Hydrogen, Fuel Cells & Infrastructure Technologies Program
2005 Annual Review
Washington, DC, May 23-27, 2005

Direct Methanol Fuel Cells

Rajesh Bashyam, Eric Brosha, Christine Hamon, Yu Seung Kim
Jong-Ho Choi, Manoj Neergat, Barbara Piela, Piotr Piela
Bryan Pivovar, Gerie Purdy, John Ramsey, Mahlon Wilson
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DOE Program Manager:	**Nancy Garland**	*Project ID*
LANL Program Manager:	**Ken Stroh**	*FC - 34*

Selected Collaborations & Interactions (ⓒ)

- *Catalyst Research & Development*

  E-TEK / de Nora North America, Dr. Emory de Castro *– metal blacks and carbon-supported catalysts for DMFC anode and cathode*
  University of Illinois, Professor Andrzej Wieckowski *– fundamental electrocatalysis; methanol-tolerant cathode catalysts (chalcogenides)*
  University of New Mexico, Professor Plamen Atanassov *– methanol-tolerant cathode catalysts (metalloporphyrins)*
  Université de Poitiers, Professor Nicolas Alonso-Vante *– chalcogenides as alternative catalyst for DMFC cathodes*

- *Membranes / Membrane-Electrode Assemblies*

  Virginia Polytechnic, Professor James McGrath *– alternative polymers and MEAs with significantly improved selectivity and durability*
  Mesoscopic Devices, Inc., Valerie Hovland *– catalysts, membranes, MEAs, and feed schemes for mixed-reactant fuel cells*

- *DMFC Stacks & Sensors*

  Mesoscopic Devices, Dr. Jerry Martin *– DMFC stack for portable power applications*
  Ball Aerospace, Dr. Jeff Schmidt *– methanol concentration sensors*

- *Technology Transfer*

  Government use license issued in FY 2005 *(two such licenses in FY 2004)*

Project Objective & Focus

Objective

Develop materials and design operating conditions for direct methanol fuel cells that would enable maximum specific power, energy conversion efficiency and cell performance durability at a tolerable cost.

Focus

- **Precious and non-precious catalysts for DMFC anode and cathode; highly-selective Nafion® and alternative membranes; membrane-electrode assembly design & structure; novel methods of fuel cell diagnostics**
- **DMFC performance durability: factors impacting performance loss; new techniques and materials for improved cell life-time**
- **Assistance to the industry in commercialization of DMFC systems for portable power (catalysts, MEAs, sensors, fuel cell stack prototypes, technology transfer)**

Funding & Milestones

Funding

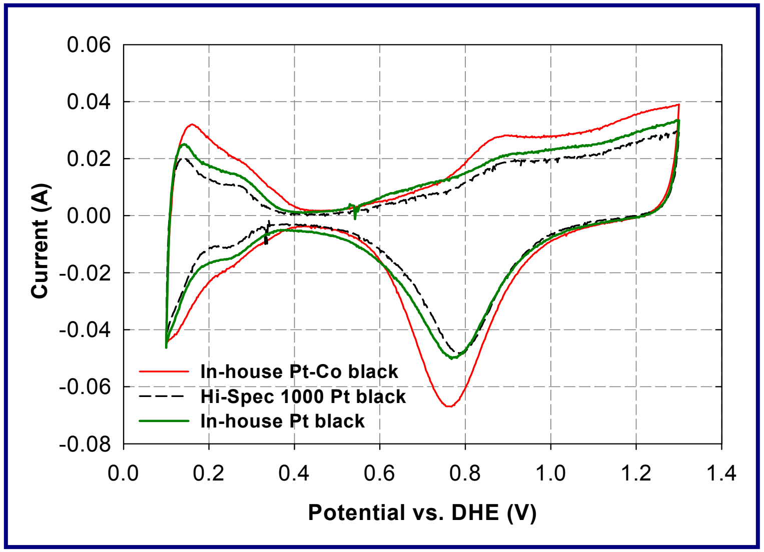
FY 2004	$300K
FY 2005	$350K
FY 2006	*uncertain again?*

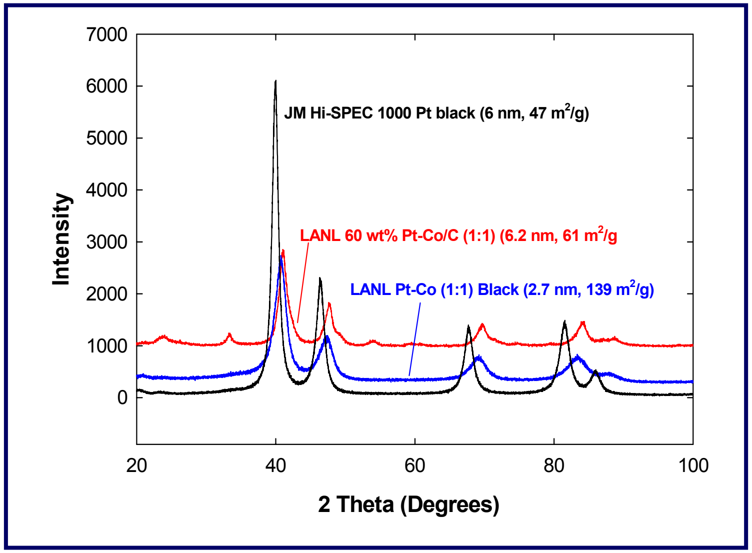
2005 Milestones

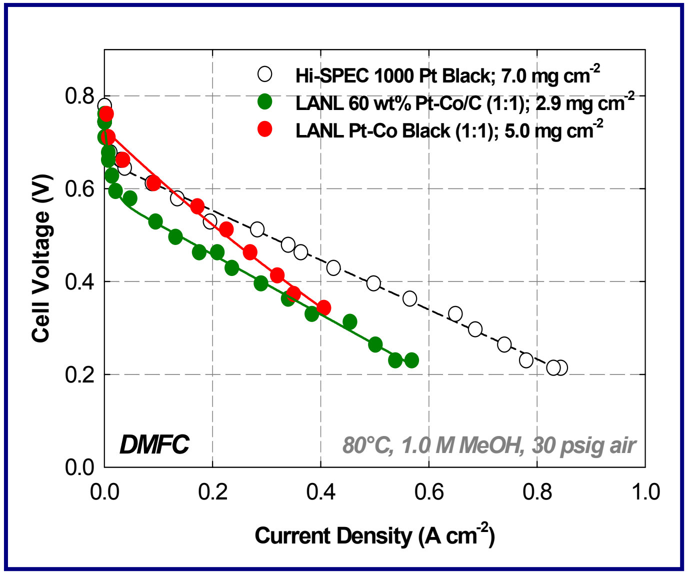
- **Demonstrate new unsupported DMFC cathode catalyst with average particle size reduced by at least 40% and performance superior to the best commercial cathode catalysts.** ***– December 2004***
- **Significantly minimize or eliminate ruthenium crossover in operating DMFC.** ***– March 2005***
- **Complete preliminary study and demonstrate performance of methanol-tolerant cathode catalyst.** ***– May 2005***
- **Complete comparative performance study of regular and novel Nafion®-based MEAs.** ***– September 2005***

Electrocatalysis Research

Pt-Co Binary Cathode Catalysts: Particle Size & Performance Evaluation





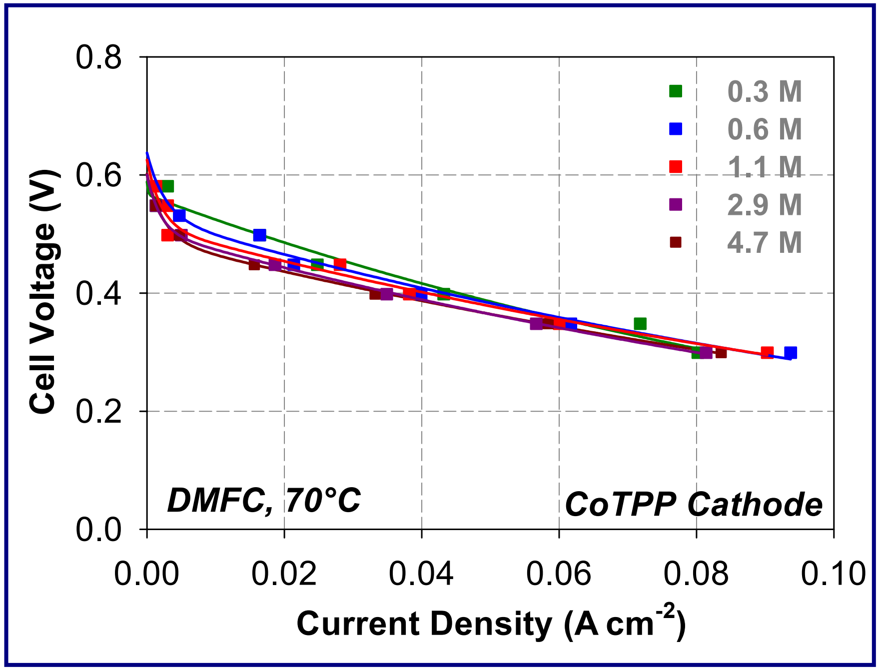


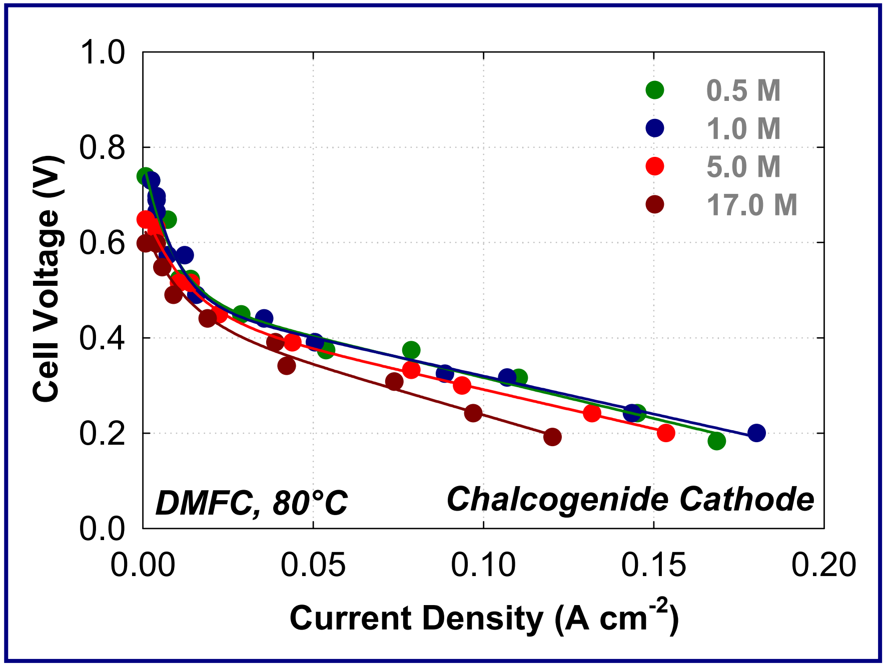
- ***Uniquely high metal loading for a Pt-Co/C catalyst (60 wt%); low particle size (6.2 nm)***
- ***HIGHLIGHT: Very small average particle size of unsupported Pt-Co catalyst – 2.7 nm***
- ***Pt-Co matches Pt-black performance at high cell voltage but catalyst utilization needs improvement***

55% particle size reduction relative to Pt-black exceeds "particle-size" 2005 Milestone (40%)

Electrocatalysis Research

*Methanol-Tolerant Cathode Catalysts *)*



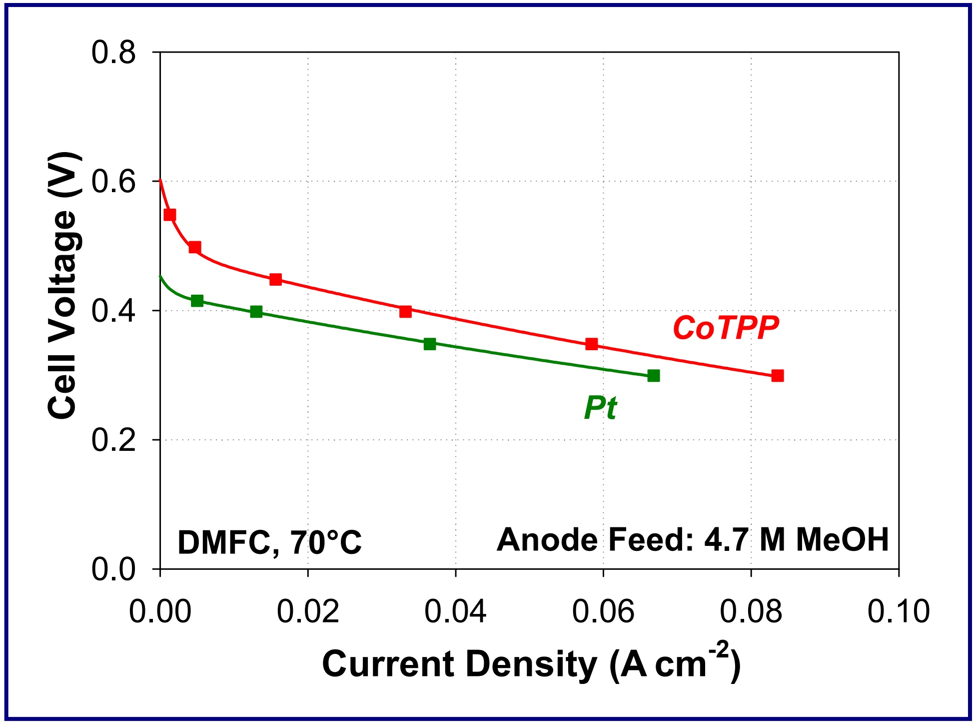


- ***Identified two classes of methanol-tolerant materials: (i) metallorganic porphyrins (CoTPP, CoTMPP, Co/Fe(1:1)CoTPP) and (ii) chalcogenides***
- ***HIGHLIGHT: Very good performance and full methanol tolerance achieved with CoTPP catalyst up to ~ 5.0 M in the anode methanol concentration; chalcogenide catalyst found almost fully tolerant to 17 M methanol (i.e. to the stoichiometric 1:1 methanol-to-water molar ratio at the anode)***

**) Collaboration with the University of New Mexico, University of Illinois, and Université de Poitiers* C

Electrocatalysis Research

Methanol Tolerant Cathode Catalyst vs. Pt Black Catalyst



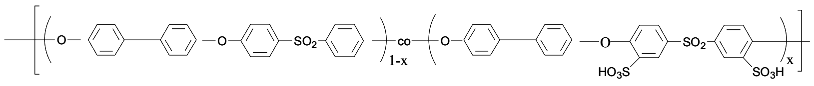
- ***HIGHLIGHT:* *In cells with relatively high MeOH anode concentration, non-precious metal CoTPP catalyst easily outperforms the stat-of-the-art Pt black catalyst*** *(2 mg cm^2 loading used with both cathode catalysts)*

2005 Milestone Accomplished & Exceeded

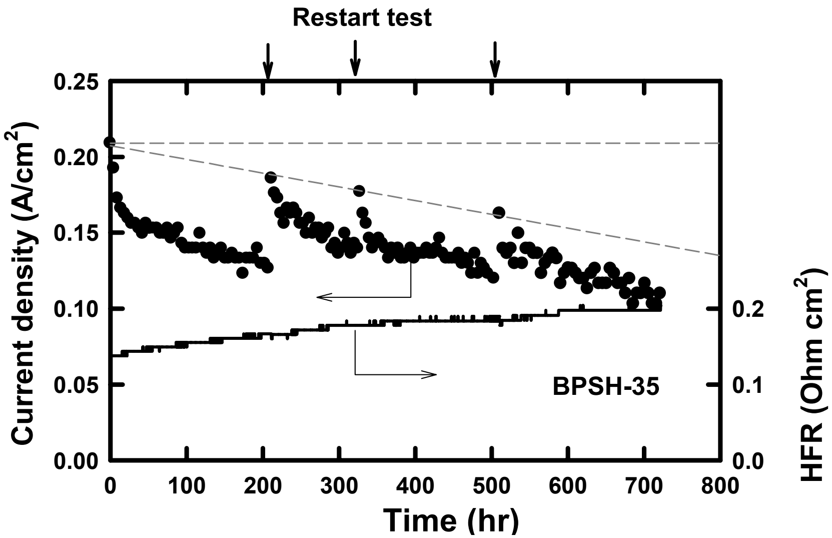
Membrane & MEA Research

New Hydrocarbon-Based Membrane with Better Long-Term Interfacial Stability

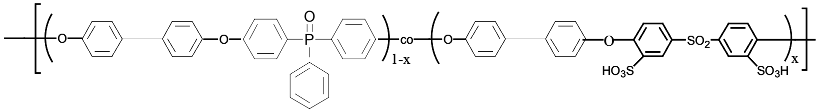
FY 2004 BPSH-35



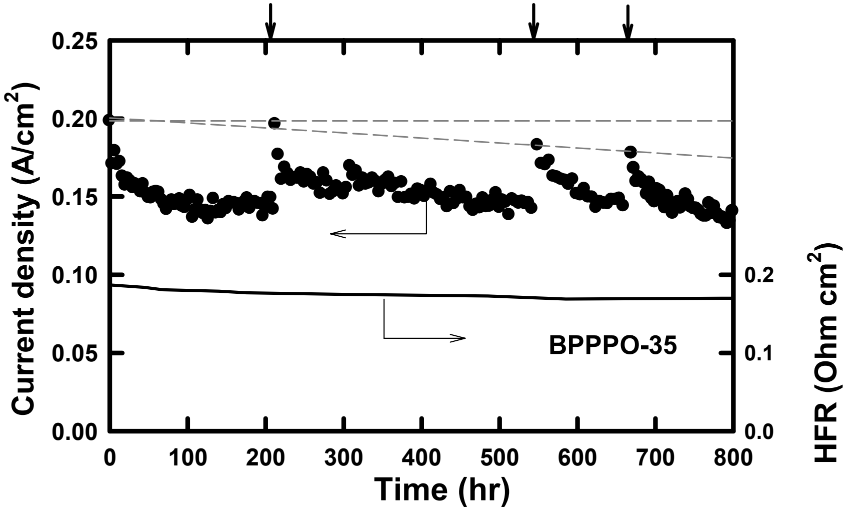
- ***Conductivity:*** ***80 mS/cm***
- ***Methanol permeability:*** ***$1.4\ 10^{-6}\ cm^2/s$***
- ***Water uptake:*** ***51 vol.%***
- ***700 h performance loss:*** ***$68\ mA/cm^2$***



FY 2005 BPPPO-35



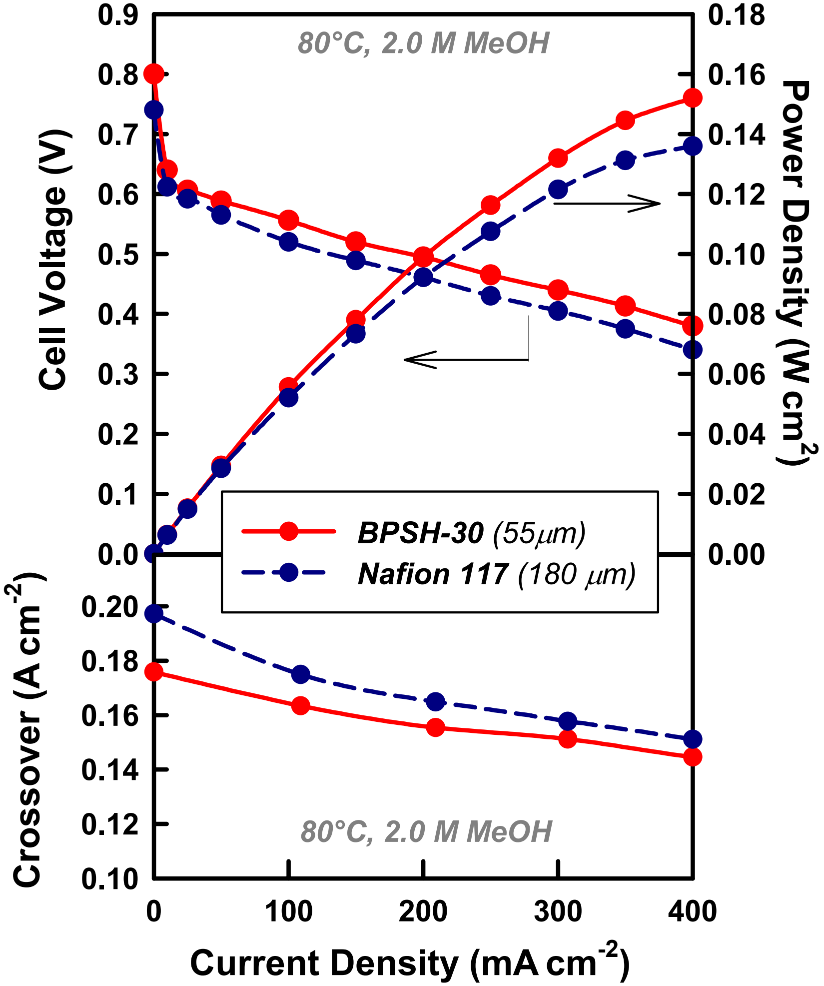
- ***Conductivity:*** ***50 mS/cm***
- ***Methanol permeability:*** ***$0.7\ 10^{-6}\ cm^2/s$***
- ***Water uptake:*** ***35 vol.%***
- ***700 h performance loss:*** ***$21\ mA/cm^2$***



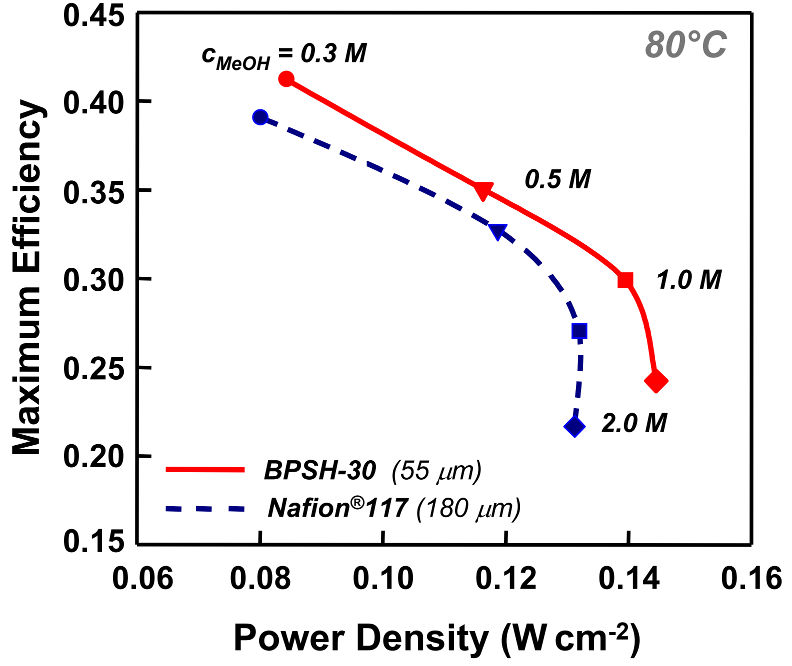
HIGHLIGHT: Major improvement to the durability of alternative membranes

Membrane & MEA Research

Alternative Hydrocarbon-Based Membrane vs. Nafion®117



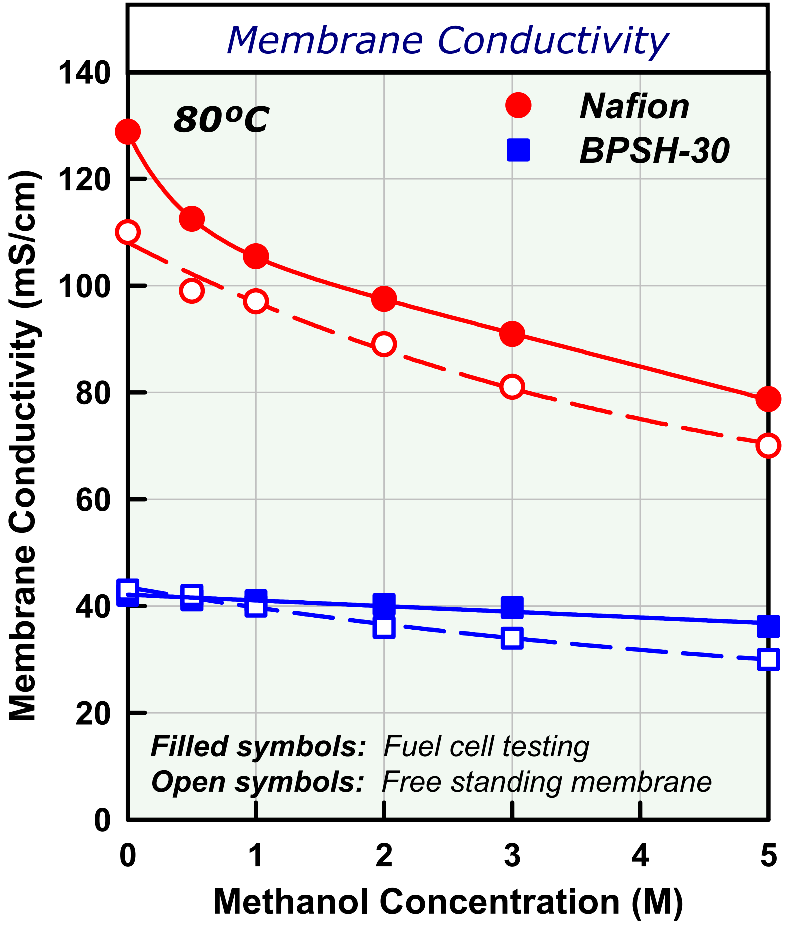
***HIGHLIGHT:* Improved energy conversion efficiency of an optimized BPSH-30 based MEA over a Nafion®-117 based MEA.**



Ⓒ ***LANL-Virginia Tech Collaboration***

Membrane & MEA Research

Impact of Methanol Concentration on MEA Properties



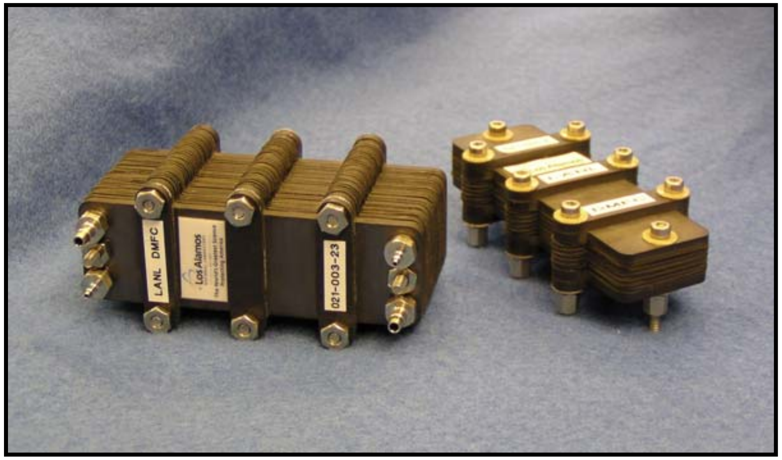
Conductivity and Interfacial Resistance

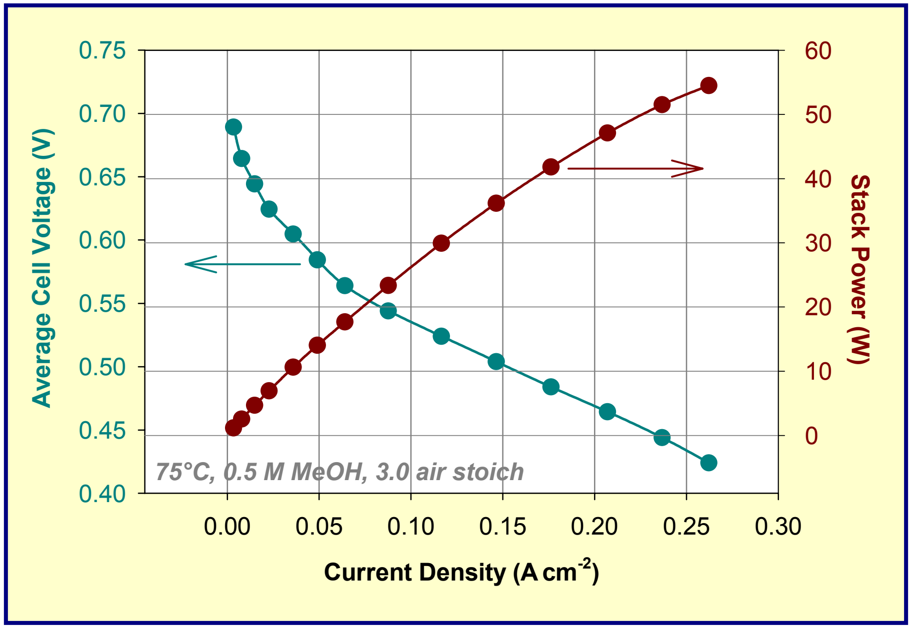
Anode Feed	Conductivity (mS/cm)	Interfacial Resistance (mΩ cm²)
H_2	128	24
0.5 M MeOH	111	29
1.0 M MeOH	105	35
2.0 M MeOH	97	41
5.0 M MeOH	78	57

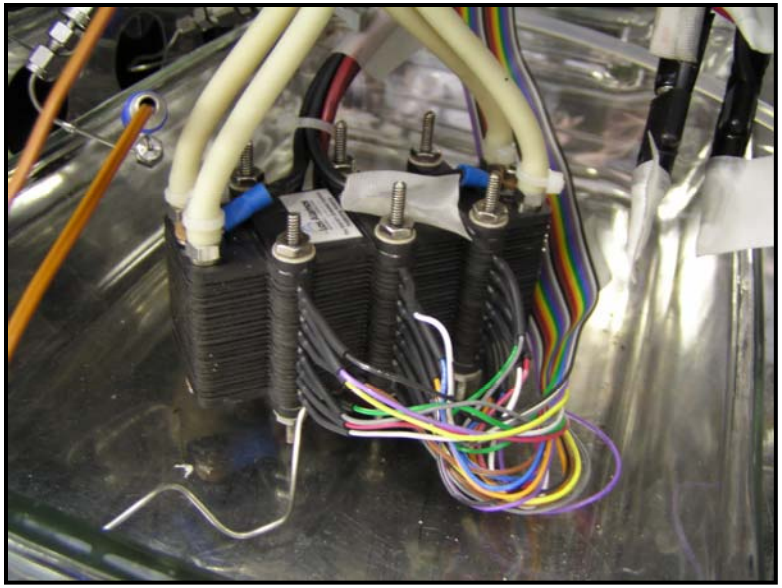
HIGHLIGHT:* *Significant drop in the membrane & MEA conductivity (30%) and increase in membrane-electrode interfacial resistance (50%) observed upon a ten-fold change in methanol concentration, from 0.5 M to 5.0 M.

High Specific-Power Stack for Portable Applications *)

Stack Performance vs. DOE Technical Targets







Number of cells:	***25***
Cell active area	***19.6 cm²***
Nominal power:	***25 W*** *(@ 0.55 V/cell)*
Maximum power:	***85 W***
Total mass:	***250 g***
***Specific power** (6-cell):*	***340 W/kg***
***Specific power** (25-cell):*	***230 W/kg***

*) *High specific-power stack project supported by LANL Tech Maturation Fund & Mesoscopic Devices, Inc.* C

High Specific-Power Stack for Portable Applications

Stack Performance vs. DOE Technical Targets

- ***Maximum specific power of a six-cell stack demonstrated to date: 340 W/kg***
- ***Expected maximum specific power of 25-cell stack: 400 W/kg***

HIGHLIGHT

Current LANL stack performance practically assures meeting DOE's 2006 & 2010 specific power and power density targets for consumer electronics systems

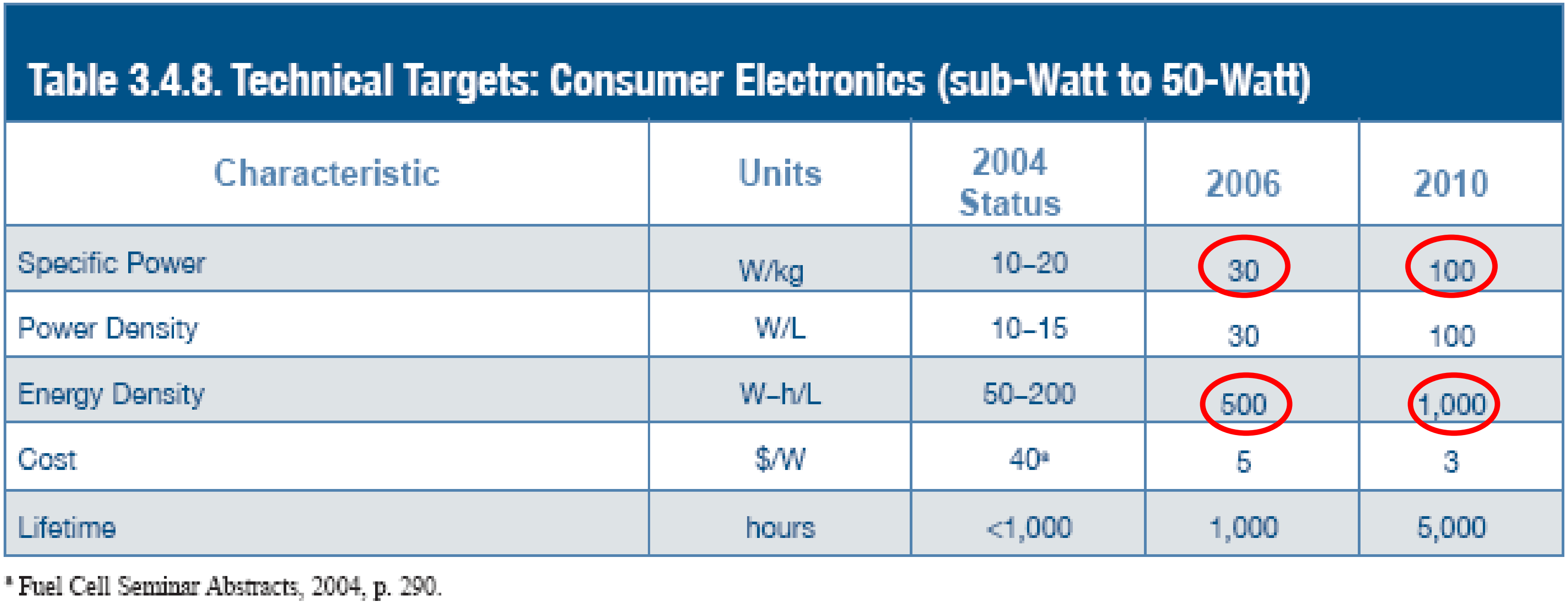


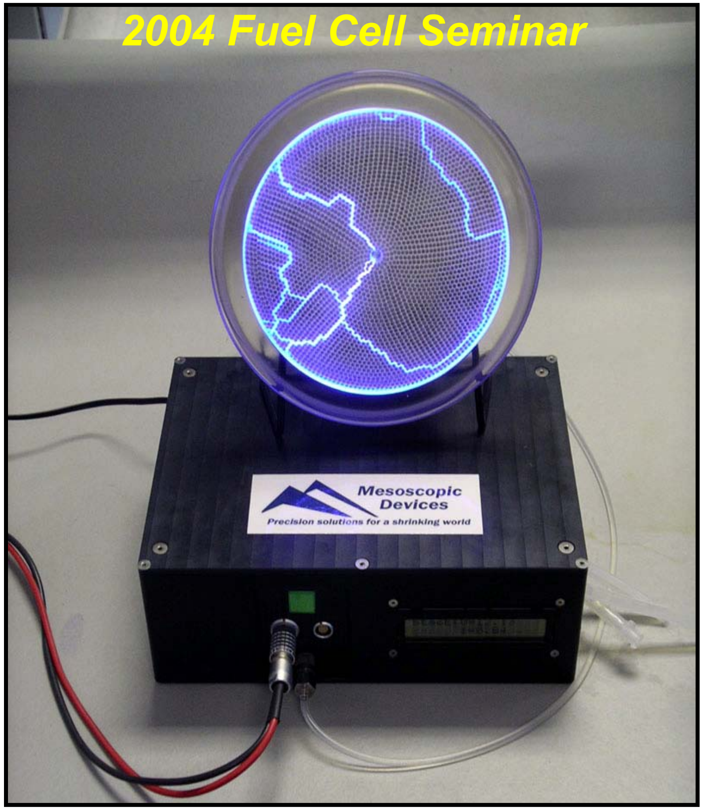
Table 3.4.8. Technical Targets: Consumer Electronics (sub-Watt to 50-Watt)

Characteristic	Units	2004 Status	2006	2010
Specific Power	W/kg	10–20	30	100
Power Density	W/L	10–15	30	100
Energy Density	W-h/L	50–200	500	1,000
Cost	$/W	40[a]	5	3
Lifetime	hours	<1,000	1,000	5,000

[a] Fuel Cell Seminar Abstracts, 2004, p. 290.

Collaboration with Mesoscopic Devices, Inc. (ⓒ)

High Power-Density Active DMFC Stack & Methanol Concentration Sensor

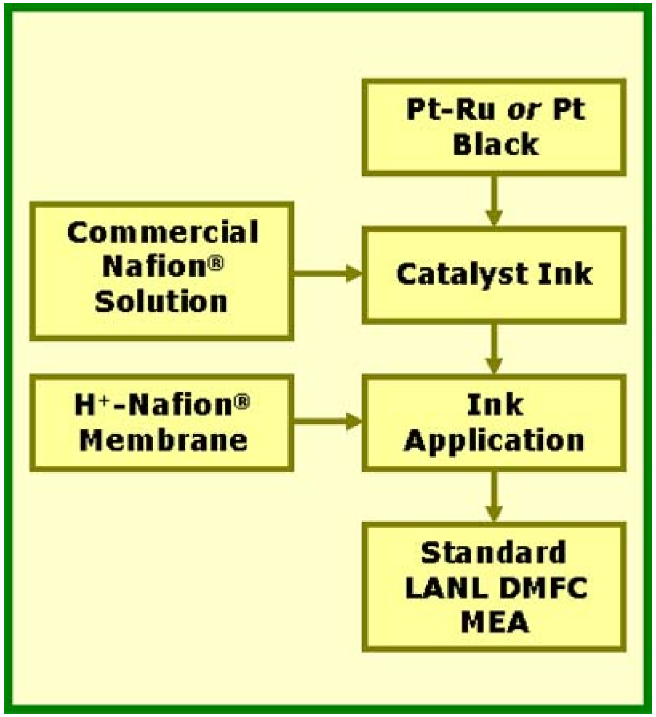


High-power density DMFC stack developed at LANL, integrated by Mesoscopic Devices into 20 W prototype portable power system for the US Government

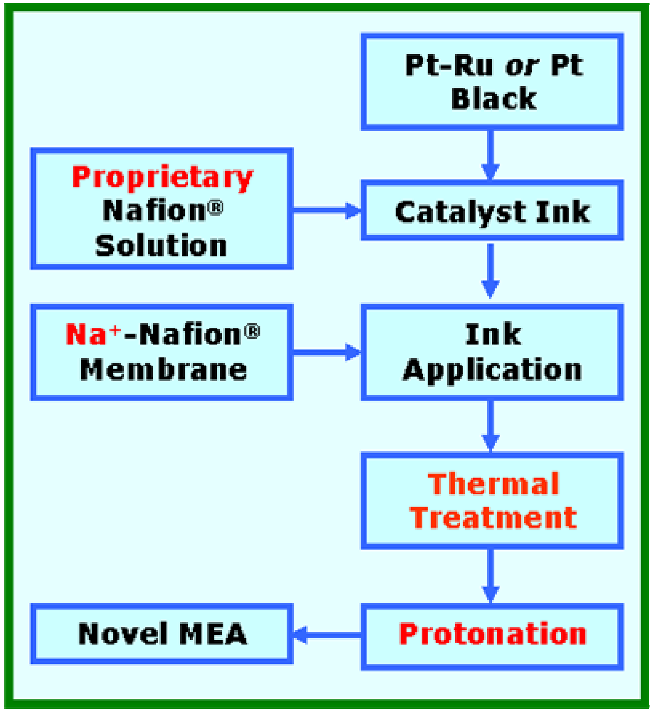
Durability Research

New Approach to Making Nafion®-Based MEAs

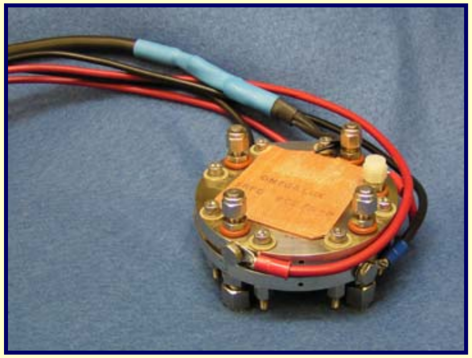
Standard Approach



Novel Approach

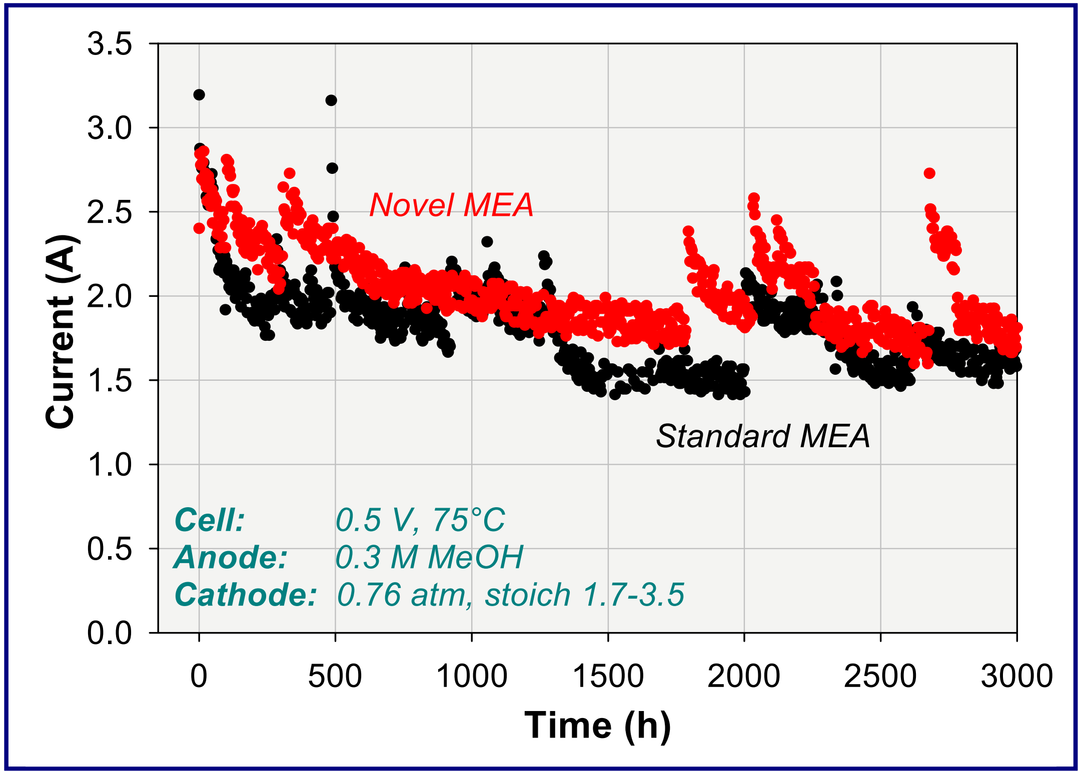


- **Membrane:** *Nafion® 117*
- **Anode:** *Pt-Ru black, 0.3 M MeOH*
- **Cathode:** *Pt black, 0.76 atm (ambient), 1.7 - 3.5 air stoich*
- ***Cell Voltage:*** *0.50 V*
- **Temperature:** *75°C*
- **Cell:** *22 cm²*
- **Life Test: 3000 hours**



Durability Research

Novel MEA – Much Improved Stability



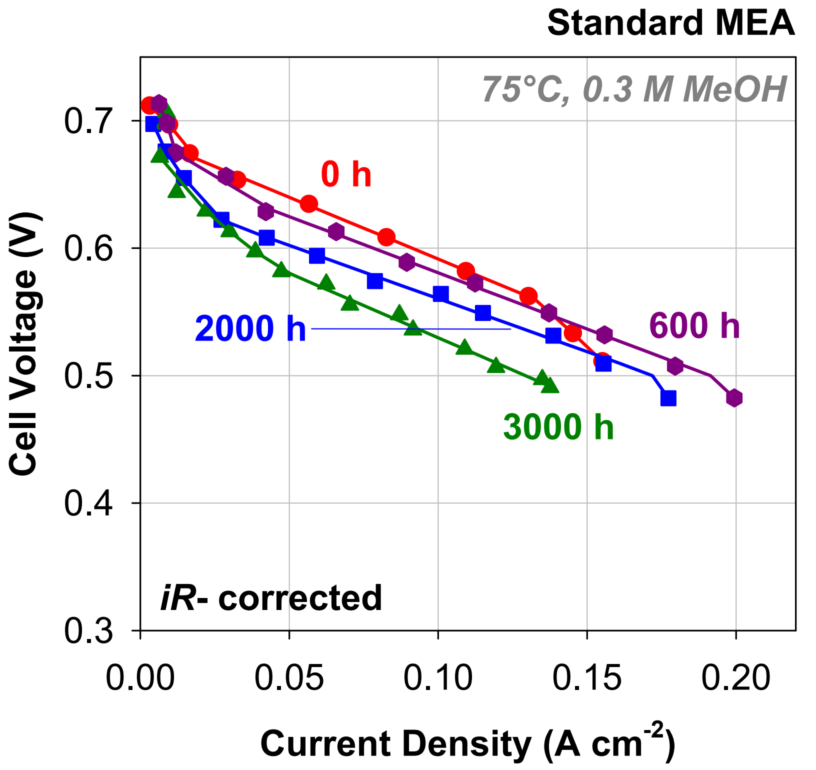
HIGHLIGHT:* *Major improvement to long-term stability achieved with the novel MEA; non-recoverable performance loss over 3000 hours:

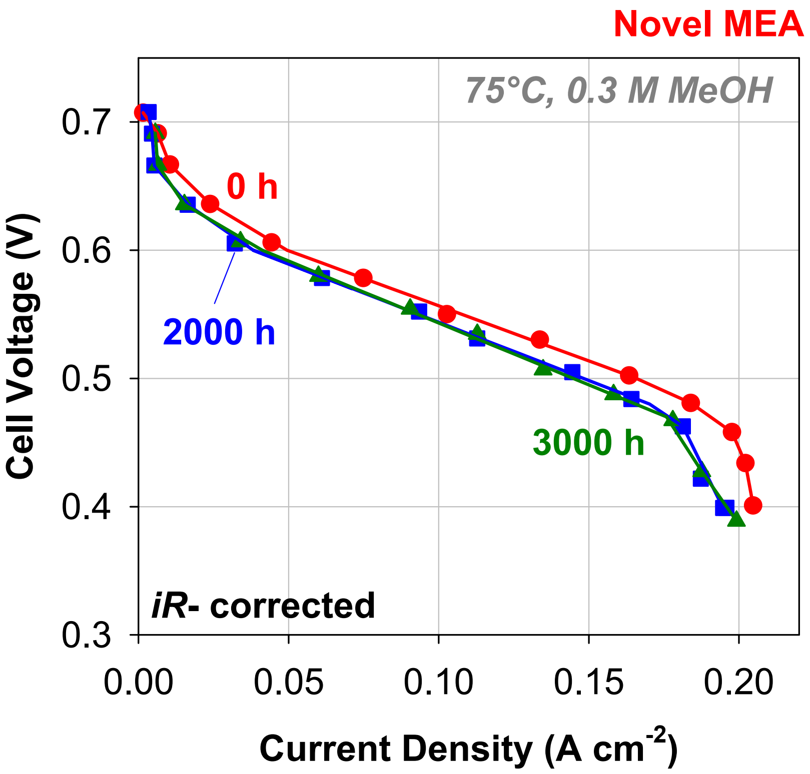
Standard MEA – ~ 40%
Novel MEA – ~ 15%

2005 Milestone Accomplished

Durability Research

Performance Loss vs. Time





Overall performance loss:	~ ***60 mV*** *(standard MEA)*
(0 h → 3000 h)	~ ***10 mV*** *(novel MEA)*

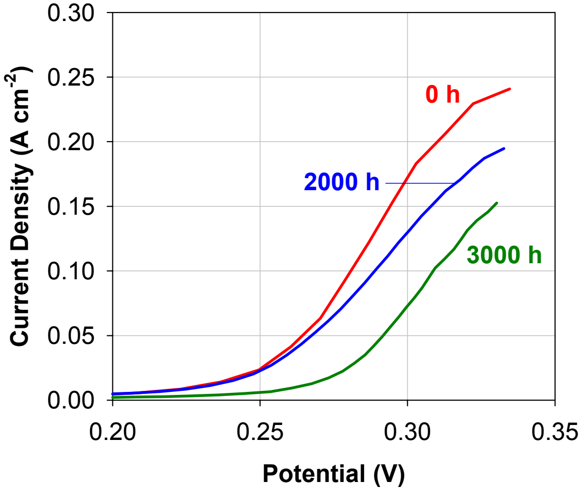
Durability Research

Breakdown of Cell Performance Loss

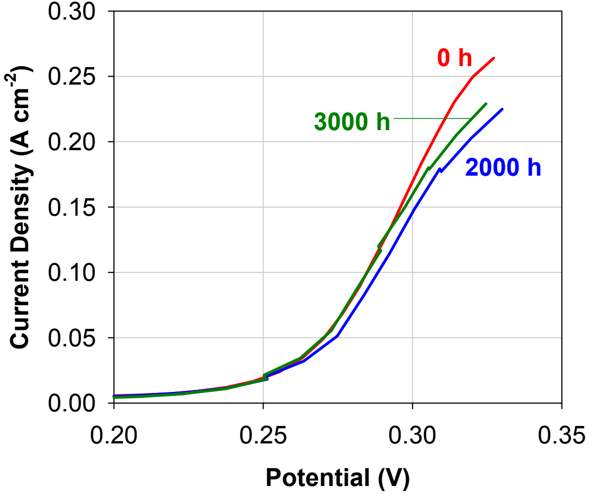
ANODE →

[Anode Polarization]

Standard MEA



Novel MEA



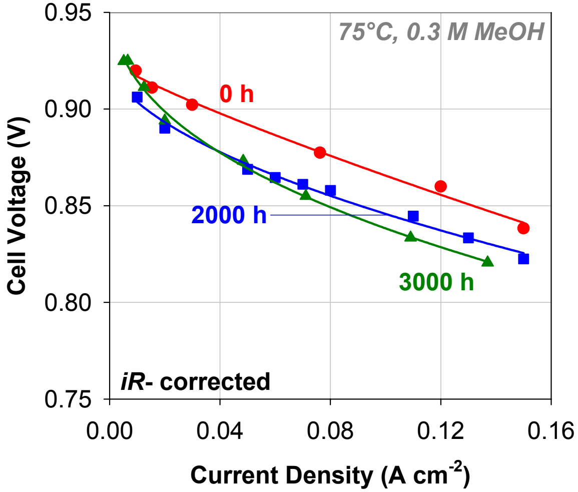
25 - 30 mV

0 - 5 mV

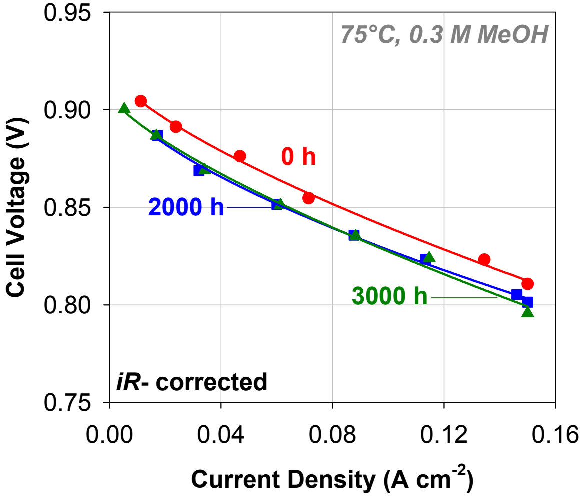
CATHODE →

[(V-i)$_{DMFC}$ + AP]

Standard MEA



Novel MEA

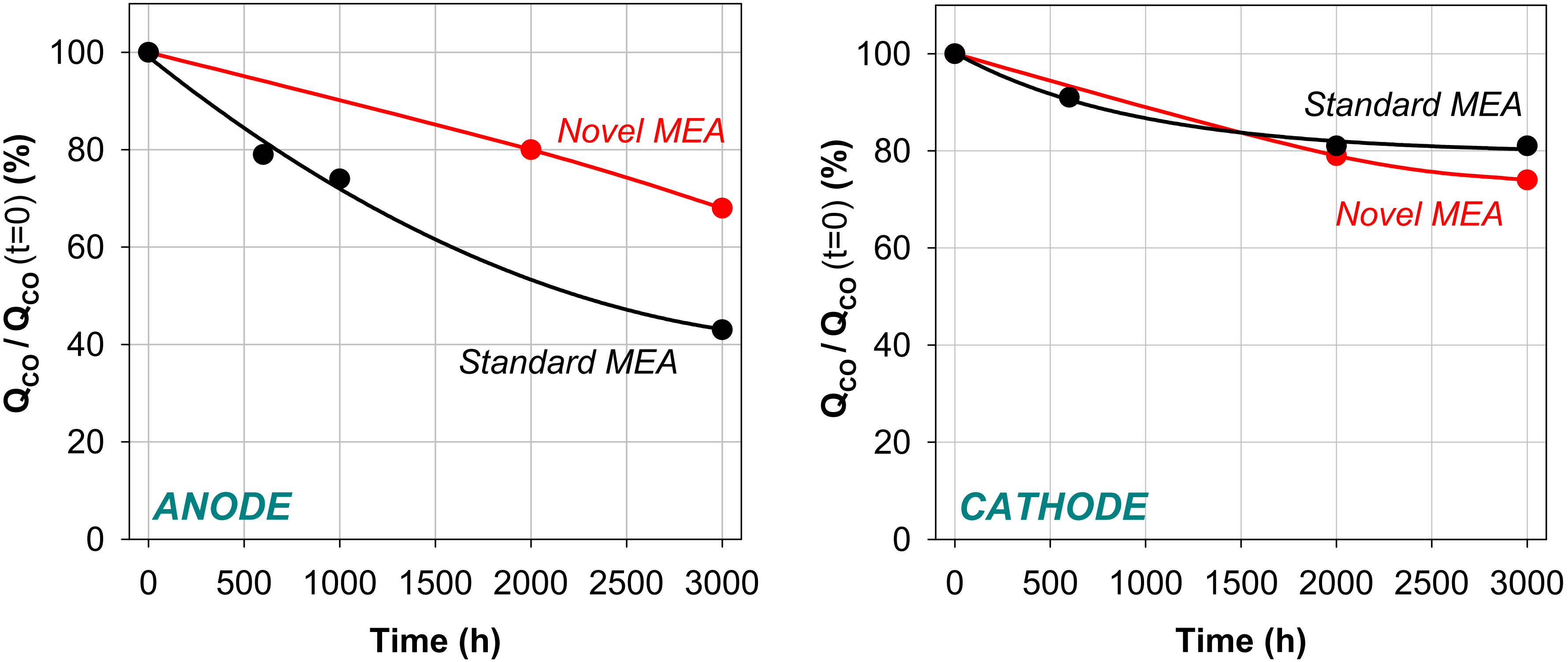


~ 25 mV

~ 10 mV

Durability Research

Anode & Cathode Surface Area Loss (from CO Stripping)



Anode surface area loss:

~ *58%* *– standard MEA*

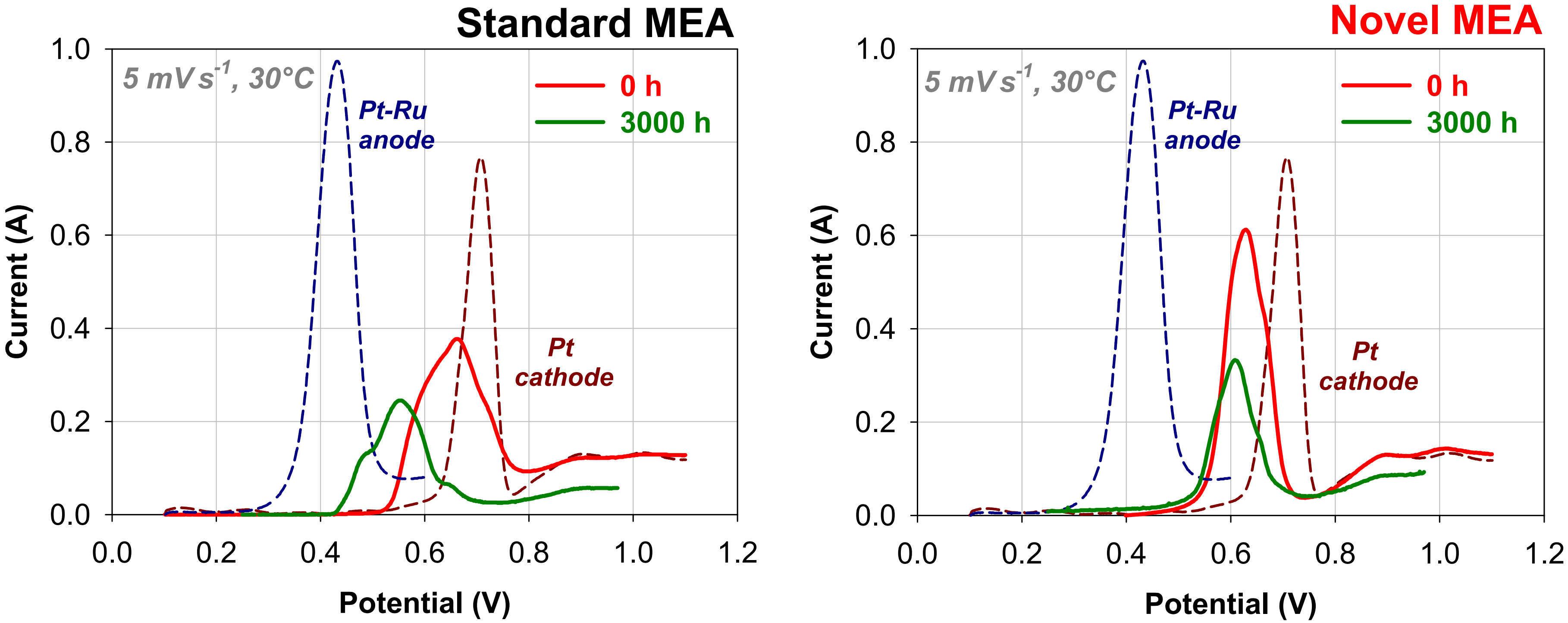
~ *32%* *– novel MEA*

Cathode surface area loss:

20-25% *– both MEAs*

Durability Research

Impact of Ruthenium Crossover



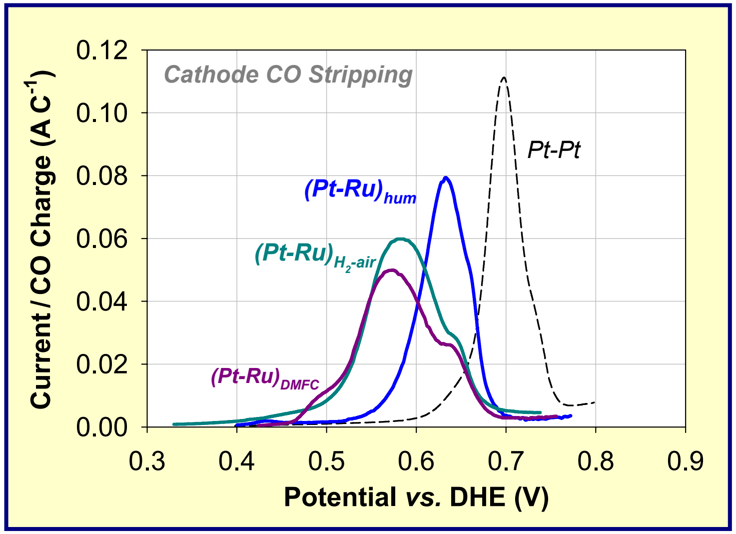
Shift in CO stripping: **~ 100 mV** *(standard MEA)*

(0 h → 3000 h) **~ 20 mV** *(novel MEA)*

HIGHLIGHT: ***Novel MEA fabrication approach leads to significant improvement in the anode stability and diminished ruthenium crossover – the most likely cause of standard cathode performance degradation***

Ruthenium Crossover

Initial Impact on Pt-Black Cathode & DMFC Performance



(Pt-Ru)° (as prepared)

↓ *3-hour air-free cell humidification*

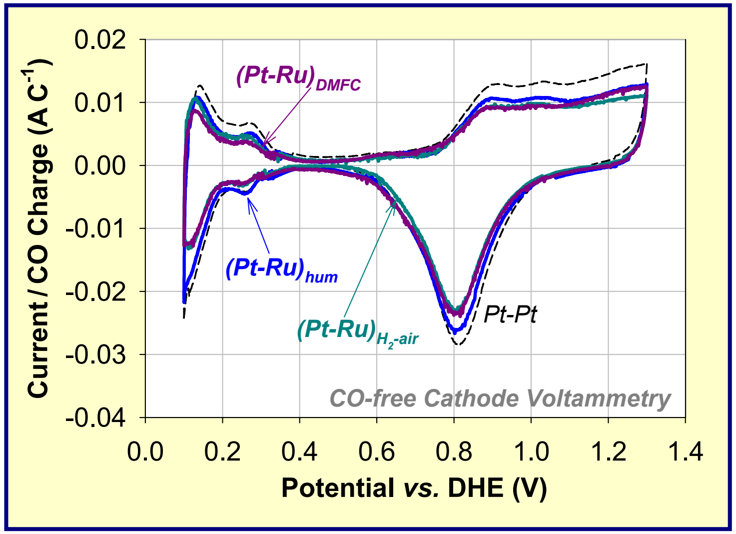
(Pt-Ru)$_{hum}$

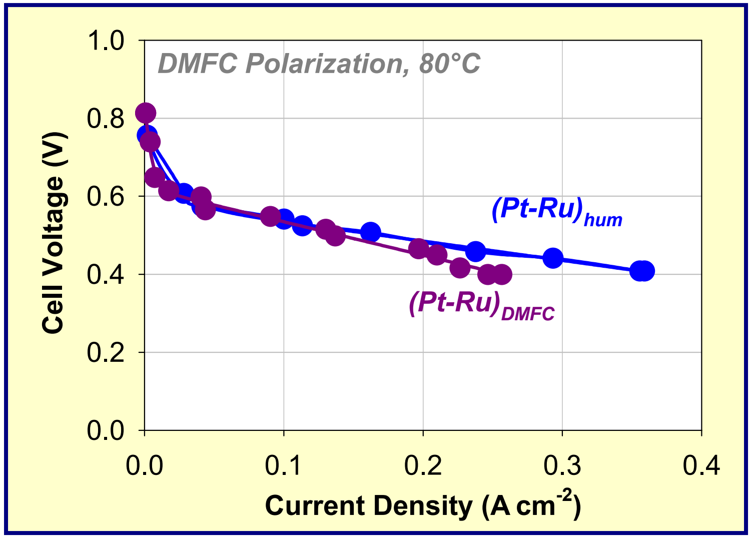
↓ *3-hour H_2-air PEM: 0.5 V, 80°C*

(Pt-Ru)$_{H2\text{-air}}$

↓ *24-hour DMFC: 0.5 V, 80°C*

(Pt-Ru)$_{DMFC}$

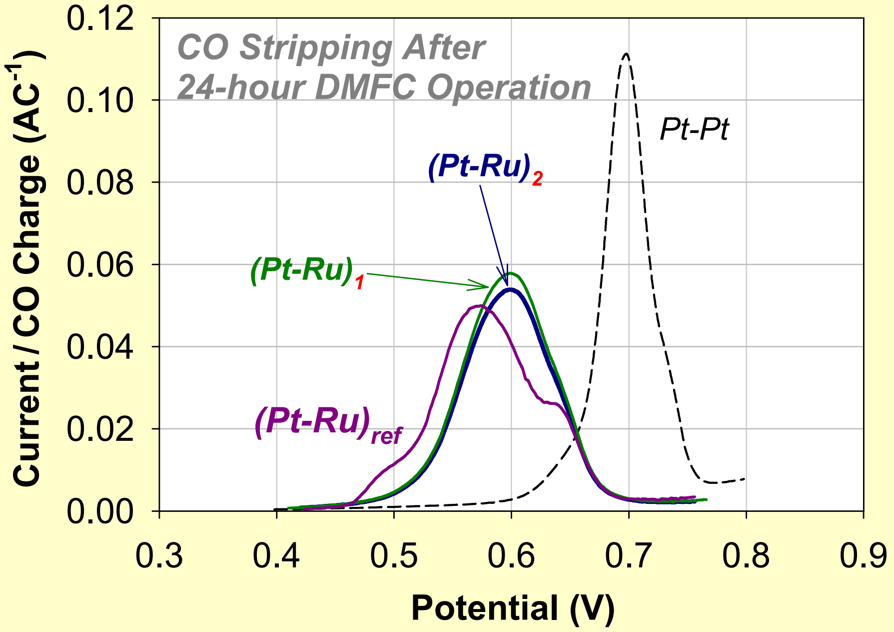




Significant 20 mV cathode loss after short 24-hour DMFC test!

Ruthenium Crossover

Pt Black Cathode Response Acid Pretreatment



Pt-Pt – *cell with Pt anode*

$(\text{Pt-Ru})_{ref}$ – *untreated anode catalyst*

$(\text{Pt-Ru})_1$ – *acid treated MEA*

$(\text{Pt-Ru})_2$ – *acid treated anode catalyst*

Acid Pretreatment

- ***Two-hour boil in 2.0 M H_2SO_4***
- ***$(\text{Pt-Ru})_1$*** – *membrane + anode catalyst (no cathode catalyst applied)*
- ***$(\text{Pt-Ru})_2$*** – *PtRu anode catalyst*

***HIGHLIGHT:* Developed four methods for minimizing Ru crossover; negative shift in CO stripping peak potential reduced by 30–130 mV.**

(1) Acid pretreatment of anode catalyst
(2) Acid pretreatment of MEA (before cathode catalyst applied)
(3) High-temperature MEA post-treatment via "decal transfer"
(4) Novel high-temperature Nafion®-based MEAs (cf. Durability)

2005 Milestone Accomplished

Technical Accomplishments & Progress *(Highlights)*

Electrocatalysis:

- ✓ **Fabricated and characterized Pt-Co black catalyst with average particle size reduced by 55% relative to the best Pt blacks for DMFC cathodes. – *Exceeded particle-size goal of Milestone #1*** *(current focus on catalyst performance)*
- ✓ **Developed MEAs with two types of cathode catalysts tolerant to 5-17 M MeOH; demonstrated high ORR activity and respectable performance durability of both catalyst types. – *Milestone #2 achieved & exceeded***

Membrane & MEA:

- ✓ **Demonstrated BPSH-based MEA with better conversion efficiency than Nafion®**
- ✓ **Developed new hydrocarbon-based BPPPO MEA with remarkable long-term stability (10% performance loss over 800 hours)**

Durability Research:

- ✓ **Developed four methods for significantly lowering Ru crossover. – *Milestone #3 achieved*** *(current focus on eliminating crossover altogether)*
- ✓ **Introduced novel Nafion®-based MEA with 15% performance loss over 3000-hour operation (*vs.* 40% loss with the standard MEA); finished detailed comparative performance loss study of both MEAs. – *Milestone #4 achieved & exceeded***

High Specific-Power Stack for Portable Applications:

- ✓ **Stack designed, built & integrated into a practical system; licensed to industry**

Selected Reviewers' Comments

- ✓ ***"Very solid progress on many fronts, including a serious attempt to move towards practical deployment. Balanced, fundamental work." "Excellent technical progress." "Can't argue with success."***

- ✓ ***"Focus on fundamentals. Develop stacks for analytical purpose only."*** *Significant stack effort in the DOE program was abandoned over a year ago. Since then, we focused even more on key fundamental issues for the DMFC technology, such as performance durability, alternative membranes & MEAs, methanol-tolerant cathodes. Limited effort has continued in high specific-power DMFC stack (non-DOE-funding). Small stacks have been used for more efficient acquisition of life test data, in durability studies in particular.*

- ✓ ***"Need to find strong industrial partners who could capitalize on impressive work at National Lab level"*** *In FY 2005, we have intensified efforts to establish collaboration, including IP transfer, with major players in the portable power industry. Negotiations are pending. In FY 2004 & 2005 LANL awarded three government use licenses in DMFC technology.*

- ✓ ***"Portable applications are key." "Needs more funding." "Ensure that DOE sustains a program that focuses also on portable applications."***

Research Plans

Remainder of FY 2005

- **Improve oxygen reduction activity of highly-dispersed Pt-Co blacks.**
- **Further minimize ruthenium crossover in DMFCs by combining various methods of Ru stabilization identified to date.**

FY 2006 Objectives *(crucial to the success of DMFCs for portable power)*

- **Develop alternative membrane based MEAs enabling the use of methanol feed concentration as high as 5.0 M without a loss in cell performance and performance durability.**
- **Through fundamental mechanistic research, eliminate ruthenium crossover from direct methanol fuel cells.**
- **Improve performance of DMFC anode and cathode by developing better "secondary" catalyst structures.**
- **Explore mixed-conducting intercalated nanocomposites as DMFC cathode materials with potentially high catalytic activity, full methanol tolerance and good stability.**

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Los Alamos National Laboratory
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LANL Program Manager:	**Ken Stroh**	*FC - 34*

Publications

1. “Researchers redefine the DMFC roadmap,” P. Piela and P. Zelenay, *The Fuel Cell Review*, 1, 17-23 (2004).

2. “Ruthenium Crossover in the Direct Methanol Fuel Cell with a Pt-Ru Anode,” P. Piela, C. Eickes, E. Brosha, F. Garzon and P. Zelenay, *J. Electrochem. Soc.*, 151, A2053-A2059 (2004).

3. Direct Methanol Fuel Cell Performance of Disulfonated Poly(Arylene Ether Benzonitrile) Copolymers, Yu Seung Kim, Mike Sumner, William Harrison, James E. McGrath, Bryan Pivovar, *J. Electrochem. Soc*. 151, 12, A2150 (2004).

4. “Sulfonated Poly(Arylene Ether Sulfone) Copolymer Proton Exchange Membranes: Composition and Morphology Effects on the Methanol Permeability,” Yu Seung Kim, Michael Hickner, Limin Dong, Bryan Pivovar and James E. McGrath, *Journal of Membrane Science*, 243, 317-326 (2004).

5. “New Proton Conducting Sulfonated Poly(Arylene ether) Copolymers Containing Aromatic Nitriles,” M.J. Sumner, W.L. Harrison, R.M. Weyers, Y.S. Kim, J.E. McGrath, J.S. Riffle, A. Brink, M.H. Brink, *Journal of Membrane Science*, 239, *2*, 199-211 (2004).

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7. “Alternative Polymer Systems for Proton Exchange Membranes (PEMs),” Michael Hickner, Hossein Ghassemi, Yu Seung Kim, Brian Einsla, and James E. McGrath, *Chemical Reviews*, 104, 4587-4612 (2004).

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1. 206th Meeting of the Electrochemical Society, Honolulu, Hawaii, October 3 – 8, 2004. Title: “Novel Process for Improved Long-Term Stability of DMFC Membrane-Electrode Assemblies,” C. Hamon, G. Purdy, Y.S. Kim, B. Pivovar and P. Zelenay*.

2. 206th Meeting of the Electrochemical Society, Honolulu, Hawaii, October 3 – 8, 2004. Title: “Direct Measurement of *iR*-Free Individual-Electrode Overpotentials in PEFC,” P. Piela, T. Springer, M. Wilson, J. Davey and P. Zelenay*.

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4. The Importance of Interfaces in Membrane Optimization for DMFCs, Yu Seung Kim, J. E. McGrath, B. S. Pivovar, 206th Meeting of the Electrochemical Society, Oct. 3-8 (2004). (paper no. 1471).

5. The Effect of Methanol Concentration on Membrane Conductivity and Interfacial Resistance in DMFCs, Yu Seung Kim and Bryan S. Pivovar, 206th Meeting of the Electrochemical Society, Oct. 3-8 (2004). (paper no. 1472).

6. Zirconium Phenylphosnate/Poly(arylene ether sulfone) Composite Membranes for Proton Exchange Membrane Fuel Cells, M. Hill, B. Einsla, Y. S. Kim, J. McGrath, 206th Meeting of the Electrochemical Society, Oct. 3-8 (2004). (Paper no. 1909).

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8. Sulfonated Poly(arylene ether sulfone) as Candidates for Proton Exchange Membranes: Influence of Substitution Position on Membrane Properties, J. E. McGrath, W. L. Harrison, B. Einsla, N. Arnett, Y. S. Kim, B. Pivovar, 206th Meeting of the Electrochemical Society, Oct. 3-8 (2004). (Paper no. 1973).

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9. Influence of Membrane-Electrode Interface on Long-Term Performance of Direct Methanol Fuel Cells, Yu Seung Kim, Bryan Pivovar, Fuel Cell Seminar, San Antonio, TX, Nov.1-5, (2004).

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11. 2004 International Taipei Power Forum & Exhibition, Taipei, Taiwan, December 1 – 3, 2004. Title: "DMFC Research and Design Trends in Los Alamos National Laboratory and Other US Fuel Cell Centers," P. Zelenay* (invited keynote lecture).

12. Industrial Technology Research Institute, Hsinchu, Taiwan, December 6, 2004. Title: "Selected Aspects of Direct Methanol Fuel Cell Research at LANL," P. Zelenay* (invited lecture).

13. Industrial Technology Research Institute, Hsinchu, Taiwan, December 7, 2004. Title: "Direct Measurement of *iR*-free Individual Electrode Overpotentials in PEFC," P. Zelenay* (invited lecture).

14. Samsung Advanced Institute of Technology, Suwon, Korea, December 9, 2004. Title: "Direct Methanol Fuel Cell Research at Los Alamos National Laboratory," P. Zelenay* (invited lecture).

15. Catalysis Club of Chicago, Chicago, Illinois, January 10, 2005. Title: "Electrocatalysis: The Key to Polymer Electrolyte Fuel Cell Success," P. Zelenay* (invited lecture).

16. Tactical Power Sources Summit, Arlington, Virginia, February 1 – 2, 2005. Title: "Research and Design Trends in Direct Methanol Fuel Cells for Portable Power," J. Ramsey* and P. Zelenay (invited keynote lecture).

Conference Presentations III

17. Direct Methanol Fuel Cell Performance of Partially Fluorinated Disulfonated Poly(arylene ether sulfone) Random (Statistical) Copolymers, M. Hill, B. R. Einsla, Y.S. Kim, W. Harrison, B. S. Pivovar, and J.E. McGrath, Advances in Materials for Proton Exchange Membrane Fuel Cell Systems 2005, Asilomar Conference Grounds, Pacific Grove, CA, Feb. 20-23 (2005).

18. Disulfonated Poly(Arylene Ether Benzonitrile) Copolymers (PAEB) for Proton Exchange Membrane Fuel Cells (PEMFC), M. Sankir, Y. S. Kim, J. E. McGrath, Advances in Materials for Proton Exchange Membrane Fuel Cell Systems 2005, Asilomar Conference Grounds, Pacific Grove, CA, Feb. 20-23 (2005).

19. Optimizing Alternative Membranes in DMFCs – Actual Performance Improvements, B. S. Pivovar, Y. S. Kim, Advances in Materials for Proton Exchange Membrane Fuel Cell Systems 2005, Asilomar Conference Grounds, Pacific Grove, CA, Feb. 20-23 (2005).

20. Small Fuel Cells 2005, Washington, DC, April 27 – 29, 2005. Title: “Advancements in DMFC MEAs and Stacks for Portable Power Applications,” P. Zelenay* and J. Ramsey (invited lecture).

Hydrogen Safety

The most significant hydrogen hazard associated with this project is:

Leak in the hydrogen supply resulting in accumulation of the gas in the room, which could then lead to explosion upon ignition.

Hydrogen Safety

Our approach to dealing with this hazard is as follows:

- ***Hydrogen sensors, interlocked with the hydrogen gas supply, have been installed in the laboratories with hydrogen supply from gas cylinders or from a hydrogen generator.***
- ***Hydrogen sensors have been installed at just below the ceiling where gas accumulation is most severe; also, two sensors are installed in every room for redundancy; the alarm is set off at 10% of Lower Flammability Limit (LFL).***
- ***In laboratories that use bottled hydrogen, only a single cylinder is used at any given time; the cylinder size is limited to ensure that the LFL is not exceeded even upon complete release of a full cylinder.***
- ***All work has been reviewed and approved through Los Alamos National Laboratory's safety programs:***
  - ***Hazard Control Plan (HCP) - hazard based safety review***
  - ***Integrated Work Document (IWD) - task based safety review***
  - ***Integrated Safety Management (ISM)***