2014 Hydrogen and Fuel Cells Program Annual Merit Review and Peer Evaluation Meeting Washington, DC – June 16-20, 2014

Advanced Materials and Concepts for Portable Power Fuel Cells

Piotr Zelenay

Los Alamos National Laboratory Los Alamos, New Mexico 87545

Project ID: FC091

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Overview

Timeline

- Project start date: September 1, 2010
- Project end date: August 31, 2014

Budget

- **FY13 DOE funding:** \$1,048K
- Planned FY14 funding: \$810K
- Total DOE project value: \$3,825K
- Cost share:

Barriers

- A. Durability (catalyst; electrode)
- B. Cost (catalyst; membrane; MEA)
- C. Electrode Performance (fuel oxidation kinetics)





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Relevance: Objective & Targets

Objective: Develop advanced materials (catalysts, membranes, electrode structures, membraneelectrode assemblies) and fuel cell operating concepts capable of fulfilling cost, performance, and durability requirements established by DOE for portable fuel cell systems; assure path to large-scale fabrication of successful materials

Technical Targets: Portable Power Fuel Cell Systems (< 2 W; 10-50 W; 100-250 W)				
Characteristics	Units	2011 Status	2013 Targets	2015 Targets
Specific power	W/kg	5; 15; 25	8; 30; 40	10; 45; 50
Power Density	W/L	7; 20; 30	10; 35; 50	13; 55; 70
Specific energy	Wh/kg	110; 150; 250	200; 430; 440	230; 650; 640
Energy density	Wh/L	150; 200; 300	250; 500; 550	300; 800; 900
Cost	\$/W	150; 15; 15	130; 10; 10	70; 7; 5
Durability	Hours	1,500; 1,500; 2,000	3,000; 3,000; 3,000	5,000; 5,000; 5,000
Mean time between failures	Hours	500; 500; 500	1,500; 1,500; 1,500	5,000; 5,000; 5,000

Original project technical targets (to be relaxed given modified targets above):

• System cost target: \$3/W

• **Performance target:** Overall fuel conversion efficiency (η_{Σ}) of 2.0-2.5 kWh/L

For methanol fuel:

(1) 2.0-2.5 kWh/L $\rightarrow \eta_{\Sigma} = 0.42-0.52$ (1.6-2.0× improvement over the state of the art, ~ 1.250 kWh/L) (2) If $\eta_{\text{fuel}} = 0.96$, $\eta_{\text{BOP}} = 0.90$, $V_{\text{th}} = 1.21$ (at 25°C)

$$V_{cell} = V_{th} [\eta_{\Sigma} (\eta_{fuel} \eta_{BOP})^{-1}] = 0.6 V$$



• **DMFC anode research** (JMFC, LANL, BNL):

(i) new catalysts with improved activity and reduced cost; (ii) durability improvements

- Innovative electrode structures for better activity and durability (UD)
- Hydrocarbon membranes for lower MEA cost and enhanced fuel cell performance (VT, LANL):
 (i) block copolymers; (ii) copolymers with cross-linkable end-groups
- Alternative fuels for portable fuel cells:
 - (i) dimethyl ether research (LANL); (ii) ethanol oxidation electrocatalysis (BNL)
- Advanced materials characterization (ORNL, BNL, LANL); MEA performance testing, including durability (LANL, JMFC, SFC); ten-cell stack (SFC)

Date	Milestones	Status	Comment
Dec 13	Synthesize multiblock copolymer based on tetramethyl bisphenol A capable of delivering > 200 mA cm ⁻² at 0.50 V (DMFC, 75°C).	Complete	> 200 mA cm ⁻² at 0.50 V achieved in a DMFC operating with TM-based multiblock copolymer at a cell temperature of 25°C
Mar 14	Improve mass activity of the precious metal catalyst of DME oxidation from the present-day 37 A g^{-1} to 50 A g^{-1} at 0.50 V (DDMEFC, 80°C).	Complete	Mass activity of 55 A g ⁻¹ (0.230 A cm ⁻²) achieved with LANL-developed ternary $Pt_{55}Ru_{35}Pd_{10}/C$ catalyst at low Pt cathode loading (2.0 mg cm ⁻²).
Jun 14	Achieve the DMFC performance goal of 150 mA cm ⁻² at 0.6 V with low Pt loadings (< 1.0 mg cm ⁻² at anode).	Pending	Presently 40 mV away from target (150 mA cm ⁻² at 0.56 V, 90°C).
Sep 14	Complete testing of a short stack utilizing fully optimized new components developed in the project.	Complete	10-cell DMFC stack utilizing JMFC's "advanced anode catalyst" (AAC) tested by SFC Energy for over 2,500 h; performance loss below that of a commercial stack with 2× higher PtRu loading.



MeOH Oxidation: Advanced Anode Catalyst (AAC) Development

Main activities: (i) further development of AAC (advanced anode catalyst) to meet the project milestone (0.15 A cm⁻² at 0.60 V) and (ii) stack demonstration (in collaboration with SFC Energy)

Strategy: (i) increase AAC loading from 1.0 to 2.0 mg_{Pt} cm⁻² (backup slide); (ii) lower carbon content to thin electrode layer; (iii) optimize Pt-to-Ru ratio to lower onset potential of MeOH oxidation; (iv) modify cell operating conditions (temperature, concentration)

Catalyst	Pt:Ru atomic ratio	Surface area by CO _{ads} (m² g ⁻¹)
HiSPEC [®] 12100	1:1	37
PtRu/C advanced anode catalyst (AAC)	1:4	20
PtRuC (new catalyst)	1:6	29

- No significant gain in AAC performance brought about by catalyst loading increase (backup slide)
- A 33% increase in wt% of metal (Pt+Ru) in AAC having no effect on catalytic activity
- Pt-to-Ru 1:4 atomic ratio: Optimum combination of fast dehydrogenation, efficient CO removal and low Ru crossover to cathode
- Highlight: Ten 50 cm² MEAs with optimized AAC prepared and supplied to SFC Energy for final stack testing







MeOH Oxidation: Testing Under Different Conditions

Anode: AAC, 1.0 mg_{Pt} cm⁻², 0.5 - 0.6 M MeOH; Cathode: Pt/C, 1.5 mg_{Pt} cm⁻², air (fuel cell), H₂ (anode polarization); Membrane: Nafion[®] 115; Cell: 80 - 88°C



- An increase in cell temperature to 88°C bringing a gain of 20 mV at 150 mA cm⁻²
- Highlight: Improvement in high current density observed without any additional methanol crossover loss with 0.6 M
- A decrease in c_{MeOH} from 0.6 M at 80°C (0.5 M at 88°C) to 0.4 M resulting in mass transport limitations at a current density of 150 mA cm⁻²

Cell performance achieved: 0.56 V at 0.150 A cm⁻² (88°C)

(at present, only 0.04 V away from target)



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JMX

Johnson Matthey Fuel Cells

the power within

Ten-Cell Direct Methanol Fuel Cell Stack

SFC EFOY Fuel Cell System 10-Cell DMFC Stack with JMFC AAC-Anode CCMs



U cell [mV] • Ucell avg filtered [mV] • Power stack [W] • Power out [W]



- Maximum stack voltage reached 70 h of operation
- Pump failure after *ca.* 2,500 h apparently affecting performance at longer times
- Highlight: Decay rate of 19 µV/(h per cell), slightly lower than in commercial MEAs with much higher Pt loadings

Stack milestone completed

AAC – promises to enable DMFCs for higher power applications



MeOH Oxidation: PtRu Nanostructures (SEM, TEM, HADDF-STEM)

PtRuNTs PtRuCuNWs HRTEM TEM SEN **TEM** 200 nm 0.2 HAADF-STEM HAADF-STEM Pt Ru Pt Ru Cu Cu 40 nm 40 nm 40 nm 40 nm Ru Ru ЭАК DGE National Laboratory 40 nm 40 nm

PtRuCuNWs: Cu-rich core, PtRu-rich shell





MeOH Oxidation: Innovative PtRu Nanostructure Catalysts



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Decoration of Expanded Pt Monolayer with Ru for Improved MeOH Oxidation Activity



Ru Nanoclusters	Onset Potential at 0.05 mA (V)*	i _f ∕i _b
Pt _{ML} /Au	0.288	1.2
Ru _{ML} /Pt _{ML} /Au	0.185	3.1
Ru _{2ML} /Pt _{ML} /Au	0.133	2.9
(Pt₃Ru₁) _{ML} /Au	0.207	1.3

* Ag/AgCl potential scale

- CO₂ the only observed product of methanol oxidation
- Ru deposition causing a shift in the onset potential of CO oxidation to *ca*.
 0.30 V *vs*. RHE (25°C)
- Different Ru quantities allowing finetuning MeOH oxidation activity

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DMFC Hydrocarbon Membranes

Technical Barrier	Related Property	Approach
High cell resistance	Proton conductivity	Multi-block copolymer
Low fuel efficiency	Methanol permeability	Nitrile group
Durability	Water uptake	Tetra methyl (TM) bisphenol A



* Kim et al., J. Electrochem. Soc. 2010, 157, B1616-B1623



DMFC Membranes: FY14 Multi-block Copolymers







- Successfully synthesized high molecular weight BisA, DM and TM multi-block copolymers
- TM-based copolymer shows more ordered domain structure (SAXS), higher elongation (stress-strain curve), and lower methanol permeability (back-up slide)
- Highlight: Reached DMFC performance of > 200 mA cm⁻² at 0.50 V (75°C)

December 2013 milestone achieved



DMFC Membranes: Progress in Multi-block Copolymer Research



- Extensive research performed towards reducing methanol crossover and water uptake
- Highlight: Much better performance with 60 μm TM-based copolymer MEA than with Nafion[®] 115 MEA (the industry standard); lower HFR, methanol crossover, and water uptake demonstrated at the same time

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Ethanol Oxidation: Improved Synthesis of Ternary Catalysts



Ethanol Oxidation: Alternative Oxide Catalysts & Intermetallic Pt_{ML} Support



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Intermetallic vs. Core-Shell (PdAuCo Support)



- Highlight: Several oxides, IrO_x in particular, identified as promising replacement for insufficiently stable SnO₂ in ternary catalysts of EtOH oxidation
- CeO₂ considerately enhances activity of Pt_{ML}/Pd/C in ethanol oxidation (also methanol oxidation, not shown)
- Pt_{ML}/PdAuCo shows high ethanol oxidation activity relative to Pt; intermetallic PdAuCo exhibiting better performance as a Pt_{ML} support than core-shell PdAuCo



DME Oxidation: MEA and Fuel Delivery System Optimization





- MEA and fuel delivery system optimization carried out with commercial PtRu/C HiSPEC[®] 12100 catalyst
- Highlight: FY13 to FY14 DDMEFC performance increase from 0.095 A cm⁻² to 0.215 A cm⁻² at 0.5 V, in spite of lowering the PGM anode loading by 25% in FY14



DME Oxidation: Development of Ternary PtRuPd/C Catalyst

PtRuPd catalyst:	Pt – primary DME adsorption and dehydrogenation
	Ru – source of surface oxidant for CO removal
	Pd – C-O and C-H bond scission catalyst

Electrode: Pt₈₀X₂₀/C (X = Pd, Ru) or HiSPEC[®] 9100 on GCE (0.196 cm²); 60 µg_{PGM} cm⁻²; Solution: DME-saturated (0.74 M), 0.1 M HClO₄; Cell: 5 mV s⁻¹; 60°C 0.14 PtPd PtRu 0.12 Pt Current density (mA cm Ecsa) °C0 ™ 0.10 . ^{зое} ³⁰⁸ СО 0.08 0.06 0.04 0.02 CO_{ads}, CH_{ads} 0.00 0.5 0.6 0.7 0.8 0.3 0.4 Potential (V vs. RHE)

Highlight: In agreement with proposed mechanism, (i) Pd addition results in higher current density with the same onset potential as observed with Pt; (ii) Ru leads to lower onset potential of DME oxidation

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Catalyst	Crystallite size (nm)	Lattice parameter (Å)
Pt ₈₀ Pd ₂₀ /C	1.9	3.93
Pt ₈₀ Ru ₂₀ /C	2.7	3.87
Pt/C (HiSPEC® 9100)	3.7	3.92

DME Oxidation: Development of Ternary PtRuPd/C Catalyst



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DME Oxidation: Progress at LANL FY11 - FY14

FY	How Progress Has Been Made	
2011	Pt-to-Ru ratios screened and Pt ₅₀ Ru ₅₀ selected	
2012	DME-to-water stoichiometry ratio and membrane optimized	
2013	New ternary $Pt_{45}Ru_{45}Pd_{10}/C$ catalyst developed replacing the $Pt_{50}Ru_{50}$ black anode catalyst	
2014	Ternary catalyst further optimized to Pt ₅₅ Ru ₃₅ Pd ₁₀ /C; MEA and fuel cell operating conditions improved	



Highlight: DDMEFC performance improved by a factor of at least **2.5**× over the project duration with concurrent significant reduction in precious metal loading



DME Oxidation: DDMEFC and DMFC Performance Comparison

Anode: 4.0 mg_{metal} cm⁻² PtRuPd/C, HiSPEC[®] 12100, 40 sccm DME gas, 26 psig, 1.8 mL/min 0.5 M or 1.0 M MeOH, 0 psig Cathode: 2.0 mg cm⁻² Pt/C HiSPEC[®] 9100, 100 sccm air, 20 psig; **Membrane**: Nafion[®] 212 (DME), Nafion[®] 115 (MeOH); **Cell**: 80°C



Performance of the latest-generation DDMEFC slightly exceeding that of the state-of-the-art DMFC

(Both systems operating under their respective optimum conditions)



Collaborations

- Seven organizations with complementary skills and capabilities in catalyst development, electrode-structure design, materials characterization, MEA fabrication, and portable fuel cell development and commercialization:
 - ✓ Los Alamos National Laboratory (LANL) *direct DOE EERE contract*
 - Brookhaven National Laboratory (BNL) direct DOE EERE contract
 - University of Delaware subcontract to LANL
 - Virginia Tech subcontract to LANL
 - Johnson Matthey Fuel Cells subcontract to BNL
 - ✓ SFC Energy *subcontract to BNL*
 - ✓ Oak Ridge National Laboratory *no cost partner*
- Collaborations outside Fuel Cell Technologies Program:
 - Oorja Fuel Cells, Fremont, California, USA reduction in DMFC cost for applications in excess of 1.0 kW in power; two LANL DMFC patents licensed by Oorja in April 2014

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For Immediate Re	elease:
Media Contact:	
Daniela Jaramillo	
djaramillo@oorja	fuelcells.com
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- University of Warsaw, Warsaw, Poland development of components for direct dimethyl ether fuel cell (common program development phase)
- Danish Technical University (DTU) direct dimethyl fuel cell electrocatalysis (joint research proposal to be submitted in summer 2014)



Responses to Previous Year Reviewers' Comments

"Very good progress was made in anode catalyst research. On the membrane research, it would be very useful to check how the TM membrane/electrode interface changes in durability test, to validate the hypothesis that matching water uptake to Nafion® can improve interface durability. Also, it is perhaps more useful to have the membrane swelling ratio data."

The data showed in our 2013 presentation was preliminary. Since then we have completed a thorough interfacial durability study of TM membrane/electrode, the results of which have been presented this year. While we plan to measure the swelling ratio of the multi-block copolymers, the relevance of the swelling ratio to interfacial durability may be questionable.

"Lack of long-term testing; 1000 hours is only a 2 month test; this is the fourth year of the program; this should have been done for all of their promising catalysts."

Most durability testing in the project has been performed using potential/voltage cycling (accelerated stress testing) to avoid tying either electrochemical or fuel cell test equipment to one cell for prolonged times. However, the most promising DMFC anode catalyst, Johnson Matthey's Fuel Cell's advanced anode catalyst (ACC), underwent this year testing in a 10-cell SFC Energy stack for *ca.* 3,500 hours. In spite of a much lower catalyst loading (approximately half of that used in SFC's commercial products), the catalyst showed lower rate of performance degradation than methanol oxidation catalysts used in commercial systems.

"Given team's strength in direct fuel cell systems, the team should drill down to the fundamentals of DME fuel system and determine the viability of the success of DME technology as a competitive technology to DMFC."

As highlighted in this year's presentation, advancements in DME research (both electrocatalysis and fuel delivery system) have been central to the project effort at Los Alamos. Test data shows that DME fuel cells can match, and in some cases outperform, the state-of-the-art DMFCs.



Direct methanol and dimethyl ether fuel cells:

- Further improvements to mass activity of both anode and cathode catalysts by modifications to existing catalyst formulations or development of new catalysts
- Reduction in cost to allow application to high-power systems (for example, by lowering catalyst cost and improving performance durability)
- Increase in fuel conversion efficiency via progress in anode catalysis (new formulations), reduction in fuel and anode-component crossover and enhancements in cathode tolerance to fuel/anode-component crossover
- Development of alternative membranes with improved stability and reduced permeability of fuels and anode catalyst component(s)

Ethanol as alternative fuel for direct fuel cells:

- Development of active and chemically stable catalyst with high selectivity for 12-electron oxidation of ethanol to CO₂ (in fuel cell type electrodes)
- Forsaking anode catalyst formulations that rely on intrinsically unstable components (including some core-shell formulations)
- Leveraging of DMFC/DDMEFC experience in the development of membranes with reduced crossover and in addressing effects of crossover on cathode performance



Direct methanol fuel cells:

- Complete current catalyst development efforts to meet last remaining project milestone (0.15 A cm⁻² at 0.60 V)
- Develop methanol oxidation catalysts free of intrinsically unstable components; possible formulations include two-dimensional platelets containing low-coordination atoms of precious metals and Au-core nanoparticles as supports
- Develop inks, GDL treatments, optimize and assure manufacturability by a scalable production process for advanced anode catalyst (AAC), targeting 50% Pt reduction in two years without performance/durability penalty (30% Pt reduction in FY15) and 500 W system

Direct DME fuel cells:

- Complete development and optimization of the ternary PtRuPd catalyst for DME oxidation
- Implement multiblock copolymer membranes in DDMEFC-type MEAs
- Complete detailed study of DME crossover and its impact on DDMEFC performance

Ethanol oxidation catalysis:

- Develop new-generation catalysts of ethanol oxidation, for example, catalysts on composites of stable oxides and lattice-expanded nanoparticles of precious metals; use in-fuel-cell stability and 12-electron selectivity and primary performance and selection criteria
- Perform assessment of DEFC viability at the present state of ethanol oxidation catalysis



Summary

- JMFC's advanced anode catalyst (AAC) reached 0.150 A cm⁻² at 0.56 V with low anode loading of 1.0 mg_{Pt} cm⁻² (total 2.5 mg_{Pt} cm⁻²).
- AAC was used in CCMs prepared JMFC for a 10-cell SFC Energy stack; in spite of much lower loading, ACC showed better stability over 2500 hours of stack operation than commercial catalysts.
- Progress in methanol catalysis is viewed by SFC Energy as enabling for higher power DMFC applications, power generators in particular (presently off-limits due to the prohibitive catalyst cost).
- High current density of 0.200 A cm⁻² at 0.50 (75°C) was achieved with TM-based multi-block copolymer; a 60-μm TM-based MEA showed lower resistance, methanol crossover and water uptake than a Nafion[®] 115-based MEA – a DMFC industry standard.
- Significant progress in DME electrocatalysis with the ternary PtRuPd/C was demonstrated resulting in a DDMEFC current density of 220 A cm⁻² (55 A g⁻¹ mass-specific activity of the anode catalyst) – a 2.5-fold improvement in activity since project inception.
- LANL DDMEFC was demonstrated to match state-of-the-art DMFC across the entire range of current densities.
- Recent advancements in ethanol oxidation electrocatalysis at BNL (Pt_{ML}/Au/C catalyst) led to *ca.* 200 mV reduction in overpotential of EtOH oxidation relative to Pt/C; LANL fuel cell testing is upcoming.









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- ethanol and methanol anode catalyst research

Radoslav Adzic (PI), Meng Li, Miomir Vukmirovic, Kotaro Sasaki

anode catalyst and membrane research; characterization

<u>Piotr Zelenay</u> (Project Lead), Hoon Chung, Joseph Dumont, Qing Li, Yu Seung Kim, Ulises Martinez, Yun Xu, Gang Wu

nanostructure catalyst structures

Yushan Yan (PI), Jie Zheng

hydrocarbon membrane research

James McGrath (PI), Jarrett Rowlett, Andy Shaver

methanol anode catalyst research; MEA integration

<u>Alex Martinez Bonastre</u> (PI), Willie Hall, Graham Hards, Emily Price, Jonathan Sharman, Geoff Spikes

- MEA integration and testing; final deliverable

Christian Böhm (PI)

microscopic characterization (no-cost partner)

Karren More (PI), David Cullen

Technical Backup Slides

MeOH Oxidation: AAC Anode; Effect of Annealing Temperature and Loading

Catalyst	CO metal area / m² g _{cat} -1
AAC	20
High metal content AAC @ T1	20
High metal content AAC @ T2	21
High metal content AAC @ T3	27
High metal content AAC @ T4	30

Anode: AAC, **1.0** mg_{Pt} cm⁻², 0.5 - 0.6 M MeOH; **Cathode:** Pt/C, 1.5 mg_{Pt} cm⁻² air; **Membrane:** Nafion[®] 115; **Cell:** 80°C



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Anode: AAC@T2, 2.0 mg_{Pt} cm⁻², 0.5 - 0.6 M MeOH; Cathode: Pt/C, 1.5 mg_{Pt} cm⁻² air (fuel cell), H₂ (anode polarization); Membrane: Nafion[®] 115; Cell: 80°C



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Johnson Matthey Fuel Cells

the power within

- Increase in annealing temperature helps performance of high metal-content (low carboncontent) catalysts (via increase in surface area), but not beyond that of low metal-content AAC
- Only small performance gain achieved with 2.0 mg_{Pt}/cm² (noticeably lower Pt mass activity)



DMFC Membranes: FY14 Multi-block Copolymers



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DME Oxidation: MEA and Fuel Delivery System Optimization

Anode: 4.0 or 3.0 mg_{PGM} cm⁻², PtRu/C (HiSPEC[®] 12100), 40 sccm DME gas, 26 psig; **Cathode:** 4.0 mg cm⁻² Pt black, 500 sccm air, 20 psig; **Membrane:** Nafion[®] 212; **Cell:** 80°C



• MEA and fuel delivery system optimization carried out with commercial PtRu/C HiSPEC[®] 12100 catalyst

 Highlight: Mass-specific activity of 67 A g⁻¹ reached with PtRu/C – the highest ever recorded, but obtained with a relatively high cathode loading of 4.0 mg_{Pt} cm⁻² and very high air flow (500 sccm)

