Power for the Real World

High Performance, Durable, Low Cost Membrane Electrode Assemblies for Transportation Applications

> Andrew Steinbach 3M Company June 10th, 2015



Project ID: FC104

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

Project Overview

Timeline

- Project start: 9/1/12
- Project end: 8/30/15

Budget

- Total DOE Project Value: \$4.606MM*
- Total Funding Spent: \$3.691MM*
- Cost Share Percentage: 20%

* Includes DOE, contractor cost-share, and FFRDC funds, as of 2/28/15.

Barriers

- A. MEA Durability
- B. Stack Material & Mfg Cost
- C. MEA Performance

Partners

- Johns Hopkins Univ. (J. Erlebacher)
- Oak Ridge Nat'l Lab. (D. Cullen)
- Lawrence Berkeley Nat'l Lab.(A. Weber)
- Michigan Technological Univ. (J. Allen)
- Freudenberg FCCT (V. Banhardt)
- Argonne Nat'l Lab. (R. Ahluwalia)
- Los Alamos Nat'l Lab. (R. Mukundan, R. Borup)
- General Motors (B. Lakshmanan)

Objective and Relevance

Overall Project Objective: Development of a durable, low-cost, robust, and high performance membrane electrode assembly (MEA) for transportation applications, able to meet or exceed the DOE 2020 MEA targets.

	Primary Objectives and	Barriers	MEA, Catalyst Targets Addressed			
	Approaches This Year	Addressed	2020 Target	Target Values	Obj.	
1.	Improve MEA Robustness for Cold		0/AT	1.45kW / °C	3.4	
	Startup and Load Transient via	B. Cost				
	Materials Optimization,	C. Performance				
	Characterization and Modeling.		Cost	\$7 / kW	3,4	
2.	Evaluate Candidate MEA and	A. Durability				
	Component Durability to Identify		Durability with	5000 hours w/		
	Gaps; Improve Durability Through		cycling	< 10% V loss	2,3,4	
	Material Optimization and Diagnostic					
	Studies.		Performance	0.300A/cm ²	3,4	
3.	Improve Activity, Durability, and Rated	A. Durability	@ 0.8V			
	Power Capability of Pt ₃ Ni ₇ /NSTF	B. Cost	Performance	1W/cm ²	3,4	
	Cathodes via Post-Process	C. Performance	@ rated power			
	Optimization and Characterization.					
4.	Integrate MEAs with High Activity,	A. Durability	PGM Content	0.125g/kW		
	Rated Power, and Durability with	B. Cost	(both electrodes)	$0.125 \text{mg}_{\text{res}}/\text{cm}^2$	3,4	
	Reduced Cost.	C. Performance		OPGINI, *		

Approach, Milestones, and Status v. Targets

Approach: Optimize integration of advanced anode and cathode catalysts, based on 3M's <u>nanostructured thin film</u> (NSTF) catalyst technology platform, with next generation PFSA PEMs, gas diffusion media, cathode interfacial layers, and flow fields for best overall MEA performance, durability, robustness, and cost.

- 1. Place appropriate emphasis on key commercialization and DOE barriers.
- 2. Through advanced diagnostics, identify mechanisms of unanticipated component interactions resulting from integration of low surface area, low PGM, high specific activity electrodes into MEAs.

МС	Q	Project Milestone	0/ Complete	Status Against DOE 2020 Targets		
ID	T R	MS 1.2, 2.2, 4.2, and 5.2 based on Achievement of Multiple Project Goals (See Backup Slides)	(Apr. '15)	Characteristic	2020 Targets	Status, '14 / '15
BUDGET PERIOD 2 (June '14-Aug. '15)			$O/\Delta T (kW / °C)$	1.45	1.45	
1.2	11	Comp. Cand. Meet Project Perf./Cost Goals.	97%		<u>(@,8kW/g</u>)	(@ 6.2/6.5 kW/g)
2.2	11	Comp. Cand. Meet Project Cold-Start Goals.	50% (2 of 4)	Cost $(\$/kW)$	7	$6/5^*$
5.2	11	Comp. Cand. Meet Project Durability Goals.	82% (9 of 11)			$35/g_{Pt}; 0.692V$
4.2	11	Best of Class MEA Meets All Perf./Cost,	80%	Durability with cycling (hours)	5000	NA (In progress)
		Cold-Start, and Durability Project Goals		Performance @ 0.8V (mA/cm ²)	300	125 / 304*
3.2	12	Validation of Integrated GDL/MEA Model With ≥ 2 3M MEAs (Different Anode GDLs).	30%	Performance @ rated power (mW/cm ²)	1000	796 / 855* (0.692V, 1.45kW/°C)
6.3	12	BOC MEA: Short Stack Eval. Complete.*	10%	PGM total content $(\alpha/kW (rated))$	0.125	$0.162 / 0.155^{*}$
0	12	Final Short Stack to DOE. *	0%	PGM total loading		(0.0)2 V, 1.43KW/ C)
	*: Wo	ork contingent upon achievement of 3 operational robustne (US DRIVE FC Tech Team draft protocol).	(mg PGM / cm ² electrode area) *: 2015 values from 2013	0.125 5(Mar.) Best of	0.129 / 0.133*	
				includes a cathode interlayer with 15µg-Pt/cm ²		

Improved Activity, Rated-Power Capable ORR Catalysts (Task 1.1): JHU Chemical Dealloying Process Development for Pt₃Ni₇/NSTF



PEM Impact on

Durable, Improved Conductivity PEMs (Task 1.3):

3M-S Integration with NSTF Electrodes – Major Challenge Resolved



- As 3M-S PEM was varied, H₂/Air performance varied unexpectedly; observed with multiple experimental lots.
- Key issue preventing integration of durable, conductive 3M-S PEM.





Best of Class Component Integration (Task 4.1): 2015(Jan.) 3M NSTF Best of Class MEA



1) Increase 0.80V H₂/Air Activity (+10% Absolute Activity) 2) Reduce HFR 14mohm-cm² (Reduce interface R and Ni leaching)

Q/ ΔT kW/°C ≤ 1.45 1.45 (@ 7.3kW/g, 90°C)rformance @ ated powermW/cm² ≥ 1000 861 (@0.692V)ecific Powerg/kW ≤ 0.125 0.137 (@0.692V)GM Contentmg/cm² ≤ 0.125 0.118Key Improvements Over 20120.118Key Improvements Over 2012Improved dealloyed Pt ₃ Ni ₇ /NSTF – high J, 0.39A/mg mass activity (MEA), and reduced loading (0.103mg _{Pt} /cm²).725 EW PFSA, 3M-S 14µ PEM – ORR suppression minimized.Improved H ₂ /Air kinetics - <i>FF</i> flooding minimized via low RH.Minimized anode Pt (0.015mg/cm²).Narrower flow field land/channels.	erformance @ 0.80V	mA/cm ²	≥300	280					
rformance @ ated power $mW/cm^2 \ge 1000$ 861 (@0.692V)ecific Power g/kW ≤ 0.125 0.137 (@0.692V)GM Content $mg/cm^2 \le 0.125$ 0.118 Key Improvements Over 2012Improved dealloyed Pt ₃ Ni ₇ /NSTF – 	Q/ΔT kW/°C ≤ 1.45 (@ 7.3kW/g 90°C)								
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Narrower flow field land/channels.	Minimized anode Pt (0.015mg/cm ²).								

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Unit

Target

Value

Anode GDL for Improved Operational Robustness (Task 2.1):

Structured Freudenberg Papers; Thickness; Hydrophobic Treatment



Interlayer for Improved Operational Robustness (Task 2.2): Cathode Interlayer Design Factors (Pt wt%; Scale-up; carbon type; HT)



Interlayer for Improved Operational Robustness (Task 2.2):

Electrocatalyst and Support Cycle Durability Evaluation



Durability of Interim Downselect IL Likely Sufficient to Achieve DOE Targets, but Insufficient to Maintain Operational Robustness – Development Continues w/ Higher Durability IL (type B).

Best of Class Component Integration (Task 4.1): Integration of Improved Anode GDL, Cathode Interlayer w/ Best of Class CCM



Cold Start Modeling (Task 3): X-Ray CT Provides Unique Insight of Liquid Water Transport within Anode Backings (LBNL, I. Zenyuk)



Cold Start Modeling (Task 3): Integration of Michigan Technological University GDL Pore Network Model and LBNL MEA Model



Cold Start Modeling (Task 3): Integration of MTU GDL Pore Network Model and LBL MEA Model



MEA Rated Power Durability (Task 5): Improved PEM; Impact of Cell V

Baseline: 0.05/0.15PtCoMn/NSTF, 825EW 20µ PEM, Mod. Tech Team Load Cycle (90°C)



MEA Rated Power Durability (Task 5): Rated Power Loss Due to ORR Activity Loss; ORR Activity Loss Due to Two Factors (Cathode ECSA, PEM Decomposition)



Response To Reviewers' Comments

Addressing NSTF MEA Operating Condition Sensitivity and Project Approach

- "The Pt/C interlayer is of limited value because there are additional process costs and durability issues with inclusion of such a layer. ... Although the ... anode ... (GDL) designs have demonstrated improvements, MEA temperature performance is still significant and ... difficult to incorporate "as-is" in an automotive application.
- "NSTF has serious issues that must be addressed ... difficulty to break in, high sensitivity to contaminants, extreme sensitivity to low temperatures, and durability. The only way to address ... is to make some serious changes to the electrode configuration. Instead, this project is focused on minor changes that are having only a minor impact"
- The anode GDL and cathode interlayer approach has:
 more than doubled the stable operating temperature range in high heat capacity single cells; stack testing is needed to determine if sufficient.
 demonstrated high durability (rated power performance, mass activity) in ASTs, similar to NSTF MEAs w/o ILs. Enhanced operational robustness durability is being actively addressed.
- •Changes to electrode configuration could feasibly resolve operating condition sensitivity, but raises host of other issues (e.g. O₂ transport through ionomer film issue which limits min. PGM with traditional dispersed electrodes). Large project well beyond 2011 FOA scope.



Collaborations

- **3M** Project management; Materials and process optimization; MEA integration
 - A. Steinbach, D. van der Vliet, C. Duru, D. Miller, I. Davy (Core)
 - Cathode Integration: A. Hester, D. Lentz, S. Luopa, D. Tarnowski, B. Smithson, C. Studiner IV, A. Armstrong, M. Stephens, J. Bender, M. Brostrom
 - PEM Integration: M. Yandrasits, D. Peppin, G. Haugen, R. Rossiter
 - Anode GDL/Cathode IL: M. Pejsa, A. Haug, J. Abulu, J. Sieracki
 - Durability: A. Komlev
- Michigan Technological University GDL char. and PNM modeling; model integration
- <u>J. Allen</u>, E. Medici, V. Konduru, C. DeGroot
- Johns Hopkins University Pt₃Ni₇/NSTF dealloying method studies
 - J. Erlebacher

Lawrence Berkeley National Laboratory – GDL char. and MEA modeling; model integration

- <u>A. Weber</u>, J. MacDonald, I. Zenyuk, A. Kusoglu, S. Shi
- Oak Ridge National Laboratory Materials characterization (TEM, XPS)
- <u>D. Cullen</u>, H. Meyer III
- Los Alamos National Laboratory Accelerated Load Cycle Durability Testing
- <u>R. Borup</u>, R. Lujan, R. Mukundan
- Argonne National Laboratory NSTF HOR/ORR kinetic modeling, ORR activity/perf. modeling
- <u>R. Ahluwalia</u>, X. Wang, J-K Peng
- **General Motors** Stack Testing
- <u>B. Lakshmanan</u>

Remaining Barriers

- A. 2015(Mar.) Best of Class MEA does not achieve the DOE 2020 total loading and specific power targets, in part due to cathode interlayer PGM content.
- B. Enhanced robustness achieved w/ cathode interlayer is insufficiently stable under ASTs.
- C. 2015(Mar.) BOC MEA is likely not sufficiently durable to achieve MEA load cycle durability targets (maintain >15mA/cm² ORR act. after 5k hours).
 - 1. Pt₃Ni₇/NSTF cyclic durability insufficient
 - 1. Specific activity, rated power loss due to Ni leaching
 - 2. Specific area loss nanoporosity coarsening.
 - 2. PEM factors influencing rated power durability not yet fully eliminated.
- D. Operational robustness of 2015(Mar.) BOC MEA has not been demonstrated to be acceptable for automotive traction applications.

Key Future Work – FY15 (Through Aug. '15)

- A. Integrate experimental NSTF cathodes with higher mass activity (developed outside this project) to allow requisite 15µg/cm² PGM reduction to achieve total PGM target and approach specific power targets.
- B. Improve operational robustness durability by
 - 1. Integrate higher durability "type B" interlayers to maintain operational robustness through ASTs (AST evaluation in progress).
 - 2. Incorporate new anode GDLs w/ X2 backing and improved MPL (evaluation in progress)
- C. Improve load cycle durability by integration of higher durability NSTF cathodes and experimental PEMs with reduced degradation contaminant impact.
 - Experimental NSTF nanoporous electrode with ~50% lower specific area loss through 30k cycles developed (outside project). Dealloying optimization in progress, then integrate into BOC.
 - 2. Experimental PEMs which have demonstrated 30% lower rated power degradation rate will be integrated into BOC format.
- D. Conduct short stack testing to evaluate operational robustness of project BOC MEAs (under consideration by project team).

Summary

Operational Robustness (Cold Start; Load Transient)

- Integrated new anode GDL and cathode interlayer (@ 15µg_{Pt}/cm²) w/ Best of Class CCM, resulting in high rated power performance and 1A/cm² operation at 40°C.
- Modeling and characterization confirms banded anode GDL structure approach;
 PNM/MEA model integration in progress and is consistent with experiment.

Durability (MEA Load Cycling; Electrocatalyst/Support ASTs)

- Rated power loss mechanism confirmed and a material approach has shown 30% improvement in V loss rate.
- NSTF MEAs w/ interlayer (likely) pass DOE Electrocatalyst, Support durability ASTs but operational robustness diminished. High durability IL integration in progress.

Power, Cost (Cathode Post Processing; Best of Class MEA Integration)

- Dealloying scale-up feasibility complete process in control, factors understood.
- 3M-S integration complete key material, process factors identified and validated.
- MEA integration
 - substantial gains in specific power (up to 70% kW/g v. pre-proj.) due to improved absolute performance and PGM reduction.
 - DOE 2020 targets for loading, rated power approached

Technical Back-Up Slides

Project Goal Table

Table 11. Performance, Cost, Durability Targets, Current Project Status, and Go/No-Go and Goal Criteria							
Performance at ¹ / ₄ Power. Performance at rated nower, and O/AT Targets							
Goal ID	Project Goals (units)	Target Value	Status (NEW)	<mark>G/NG</mark> or Interim Goal Value			
1		0.300	0.304 ^A	0.250			
	Performance at 0.80V (A/cm ²); single cell, \geq 80°C cell	NA	NA	≥0.300			
	temperature, 50,100,150kPag, respectively.	NA	NA	≥0.300			
2	Performance at Rated Power, $Q/\Delta T$: Cell voltage at 1.41A/cm ² (Volts); single cell, \geq 88°C cell temperature, 50kPag [*]	0.709	0.672 ^A	0.659			
	Cost Targets						
3	Anode, Cathode Electrode PGM Content (mg/cm ²)	≤ 0.125	0.133 ^A	0.135			
4	PEM Ionomer Content (effective ion. thickness, microns)	≤16	12 ^A	20			
Transient response (time from 10% to 90% of rated power), Cold start up time to 50% of rated power at -							
	20°C, +20°C), and Unassisted start.						
5	Transient response (time from 10% to 90% of rated power); single cell at 50°C, 100% RH (seconds)	≤ 1	PASS (0%RH) ^F	5			
6	Cold start up time to 50% of rated power at +20°C; evaluated as single cell steady state J at 30°C (A/cm ²)	≥ 0.8	0.7 ^B	0.6			
7	Cold start up time at -20°C; short stack (seconds)	≤ 3 0	27 ^C	30			
8	Unassisted start from -40°C (pass/fail); short stack	Pass at	Pass at	Pass at			
		-40°C	-20°C ^C	-30°C			
MEA	MEA Durability with cycling, Electrocatalyst Cycle, Catalyst Support Cycle, MEA Chemical Stability, and Membrane Mechanical Targets						
9	Cycling time under 80°C MEA/Stack Durability Protocol with ≤ 30mV Irreversible Performance Loss (hours)	≥ 5000	600 ^{D,**}	2500			
10	Table D-1 Electrocatalyst Cycle and Metrics (Mass activity %	≤-40	-66±4	≤-40			
	loss; mV loss at 0.8A/cm ² ; % initial area loss)	≤-30	-13±15	≤- 30			
		≤-40	-28±4 ^E	≤-40			
11	Table D-2 Catalyst Support Cycle and Metrics (Mass activity %	≤-40	-40±7	≤-40			
	loss; mV loss at 1.5A/cm ² ; % initial area loss)	≤-30	-11 ± 3 (0.8)	≤-30			
		≤-40	-19±3 ^E	≤-40			
12	Table D-3 MEA Chemical Stability: 500 hours (H ₂ crossover	≤2	3.7±0.3	≤2			
	(mA/cm ²); OCV loss (% Volts); Shorting resistance (ohm-cm ²))	≤-20	-2	≤-20			
		>1000	971±98 ^E	>1000			
13	Table D-4 Membrane Mechanical Cycle: 20k Cycles (H ₂ crossover (mA/cm ²); Shorting resistance (ohm-cm ²))	≤2 >1000	20.1k cycles ^G	≤3 >500			

A: Mean values for duplicate or singular 3M 2015(Mar.) Best of Class NSTF MEAs: Anode=0.015PtCoMn/NSTF, Cathode= 0.103Pt₃Ni₇/NSTF + 0.015Pt/C Interlayer, (0.133m_{PGM}/cm² total), 3M-S 725EW 14µ PEM, Baseline 2979/2979 GDLs, 3M "FF2" flow fields, operated at 90°C cell temperature with subsaturated inlet humidity and anode/cathode stoichs of 2.0/2.5 and at stated anode/cathode reactant outlet pressures, respectively. B: Mean values for duplicate 3M NSTF MEAs: Anode=0.05PtCoMn/NSTF, Cathode=0.15PtCoMn/NSTF, (0.15mg_{PGM}/cm² total), 3M 825EW 24µ PEM, "X2"/2979 GDLs, Baseline Quad Serpentine Flow Field. C: OEM Stack testing results with 3M NSTF MEAs: Anode=0.10PtCoMn/NSTF. Cathode=0.15PtCoMn/NSTF. (0.25mg_{PGM}/cm² total), 3M ionomer in supported PEM, Baseline 2979/2979 GDLs. OEM-specific enabling technology. D: Mean or singular values for 3M NSTF MEAs: Anode=0.05PtCoMn/NSTF, Cathode=0.15PtCoMn/NSTF, (0.20mg_{PGM}/cm² total), 3M supported 825EW PEM, Baseline 2979/2979 GDLs, Baseline Quad Serpentine Flow Field. Values with estimated standard deviation error tested in duplicate. E: Value for Replicate 3M NSTF MEAs. Anode: 0.05PtCoMn/NSTF. Cathode=0.107 or 0.125 Pt₃Ni₇/ NSTF(Dealloy+SET), 3M 825EW 24µ PEM w/ or w/o additive, Baseline 2979/2979 GDLs, w/ or w/o Edge Protection, Quad Serpentine Flow Field. F: Mean values for duplicate 3M NSTF MEAs: Anode=0.05PtCoMn/NSTF. Cathode=0.15PtCoMn/NSTF. (0.15mg_{PGM}/cm² total), 3M 825EW 24µ PEM, "X2"/2979 GDLs, Baseline Quad Serpentine Flow Field. 0.03mgPt/cm² Cathode Interlayer. G: 2015(Jan). Best of Class PEM and GDLs Only. *: Cell performance of 0.709V at 1.41A/cm² with cell temperature of \geq 88°C simultaneously achieves the Q/ Δ T and rated power targets of 1.45kW/°C and 1000mW/cm², respectively. **: Single sample result. MEA failed prematurely due to experimental error. Notes Goal 9 is addressed in Task 5. Currently using higher T accelerated testing prior to evaluating at 80C. Goal 8 requires stack testing to achieve – contingent upon passing robustness criteria. Goal 10 requires cathode w/ improved durability – out of proj. scope but is in progress at 3M.

Cold Start Modeling (Task 3): MTU PNM Predicts Improved Low J Response of Banded Anode Papers – Maintains Higher Permeability



Best of Class Component Integration (Task 4.1): Strong Kinetic Response to Low RH – Due to FF Flooding (FF2, highly parallel)



relatively higher RH)

With 2015 BOC, performance maximized and

68°C inlet dewpoint at low J v. 84°C at high J.

stabilized with substantial RH reduction

MTU – LBL models

Double-trap kinetic model based on Wang and Adzic [1] formulation:



Concentrated species theory for diffusion Darcy's law for liquid and gas (convection) Ohm's law for ionic and electric currents Electro-osmosis and back-diffusion for membrane

[1] J. X. Wang, J. Zhang and R.R. Adzic, J. Phys. Chem. A (2007) [2] A.Z. Weber and J. Newman, JES 151 (2004)

Standard cell potential Equilibrium H₂O content membrane, liquid, vapor

[3] E. F. Medici, and J. S, Allen, IJHMT (2013)

Pore Network model of GDL [3]:

Pore Size, LogR (R,nm)

Robustness Metric Testing

Table 3. Robustness Criteria Needed for Stack Testing at GM Demonstration of the three robustness criteria to occur in subscale (e.g. 50cm²) hardware with stack candidate materials. Evaluation to occur at 3M. Status Status (1x X2 GDL, (2x X2 GDL, Target Criteria name Description Value Int. DS Cathode Int. DS Cathode IL IL(0.03mg/cm2))(0.015 mg/cm2))~ 0 (w/ 150kPa 0.38 Cold Operation Stack voltage at 30°C as a fraction of the stack voltage at > 0.3 80°C operation at 1.0 A/cm², measured using the protocol anode) for a polarization curve found in Table 3. A 25°C dew point is used only for 30°C operation. 0.29 w/ 100kPa Hot Operation 0.9 Stack voltage at 90°C as a fraction of the stack voltage at > 0.3 1.0 (performance 80°C operation at 1.0 A/cm², measured using the protocol increased) for a polarization curve found in Table 3. A 59°C dew point is used for both 90°C and 80°C operations. Cold Transient Stack voltage at 30°C transient as a fraction of the stack > 0.3 ~0 w/150kPa 0.38 voltage at 80°C steady-state operation at 1.0 A/cm², anode measured using the protocol for a polarization curve found "almost" achieved in Table 3. A 25°C dew point is used only for 30°C operation. 30°C transient operation is at 1 A/cm² for at @ 100kPa anode least 15 minutes then lowered to 0.1 A/cm² for 3 minutes without changing operating conditions. After 3 minutes, the current density is returned to 1 A/cm². The voltage is measured 5 seconds after returning to 1 A/cm^2 . CCM: 0.05PtCoMn/0.15PtCoMn, 3M 20u 825EW Anode GDL: 1 or 2 X2 w/ 3M Hydrophobic Treatment+ MPL (interim DS). Cathode IL: 2979 + "B" IL (interim DS)