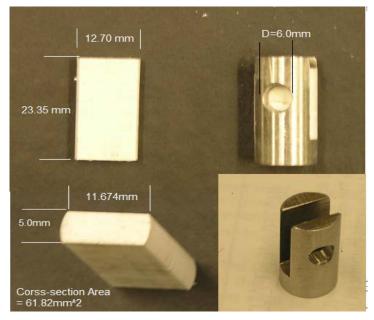
ENG deposits in the MOF-5 have a horizontal orientation preference



## Progress: Anisotropic Effects of MOF-5 & ENG

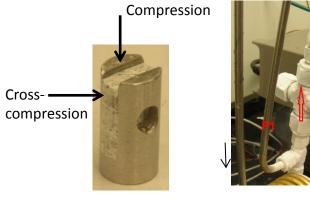
#### FMEA Task: Non-homogenous Bed Failure Mode - Evaluate pellet variation

## Anisotropic permeation evaluation was studied with specialized pellets



MOF-5+5% ENG, D=0.4 g/cc permeability in the cross-compression direction are listed in red to compare with the compression direction in black.

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#### **Table 2.**MOF-5+5wt % ENG sample density vs Darcy permeability & diffusivity

		-			<u> </u>
MOF	7-5 +5% ENG at 296	K	MOF-5 +5% ENG at 77 K		
D (gcm <sup>-3</sup> )	$\kappa$ , $\kappa_c$ (Darcy)	$D_{f}$ (m <sup>2</sup> s <sup>-1</sup> )	D(gcm <sup>-3</sup> )	$\kappa$ , $\kappa_c$ (Darcy)	$D_{f}$ (m <sup>2</sup> s <sup>-1</sup> )
0.3057	0.1896, 0.1743	2.086×10-3	0.2914	0.0486, 0.0465	1.373×10-3
0.3096	0.1741, 0.1717	1.942×10-3	0.3244	0.0209, 0.0193	5.877×10-4
0.3244	0.0796, 0.0689	9.028×10-4	0.3587	0.0081, 0.00679	2.292×10-4
0.3587	0.0581, 0.0461	6.314×10 <sup>-4</sup>	0.3600	0.0094, 0.00809	2.671×10 <sup>-4</sup>
0.3981	0.0278, 0.0185	3.059×10 <sup>-4</sup>	0.3974	0.0044, 0.0032	1.258×10 <sup>-4</sup>
0.4040	0.0409 0.0214	4.524x10 <sup>-4</sup>	0.3981	0.0046, 0.0036	1.321×10-4
0.4510	0.0160, 0.0087	1.764×10 <sup>-4</sup>	0.404	0.0111 0.00825	3.610x10-4
0.4908	0.0114, 0.0020	1.275×10 <sup>-4</sup>	0.4317	0.0041, 0.0031	1.169×10 <sup>-4</sup>
0.4933	0.0081, 0.0036	8.929×10-5	0.4510	0.0029, 0.0020	8.314×10-5
0.4942	0.0064, 0.0029	7.053×10-5	0.4933	0.0026, 0.0018	7.411×10-5
			0.4942	0.0022, 0.0015	5.971×10-5

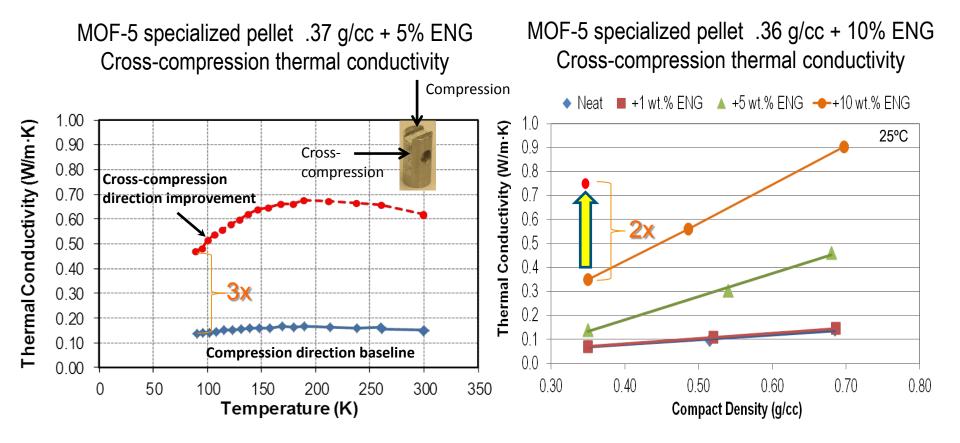
Specialized anisotropic pellet has a ~2x improvement in the permeability





## Progress: Anisotropic Effects of MOF-5 & ENG

FMEA Task: Non-homogenous Bed Failure Mode - Evaluate pellet variation



Specialized anisotropic pellet has a ~2x to 3x improvement in the conductivity

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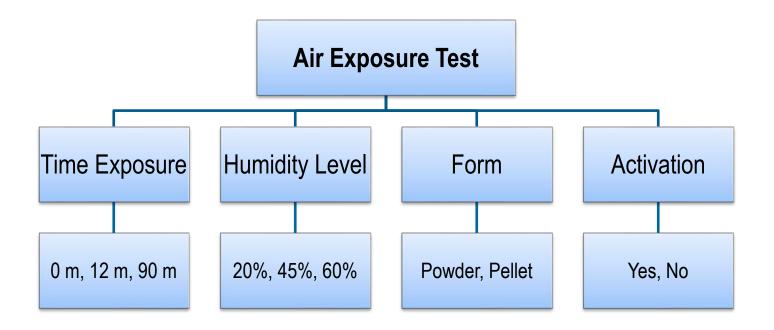
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# **Progress:** Humidity and Air Exposure Testing

FMEA Task: Air exposure & In-service Activation Failure Mode

Design of experiments testing initiated to evaluate the hydrogen uptake effect



Ford Lab - typical climate control over 24 hr period Humidity Level: 46.1%  $\sigma$  = .06% Temperature: 22.2 C  $\sigma$  = .3 C

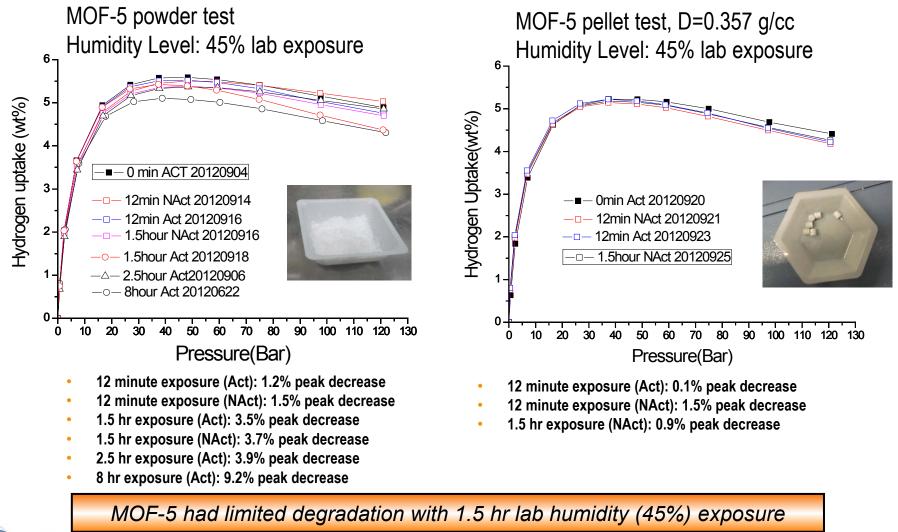


## **Progress:** Humidity and Air Exposure Testing



#### FMEA Task: Air exposure & In-service Activation Failure Mode

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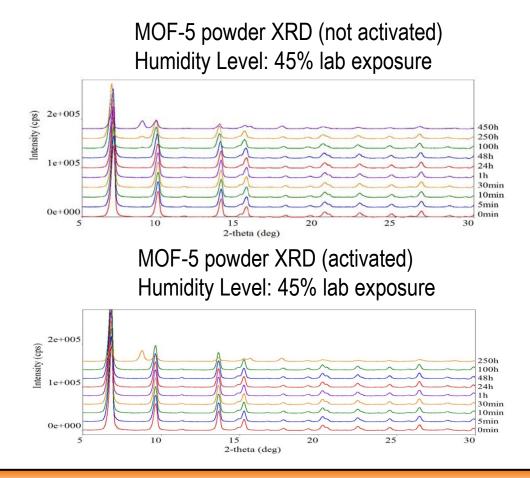




# **Progress:** Humidity and Air Exposure Testing

#### FMEA Task: Air exposure & In-service Activation Failure Mode

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XRD lab humidity results support the hydrogen uptake measurements





# Progress: MOF-5 Dust Ignition Safety Testing

#### FMEA Task: Containment Failure Mode – Material handling or rupture with ignitable dust mixture

Assumption: Worst case scenarios Evaluation: Safe handling of MOF-5, tank operation and rupture

#### **Experiments and results:**

1) <u>D</u>ifferential <u>S</u>canning <u>C</u>alorimetry:

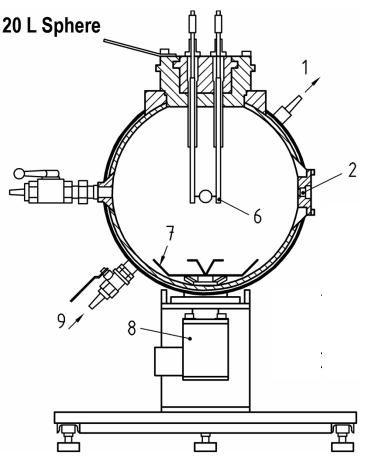
No chemical reaction between MOF-5 and hydrogen (energy release or onset temp)

2) Dust Explosibility Tests

The Hartman tube test did not ignite the MOF-5 with about 4 J of energy. The 20 L sphere dust test did result in ignition with a high energy level of 2 kJ. MIE is between these values with the exact value to be determined with further

Reference Information: - Static electric spark is typically 22 mJ

Material	Minimum Ignition Energy
Aluminum Dust	15 mJ
Magnesium Dust	40 mJ
Coal Dust	30 to 60 mJ
Grain-based Flour	240 mJ



Analyses done by BASF's safety engineering group in Ludgwigshafen, Germany in an accredited laboratory according to DIN EN ISO/ IEC 17025. All standard test methods are performed according to official guidance documents.



# **Summary:** Phase 2 SMART Milestones and Tasks



MOF-5 Material Development Tasks	<u>Status</u>
Density of $\ge 0.3$ g/cc with total capacity: $\ge 11\%$ and $\ge 33$ g/l	<ul> <li>✓ Demonstrated a theoretical total capability of ≥ 33 g/l for densities of ≥ 0.3 g/cc and potential for 11 wt %</li> </ul>
Thermal Conductivity of ≥ 0.5 W/m-K at 5-60 bar and 80-160 K	<ul> <li>✓ Demonstrated thermal conductivity of ≥ 0.5 W/m-K can be approached with 10% ENG at ~100 to 150 K</li> </ul>
Demonstrate effective kinetics for 3 minute fill of 5.6 kg	<ul> <li>Conducted sub-scale cycle test that provided effective kinetics with the potential of a 3 minute fill</li> </ul>
Demonstrate permeation with flow rate of 1 m/s and pressure drop of 5 bar	<ul> <li>Provided permeation data that indicates a <u>projected</u> pressure drop of 3.6 bar at 77 K for .3 g/cc</li> </ul>
MOF-5 Material FMEA Tasks	<u>Status</u>
Non-homogenous bed evaluation	<ul> <li>Completed microscopy analysis and evaluated potential to optimize with anisotropic properties</li> </ul>
Air exposure & in-service activation	<ul> <li>Initiated a design of experiments for humidity exposure</li> </ul>
Cycling over lifetime	<ul> <li>Confirmed over 390 cycles without degradation</li> </ul>
Impurity effects	<ul> <li>Planned a design of experiments for impurity exposure</li> </ul>
Safety Assessment	<ul> <li>Completed ignition and internal pressure evaluation</li> </ul>



#### **Future Work:** Complete Neutron Imaging Analysis BASE

## Completed in situ neutron imaging of MOF-5 pellets for model validation and mass transport analysis (1st Quarter 2013).

2200-3284

0-0.043Bar

37500-37994

### Results and Next Steps:

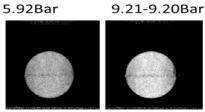
- Characterized transient behavior 1 associated with recharge and discharge as a function of rate and degree of fill.
- 2. Evaluated multiple orientations and ENG levels for anisotropic effects.
- Calculate intensity and validate the 3. mass transport models.



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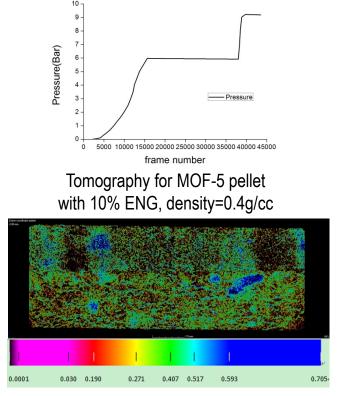
7405-8198

1.0-1.3Bar

41000-42000



#### Pressure vs. Frame number



The ENG particle (density=2.2g/cc) should have attenuation coefficient µ=0.608, which corresponds to dark blue

# Future Work: Technical gaps & near-term plans



#### 1. Demonstrate engineering concepts

- Scale-up and characterization of MOF-5 materials based on production manufacturing process
- Synthesis of 10 kg MOF-5 for system testing at UQTR and pucks for MATI system at OSU

#### 2. Required material properties

- Alternative approaches to enhanced thermal conductivity
- Robustness and failure mode testing:
  - o Powders vs pellets handling and alternative loading configurations
  - $\circ$  Clean vs. "dirty" H<sub>2</sub> (impurities other than humidity based on SAE J2719)
  - Failure mode and degradation mechanisms (i.e. thermal and cycle effects)
- Evaluate theoretical potential of MOFs and predict expected material properties

#### 3. Develop and validate engineering models

- Validate and refine integrated framework model based on system testing
- Translate sub-scale system test results to full scale vehicle simulations
- Identify cost/function benefits for system associated with the integration of components

#### Phase 3 SMART Milestones

- Report on the ability to enhance thermal conductivity beyond 10% ENG at operating temperatures
- Report on the degradation and failure modes associated with real-world operating conditions
- Determine the parameters and viability of a production on-board sorbent system

# **Collaborations:** HSECoE Partners





- SRNL (federal lab collaborator): team lead for sorbent (bed) transport phenomena, adsorbent system modeling, and center management
- Universite du Quebec a Trois-Rivieres (university collaborator): adsorption system test bench and MOF-5 isotherm validation
- GM (industrial collaborator): sorbent materials operating parameters, sorbent system modeling, and helical coil heat exchanger development
- Oregon State University (university collaborator): development of microchannel internal bed heat exchanger and combustors
- Hexagon Lincoln (industrial collaborator): pressure vessel development for hydrogen storage system concepts
- PNNL (federal lab collaborator): team lead for cost modeling, bill of materials, and materials operating requirements
- UTRC (industrial collaborator): material particulate testing, MOF-5 thermal conductivity measurements, and on-board system modeling
- NREL (federal lab collaborator): vehicle level modeling, wells-to-wheels analysis, MOF-5 isotherm validation, and low temperature isotherms
- JPL (federal lab collaborator): insulation development and cryogenic parameter evaluation

Interactions include monthly team meetings (sorbent system, material operating req., system modeling), regular data and information exchanges, and <u>nine</u> HSECoE face-to-face meetings

# **Technical Back-up Slides**



## General FMEA Overview and Approach

#### The FMEA is based on the required system functions from the technical targets.

Storage Parameter	Units	2010	2017	Ultimate
System Gravimetric Capacity: Usable, specific-energy from H <sub>2</sub> (net useful energy/max system mass) <sup>a</sup>	kWh/kg (kg H₂/kg system)	1.5 (0.045)	1.8 (0.055)	2.5 (0.075)
System Volumetric Capacity: Usable energy density from H <sub>2</sub> (net useful energy/max system volume)	kWh/L (kg H <sub>2</sub> /L system)	0.9 (0.028)	1.3 (0.040)	2.3 (0.070)
Storage System Cost <sup>b</sup> : • Fuel cost °	\$/kWh net (\$/kg H <sub>2</sub> ) \$/qge at pump	TBD (TBD) 3-7	TBD (TBD) 2-4	TBD (TBD) 2-4
Durability/Operability: • Operating ambient temperature <sup>d</sup> • Min/max delivery temperature • Operational cycle life (1/4 tank to full) <sup>e</sup> • Min delivery pressure from storage system; FC= fuel cell, ICE= internal combustion engine • Max delivery pressure from storage system <sup>1</sup>	°C °C Cycles bar (abs) bar (abs)	-30/50 (sun) -40/85 1000 5 FC/35 ICE 12 FC/100 ICE	-40/60 (sun) -40/85 1500 5 FC/35 ICE 12 FC/100 ICE	-40/60 (sun) -40/85 1500 3 FC/35 ICE 12 FC/100 ICE
<ul> <li>Onboard Efficiency</li> <li>"Well" to Powerplant Efficiency</li> </ul>	% %	90 60	90 60	90 60
Charging / Discharging Rates: • System fill time (5 kg) • Minimum full flow rate • Start time to full flow (20°C) <sup>9</sup> • Start time to full flow (-20°C) <sup>9</sup> • Transient response 10%-90% and 90% - 0% <sup>h</sup>	min (kg H₂/min) (g/s)/kW s s s	4.2 (1.2) 0.02 5 15 0.75	3.3 (1.5) 0.02 5 15 0.75	2.5 (2.0) 0.02 5 15 0.75
Fuel Purity (H <sub>2</sub> from storage) <sup>i</sup> :	% H <sub>2</sub>	SAE J2	719 and ISO/PDT (99.97% dry basi	
Environmental Health & Safety: • Permeation & leakage <sup>J</sup> • Toxicity • Safety • Loss of useable H <sub>2</sub> *	Scc/h - (q/h)kq H2 stored	Meets o	or exceeds applicable	standards 0.05

Cost of Ownership (Provide a competitive system)

Accept Fuel (Fill storage system)

Deliver Fuel (Supply H<sub>2</sub> from storage system)

Store Fuel (Manage H<sub>2</sub> in the system)



## General FMEA Overview and Approach

X

Seventy				
Effect	Ranking			
Hazardous without warning	10			
Hazardous with warning	9			
Very High	8			
High	7			
Moderate	6			
Low	5			
Very Low	4			
Minor	3			
Very Minor	2			
None	1			

Sovarity

# Occurrence Probability Ranking

X

Probability of Failure	Ranking
Very High: Persistent	10
Failures	9
High: Frequent	8
Frequent Failures	7
Moderate:	6
Occasional Failures	5
	4
Low:	3
Relatively Few Failures	2
Remote:	1
Failure is Unlikely	

# Detection=Likelihood<br/>of DetectionRanking<br/>10Absolute<br/>Uncertainty10Very Remote9Remote8Very Low7

Low

Moderate

Moderately High

High

Very High

Almost

Certain

RPN

)	Ranking	Risk
	10	Priority Number
;	9	
	8	
	7	
	6	
	5	
	4	
	3	

2

1

