

## V.G.3 Advanced Materials and Concepts for Portable Power Fuel Cells

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Project Start Date: September 2010

Project End Date: August 2014

- Design and implement innovative electrode structures with better activity and durability in portable power fuel cell systems.
- Develop new hydrocarbon membranes based on (i) multiblock copolymers and (ii) copolymers with cross-linkable end-groups to assure lower MEA cost and enhanced fuel cell performance.
- Develop and demonstrate new oxidation electrocatalysts for two alternative fuels: ethanol (EtOH) and dimethyl ether (DME); evaluate viability of portable power systems based on alternative fuels to methanol.

### Technical Barriers

This project addresses the following technical barriers in the Fuel Cells section 3.4.5 of the Fuel Cell Technologies Multi-Year Research, Development and Demonstration Plan [1]:

- (A) Durability (catalysts, membranes, electrode layers)
- (B) Cost (catalysts, MEAs)
- (C) Performance (catalysts, membranes, electrodes, MEAs)

### Technical Targets

Portable fuel cell research in this project focuses on the DOE technical targets specified in Tables 3.4.7a, 3.4.7b, and 3.4.7c in Section 3.4.4 (Technical Challenges) of the Multi-Year Research, Development and Demonstration Plan [1]. Table 1 summarizes the latest DOE performance targets for portable power fuel cell systems in three power ranges.

Using DOE's Table 3.4.7 as guidance relevant to portable power systems, the following specific project targets have been devised:

- System cost target: \$5/W
- Performance target: Overall fuel conversion efficiency ( $\eta_{\Sigma}$ ) of 2.0-2.5 kWh/L (per liter of fuel)
- In the specific case of a DMFC, the above assumption translates into a total fuel conversion efficiency ( $\eta_{\Sigma}$ ) of 0.42-0.52, corresponding to a 1.6-to-2.0-fold improvement over the state of the art (*ca.* 1.250 kWh/L). Assuming fuel utilization ( $\eta_{\text{fuel}}$ ) and balance-of-plant efficiency ( $\eta_{\text{BOP}}$ ) of 0.96 and 0.90, respectively (efficiency numbers based on information obtained from DMFC systems developers), and using theoretical voltage ( $V_{\text{th}}$ ) of 1.21 V at 25°C, the cell voltage ( $V_{\text{cell}}$ ) targeted in this project can be calculated as:  $V_{\text{cell}} = V_{\text{th}} [\eta_{\Sigma} (\eta_{\text{fuel}} \eta_{\text{BOP}})^{-1}] = 0.6-0.7$  V

### Objectives

The main objective of this project is to:

- Develop advanced materials, catalysts, membranes, electrode structures, membrane-electrode assemblies (MEAs), and operating concepts for fuel cells that would help meet cost, performance, and durability requirements established by DOE for portable fuel cell systems; assure path to large-scale fabrication of successful materials.

### Fiscal Year (FY) 2012 Objectives

- Develop direct methanol fuel cell (DMFC) anode catalysts with enhanced activity, improved durability, and reduced cost.

TABLE 1. DOE Performance Targets for Portable Power Fuel Cell Systems in Three Power Ranges

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

Thus, the ultimate target of the materials development effort in the DMFC part of this project is to assure an operating single fuel cell voltage of at least 0.6 V. Very similar voltage targets have been calculated for fuel cells operating on two other fuels, EtOH and DME.

- A new ternary PtRuPd catalyst of DME oxidation synthesized and shown to perform better than a “standard” binary PtRu catalyst in electrochemical-cell testing.



## FY 2012 Accomplishments

- PtRu “advanced anode catalyst” of methanol oxidation demonstrated with performance exceeding that of the HiSPEC<sup>®</sup> 12100 benchmark by 40 mV; the catalyst synthesis successfully scaled up to 100 g.
- A ternary PtRuSn/C catalyst synthesized with methanol oxidation combining unique activity of PtSn/C at low overpotentials with superior performance of PtRu/C at high overpotentials; mass activity exceeding 500 mA/mg<sub>Pt</sub> at 0.35 V (higher than that of the most active thrifted PtRu catalysts).
- Onset potential of methanol oxidation improved by 30 mV with PtRu/CuNWs relative to the HiSPEC<sup>®</sup> 12100 benchmark durability on par with the benchmark catalyst.
- DMFC fuel utilization milestone of ≥95% at peak power achieved with 6F25BP75PAEB-BPS100 copolymer.
- DMFC accelerated performance degradation with increasing feed concentration of methanol shown to be associated with significant formation of cracks in the anode and cathode catalyst layers.
- Several carbon-supported Pt<sub>ML</sub>/Au and Pt<sub>ML</sub>/Pd catalysts demonstrated with the onset potential of ethanol oxidation in an electrochemical cell near 0.20 V vs. reference hydrogen electrode (RHE) at room temperature.
- Excellent DEFC anode activity shown with two ternary catalysts with the onset potential of ethanol oxidation very close to the thermodynamic value of ca. 0.04 V at 80°C.
- 250 mA cm<sup>-2</sup> at 0.40 V achieved in the DME fuel cell, exceeding the FY 2011 performance at 0.50 V by ca. 65%.

## Introduction

This multitask, multi-partner project targets advancements to portable fuel cell technology through the development and implementation of novel materials and concepts for (i) enhancing performance, (ii) lowering cost, (iii) minimizing size, and (iv) improving durability of fuel cell power systems for consumer electronics and other mobile and off-grid applications. The primary focus of the materials research in this project is on electrocatalysts for the oxidation of methanol, EtOH, and DME; on innovative nanostructures for fuel cell electrodes; and on hydrocarbon membranes for lower MEA cost and enhanced fuel cell performance (fuel crossover, proton conductivity). In parallel with new materials, this project targets development of various operational and materials-treatment concepts, concentrating among others on improvements to the long-term performance of individual components and the complete MEA.

## Approach

The two primary research goals of this project are (i) the development of binary and ternary catalysts for the oxidation of methanol, ethanol, and DME, and (ii) synthesis of hydrocarbon polymers (multiblock copolymers, copolymers with cross-linkable functional groups) for lower cost and better fuel cell performance through reduced fuel crossover and increased protonic conductivity. Better understanding of the key factors impacting the performance of both catalysts and polymers is also pursued through a major characterization effort including X-ray absorption spectroscopy, X-ray photoelectron spectroscopy, nuclear magnetic resonance, and transmission electron microscopy.

Development of new catalysts and polymers is closely tied to novel electrode nanostructures tailored to minimize precious metal content, maximize mass activity, and enhance durability. The electrode-structure component of the effort concentrates on two groups of materials: (i) solid-metal nanostructures (e.g., nanowires and nanotubes) and (ii) carbon-based nanostructures acting as supports for metal catalysts.

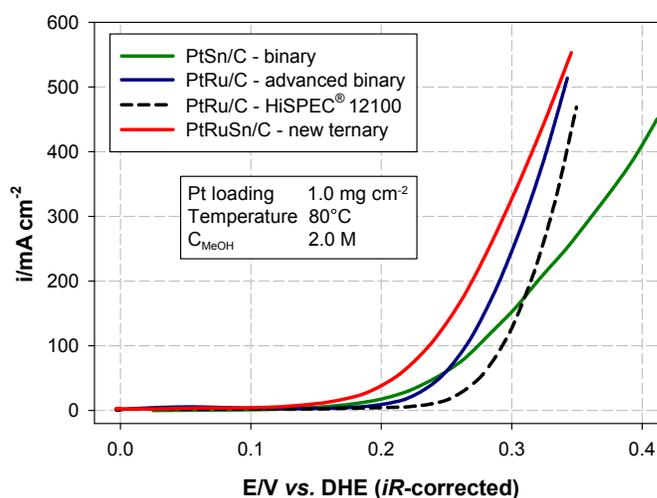
In addition to short-term testing and initial performance assessment, the catalysts, membranes, supports, electrode structures, and MEAs developed in this project are subject to long-term performance (durability) testing. Performance-limiting factors and degradation mechanisms are being identified and, if possible, addressed. Fabrication and scale up of viable catalysts, membranes, and supports is also being tackled through collaboration between partners in this project.

## Results

**DMFC Catalysts** — Development of new methanol oxidation catalysts continued in FY 2012 through “thrifting” of both precious metals, Pt and Ru, in the binary PtRu catalysts. A Variation 4 advanced anode catalyst (AAC) was synthesized and tested in the DMFC anode at 80°C, showing ca. 40 mV activity improvement relative to the benchmark HiSPEC® 12100 catalyst. The synthesis of the Variation 4 AAC was successfully scaled up to a 100-gram batch without a performance loss, in spite of a slightly lower specific surface area of the catalyst from the large batch. The DMFC anode research is on track to reaching the target of improved activity of thrifted PtRu catalysts without a durability loss and to achieving the project catalyst activity goal of 150 mA/cm<sup>2</sup> at 0.60 V (the DMFC voltage target).

An activity advantage of PtSn/C catalysts, PtSn catalyst with an atomic Pt-to-Sn ratio of 3:1 in particular, relative to PtRu/C was confirmed in the kinetic region (at current densities up to 150 mA/cm<sup>2</sup>). At the same time, the PtSn/C catalyst activity was found to be limited at potentials higher than 0.2 V due to the formation of a surface SnO<sub>2</sub>, resulting in a decrease in the OH availability for the oxidation of surface CO. This drawback of the PtSn catalyst was the reason of a “no-go” decision for further research on binary PtSn catalysts.

However, in an attempt to combine the unique activity of the PtSn catalyst at low methanol oxidation overpotentials with the superior performance of PtRu binary catalysts at high current densities, the effort shifted to the development of a PtRuSn/C ternary catalyst. Four different synthesis approaches were used, of which one proved particularly successful yielding a catalyst with significantly higher methanol oxidation activity in the entire range of the DMFC anode potentials than that of the most active “thrifty” PtRu catalysts and the benchmark HiSPEC® 12100 catalyst



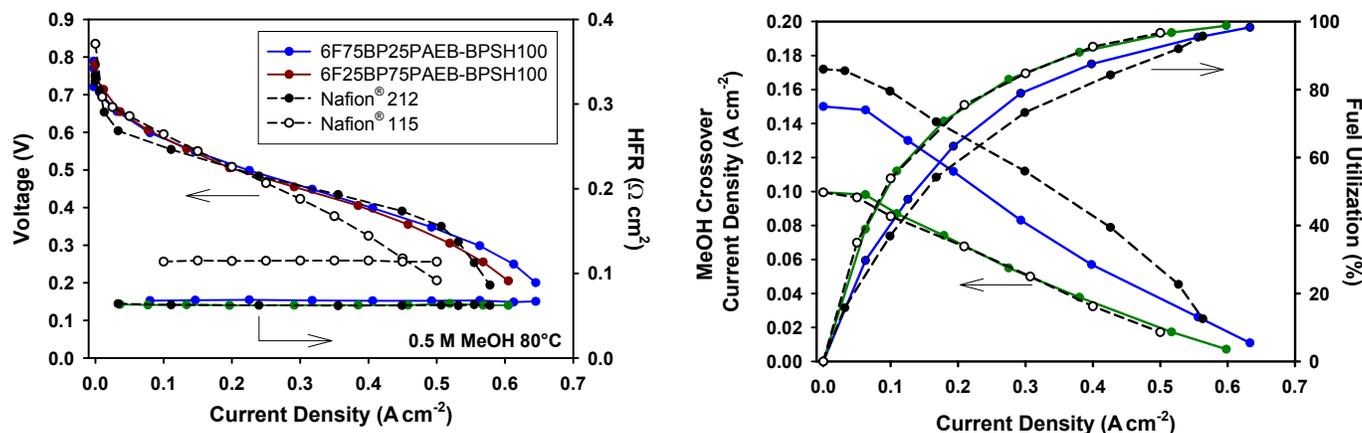
**FIGURE 1.** DMFC anode polarization plots recorded with a new ternary PtRuSn/C catalyst. Polarization plots for an advanced binary PtRu/C, a binary PtSn/C and a benchmark HiSPEC® 12100 PtRu catalyst shown for reference.

(Figure 1, red curve). The mass activity of 500 mA/mg<sub>Pt</sub> at 0.35 V was reached with the new ternary catalyst, exceeding by 150% the interim mass-activity target of 200 mA/mg<sub>Pt</sub> at 0.35 V. Future research will focus on further improvements in the PtRuSn/C catalyst activity and on assuring its durability under the operating conditions of a DMFC.

**Innovative Electrode Structures** — PtRu and PtSn nanowire catalysts for methanol oxidation were obtained using Cu nanowire (CuNWs) supports. The onset potential of methanol oxidation in an electrochemical cell at a room temperature was improved by 20 and 30 mV with PtSn/CuNWs and PtRu/CuNWs relative to the benchmark PtRu/C catalyst (HiSPEC® 12100), respectively. Performance stability of both catalysts was demonstrated to be on par with the benchmark catalyst.

**Multiblock Copolymers for Reduced MeOH Crossover** — Highly conductive multiblock copolymers were prepared using telechelic block polysulfone ether polymer (BPSH)-100 oligomers. The block size of these polymers varied between 7,000 and 15,000 g. The copolymers showed much reduced methanol permeability relative to previous-generation multiblock materials (no more than 10-15% higher than that of the reference Nafion® perfluorosulfonic acid polymer). Thanks to their high protonic conductivity the multiblock copolymer membranes were found to outperform Nafion® 212 in DMFC testing. DMFC current densities in excess of 0.28 A/cm<sup>2</sup> at 0.5 V (a membrane performance milestone) were demonstrated with three out of 11 multiblock copolymers synthesized.

In order to further reduce methanol permeability of the copolymers, biphenyl (BP) groups were introduced into the polymer backbone and ratio of BP to 6F-BPA was varied. Small angle X-ray scattering profiles obtained with different



**FIGURE 2.** DMFC polarization plots (left) and methanol crossover and fuel utilization plots (right) for two multiblock copolymers and reference Nafion® 212 and 115 membranes;  $c_{\text{MeOH}} = 0.5 \text{ M}$ ; cell temperature  $80^\circ\text{C}$ .

copolymers indicated an increase in the interdomain distance with a decrease in the 6F-BPA content, accompanied by the formation of a less ordered structure of copolymers and drop in methanol permeability. A 55% reduction in methanol crossover relative to Nafion® was measured with the least permeable copolymer, containing 25% of 6F-BPA groups (versus 75% of BP groups).

Fuel cell test data attested to improved MEA performance of multiblock-copolymer membranes relative to Nafion® at DMFC voltages higher than ca. 0.55 V (Figure 2, left), with similar resistance of the hydrocarbon and Nafion® membranes maintained across the entire range of fuel voltages. A fuel utilization of 95% was achieved with a multiblock copolymer at the peak DMFC power point (Figure 2, right).

**DMFC Performance Degradation**— The impact of the feed concentration of methanol on the rate of DMFC performance degradation was studied at four MeOH concentrations, 0.5, 1.0, 2.0, and 4.0 M. The unrecoverable DMFC performance loss was found to significantly increase with methanol concentration. At the same time, the fraction of the overall performance loss that could be recovered noticeably decreased. A relatively small unrecoverable performance loss of 3% after a 100-hour test at 0.40 V was measured only with 0.5 M MeOH.

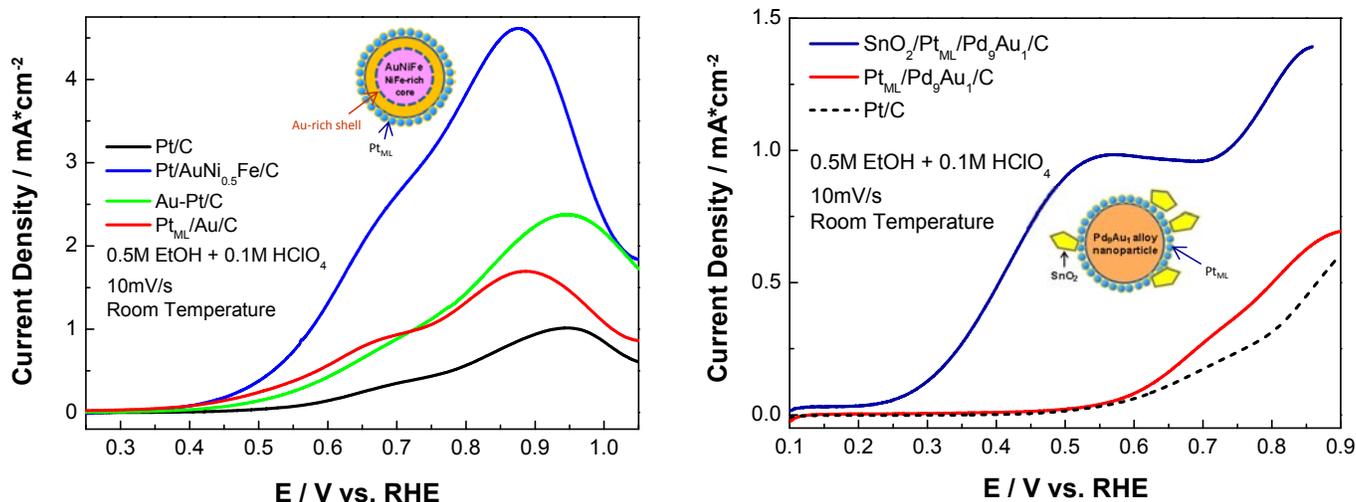
Post-mortem X-ray tomography of MEAs revealed cracking of both the anode and cathode catalyst layers that substantially increased with the feed concentration of methanol. The cathode was found more vulnerable to cracking at high methanol concentrations, with more than 9% of the surface covered by the cracks after a 100-hour life test with 4.0 M methanol. Once (and if) unequivocally correlated to DMFC performance loss crack formation may require development of an effective mitigation strategy.

**EtOH Oxidation Catalysts**— In the part of research involving well-defined surfaces, the lattice expansion in Pt<sub>ML</sub> supported on Au(111) was found to result in significantly enhanced EtOH oxidation current relative to Pt(111), with indications of improved selectivity in CO<sub>2</sub> generation. “Engineering” of the Pt<sub>ML</sub>/Au(111) surface led to an additional shift in the onset EtOH oxidation potential to below 0.2 V vs. RHE at the Sn(OH)<sub>x</sub>/(Pt<sub>3</sub>Ir<sub>1</sub>)<sub>ML</sub>/Au(111) catalyst. While these effects pave the road for further improvements in ethanol-oxidation electrocatalysis they also highlight the need for cost-effective core materials.

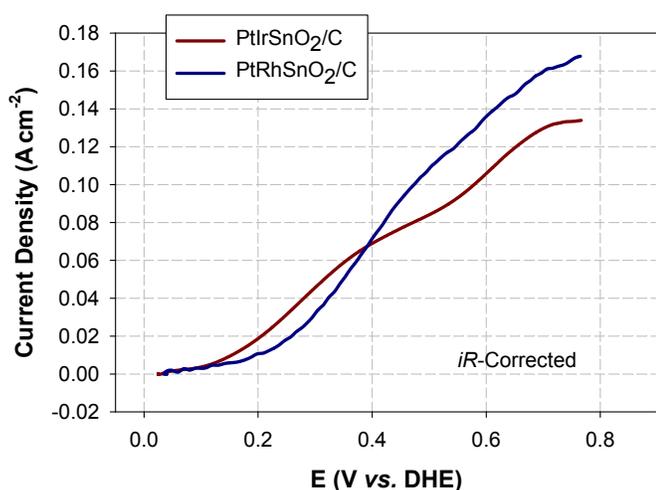
In the part of research focusing on highly DEFC-relevant carbon-supported catalysts, very promising activity was demonstrated using a Pt<sub>ML</sub>/AuNi<sub>0.5</sub>Fe/C catalyst with reduced noble metal loading in the nanoparticle core (Figure 3, left). A SnO<sub>2</sub>/Pt<sub>ML</sub>/Pd<sub>9</sub>Au<sub>1</sub>/C catalyst (Figure 3, right) was found to exhibit the lowest onset potential of EtOH oxidation among carbon-supported catalysts, comparable to that measured with the most active single-crystal catalysts (high CO<sub>2</sub> yields are also likely). In situ infrared reflection-absorption spectroscopy (IRRAS) and on-line differential electrochemical mass spectroscopy (DEMS) are close to being completed for the study of substrate-induced change in Pt<sub>ML</sub>'s selectivity for the oxidation of EtOH.

Finally, excellent activity was demonstrated with two ternary catalysts developed in FY 2011. The onset potential of EtOH oxidation measured in a DEFC at  $80^\circ\text{C}$  with the PtIrSnO<sub>2</sub> and PtRhSnO<sub>2</sub>/C anode catalysts was very close to the thermodynamic value of ca. 0.04 V (Figure 4). However, the DEFC performance was significantly below that expected based on the activity of both anodes due to the cathodes contamination by crossover anode species. Reduction in the non-noble metal migration from the anode is required.

**DME Fuel Cell Research**— It was determined that the DME-to-H<sub>2</sub>O ratio of 1.4:1 used previously for in the DME fuel cell (with anode humidifier at  $85^\circ\text{C}$ ) was much



**FIGURE 3.** Ethanol oxidation plots on carbon-supported Pt<sub>ML</sub>/Au (left) and Pt<sub>ML</sub>/Pd catalysts (right) in an aqueous 0.1 M HClO<sub>4</sub> electrolyte electrochemical cell at room temperature;  $c_{\text{EtOH}} = 0.5$  M. Catalysts structures shown in the insets.



**FIGURE 4.** Direct EtOH fuel cell anode polarization plots recorded with two carbon-supported ternary catalysts, PtIrSnO<sub>2</sub>/C and PtRhSnO<sub>2</sub>/C at 80°C. Anode: 1.0 mg/cm<sup>2</sup><sub>metal</sub> 12 wt% PtIrSnO<sub>2</sub>/C or 13% PtRhSnO<sub>2</sub>/C, 0.5 M EtOH, 1.8 ml/min; cathode: 4.0 mg/cm<sup>2</sup> Pt black, 200 sccm H<sub>2</sub>; membrane: a triple Nafion® 212 sandwich.

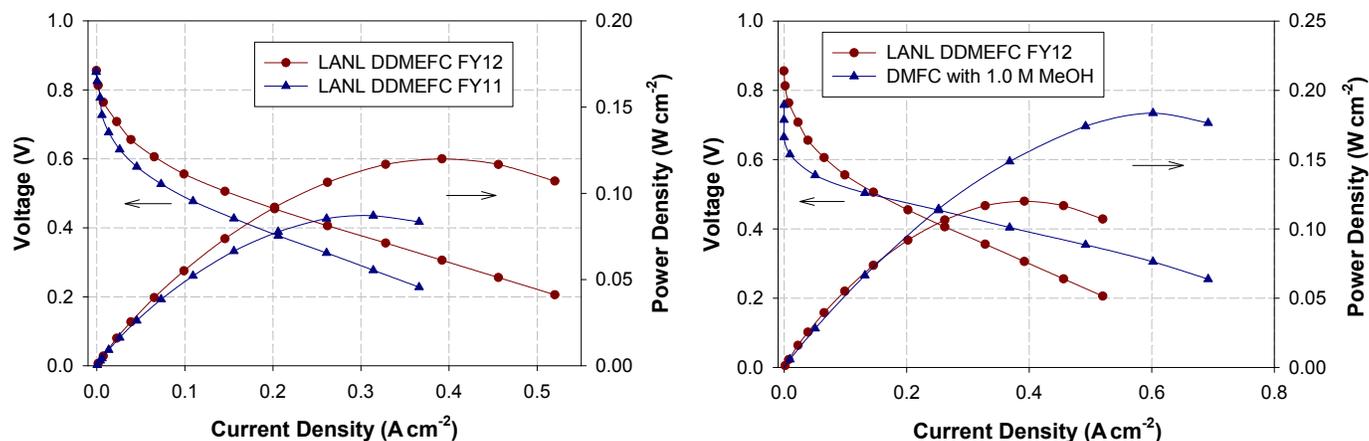
larger than required by the reaction stoichiometry (1:3) and possibly resulted in a water deficiency at the anode. DME fuel cell performed better with the molar DME-to-H<sub>2</sub>O ratio closer to stoichiometric. As a result, a gas-fed DDMFC with the anode humidifier maintained at 110°C was found to outperform the liquid-fed DME fuel cell. Unlike DMFC performance, the internal resistance-corrected direct DME fuel cell performance was found to be independent of the membrane thickness, attesting to a relatively low fuel crossover and/or lower activity of the Pt cathode in DME than MeOH oxidation at high potentials.

The direct DME fuel cell performance reached current density of more than 250 mA/cm<sup>2</sup> at 0.40 V, exceeding previous-year performance by ca. 65%. At voltages higher than 0.49 V, the direct DME fuel cell performance was found superior to that of a corresponding DMFC, mainly due to reduced effect of DME crossover on the cathode activity compared to that of methanol crossover (Figure 5). Based on those results a “go” decision was made for further DME research.

Finally, a new ternary PtRuPd catalyst was synthesized, with Pd added to aid in the C-O bond cleavage during DME oxidation. The catalyst, which exhibited significant activity in testing performed in an electrochemical cell, will be next optimized for maximum activity and stability under DME fuel cell operating conditions.

## Conclusions

- The latest PtRu “advanced anode catalyst” exceeded performance of the HiSPEC® 12100 benchmark by 40 mV; the catalyst synthesis was successfully scaled up to 100 g; a “no-go” decision was made for further PtSn catalyst research; the effort was redirected towards PtRuSn catalysts that already showed very promising activity in methanol oxidation.
- PtRu/CuNW catalyst was synthesized with a ca. 30 mV improvement in the onset potential of methanol oxidation relative to the HiSPEC® 12100 benchmark.
- Multiblock copolymers, e.g. 6F25BP75PAEB-BPS100, allowed for up to 55% reduction in methanol crossover relative to the Nafion® 212 benchmark; fuel utilization up to 95% was reached with 0.5 M methanol feed near the peak-power point.



**FIGURE 5.** Polarization and power-density plots depicting progress in direct DME fuel cell performance at LANL (left) and performance comparison between direct DME fuel cell and DMFC at 80°C (right). Anode: 6 mg/cm<sup>2</sup> Pt<sub>50</sub>Ru<sub>50</sub> black, 40 sccm DME gas (direct DME fuel cell), 30 psig or 1.0 M MeOH (DMFC); cathode: 4 mg/cm<sup>2</sup> Pt black, 20 psig (direct DME fuel cell) or 0 psig (DMFC), 500 sccm air; membrane: Nafion® 212.

- High feed concentration of methanol was found to accelerate DMFC performance degradation strongly and lead to significant cracking of both the anode and the cathode.
- Pt<sub>ML</sub> catalysts with expanded lattice and “engineered” catalysts were found capable of delivering the onset potential of EtOH oxidation of ca. 0.2 V vs. reference hydrogen electrode (room temperature).
- Both PtIrSnO<sub>2</sub>/C and PtRhSnO<sub>2</sub>/C ternary catalysts showed capability of oxidizing EtOH in an MEA at 80°C at potentials close to the thermodynamic value of 0.04 V.
- DME performance was improved by 65% relative to the previous year, resulting in a “go” decision for further DME research; a new PtRuPd/C catalyst showed promise in aiding the C-O bond cleavage.

## Future Directions

- Methanol oxidation catalysis: Further develop PtRuSn ternary catalysts to improve the kinetic performance at low Pt loadings; develop protocols for stack testing under 75-80°C, 0.5 M methanol conditions; evaluate stability and durability of new methanol oxidation catalysts; meet durability milestone (durability of thrifed PtRu catalyst matching that of HiSPEC® 12100 without activity loss); carry out breakdown of performance losses in DMFCs and initiate development of mitigation strategies; optimize accelerated corrosion test to mimic decay mechanisms in long-term stack testing.
- Innovative membranes and electrode structures: Continue reducing methanol crossover by introducing hydroquinone into multiblock copolymers; improve durability of alternative membranes in the presence of higher concentrations of MeOH; develop PtSn/CuNW

structure to achieve the onset potential of methanol oxidation of 0.29 V and 20% improvement in platinum group metal mass activity of innovative nanostructure catalysts.

- EtOH oxidation catalysis: Establish methodology for the synthesis of Pt<sub>ML</sub>-nanoparticle catalysts with cost-effective core materials for the deposition of Pt<sub>ML</sub> and active promoters (SnO<sub>x</sub>, SnO<sub>2</sub>, Ru, etc.); scale up the synthesis; implement in situ IRRAS and on-line DEMS to determine substrate-induced selectivity of Pt<sub>ML</sub>s in EtOH (and methanol) oxidation and EtOH oxidation at ternary PtRhSnO<sub>2</sub>/C and PtIrSnO<sub>2</sub>/C catalysts; determine the mechanism of cathode performance loss in direct ethanol fuel cells operating with ternary anode catalysts; develop a mitigation strategy.
- DME research: Develop a model of DME oxidation and catalyst requirements; optimize the ternary PtRuPd catalyst for maximum activity and stability at the DME fuel cell anode.

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