New Fuel Cell Membranes with Improved Durability and Performance

Mike Yandrasits
3M Energy Components Program
June 8th, 2017
Overview

Timeline
• Start October 1st, 2013
• End June 30th, 2017
• 93% complete

Barriers
Durability
Performance
Cost

Budget
• Total Project funding $4.2 million
  - $3.1 million - DOE
  - $1.1 million - contractor cost share (26%)
• DOE Funding to date (through March 2016)
  - $2,725,188 (88%)

Partners
3M Company M. Yandrasits (Project lead)
General Motors C. Gittleman
Vanderbilt University Professor P. Pintauro
Objective: Meet all of the DOE Fuel Cell Technologies Office Multi-year RD&D Plan membrane performance, durability and cost targets simultaneously with a single membrane.

Collaborations: Flow Of Samples & Information

- Task 1: Ionomer development
- Task 2: Nanofiber development
- Task 3: Ionomer and Membrane Testing
- Task 4: MEA Fabrication and Fuel Cell Testing
- Task 5: Stack Testing

Green shading indicates approximate task completion

General Motors,
- Chemical and mechanical property testing
- Single cell performance testing
- Stack testing
- Post mortem analysis

Vanderbilt University
- Electrospinning expertise
- Dual fiber electrospinning

Objective: Meet all of the DOE Fuel Cell Technologies Office Multi-year RD&D Plan membrane performance, durability and cost targets simultaneously with a single membrane.

Work completed in 2016
# Milestone Summary

<table>
<thead>
<tr>
<th>Milestone</th>
<th>Requirement</th>
<th>Date Completed</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ionomer conductivity</td>
<td>Jan, ‘14</td>
<td>✔</td>
</tr>
<tr>
<td>2</td>
<td>Nanofiber down select</td>
<td>Apr, ‘14</td>
<td>✔</td>
</tr>
<tr>
<td>3</td>
<td>Electrospin Ionomer</td>
<td>May, ‘14</td>
<td>✔</td>
</tr>
<tr>
<td>4 Go/No Go</td>
<td>Durability &amp; performance</td>
<td>Oct, 14</td>
<td>✔</td>
</tr>
<tr>
<td>5</td>
<td>Ionomer conductivity</td>
<td>Mar, ‘15</td>
<td>✔</td>
</tr>
<tr>
<td>6</td>
<td>Fiber surface treatment selection</td>
<td>Apr, ‘15</td>
<td>✔</td>
</tr>
<tr>
<td>7</td>
<td>Durability &amp; ASR</td>
<td>Jun, ‘15</td>
<td>✔-✔</td>
</tr>
<tr>
<td>8 Go/No Go</td>
<td>Durability, ASR, short res. H₂&amp;O₂ crossover, &amp; cost</td>
<td>Sep, ‘15</td>
<td>✔-✔</td>
</tr>
<tr>
<td>9</td>
<td>Produce membrane for stack testing (&gt;20 meters)</td>
<td>March ‘16</td>
<td>✔</td>
</tr>
<tr>
<td>10</td>
<td>Begin Stack Testing</td>
<td>June, ‘16</td>
<td>✔</td>
</tr>
<tr>
<td>11</td>
<td>Post Mortem Analysis, Determine Failure Mode</td>
<td>Nov. ‘16</td>
<td>✔</td>
</tr>
<tr>
<td>12</td>
<td>Deliver MEAs to DOE, Complete 2,000hrs stack testing</td>
<td>Nov. ‘16</td>
<td>✗</td>
</tr>
</tbody>
</table>

*Full Milestone List in Back-Up Slides*
Membrane For Milestones 8 and 10

\[
\left(\text{CF}_2\text{CF}_2\right)_n\text{CF}_2\text{CF} + \text{H} - \text{SO}_2\text{NSO}_2\text{C}_3\text{F}_6\text{SO}_3\text{H}
\]

Perfluoro imide acid (PFIA) ionomer

Nanofiber support made using fluoropolymer (FC1)

Electron microscope cross section image of composite membrane

<table>
<thead>
<tr>
<th>3M ID</th>
<th>Milestone</th>
<th>Ionomer</th>
<th>Fiber type</th>
<th>Additive</th>
<th>Fiber (vol%)</th>
<th>Thickness (um)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0513277A</td>
<td>Control</td>
<td>3M 725EW</td>
<td>B1</td>
<td>Type A</td>
<td>20.6</td>
<td>14</td>
</tr>
<tr>
<td>0515079D</td>
<td>#8</td>
<td>PFIA – Lot #1</td>
<td>FC1</td>
<td>Type A</td>
<td>18.0</td>
<td>10</td>
</tr>
<tr>
<td>05160081A, B, C, D</td>
<td>#10</td>
<td>PFIA - Lot #2</td>
<td>FC1</td>
<td>Various</td>
<td>18.0</td>
<td>10</td>
</tr>
</tbody>
</table>
## Milestone 8: 3M ID 0515079D

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Units</th>
<th>2017 &amp; 2020 Targets</th>
<th>MS#8 PFIA-S (10 um)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum oxygen cross-over</td>
<td>mA / cm²</td>
<td>2</td>
<td>0.6&lt;sup&gt;a&lt;/sup&gt;, 3.5&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>Maximum hydrogen cross-over</td>
<td>mA / cm²</td>
<td>2</td>
<td>1.9&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td>Area specific proton resistance at:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>120°C, P&lt;sub&gt;H₂O&lt;/sub&gt; 40 kPa</td>
<td>Ohm cm²</td>
<td>0.02</td>
<td>0.054</td>
</tr>
<tr>
<td>120°C, P&lt;sub&gt;H₂O&lt;/sub&gt; 80 kPa</td>
<td>Ohm cm²</td>
<td>0.02</td>
<td>0.019</td>
</tr>
<tr>
<td>80°C, P&lt;sub&gt;H₂O&lt;/sub&gt; 25 kPa</td>
<td>Ohm cm²</td>
<td>0.02</td>
<td>0.020</td>
</tr>
<tr>
<td>80°C, P&lt;sub&gt;H₂O&lt;/sub&gt; 45 kPa</td>
<td>Ohm cm²</td>
<td>0.02</td>
<td>0.008</td>
</tr>
<tr>
<td>30°C, P&lt;sub&gt;H₂O&lt;/sub&gt; up to 4 kPa</td>
<td>Ohm cm²</td>
<td>0.03</td>
<td>0.018</td>
</tr>
<tr>
<td>-20°C</td>
<td>Ohm cm²</td>
<td>0.2</td>
<td>0.2&lt;sup&gt;d&lt;/sup&gt;</td>
</tr>
<tr>
<td>Minimum electrical resistance</td>
<td>Ohm cm²</td>
<td>1,000</td>
<td>1,635&lt;sup&gt;e&lt;/sup&gt;</td>
</tr>
<tr>
<td>Cost</td>
<td>$ / m²</td>
<td>20</td>
<td>Not available</td>
</tr>
</tbody>
</table>

**Durability**

- **Mechanical**: Cycles with <10 sccm crossover 20,000 >24,000
- **Chemical**: hrs >500 614

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**Relevance:**

- Lower membrane resistance at low P<sub>H₂O</sub> and/or high T will allow for fewer cells or simplified balance of plant.
- Membrane cost, especially at low volumes, is an issue for FCEVs.

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<sup>a</sup> O₂ crossover based on DOE Table 3.4.12 indicating measurement at 0.5V
<sup>b</sup> Calculated from GM O₂ permeability data at 80°C, 100% RH, 1 atm air.
<sup>c</sup> In cell measurements at 3M 70°C, 100% RH, 1 atm.
<sup>d</sup> Calculated from in-plan data
<sup>e</sup> Data provided by GM

DOE Table in Back-up Slides
PFIA Pilot Scale

<table>
<thead>
<tr>
<th>Lot Number</th>
<th>Date</th>
<th>Titrated EW</th>
<th>Program</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>January 2015</td>
<td>660</td>
<td>DOE</td>
</tr>
<tr>
<td>2</td>
<td>December 2015</td>
<td>652</td>
<td>3M</td>
</tr>
<tr>
<td>3 and 4</td>
<td>March 2016</td>
<td>625</td>
<td>3M</td>
</tr>
<tr>
<td>5</td>
<td>Sept 2016</td>
<td>650</td>
<td>3M</td>
</tr>
</tbody>
</table>

- Five pilot scale batches complete
- High degree of conversion in each step
Milestones 10-12: Stack Testing at GM

- **Milestone 10:** Manufacture for stack testing at least 30 MEAs with a minimum cell area of 250 cm². Evaluate in fuel cells and ex situ tests. Begin stack testing.
- **Milestone 11:** Begin post mortem analysis of MEAs to determine failure mode.
- **Milestone 12:** Prepare the MEAs, the number and size to be determined by 3M and the DOE, and deliver them for testing at a DOE approved facility. Complete stack testing for a minimum of 2,000 hours.

<table>
<thead>
<tr>
<th>Membrane Types</th>
<th>Ionomer</th>
<th>Ionomer EW (g/mol)</th>
<th>Thickness</th>
<th>Support</th>
<th>3M + GM additive</th>
</tr>
</thead>
<tbody>
<tr>
<td>3M 0513277A</td>
<td>3M PFSA</td>
<td>725</td>
<td>14µm</td>
<td>HC/FC</td>
<td>1X type A, 2XB</td>
</tr>
<tr>
<td>3M 05160081A</td>
<td>3M PFIA</td>
<td>650</td>
<td>10µm</td>
<td>FC1</td>
<td>0X type A, 2XB</td>
</tr>
<tr>
<td>3M 05160081B</td>
<td>3M PFIA</td>
<td>650</td>
<td>10µm</td>
<td>FC1</td>
<td>1X type A, 2XB</td>
</tr>
<tr>
<td>3M 05160081C</td>
<td>3M PFIA</td>
<td>650</td>
<td>10µm</td>
<td>FC1</td>
<td>2X type A, 2XB</td>
</tr>
<tr>
<td>3M 05160081D</td>
<td>3M PFIA</td>
<td>650</td>
<td>10µm</td>
<td>FC1</td>
<td>2X type A, 1XB</td>
</tr>
<tr>
<td>GM state-of-art PFSA</td>
<td></td>
<td></td>
<td></td>
<td>ePTFE</td>
<td>yes</td>
</tr>
</tbody>
</table>
Milestone 12: Stack Test Data from GM

Summary
• Membranes failed prior to the 2,000 hr target (~830 hrs)
• Performance decay was observed for the PFIA that was partially recoverable
• No dependence on additive type or level

Potential reasons for early failure
• Short resistance marginal at BOL (see back-up slides)
• 3M determined PFIA lot had high iron content (~50 ppm)
• Possible defects in membrane coating
• Ionomer stability

Accomplishments and Progress
Milestone 11: Post Mortem Analysis

Leaks identified with bubble test

Optical microscope cross sections images

Summary
- Membrane thinning/erosion observed near cathode inlet of cell 7.
- Damaged areas observed in several locations.
- Cracks more severe on anode side.
A Closer Look at the OCV Testing

Unexpected OCV decay
- Impurities related to synthesis eliminated as possible cause.
- Cross-over or shorts eliminated (see 2016 AMR)
- Commercial electrodes using alloy catalyst and additional additive appear to delay the effect (see 2016 AMR)
- ~50ppm iron detected in PFIA ionomer lot (2017 AMR)
- Membrane decomposition hypothesis explored (2017 AMR)

Unexpected HFR increase
- Measurement error eliminated (data not shown).
- Formation of resistive layer (i.e. voids in support layer) eliminated (data not shown).
- Commercial electrodes (Alloy and additional additive) appear to delay the effect (see 2016 AMR)
- ~50ppm iron detected in PFIA ionomer lot (2017 AMR)
- Membrane decomposition hypothesis explored (2017 AMR)

Summary
- Ionomer decomposition hypothesis under evaluation
- OCV decay and resistance increase appear related
- High surface area catalyst with increased additives mitigate effect (See 2016 AMR and back-up slides).
OCV Testing – Low Iron Samples

• Iron Identified in PFIA lot used for MS#8 Membrane (~50ppm)
• Low Iron samples prepared and run in OCV test (~1 ppm)

Summary
• Increased degradation due to iron contamination alone is not the likely cause of the observed OCV decay or resistance increase.
Peroxide Vapor Test (30 ppm)

Four, 14 µm thick, ePTFE reinforced membranes with additive were subjected to the standard 60 hour $H_2O_2$ vapor test.

Summary

- 3M PFSA exhibited near zero acceleration at the conclusion of the test (1.2x).
- All PFIA membranes showed about a 2.3x acceleration over the course of the 60 hour test. Reason for differences in $t=0$ degradation rates are unknown.
- Membrane damage assessed by FTIR analysis of K$^+$ salts, increased COO$^-$ observed in PFIA samples.
Objective:
Subject membrane to the same conditions that result in performance decay and analyze separate membrane layers for chemical or physical changes (3-5 layers);
- Conductivity
- $^{19}$NMR
- FTIR
OCV Testing – 3 Layer Samples

Inner layer Membranes:
- 20um 725 EW no add. no support
- 20um* PFIA (CG3/4) no add. no support

Summary
- PFIA in center layer exhibited OCV decay and resistance increase
- Center membrane isolated for both PFIA and control
Multilayer OCV Test – Effluent Water

**Inner layer Membranes:**
- 20um 725 EW no add. no support
- 20um* PFIA (CG3/4) no add. no support

**Summary**
- Ion chromatography measurements of effluent water (**cathode only**) show fluoride and sulfate levels similar between PFSA and PFIA
Post Mortem Conductivity

In-plane conductivity measured using 4 point probe method

Summary
- All samples showed thinning in active area 20um → 5-15um
- Conductivity for PFSA control membranes are similar between edge and active area
- Conductivity of PFIA active area significantly reduced
Post Mortem $^{19}$F NMR and FTIR

Analysis of center layer of 3 layer OCV test (K+ or Li+ form)

Summary
- Carboxylate end groups observed in FTIR for both ionomers as expected.
- FTIR analysis of PFSA shows no meaningful change in side chain functionality.
- FTIR and $^{19}$F NMR analysis of PFIA layer show loss of the imide functionality and appearance of the amide functionality.
OCV Testing – 5 Layer Samples

All membrane layers: 14um PFIA with support and additives

Water samples retained for fluoride measurements (cathode only) were analyzed by liquid chromatography-mass spectroscopy (LC-MS)

Summary
• Layers not able to be effectively separated.
• Water analysis shows low levels of PFIA side chain fragments throughout cell lifetimes.
• Additional fragments detected but not reported here.
Summary

- Fluoride and sulfate release levels similar between PFSA and PFIA samples.
- Bond strengths and oxidative decomposition likely to be similar between sulfonic acid and imide functionality.
- **No evidence yet that PFIA is fundamentally less stable than PFSA**
- **Consequences** of decomposition are different
  - Potential catalyst poisoning (larger/different fragments)
  - Nonconductive polymer (sulfonamide polymer has very low conductivity)
Summary

• Nearly all milestones met with the exception of final stack test
• Nearly all of the DOE targets for membrane performance, and durability were met simultaneously with a single membrane.
• A new durability concern has emerged;
  – Oxidative decomposition of PFIA has new implications on MEA electrode performance and membrane conductivity.
  – Existing OCV and RH cycle accelerated stress tests did not immediately reveal the problem
  – Mechanism of degradation not fully understood
Future Work

Remainder of project

• Investigate PFIA oxidation mechanism
  – Model compound studies (isolate imide and sulfonic acid functionality)
  – Study imide only ionomers

• Evaluate the effects of peroxide stabilizing additives on PFIA stability
  – Revisit Milestone #7
  – Use mechanism learnings to develop stabilizing strategy

• Study the effects of decomposition products on catalyst activity
  – Rotating disk electrode (RDE) studies at NREL (H., Dinh, G. Bender)
  – Introduce side chain fragments into operating fuel cell

• Assess stabilization strategies by the end of 2017

• Ongoing work in fiber support development

Any proposed future work is subject to change based on funding levels
Technical Back-up Slides
### Table 3.4.12 Technical Targets: Membranes for Transportation Applications

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Units</th>
<th>2011 Status a</th>
<th>2017 Targets</th>
<th>2020 Targets</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum oxygen cross-over</td>
<td>mA / cm²</td>
<td>&lt;1</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Maximum hydrogen cross-over b</td>
<td>mA / cm²</td>
<td>&lt;1.8</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Area specific proton resistance at:</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maximum operating temperature and water partial pressures from 40-80 kPa</td>
<td>Ohm cm²</td>
<td>0.023 (40kPa)</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>80°C and water partial pressures from 25-45 kPa</td>
<td>Ohm cm²</td>
<td>0.017 (25kPa)</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>30°C and water partial pressures up to 4 kPa</td>
<td>Ohm cm²</td>
<td>0.02 (3.8 kPa)</td>
<td>0.03</td>
<td>0.03</td>
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<tr>
<td>-20°C</td>
<td>Ohm cm²</td>
<td>0.1</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>Operating temperature</td>
<td>°C</td>
<td>&lt;120</td>
<td>≤120</td>
<td>≤120</td>
</tr>
<tr>
<td>Minimum electrical resistance</td>
<td>Ohm cm²</td>
<td>–</td>
<td>1,000</td>
<td>1,000</td>
</tr>
<tr>
<td>Cost c</td>
<td>$ / m²</td>
<td>–</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>Durability d</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mechanical</td>
<td>Cycles with &lt;10 sccm crossover hours</td>
<td>&gt;20,000</td>
<td>20,000</td>
<td>20,000</td>
</tr>
<tr>
<td>Chemical</td>
<td></td>
<td>&gt;2,300</td>
<td>&gt;500</td>
<td>&gt;500</td>
</tr>
</tbody>
</table>

a: [http://www.hydrogen.energy.gov/pdfs/progress11/v_c_1_hamrock_2011.pdf](http://www.hydrogen.energy.gov/pdfs/progress11/v_c_1_hamrock_2011.pdf). Status represents 3M PFIA membrane (S. Hamrock, U.S. Department of Energy Hydrogen and Fuel Cells Program 2011 Annual Progress Report, (b: Tested in MEA at 1 atm O₂ or H₂ at nominal stack operating temperature, humidified gases at 0.5 V DC. c: Costs projected to high-volume production (500,000 stacks per year). d: [http://www.uscar.org/commands/files_download.php?files_id=267](http://www.uscar.org/commands/files_download.php?files_id=267)Protocol for mechanical stability is to cycle a 25-50 cm² MEA at 80°C and ambient pressure between 0% RH (2 min) and 90°C dew point (2 min) with air flow of 2 SLPM on both sides. Protocol for chemical stability test is to hold a 25-50 cm² MEA at OCV, 90°C, with H₂/air stoichs of 10/10 at 0.2 A/cm² equivalent flow, inlet pressure 150 kPa, and relative humidity of 30% on both anode and cathode. Based on U.S. DRIVE Fuel Cell Tech Team Cell Component Accelerated Stress Test and Polarization Curve Protocols (), MEA Chemical Stability and Metrics (Table 3) and Membrane Mechanical Cycle and Metrics (Table 4).
<table>
<thead>
<tr>
<th>MS ID</th>
<th>Full Milestone</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Measure conductivity and fuel cell performance on at least two different control PFSA membranes and initial samples of MASC ionomer membranes. Demonstrate MASC ionomer with conductivity of 0.1 S/cm or higher at 80°C and &lt;50% RH.</td>
<td>January 9, 2014</td>
</tr>
<tr>
<td>2</td>
<td>Identify one or more polymer systems for further development in a nanofiber support that provides a membrane with x-y swelling of &lt;5% after boiling in water.</td>
<td>April 8, 2014</td>
</tr>
<tr>
<td>3</td>
<td>Develop electrospinning conditions for one or more 3M ionomers that provides fiber diameter of &lt;1 micron.</td>
<td>May 22, 2014</td>
</tr>
<tr>
<td>4 - Go/No-Go</td>
<td>Develop a laboratory produced membrane using an optimized ionomer and electrospun nanofiber support that passes all of the tests shown in tables D3 (chemical stability) and D4 (mechanical stability) of the FOA while still showing performance in single cell polarization experiments above state of the art, mass produced membranes (nanofiber supported 725 EW 3M Membranes) tested in the beginning of this program (not to be less than 0.5 V at 1.5 A/cm2 at 95°C, 50%RH, 150 kPa inlet pressure, and 0.4 mg/cm2 total pgm catalyst loading).</td>
<td>October 16, 2014</td>
</tr>
<tr>
<td>5</td>
<td>Prepare at least one additional MASC polymer. Demonstrate conductivity of 0.1 S/cm or higher at 80°C and &lt;40% RH. Evaluate in a supported membrane in Fuel Cell and ex situ tests.</td>
<td>March 6th, 2015</td>
</tr>
<tr>
<td>6</td>
<td>Prepare dense electrospun films with and without surface treatment of the support polymer with a maximum void fraction of &lt;5%. Prepare and characterize the resulting nanofiber composite membranes. Determine if surface treatment impacts swell, tensile or tear properties of the membrane. Select surface treatment, if any.</td>
<td>April 3rd, 2015 - ongoing</td>
</tr>
<tr>
<td>7</td>
<td>Prepare an ionomer formulation (ionomer, stabilizing additive) with optimum performance and durability that provides &gt;500 hours in test D3 (chemical stability), and equal or better area specific resistance (ASR) to the membrane described in the Q4 milestone of the same thickness, evaluated in a 50cm2 fuel cell using the same MEA components and same support, to be used for development of the supported membrane described in milestone Q8.</td>
<td>June 30th, 2015</td>
</tr>
<tr>
<td>8 - Go/No-Go</td>
<td>Produce membrane comprising a MASC Ionomer, a nanofiber support and a stabilizing additive which meets all of the 2020 membrane milestones in Table 3.4.12 (Technical Targets: Membranes for Transportation Applications) in the DOE Fuel Cell Technologies Office Multi-Year Research, Development and Demonstration Plan, section 3.4, update July 2013.</td>
<td>September 30th, 2015</td>
</tr>
<tr>
<td>9</td>
<td>Develop a process for producing the membrane described in Milestone Q8 in quantities large enough to produce membranes for use in Milestone Q10 (at least 20 linear meters)</td>
<td>January 1, 2016</td>
</tr>
<tr>
<td>10</td>
<td>Manufacture for stack testing at least 30 MEAs with a minimum cell area of 250 cm2. Evaluate in fuel cells and ex situ tests. Begin stack testing.</td>
<td>April 1, 2016</td>
</tr>
<tr>
<td>11</td>
<td>Begin post mortem analysis of MEAs to determine failure mode.</td>
<td>July 1, 2016</td>
</tr>
<tr>
<td>12</td>
<td>Prepare the MEAs, the number and size to be determined by 3M and the DOE, and deliver them for testing at a DOE approved facility. Complete stack testing for a minimum of 2,000 hours.</td>
<td>October 1, 2016</td>
</tr>
</tbody>
</table>
MEA Resistance Quality Check. Red line is GM specification.
Summary

- Ionomer with an Equivalent weight of 450 g/mol is need to meet this target for a 10 um membrane with additive and support (see 2016 AMR).
Milestones #1 & 5

Accomplishments:
- State of the art conductivity improved by 5x at 80°C and 40% RH.
- 100mS/cm conductivity threshold moved from 80% to 40% RH compared to Nafion™.
- 100mS/cm conductivity threshold moved from 50% to 40% RH since the start of project.
**Ionomer Development**

In-Plane conductivity (4 point probe)

**Accomplishments:**
- Simple model establishes conductivity as a function of ‘apparent’ equivalent weight
- PFICE-4 conductivity is very close to ‘ionone limit’. Additional chain extension would provide little addition gains.

\[
y = 0.3236e^{-0.003x}
\]

\[
R^2 = 0.9697
\]

**Equation:**

\[
\text{Apparent EW} = \frac{1}{\frac{1}{\text{EW}} \times \left(1 - \frac{\% \text{ fiber}}{100}\right)}
\]
Accomplishments and Progress

Milestone #8 Membrane with Commercial Electrodes

- MS#8 Membrane passed 500 hrs with lab electrodes.
- OCV decay and HFR increase observed with lab electrodes.
- Commercial electrodes and additive levels appears to have eliminated OCV decay and delayed HFR increase.
- H₂ crossover constant until membrane failure.

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Electrodes</th>
<th>Lifetime (hrs) 80% OCV</th>
</tr>
</thead>
<tbody>
<tr>
<td>MS#8</td>
<td>Lab control</td>
<td>614 ± 55</td>
</tr>
<tr>
<td>MS#8</td>
<td>Commercial</td>
<td>2105 ± 851</td>
</tr>
<tr>
<td>3M Commercial</td>
<td>Commercial</td>
<td>1484 ± 209</td>
</tr>
</tbody>
</table>

OCV Accelerated Durability

- H₂ crossover constant until membrane failure.

Diagnostic Test - Hydrogen Cross Over

H₂ Crossover (mA/cm²)

- FC035405
- FC035406
- FC035407
- FC035408

- Average: 2.2
- Stdev: 0.21