VII.A.2 Advanced MEAs for Enhanced Operating Conditions

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Objective

- Develop high performance, lower cost membrane electrode assemblies (MEAs) that:
 - meet demanding system operating conditions of higher temperature and little or no humidification.
 - use less precious metal than current state-of-the-art constructions.
 - could be made by processes amenable to high volume manufacturing.

Technical Barriers

This project addresses the following technical barriers from the Fuel Cells section of the Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan:

- A. Durability
- B. Cost
- C. Electrode Performance
- D. Thermal, Air and Water Management

Technical Targets

Characteristic	Units	2010 Target	MEA, Component Status
PEM Conductivity	S/cm	0.10	~ 0. 06 @ 100°C w/80°C dew pt
PEM H ₂ X-over	mA/cm ²	5	< 2 at 70°C, fully humidified
PEM durability	hours	2000 > 80°C	4300 under 90/70/70°C load cycling
MEA total PGM Electrocatalyst	g PGM / kW rated	0.33	0.30 @ V< 0.72 volts, 270 kPa, 80°C, Cathode Stoich. = 2.5, 60/60% RH
MEA total PGM catalyst loading	mg PGM/cm ²	0.30	0.21 total anode and cathode
MEA Performance at peak power	mW/cm ²	1280	980 , w/ 0.21 mg PGM/cm ² -MEA @ 270 kPa, 80°C, Cat. Stoich. = 2.5
MEA performance at ¼ power (0.8 V)	mA/cm ²	400	160 –200 , with 0.21 mg/cm ² -MEA @ 270 kPa, 80°C, Cat. Stoich. = 2.5

Approach

- Develop advanced cathode catalysts that use less precious metal, are more durable and can be made by high volume compatible processes, building on the knowledge gained in the preceding 3M/DOE project.
- Develop new proton exchange membranes (PEMs) based on 3M's perfluorinated sulfonic acid (PFSA) ionomer to obtain enhanced oxidative stability under hotter, drier operating conditions.
- Optimize matching MEA components using the above catalysts and membrane, for use in the temperature range of 85-120°C.
- Scale up and optimize MEA component fabrication processes amenable to high volume, high quality, low cost production for selected components from the above tasks.
- Characterize selected MEAs in large area, short stacks; evaluate compressors for customized air management.
- Explore multiple approaches and develop high temperature non-aqueous electrolyte membrane technology for an operating range of 120-150°C and nearly dry conditions.

Accomplishments

- Demonstrated greater than 15x increase in lifetime under 90/70/70°C load-cycling accelerated tests with new 3M PFSA membrane compared to standard PFSA membrane.
- Determined that the 3M PFSA membrane has higher conductivity with lower water-per-sulfonate group than standard PFSA.
- Demonstrated the new 3M PFSA membrane maintains a conductivity of 25-30 mS/cm at 120°C and 80°C dewpoint.
- Coated the new 3M PEM at pilot scale level (over 1000 ft).
- Demonstrated the 3M NanoStructured Thin Film (NSTF) catalyst achieved a mass specific power density of 0.22 g-Pt/kW at ambient pressure with only 0.12 mg-Pt/cm² total per MEA, using roll-good fabricated catalysts in 50-cm² cell.
- Determined NSTF catalyst electrodes (0.1 mg-Pt/cm²) exhibit linear relation of mass transport overpotential (MTO) with electrode thickness and have < 50 mV of MTO at 2 A/cm².
- Demonstrated 3M MEAs with 3M NSTF catalysts and 3M PEM having over 1000-hour lifetime at 120° C and subsaturated H₂/air input gases.

- Demonstrated that 3M NSTF-ternary catalysts produce 75x less F⁻ than Pt/carbon dispersed catalysts at 120°C with same PEM and gas diffusion layer (GDL), giving a 10x gain in lifetime to membrane failure.
- Demonstrated that 3M NSTF catalysts are ~ 80x more resistant to loss of electrochemical surface area (ECSA) via Pt dissolution and/or support oxidation under cyclic voltammogram (CV) cycling between 0.6 - 1.2 V, compared to standard Pt/carbon dispersed catalysts.
- Demonstrated the 3M NSTF-based MEAs can operate without loss of performance with totally dry input gases at 80°C and 200 kPa.
- Demonstrated that fuel cell performance of a high temperature membrane under H₂/air at 110°C was better than PBI/H₃PO₄ with a catalyst loading of only 0.4/0.4 mg/cm² Pt/Pt, implying the 3M acid is more compatible for oxygen reduction than phosphoric acid.
- Demonstrated that pulse field gradient spin echo-diffusion measurements show lower activation barriers for H⁺ transport of new heteropolyacids and higher maximum temperatures.
- Designed and fabricated two air compressors for testing MEA specific turndown.

Future Directions

85 - 120°C MEA:

- Complete pilot-level scale-up of roll-good catalyst coated membrane (CCM) fabrication on new equipment, including statistical analyses to demonstrate uniformity and high volume capability of roll-good CCM made using down-selected NSTF ternary catalysts and 3M PEM components.
- Generate final CCMs for large area (312 cm²) short-stack testing, complete stack testing.

<u>T > 120°C MEA:</u>

• Conclude property evaluation and screening of non-PFSA based membrane material sets, including measurements of conductivity, electrolyte retention, fuel cell performance and membrane morphology characterization.

Introduction

In order for proton exchange membrane fuel cells (PEMFCs) to be commercially viable for automotive and distributed stationary applications, several issues must be addressed, including performance (efficiency and peak power), durability, and cost. The efficiency of a fuel cell is directly related to the cell operating voltage. The higher the current and power density, the smaller the stack size and cost can be. These factors are primarily affected by polarization of the cathode catalyst and stability of catalyst activity and surface area. Therefore, higher performing and more stable cathode catalysts are required to achieve the goals of increased performance with less precious metal catalyst. From a system perspective, operation at higher temperatures reduces thermal management constraints thereby increasing overall efficiency and reducing cost. Designing MEAs for hot, dry, reactant conditions requires development of new

membranes, catalysts and GDLs that are stable and durable under those conditions. The components must be matched to each other and integrated with a flow field design and air management strategy. Membranes that are stable against peroxide induced decomposition and catalysts that produce less peroxide and have stable surface areas under the demanding operating conditions are critical requirements for meeting the DOE long-term technical targets for durability. In addition, the processes for making the MEA components must be consistent with high volume production to meet the quality and cost targets. This project is directed toward development of MEAs that meet demanding system operating conditions of higher temperature and low humidification, use less precious metal than current state-of-the-art constructions, have the required stability and durability, and are made by processes amenable to high volume manufacturing.

<u>Approach</u>

The approach to higher temperature MEAs involves the development of full MEAs for the temperature range of 85-120°C and development of new electrolytes for operation at or above 120°C. For the lower temperature range, PEMs are being developed based on modifications of 3M's novel PFSA type membrane that still utilizes water for proton conduction. The 3M PEM's T_g is 25°C higher than the PFSA standard membrane (Nafion[™]) of equivalent weight, and the modulus is over ten times higher at 120°C, dry. The modifications included incorporation of functionalized additives to facilitate peroxide decomposition for better oxidative stability and enhanced water retention for higher conductivity under low humidification. The upper operating temperature for an MEA made from a PFSA membrane is about 120°C, due to performance and stability considerations. For the higher temperature range, new membrane materials and low-water based proton conduction methods, which utilize 3M perfluorinated acids, various proprietary liquids, and various inorganic additives, are being investigated. Work includes understanding how to incorporate those materials into polymer matrices to form effective membranes.

The approach to the development of advanced cathode catalysts having less precious metal than current state-of-the-art constructions is based on the novel 3M nanostructured thin film (NSTF) catalyst support and deposition system [1]. The approach builds on previous work in which multi-element cathode catalysts were found that significantly outperformed pure Pt-coated NSTF supports. The NSTF process easily generates new compositions and structures via a dry, roll-goods process amenable to high volume manufacturing [1,2,3]. The 3M NSTF catalysts exhibit 5 times higher specific activity [4], significantly reduced fluoride release and much greater surface area stability under highly oxidative conditions than commercially available, carbon-supported, dispersed catalysts. Methods for generating new catalyst constructions include pilotline production of large area catalyst formulations and a combinatorial method for generating and characterizing a wide latitude of binary and ternary compositions and structures (with subcontractor Dalhousie University).

Results

This past year, another 20 new NSTF Pt-based ternary cathode catalyst constructions were fabricated on large-area, roll-good-capable, pilot-line equipment and evaluated by various fuel cell tests for performance and stability, for a total of 113 under this project [5]. The catalysts differ by composition, non-noble metal type, and a structure factor tied to the catalyst deposition process. Based on measurements of specific activity (A/cm²-Pt), performance and durability tests, two PtCxDv ternary compositions were downselected for further refinement. In addition to our standard loading of 0.1 mg-Pt/cm², we also investigated loadings down to 0.05 mg/cm^2 on both anode and cathode. Advances in both performance with lower loadings and catalyst surface area durability at temperatures up to 120°C and high voltages were demonstrated. In the study to determine minimum precious group metal (PGM) loading, the anode loading was varied from 0.05, 0.1, 0.15 to 0.20 mg-Pt/cm², with a fixed cathode loading of 0.1 mg-Pt/cm^2 in the preferred NSTF ternary. The polarization curves substantially lie over one another, with no significant difference between any of the four anode loadings above ~ 0.6 V [5].

The mass activity for a given NSTF catalyst loading depends on the area of whisker support particles. This year, optimization of the NSTF support particles to improve the mass activity was begun, and a new ambient air pressure record performance was achieved. Figure 1 shows ambient pressure (constant flow rate) polarization curves from an MEA having 0.060 mg-Pt/cm² on each of the anode and cathode with the better-matched catalyst support system and preferred ternary.

Further understanding of how to improve high current density with ultra-low loading catalysts was achieved through studies relating mass transfer overpotential (MTO) to the NSTF electrode layer thickness. Last year, we showed it was possible to have zero MTO at 2 A/cm² under air operation and 0.1 mg/cm^2 of Pt. This year, it was determined that, for NSTF electrode layer thicknesses less than about 0.3 microns, MTO was less than about 40 mV at 2 A/cm^2 , and a linear relationship between MTO and electrode layer thickness was observed [6]. Furthermore, knowledge of the catalyst material

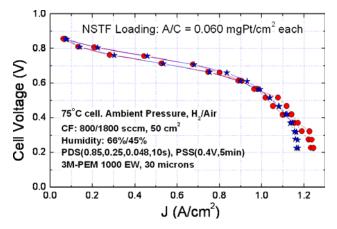


Figure 1. Ambient pressure (constant flow rate) polarization curves from an MEA having 0.060 mg-Pt/cm² on each of the anode and cathode with the better-matched NSTF catalyst support system and preferred ternary catalyst. The membrane used is a 1000 EW PEM made with the 3M PFSA ionomer.

densities and loadings combined with the scanning electron microscopy-determined layer thicknesses allowed determining that the NSTF electrode porosity is $\sim 75\%$ and filled primarily with the ionomer from the PEM [5]. The low MTO for the ultra-thin electrode layers allows the NSTF Pt specific power density to reach below 0.3 gPt/kW for cell voltages < 0.7 V. This is illustrated in Figure 2, where a pressurized (constant stoichiometric flow) polarization curve from an NSTF ternary catalyst MEA having 0.10 mg Pt/cm^2 on the cathode and 0.05mg Pt/cm^2 on the anode is plotted as mass specific power density versus cell voltage with star symbols. The ambient pressure, constant flow polarization curve from Figure 1 is similarly plotted as the square symbols. As seen, both MEAs at the conditions shown significantly exceed the DOE 2005 target and approach the 2010 target of 0.2 gPt/kW for voltages below 650 mV.

Durability studies of the NSTF catalysts and MEAs made with the catalysts begun last year were significantly extended this year. First it was found that the preferred NSTF catalyst based MEAs (using neat PFSA membranes) release very low fluoride levels, implying that the NSTF catalysts are producing lower peroxide levels than conventional Pt/carbon dispersed catalysts. For example, at 120°C, daily fluoride release rates remained constant at less than 24 nanogram/hr-cm² during 1080 hours

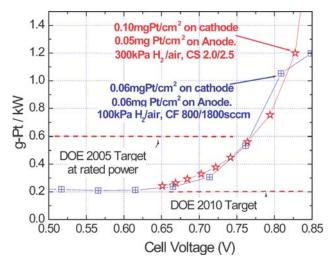


Figure 2. Mass specific power density versus cell voltage plots of NSTF MEA polarization curves compared to DOE 2005 and 2010 targets. Star symbols: pressurized (constant stoichiometric flow) polarization curve from an NSTF ternary catalyst MEA having 0.10 mg Pt/cm² ternary on the cathode and 0.05 mg Pt/ cm² on the anode. Square symbols: ambient pressure, constant flow polarization curve from MEA of Figure 1.

of testing at 120°C. Two basic reasons to expect lower peroxides with the NSTF catalysts are that they have 5 times higher specific activity and the NSTF catalysts do not contain any carbon. The exact fluoride release levels were found to be a strong function of the NSTF structure factor. The higher specific activity of the NSTF catalysts and resulting lower peroxide generation have important consequences for MEA lifetime, as discussed next.

Real time durability stress tests at 120°C and subsaturated H₂/air input streams were completed for several 100-cm² sized MEAs containing various NSTF ternary catalysts (0.1 mg-Pt/cm² cathode loadings) and thin membranes made with neat 3M ionomer. The two best performing ternaries also had the longest lifetimes, lasting over 1000 hours at 120°C (> 1800 hours total testing time) before catastrophic failure of the MEA occurred. In contrast, two MEAs with conventional Pt/carbon dispersed catalysts (0.4 and 0.8 mg Pt/cm² total per MEA) but identical membranes and GDLs as used in the NSTF MEAs, failed catastrophically after only about 100 hours. The fluoride release rates were

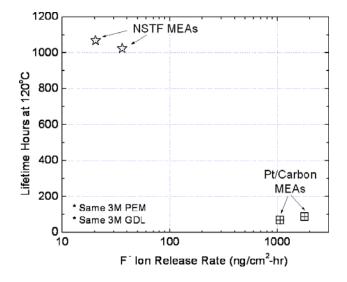


Figure 3. Plot of lifetime at 120°C versus fluoride (F⁻) release rate for two NSTF ternary catalyst based MEAs and two MEAs with Pt/carbon dispersed catalysts. The same neat 3M PEM and 3M GDL are used in each MEA. Conditions at 120°C are: 300 kPa, 0.4 A/cm², subsaturated inlets.

monitored continuously and are sufficient to explain the 15-20 times longer lifetime of the NSTF based MEAs under the identical test conditions. As shown in Figure 3, the F⁻ release rates were ~ 75 times less for the NSTF based MEAs than the conventional Pt/C dispersed catalyzed MEAs. In addition, daily measurements of the surface areas of the NSTF anodes and cathodes were observed to initially decrease but then stabilize over time to ~50% and 40%, respectively, of the initial values.

A third critical durability aspect of the NSTF catalysts quantified this year directly relates to stop/ start durability and potential loss of catalyst surface area due to agglomeration, Pt dissolution and support oxidation. An accelerated test to simulate the effects of start/stop cycling and hydrogen starvation was used in which MEAs are CV cycled between 0.6 and 1.2 V at 20 mV/sec under saturated H_2/N_2 . Periodically, the repetitive CV scanning is stopped and the catalyst surface area measured and recorded as a function of the number of completed scans to 1.2 V. Figure 4 plots the normalized surface area versus number of CV cycles at 80°C for four different NSTF catalyst based MEAs and, for comparison, the results from three conventional Pt/carbon dispersed catalyst based MEAs having four

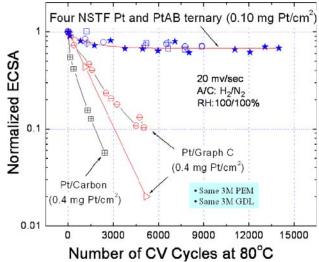
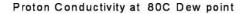
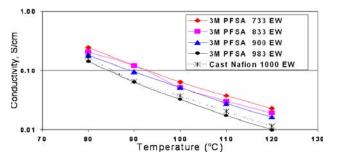


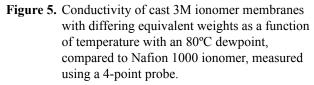
Figure 4. Normalized ECSA vs. number of CV cycles for four MEAs with low loading NSTF catalysts, and three MEAs with Pt/carbon or Pt/graphitized carbon dispersed catalysts. Membranes and GDLs were the same. CV conditions: 0.6 to 1.2 V at 20 mV/sec scan rates under H_2/N_2 (anode/cathode) with 100%/100% inlet humidification at 80°C.

times higher Pt loading. For all the MEAs, the same 3M PEM and 3M GDL were used. As seen, the NSTF catalyst ECSA plateaus to about 70% of the initial value out to 15,000 cycles, while the Pt/carbon and Pt/graphitic carbon samples maintain only 10% of their initial surface area after 2,000 and 5,000 cycles, respectively. This suggests that the NSTF catalysts will be more robust against high voltage excursions, but further improvements are desired for operation at higher temperatures. An extensive series of similar tests were also completed as a function of temperature for both NSTF and conventional Pt/carbon catalysts, and the loss of surface area with number of cycles, although still much less for the NSTF catalysts, can be much more severe at higher temperatures. In the scale-up effort, hundreds of feet of NSTF catalyst roll-goods were fabricated, and initial trials for catalyst coated membrane fabrication on a new pilot line were successfully completed.

The work on a membrane for the 85° to 120°C range has built upon a new perfluorinated sulfonic acid ionomer (PFSA) that was developed at 3M [7]. As discussed in previous reports, this polymer demonstrates high modulus, tear, and puncture

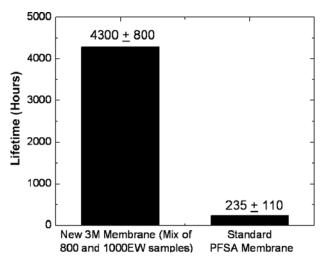


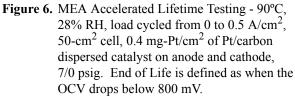




resistance (both dry and under use conditions), low levels of Fe and other metal impurities, which increases the oxidative stability of the membrane, improved oxidative stability in Fenton's type test, and low fluoride content in the water coming from the cell. More recent results indicate that these improvements result in a membrane with high conductivity at various temperatures and humidifications and a longer MEA life in accelerated testing.

The chemical structure of the new ionomer has a somewhat shorter side-chain than other commercially available perfluorinated ionomers, providing a polymer with a higher glass transition temperature (125°C vs. ca. 100°C) [5] at the same equivalent weight (EW). In the past year, this ionomer has been prepared and tested with EWs ranging from 700 to 1100. All of these polymers produced membranes with good mechanical properties. It is worth noting that, even at an EW of 700, the membrane is not soluble in water, and accelerated durability tests (90°/70°/70°C, H₂/air) of MEAs made from EWs ranging from 1100 to 700 showed no difference in lifetimes [5]. Water absorption and conductivity did increase with lower EW [5]. The conductivity as a function of temperature is shown in Figure 5 compared to a control made with Nafion 1000 ionomer. While the conductivity of the 983 EW sample is nearly identical to the Nafion 1000 sample, the number of water molecules absorbed for each sulfonic acid group at a given %RH is lower in the 3M membrane than it is in membrane made with Nafion ionomer at





this EW [5]. This means that the conductivity contribution of each absorbed water molecule is higher in the 3M ionomer than in Nafion ionomer. While the cause of this effect is not fully understood, it does seem to indicate that the polymer morphology of the 3M ionomer is different from that of Nafion ionomer.

Using stabilizing additives in the membrane and significantly lowering the number of carboxylate end groups in the polymer has resulted in membranes with substantially improved oxidative stability, as measured by Fenton's testing (1M H₂O₂, 10 ppm Fe, 90°C). Fuel cell testing under accelerated conditions showed that MEAs made from this new membrane had a greater than 15-fold increase in lifetime. This is shown in Figure 6, where the best new membrane construction lasted 4300 hours under dry, 90°C load cycling before end of life occurred, in contrast to the standard PFSA for which the average lifetime was 235 hours. The lower EW samples also provided MEAs with lower cell resistance, as measured by both AC impedance and hydrogen pump, as well as improved performance under hotter, dryer operating conditions compared to a standard PFSA ionomer.

Progress has also been made in the challenging area of high-temperature proton conducting materials, targeting operation above 120°C. An extensive library of new high-temperature

electrolytes formed from proton conducting fluorinated liquids, inorganic additives and polymer matrices were fabricated and characterized for conductivity, acid retention and fuel cell performance. While these materials still do not have proton conductivity as high as PBI-phosphoric acid membranes at 120°C, MEAs prepared from them have better fuel cell performance under H₂/air at 110°C [5]. These MEAs were made with catalyst loadings of only 0.4/0.4 mg/cm² Pt (anode/cathode). This seems to indicate that the 3M proton conducting liquids are more compatible with oxygen reduction than phosphoric acid. In a second approach to high temperature membranes, involving inorganic proton conductors, pulse field gradient spin echo-diffusion measurements of new heteropoly acids developed at the Colorado School of Mines show that they have lower activation barriers for H⁺ transport, and higher maximum temperatures [8].

Conclusions

- Significant improvements in membrane and catalyst durability were demonstrated in the past year for operation in the 85 to 120°C range.
- The 3M PFSA ionomeric membrane can be fabricated with equivalent weights optimized for performance and low humidity conditions.
- Combining the durability and performance advantages of the 3M PEM and NSTF catalyst offers real potential for achieving the DOE 2010 durability target of 5000 hours for MEAs in automotive fuel cell stacks at temperatures above 80°C.
- Performance improvements of the 3M NSTF catalyst with total MEA PGM loadings below 0.2 mg/cm² have enabled Pt specific power densities of 0.3 gPt/kW to be reached below 0.71 V.
- The 3M PEM, NSTF catalysts and CCMs can all be fabricated by high volume compatible processes.
- Development of new membrane technology for operation above 120°C without relying on aqueous proton conduction remains a considerable challenge to meet all the requirements for performance, durability and catalyst compatibility.

FY 2005 Publications/Presentations

- "Studies of Transition Metal Dissolution from Combinatorially Sputtered, Nano-Structured Pt1-x M_x (M=Fe, Ni;0<x<1) Electrocatalysts for PEM Fuel Cells," A. Bonakdarpour, J. Wenzel, D. A. Stevens, S. Sheng, T. L. Monchesky, R. Lobel, R. T. Atanasoski, A. K. Schmoeckel, G. D. Vernstrom, M. K. Debe and J. R. Dahn, J. Electrochemical Society, Vol. 152 (1), 2005, A61-A72.
- "NanoStructured Thin Film, Thin Layer Electrodes Optimized for PEM Fuel Cell Performance at High Current Density," M. K. Debe et al., 2004 Fuel Cell Seminar, San Antonio, TX, Nov. 1-5, 2004.
- "Corrosion of Transition Metals in Pt 1-xMx (M=Fe, Ni, Mn) Proton Exchange Membrane Fuel Cell Electrocatalysts," A. Bonakdarpour et al., presented at the Fall 2004 Electrochemical Society Meeting, Honolulu, HI, USA.
- "64-Channel Fuel Cell for Testing Sputtered Combinatorial Arrays of Oxygen Reduction Catalysts," D. A. Stevens et al., presented at the Fall 2004 Electrochemical Society Meeting, Honolulu, HI, USA.
- "Combinatorial PEM Fuel Cell Studies of Binary Platinum Alloys," D. A. Stevens et al., presented at the Fall 2004 Electrochemical Society Meeting, Honolulu, HI, USA.
- "The Search for Higher Temperature Proton Conductors for the PEM Fuel Cell," J. Woods Halley, Symposium on Fuel Cells, MRS Meeting, Boston, MA, December 1, 2004.
- "The Development of New Membranes for PEM Fuel Cells," S. J. Hamrock, Advances in Materials for Proton Exchange Membrane Fuel Cell Systems, Asilomar Conference Grounds, Pacific Grove, CA, February 21, 2005.
- "The Use of Heteropoly Acids in Composite Membranes for Elevated Temperature or Low Humidity PEM Fuel Cell Operation," A. Herring, J. Turner, S. Dec, J. Malers, F. Meng, J. Horan and N. Aieta, 2nd International Conference on Polymer Batteries and Fuel Cells, Las Vegas, NV, June 13th 2005.
- "Mechanical Property Measurements of PFSA Membranes at Elevated Temperatures and Humidities," M. A. Yandrasits, 2nd International Conference on Polymer Batteries and Fuel Cells, Las Vegas, NV, June 14, 2005.

[&]quot;Nafion" is a registered trademark of DuPont.

 "Prospects and Challenges for PEM Fuel Cells with a Focus on MEA Development for Automotive Applications," M. K. Debe, Invited Plenary Lecture, 2005 Annual Meeting of the North American Membrane Society, Providence, RI, June 15, 2005.

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- 2004 DOE Hydrogen, Fuel Cells, and Infrastructure Technologies Program Review Meeting, May 24 - 27, 2004, Philadelphia, PA, Presentation 54-FC4.
- Hydrogen, Fuel Cells, and Infrastructure Technologies FY 2004 Progress Report, p. 301.

- 2005 DOE Hydrogen, Fuel Cells, and Infrastructure Technologies Program Review Meeting, May 23 - 26, 2005, Arlington, VA, Presentation FC-3.
- "NanoStructured Thin Film, Thin Layer Electrodes Optimized for PEM Fuel Cell Performance at High Current Density," M. K. Debe et al., 2004 Fuel Cell Seminar, San Antonio, TX, Nov. 1-5, 2004.
- 7. "Development of a new electrolyte membrane for PEM fuel cells," L. Rivard, D. Pierpont,
 H. Freemeyer, A. Thaler and S. Hamrock, 2003 Fuel Cell Seminar, Miami Beach, FL, November 3-7, 2003, Abstract Book p. 73.
- "The Use of Heteropoly Acids in Composite Membranes for Elevated Temperature or Low Humidity PEM Fuel Cell Operation" A. Herring, J. Turner, S. Dec, J. Malers, F. Meng, J. Horan and N. Aieta, 2nd International Conference on Polymer Batteries and Fuel Cells, Las Vegas, NV, June 13, 2005.