# New Fuel Cell Membranes with Improved Durability and Performance

Mike Yandrasits

3M Energy Components Program

June 9th, 2015



FC109

# Overview

## Timeline

- Start October 1st, 2013
- End September 30th, 2016
- 50% complete

## Budget

- Total Project funding $4.2 million
  - $3.1 million - DOE
  - $1.1 million - contractor cost share (26%)
- Funding in FY 2014
  - $678,000
- Funding in FY 2015
  - $476,000 (Through March 2015)

## Barriers

Durability

Performance

Cost

## Partners

**3M Company** *M. Yandrasits (Project lead)*

**General Motors** *C. Gittleman*

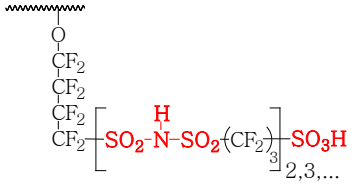
**Vanderbilt University** *Professor P. Pintauro*

# Collaborations: Flow Of Samples & Information

**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.**

Green shading indicates approximate task completion

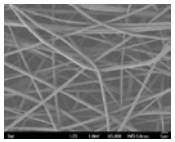
**Task 1: Ionomer development** (3M)




**Task 3: Ionomer and Membrane Testing** (3M, Vanderbilt University, GM)

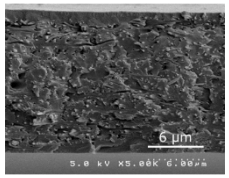
**Task 4: MEA Fabrication and Fuel Cell Testing** (3M, GM)

**Task 5: Stack Testing** (GM)

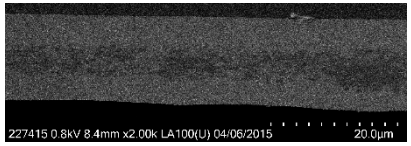


**Task 2: Nanofiber development** (3M, Vanderbilt University)

**Dual Fiber Electrospinning** (Vanderbilt University)
(ionomer and support fibers)




**Nanofiber Support** (3M)
(3M Korea and 3M St. Paul)




## General Motors,

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

## Vanderbilt University

- Electrospinning expertise
- Dual fiber electrospinning

# Milestone Summary

| Milestone | Requirement | Date Completed | Status |
|---|---|---|---|
| 1 | Ionomer conductivity | Jan, '14 | ✓ |
| 2 | Nanofiber down select | Apr, '14 | ✓ |
| 3 | Electrospin Ionomer | May, '14 | ✓ |
| 4 Go/No Go | Durability & performance | Oct, 14 | ✓ |
| 5 | Ionomer conductivity | Mar, '15 | ✓ |
| 6 | Fiber surface treatment selection | Apr, '15 | ✓ |
| 7 | Durability & ASR | Jun, '15 | Started |
| 8 Go/No Go | Durability, ASR, short res. $H_2$&$O_2$ crossover, & cost | Sep, '15 | Started |

*Reviewed in this talk*

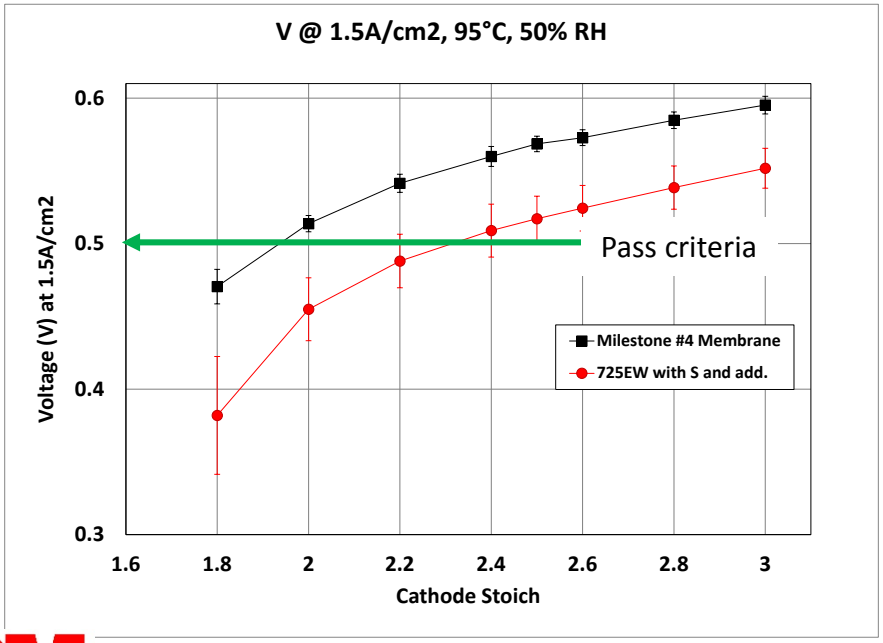
***Full Milestone List in Technical Back-Up Slides***

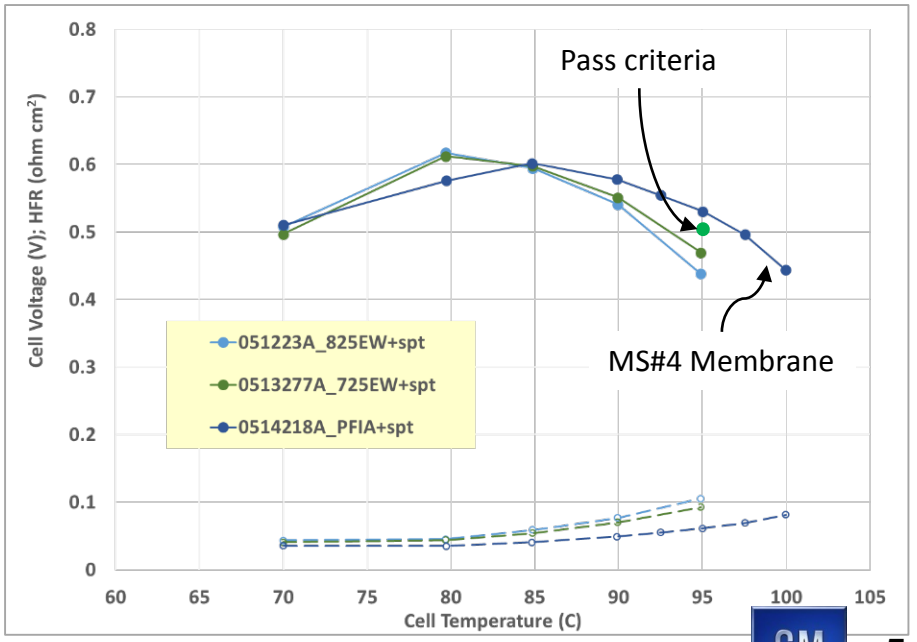
# Milestone #4 Go/No Go

MS#4: Develop a laboratory produced membrane that passes the chemical stability (***OCV hold***) and mechanical stability (***RH cycle***) tests while still showing performance in single cell above supported 725 EW 3M membrane not to be less than ***0.5 V at 1.5 A/cm2 at 95C, 50%RH***.(see detailed milestone in technical back-up slides)

| Run ID | Description | Fiber basis wt (gsm) | Fiber fraction (vol%) | Ionomer EW (g/mol) | Apparent EW of composite (g/mol) |
|---|---|---|---|---|---|
| 0514218A | PFIA, 14um,4.3gsm S-15,w/additive | 4.3 | 20.6 | 620 | 766 |

- Ionomer: Lab made PFIA
- Support: Fluoropolymer (FC1) based nanofiber made in pilot scale quantities (~100 linear meters)
- Additive: Inorganic peroxide scavenger at the same loading as 3M's commercial membrane for automotive market.



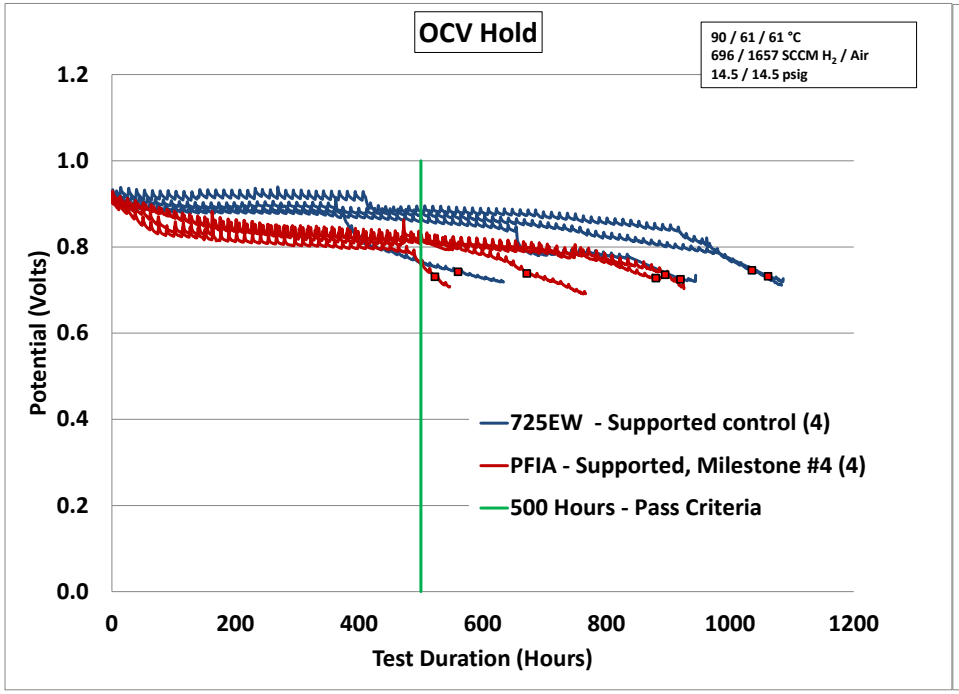



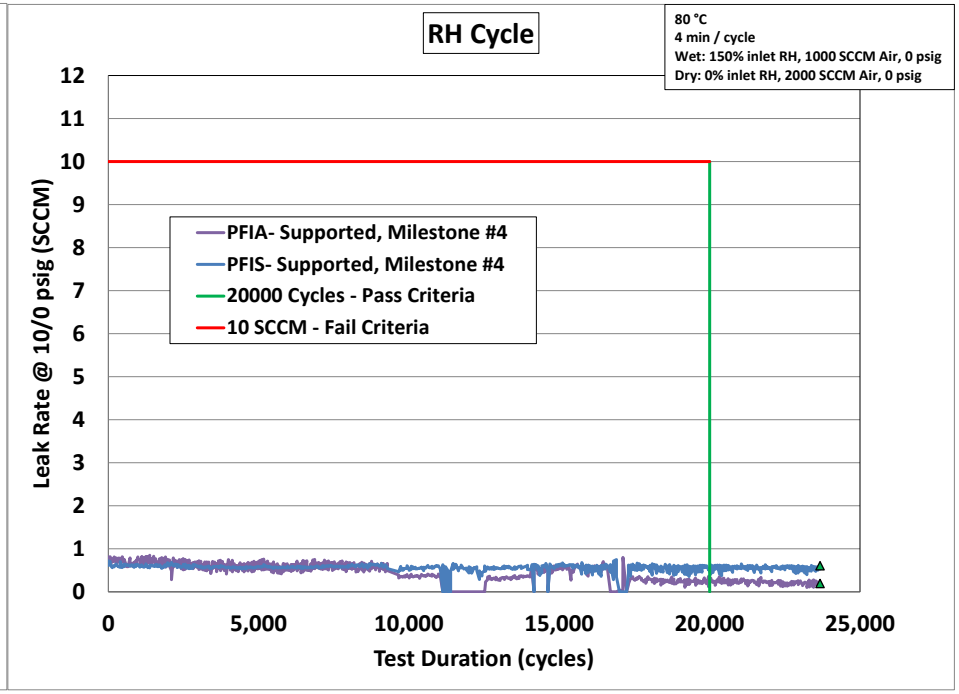
# Milestone #4 Go/No Go

| Membrane | Average lifetime (hrs) | 95% C.I. |
|---|---|---|
| 725EW - Supported Control | 894 | 226 |
| PFIA - SupportedMilestone #4 | 742 | 175 |

Accomplishments:

- High current performance targets met
- OCV target exceeded
- RH Cycle target exceeded



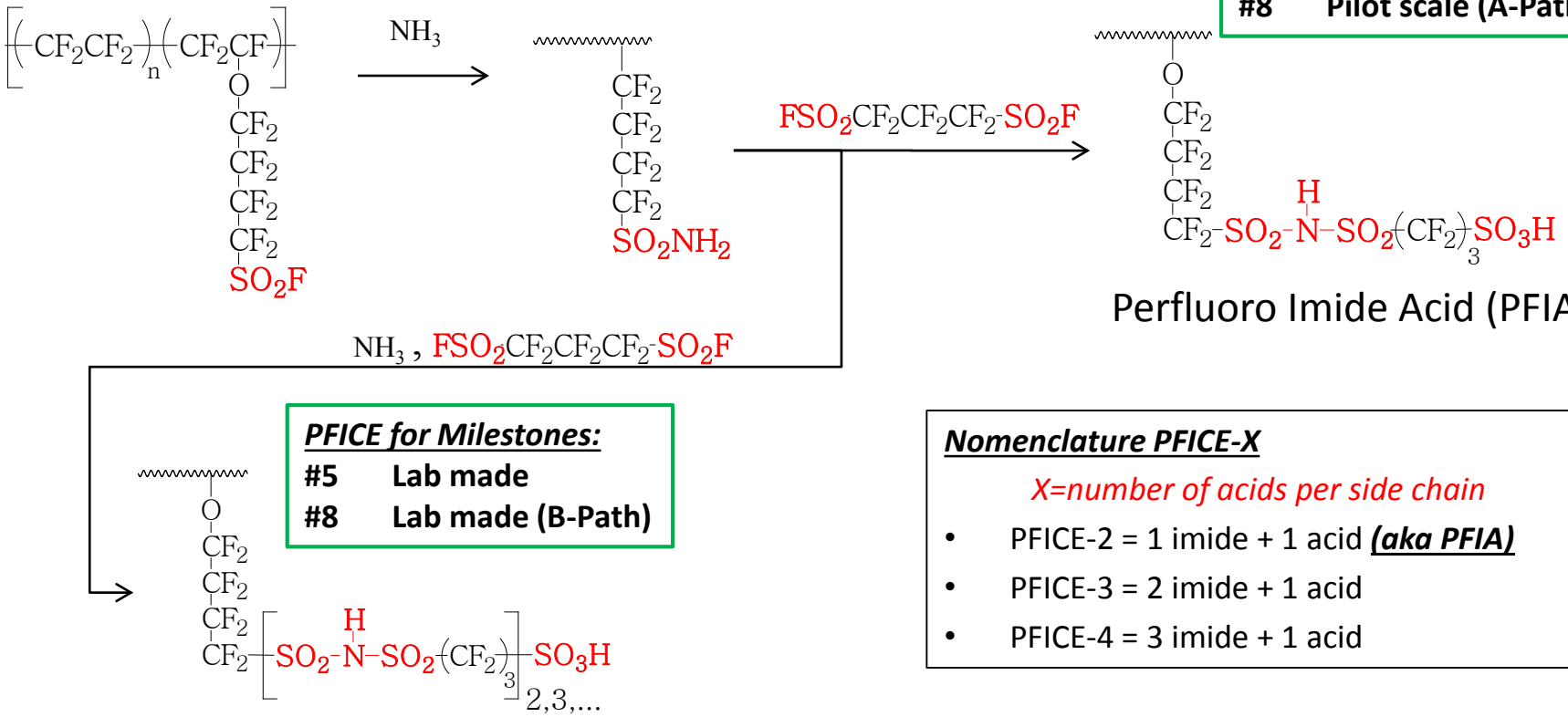

- 0.35mg/cm$^2$ total loading Pt
- ***Same level of peroxide scavenging additive as used in performance testing for both 725 control PFIA based membranes***
- Lifetimes can be increased with higher levels or different additives

- Membranes oriented with machine direction (MD) parallel to channels (most challenging configuration)
- Unsupported PFIA fails at 8,000 cycles
- Test terminated at 23,700 cycles due to equipment failure

# New Ionomers – Task 1

## Synthetic Approach

**PFIA for Milestones:**
- #4 Lab made
- #7 Pilot scale
- #8 Pilot scale (A-Path)




Perfluoro Imide Acid (PFIA)

**PFICE for Milestones:**
- #5 Lab made
- #8 Lab made (B-Path)

PFICE (Perfluoro Ionene Chain Extended)

**Nomenclature PFICE-X**

*X=number of acids per side chain*

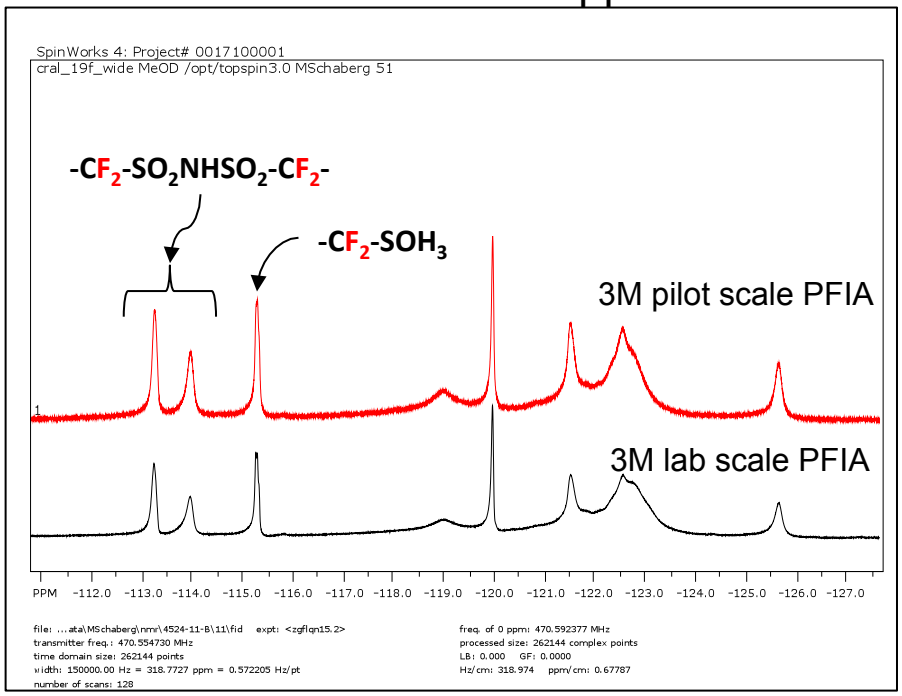
- PFICE-2 = 1 imide + 1 acid ***(aka PFIA)***
- PFICE-3 = 2 imide + 1 acid
- PFICE-4 = 3 imide + 1 acid

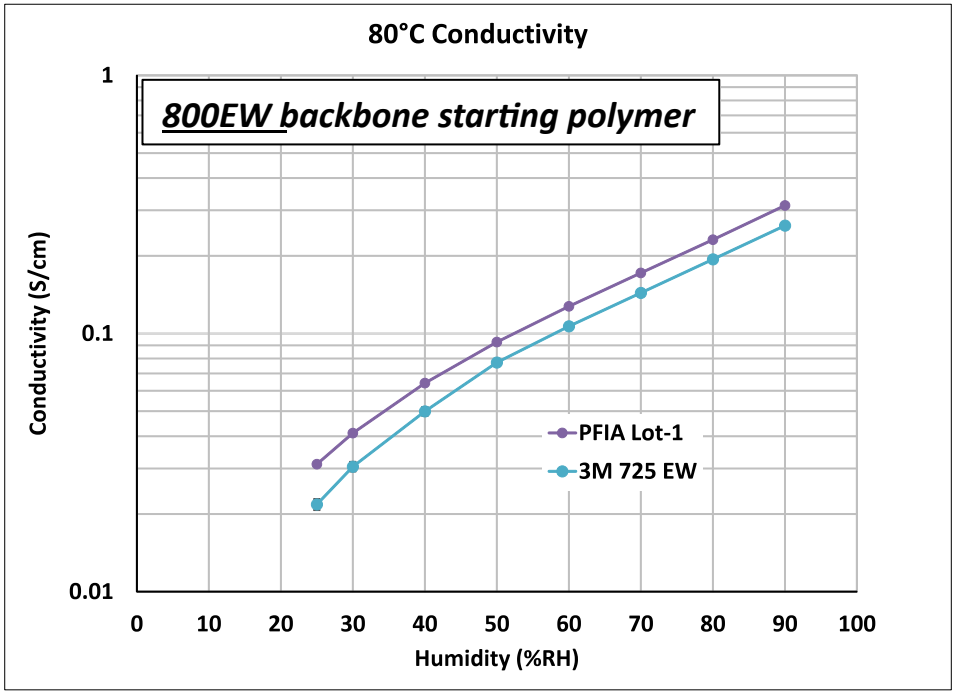
Accomplishments
- First pilot scale polymer completed (800 EW backbone starting polymer)
- A series of polymers with 2, 3, and 4 acid groups per side chain have been synthesized and characterized (700 EW backbone starting polymer)

# Task 1: Ionomer Development

***Pilot scale batch of ionomer completed in January of 2015***

- 800 EW backbone sulfonyl fluoride starting polymer
- PFIA Lot-1 EW titrated to be about 650 g/mol
- Ionomer used to fabricate membranes:
  - 20 um with no support and no additive for ionomer characterization
  - 14 um with support and additive as MS#7 candidate
  - 10 um with support and additive as MS#8 candidate



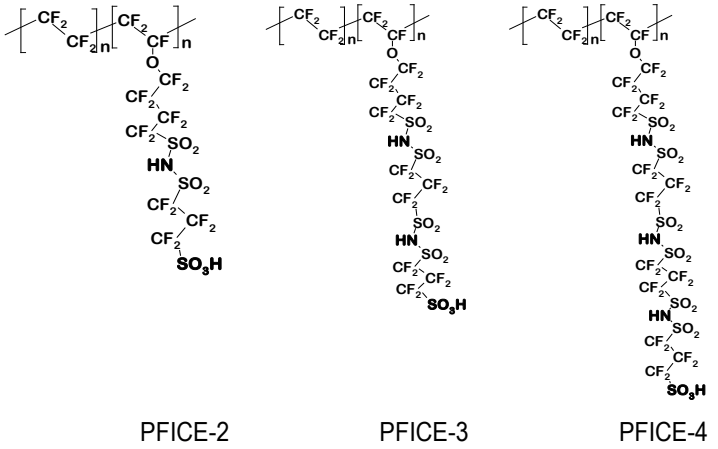



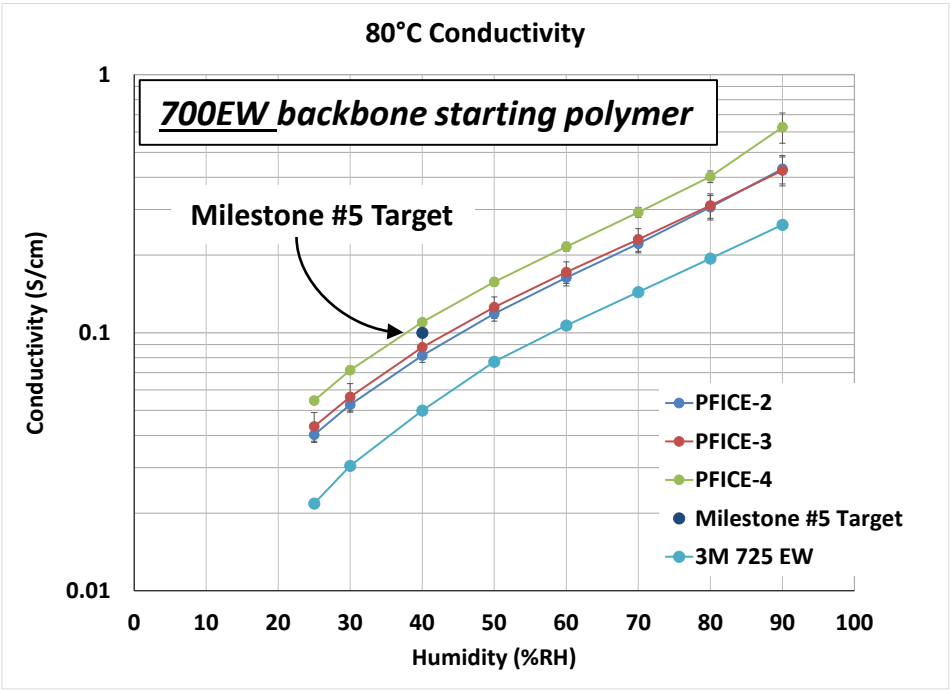

Conductivity for 20um membrane with no additives and no support

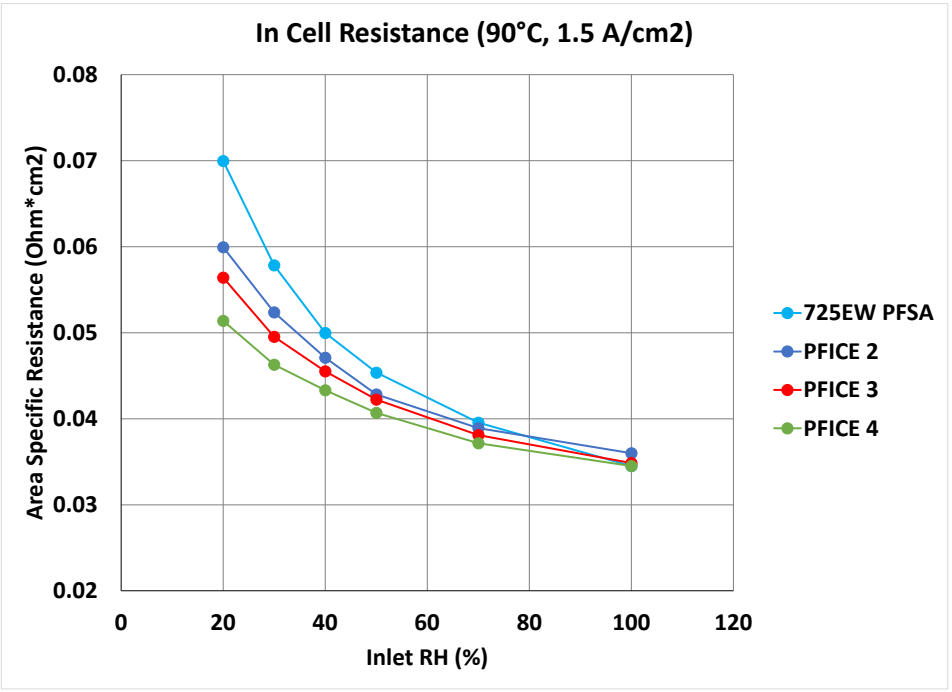
# Milestone #5

MS#5: Prepare at least one additional MASC polymer. Demonstrate conductivity of **0.1 S/cm** or higher at **80°C and <40%** RH. Evaluate in a supported membrane in Fuel Cell and ex situ tests.

| Ionomer | # Imides | Theoretical (EW) | Titration (EW) |
|---|---|---|---|
| PFICE-2 | 1 | 501 | 534 ± 7 |
| PFICE-3 | 2 | 431 | 475 ± 5 |
| PFICE-4 | 3 | 397 | 438 ± 3 |

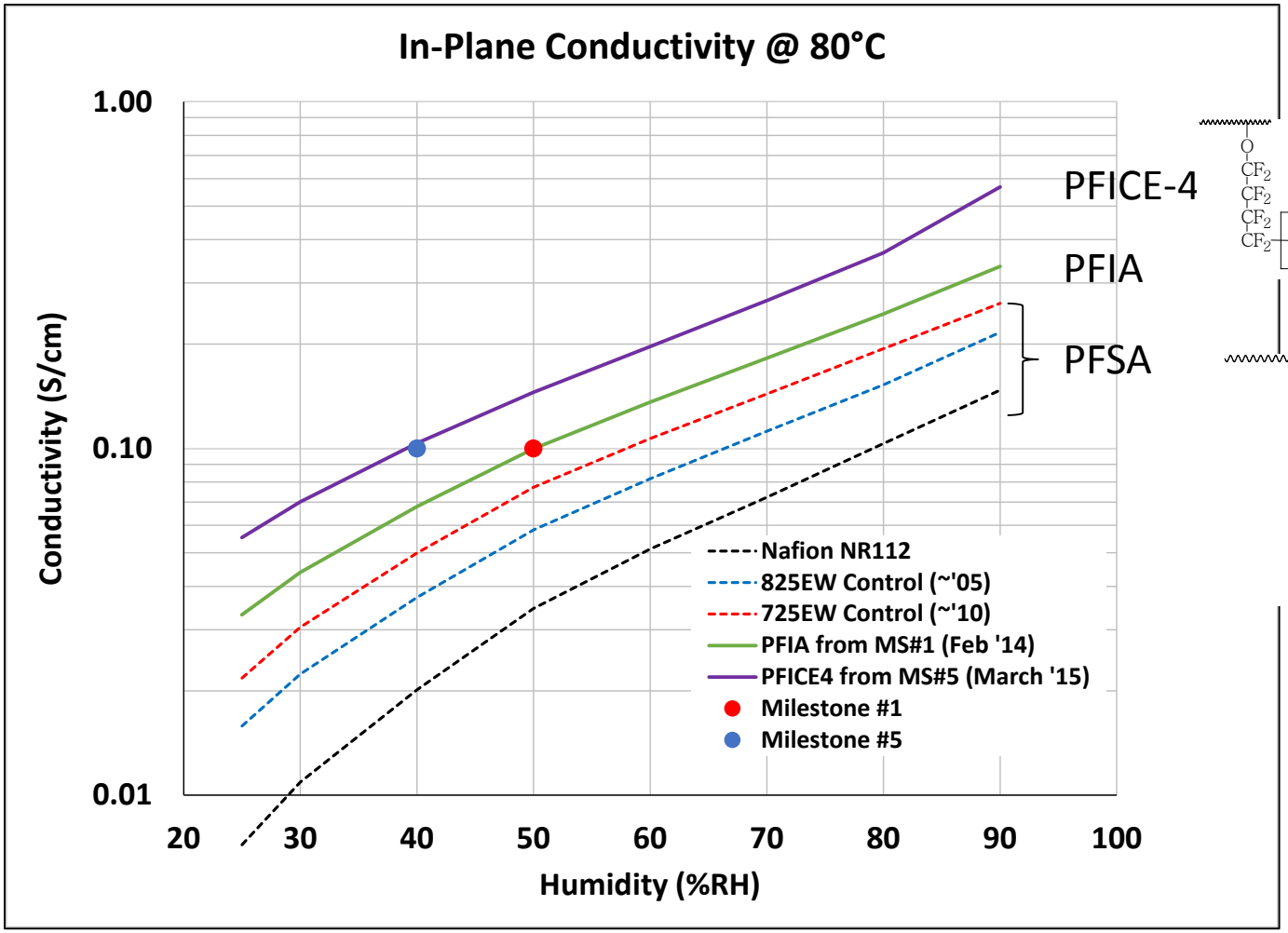



Accomplishment: Milestone #5 conductivity target met

# Milestones #1 & 5

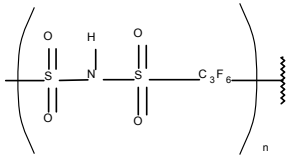



**Accomplishments:**

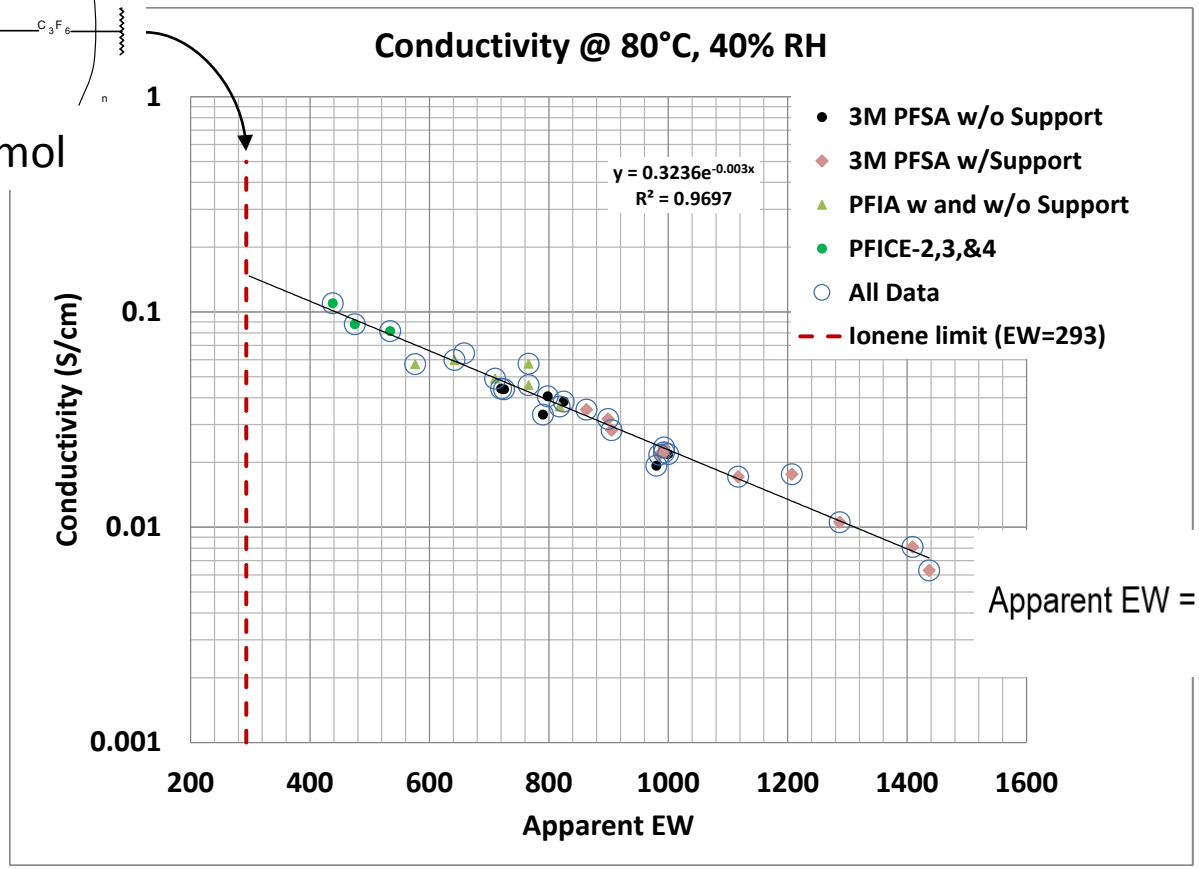
- State of the art conductivity improved by 5x at 80°C and 40% RH.
- 100mS/cm conductivity threshold moved from 80% to 40% RH compared to Nafion®.
- 100mS/cm conductivity threshold moved from 50% to 40% RH since the start of project.

# Milestone #5

In-Plane conductivity (4 point probe)



293 g/mol




$$\text{Apparent EW} = \frac{1}{\left(\frac{1}{EW}\right) * \left(\frac{(1\text{-}\% \text{ fiber})}{100}\right)}$$

**Accomplishments:**

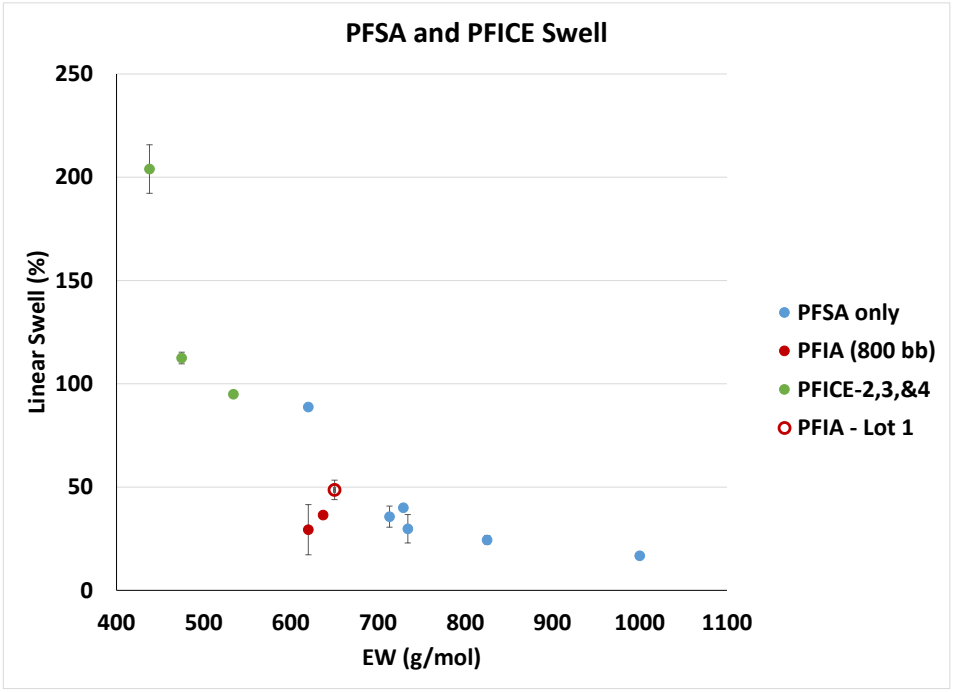
- Simple model establishes conductivity as a function of 'apparent' equivalent weight
- PFICE-4 conductivity is very close to 'ionone limit'. – Additional chain extension would provide little addition gains.

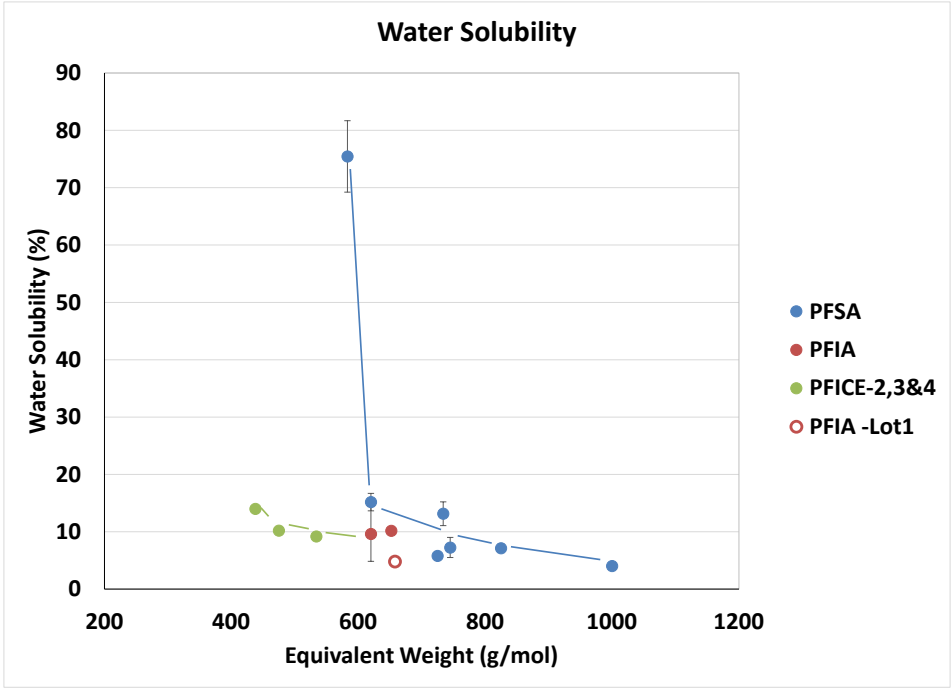
# Task 3: Ionomer and Membrane Testing

## Swell and Water Solubility

Samples boiled in water for 3hrs and measured at RT

Samples refluxed in Soxhlet extractor for 4 hrs

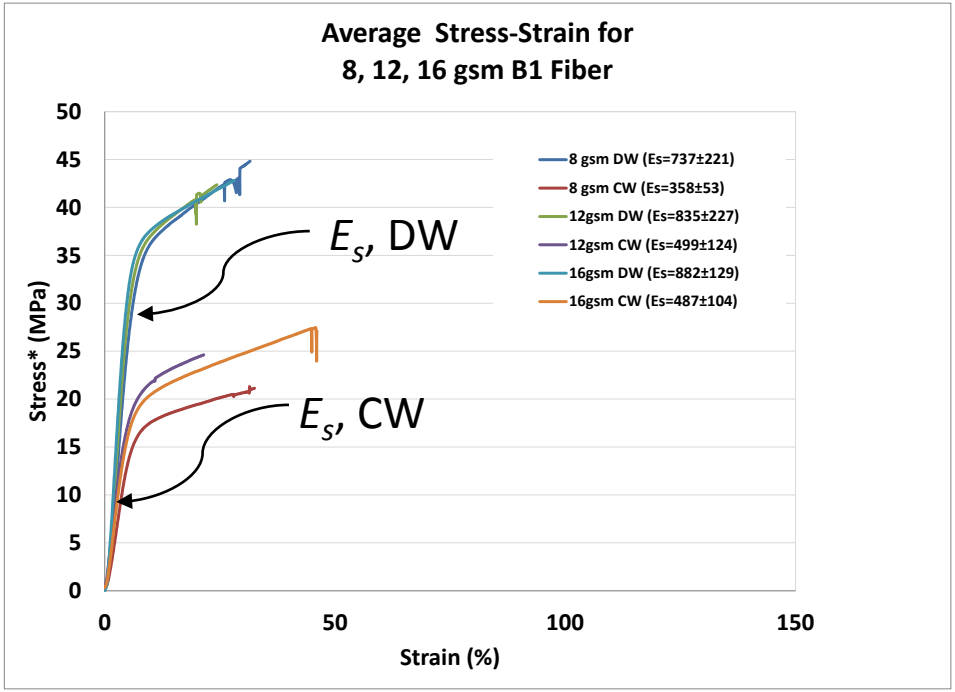


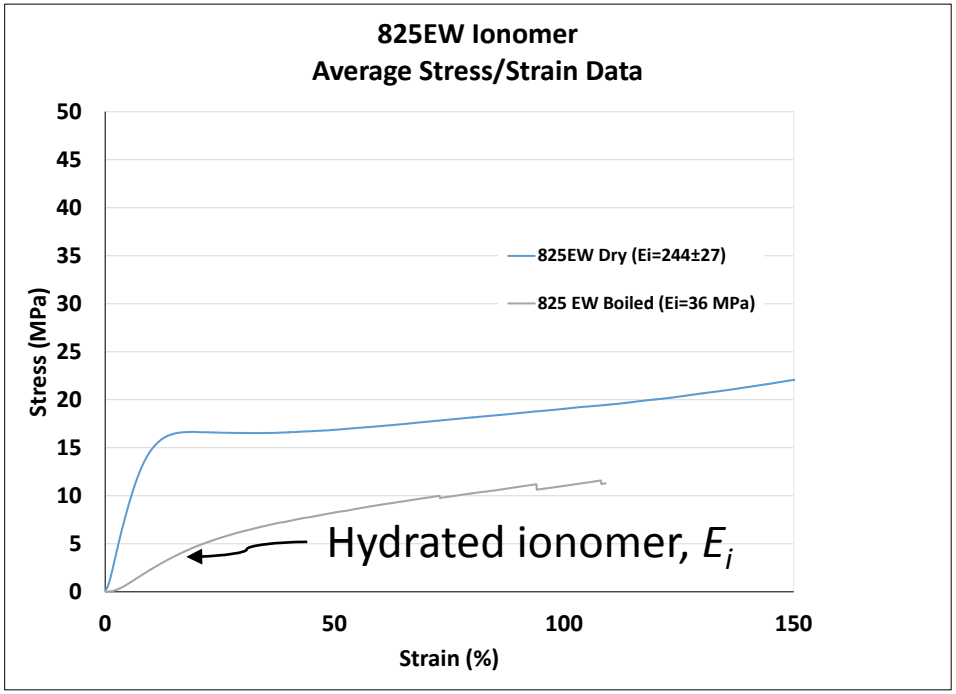




- Swell increases with decreased EW for all ionomers
- Water solubility is a key limiting factor in very low EW PFSAs
- PFIA and PFICE solubility defined primarily by polymer backbone
- PFICE polymers show low water solubility down to EW of 440 g/mol

# Task 2: Nanofiber Development

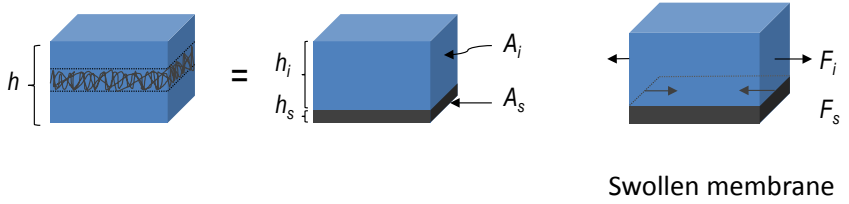
Modulus of hydrated ionomer and modulus of fiber support can be used to predict composite membrane swell




*Stress calculated from the condensed thickness of support fibers

$$\varepsilon_c = \frac{E_i*(1-f)*\varepsilon_i}{E_i*(1-f)+E_s*f}$$



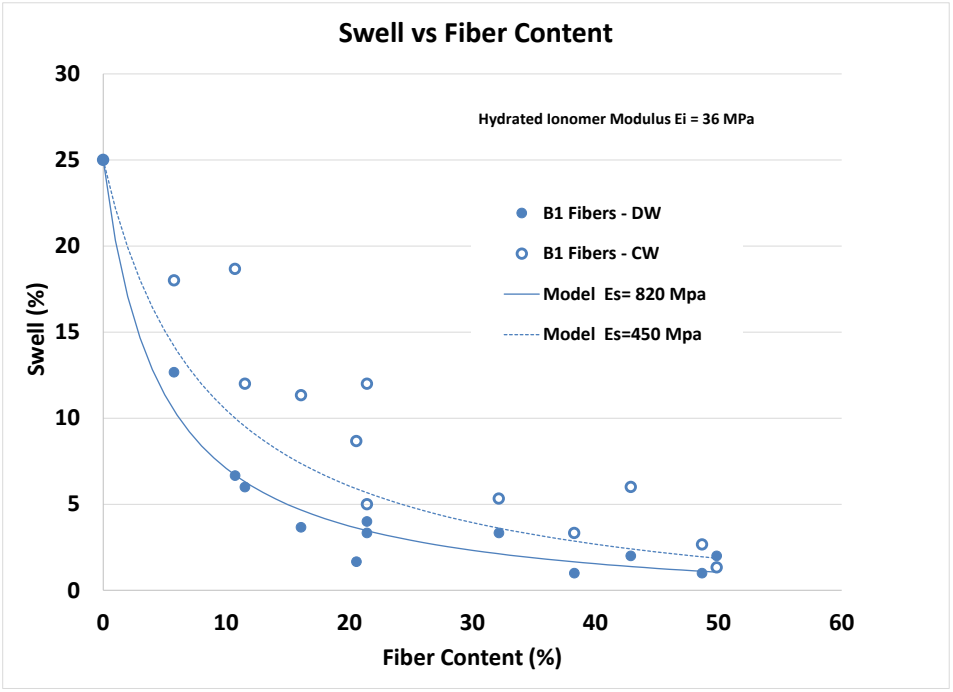

Swollen membrane

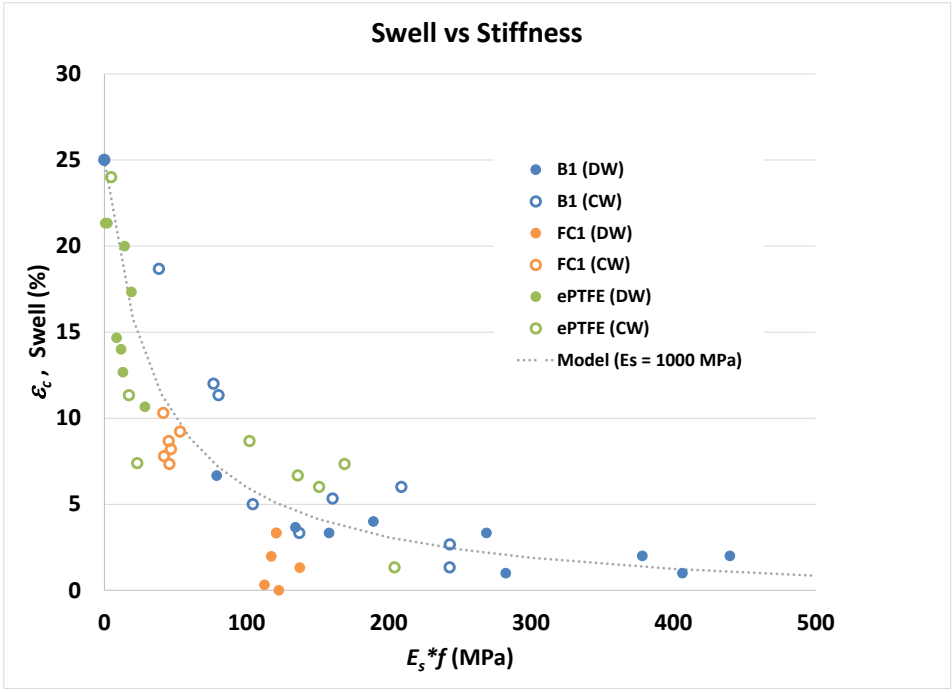
| | |
|---|---|
| $E_i$ | Modulus of ionomer at the wet condition |
| $E_s$ | Modulus of support at the dry condition |
| $\varepsilon_i$ | Swelling strain of the free-standing ionomer |
| $\varepsilon_c$ | Swell strain of the composite membrane |
| $f$ | Fiber fraction (vol%) |
| $h$ | Thickness |
| A | Area |
| $F_i$ | Force due to swelling ionomer |
| $F_s$ | Force of support resisting swell |

# Task 2: Nanofiber Development

## Swell Prediction Model

$$\varepsilon_c = \frac{E_i*(1-f)*\varepsilon_i}{E_i*(1-f)+E_s*f}$$




- Specific case where the fiber modulus is constant over a range of fiber fractions

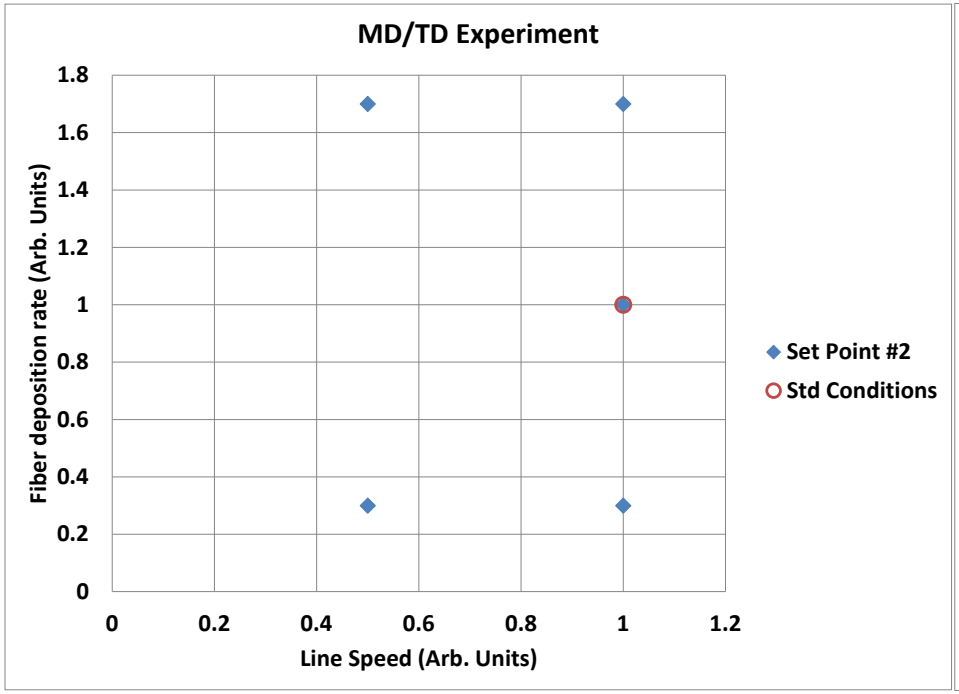
- General case where the fiber modulus and fiber fraction varies.
- $E_s*f$ represents a 'stiffness' factor

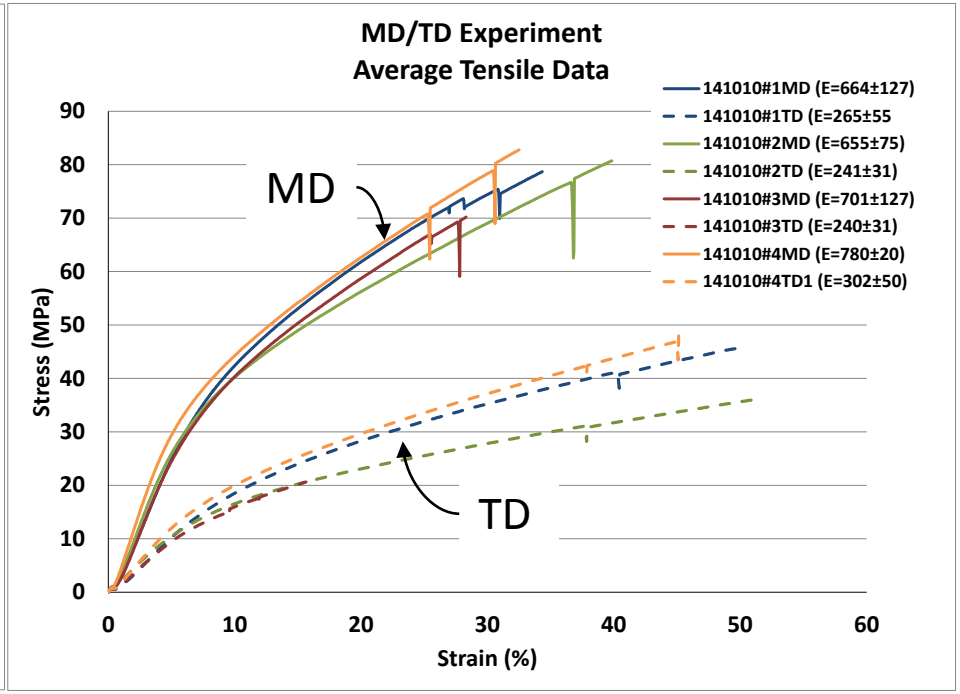
Accomplishment: Model allows for evaluating candidate nanofiber materials without the need for composite membrane fabrication and testing.

# Task 2: Nanofiber Development

Nanofiber experiments to reduce machine direction (MD) and transverse direction (TD) differences in mechanical properties

- Line speed varied between normal and ½ normal set point.
- Fiber deposition rate varied between 30% and 170% of normal condition.

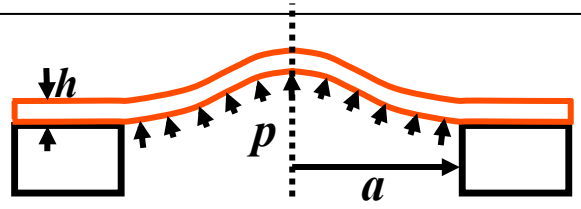



Accomplishment and result:

- Samples successfully made over a large process window.
- MD/TD differences remain despite process changes.

# Task 3: Ionomer and Membrane Testing




$$\sigma = \frac{1.724}{4}\left(\frac{E(t,T,RH)p^2a^2}{h^2}\right)^{1/3}$$

where

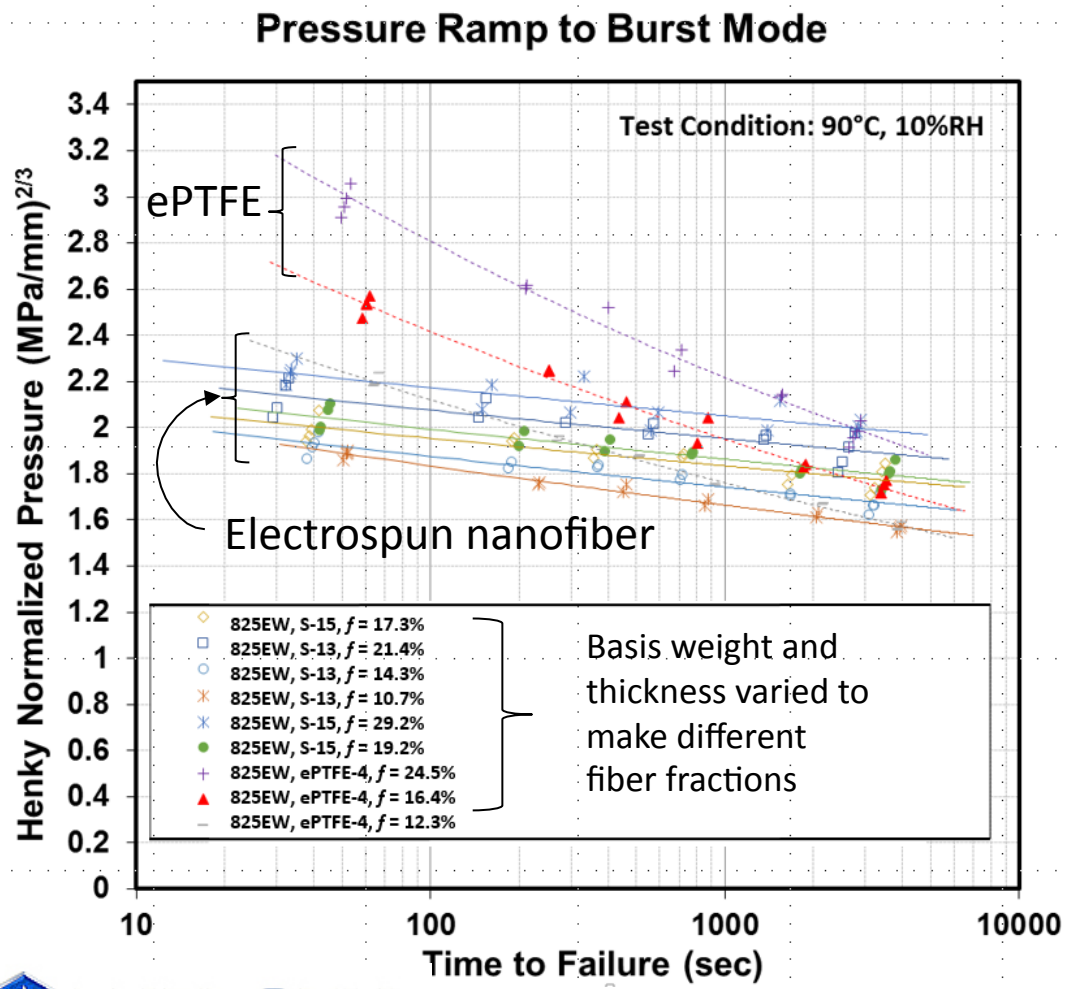
$\sigma$ is the stress at the center of blister;

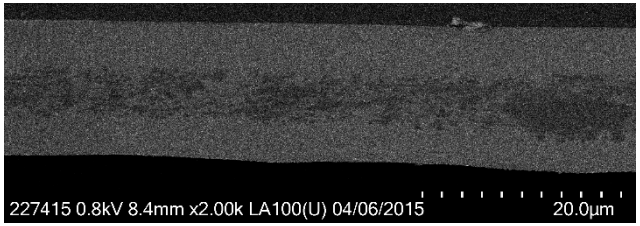
E is the relaxation modulus;

p is the applied pressure;

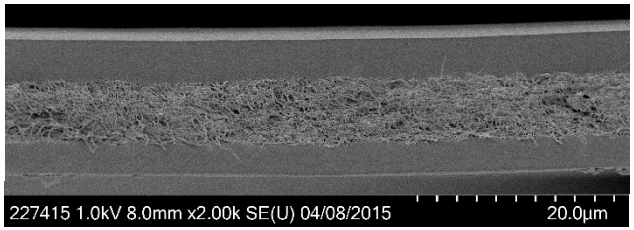
a is the radius; and

h is the thickness of blister.




Electrospun nanofiber supported membrane (~16.1% fiber vol fraction)

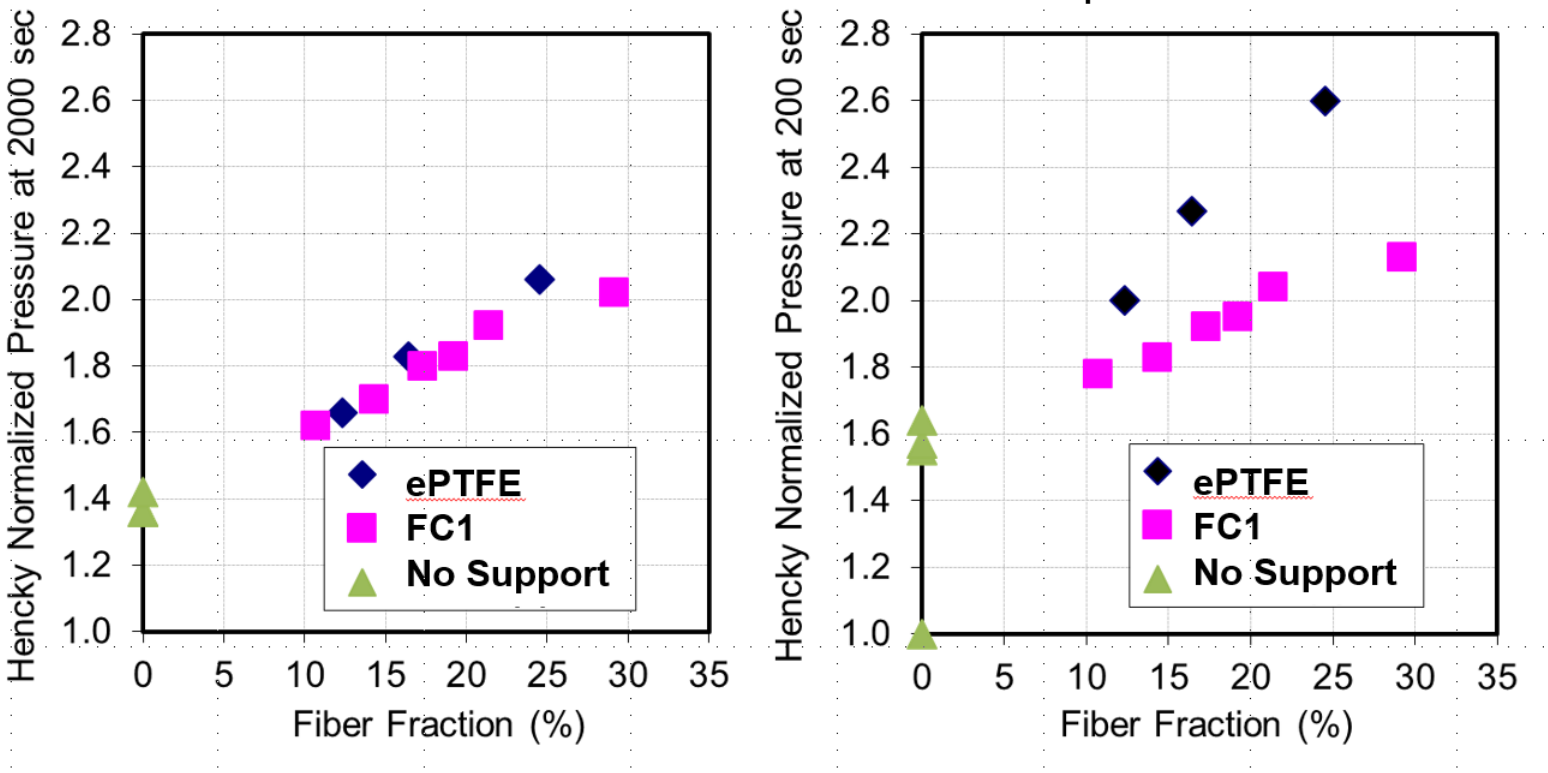



ePTFE supported membrane (~16.4% fiber vol fraction)

# Task 3: Ionomer and Membrane Testing

## Effect of Fiber Content on Blister Strength

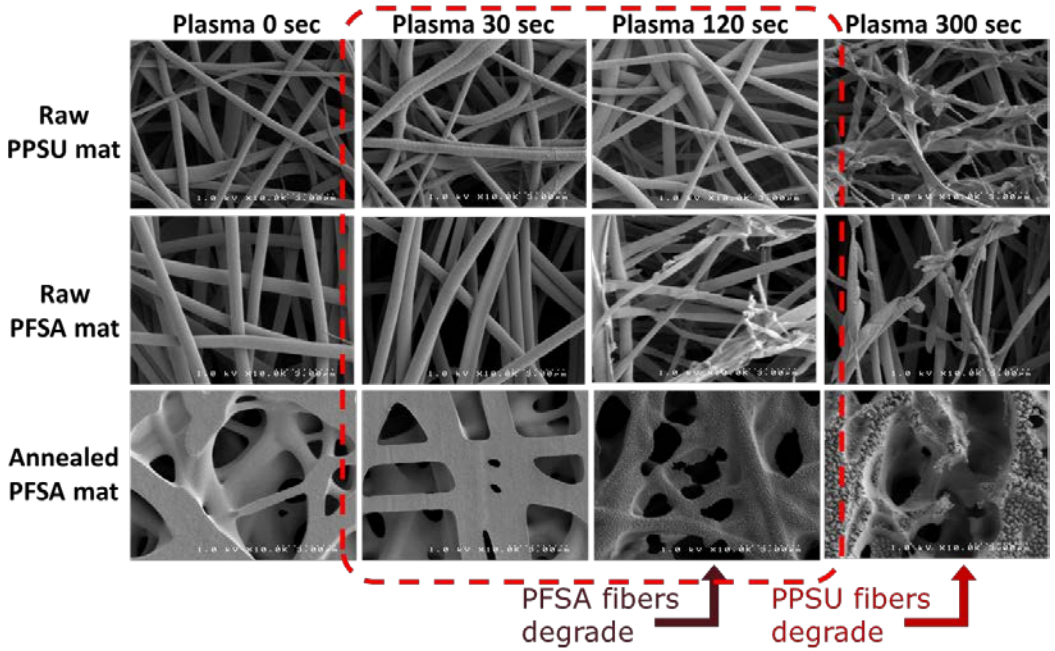
All membranes annealed at the same temperature




- Linear relationship between blister strength and fiber fraction for both support types
- At longer times, PEMs with ePTFE and FC1 nanofiber supports show similar burst strength
- At shorter times, PEMs with ePTFE show higher burst strength than those with FC1 nanofiber supports

# Milestone #6

MS#6: Prepare dense electrospun films with and without surface treatment of the support polymer with a maximum void fraction of <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.

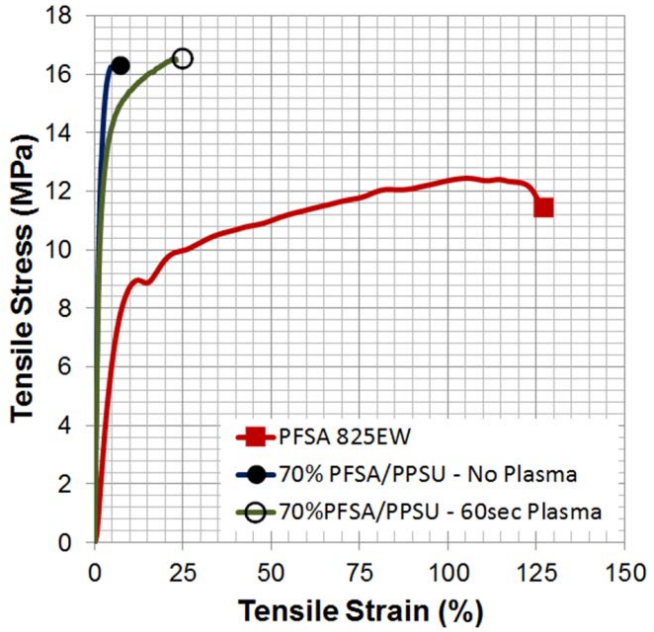



PPSU: polyphenylsulfone

Electrospun mats from PPSU and from PFSA, were subjected to oxygen plasma (Reactive Ion Etch RF 100W) for various periods (0-300 sec). Destruction of the PPSU mat is evident after 300 sec (fiber surface roughening after 30 sec) and PFSA mat degrades after 120 sec.

# Milestone #6

While plasma treated membrane showed somewhat higher conductivity its modulus was lower than that of the untreated membrane. Also the lateral swelling of the treated membrane was higher than that of untreated membrane (7.9% vs. 6.5%).




| PFSA content [vol%] | Plasma 100W | Gravimetric Swelling (wt%) | Lateral Swelling [%] | In-Plane Cond [S/cm] | Modulus (Air Dry) [GPa] |
|---|---|---|---|---|---|
| 100 | - | 58.2 | 28.9 | 0.120 | 0.17 |
| 70 | - | 39.2 | 6.5 | 0.071 | 0.78 |
| | 60 sec | 45.7 | 7.9 | 0.079 | 0.56 |

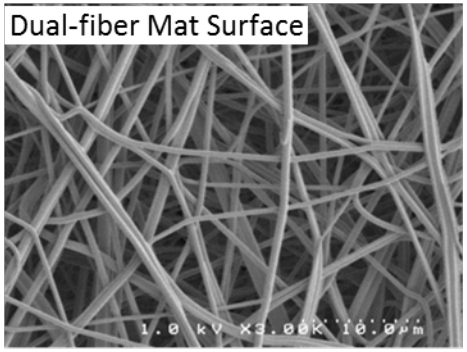
Dual fiber PFSA/PPSU (70vol% PFSA) were exposed to oxygen plasma for 30 sec each side and then densified (hotpressed at 160°C and annealed at 160°C for 1 hr). The resultant membranes were treated with boiling 1M $H_2SO_4$ for 1 hr. and then with boiling water for another 1 hr. The basic membrane characteristics are shown below.

Accomplishments

- Initial surface treatment studies have been completed.
- No surface treatment is selected at this time.
- Additional work is planned beyond Milestone #6 timing.

# Task 2.2 Membrane Development and Fabrication

Mixed-fiber mats were prepared by concurrent electrospinning PFIA and polyamide-imide (PAI, Torlon®) solutions on the same target. The mats contained 60-65vol% PFIA. The mats were densified by exposure to solvent vapor (methanol, DMF) and then annealed for 15 min. at 200°C.




**Mat densification (pore closure, ionomer annealing)**

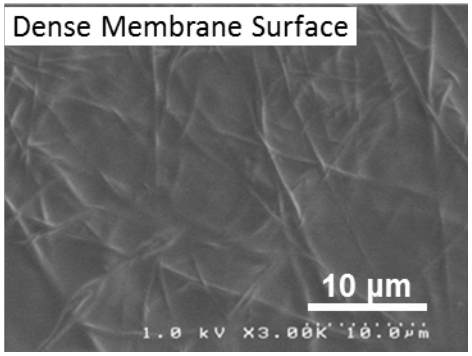
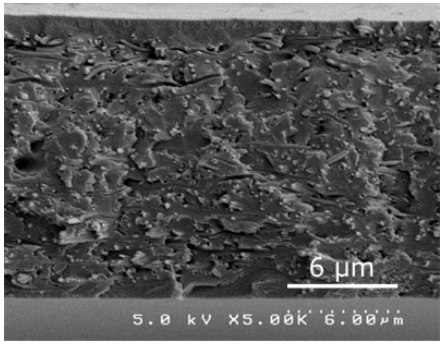



**Photo of two PFSA/PAI membranes (85vol% PFSA)**



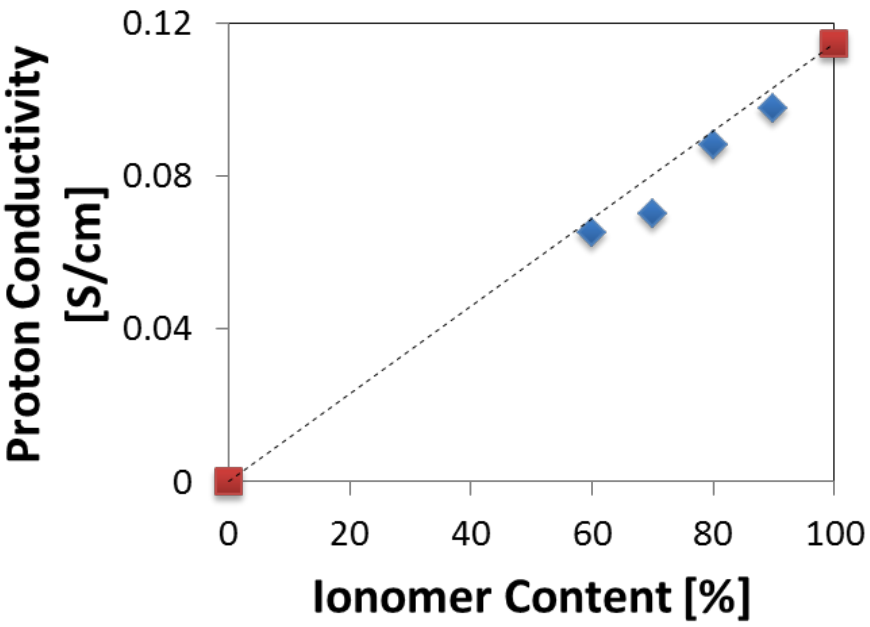

**SEM micrograph of PFSA/PAI membrane cross-section**

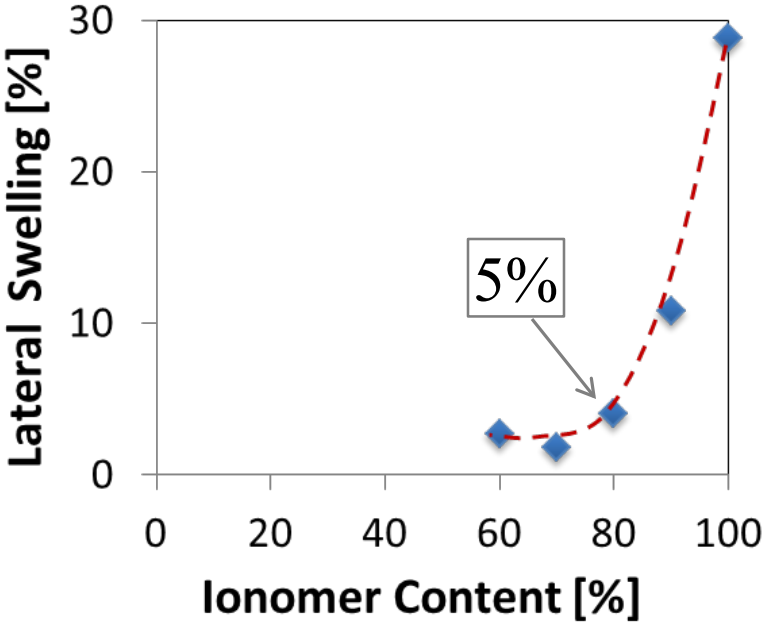
- The resultant PFIA/PAI membranes had expected proton conductivity (ca. 60% that of pristine PFIA membrane) but dramatically reduced in-plane swelling (less than 5% compared to over 40% for pristine PFIA membrane film).
- Wet dual fiber composite membranes were significantly stronger than wet pristine PFIA films, which easily broke into pieces during handling.

# Task 2.2 Membrane Development and Fabrication

## *Dual-Fiber PFSA/PAI Membranes*

- Proton conductivity of the composite PFSA/PAI membranes was linearly dependent on PFSA content and followed the law of mixtures.
- The water swelling showed non-linear dependence; membranes with PFSA content below 80vol% had lateral swelling of 5% and less.



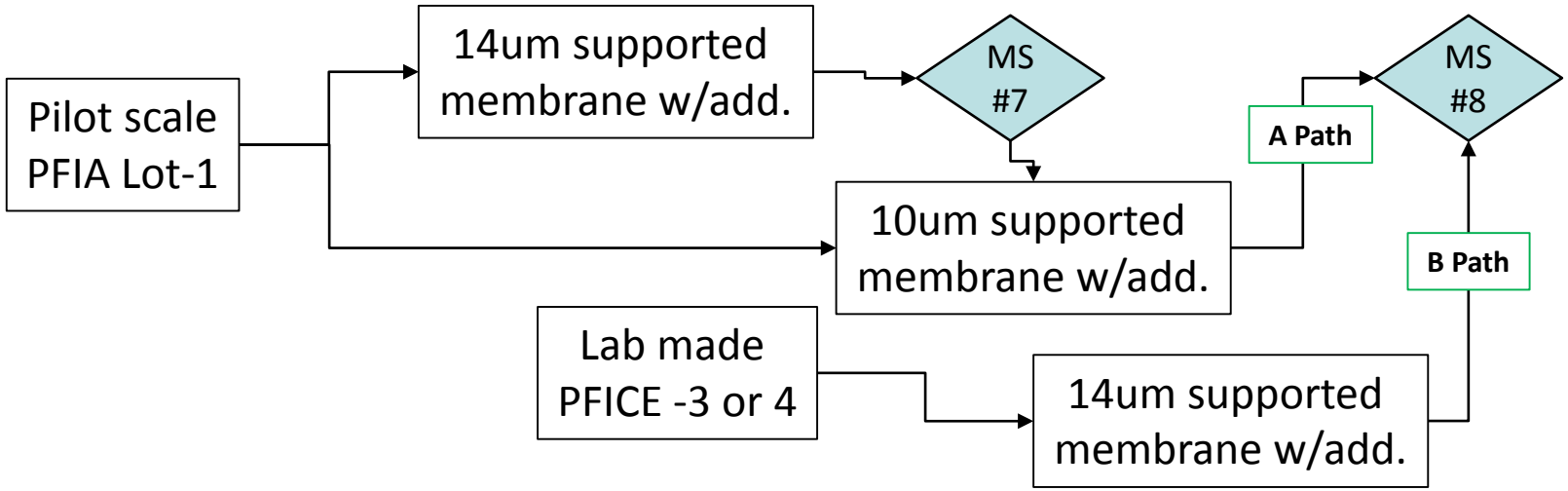




**Annealing conditions: 170°C for 2 hr**

# Milestones #7 & #8

***MS#7:*** Prepare an ionomer formulation (ionomer, stabilizing additive) with optimum performance and durability…to be used for development of the supported membrane described in milestone Q8.

***MS#8:*** Produce membrane…which meets all of the 2020 membrane milestones in Table 3.4.12….in the DOE Fuel Cell Technologies Office Multi-Year Research, Development and Demonstration Plan, section 3.4, update July 2013.



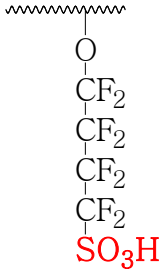

| Membrane | Composition | Fiber fraction (vol%) | Predicted swell - DW | Predicted swell - CW | Estimated ASR (Ohm*cm2) |
|---|---|---|---|---|---|
| MS#7 | 14 um, 4.3 gsm S-15, w/Add | 17.3 | 3.3 | 7.6 | **24.8** |
| MS#8 candidate | 10um, 3.2 gsm S-16, w/Add | 18.0 | 3.1 | 7.3 | **18.0** |
| DOE Req. Control | 14um, 5.4 gsm ePTFE, w/Add | 17.5 | 13.3 | 2.7 | **25.6** |

# Ionomer Cost

## *PFIA Manufacturing Cost Issues:*

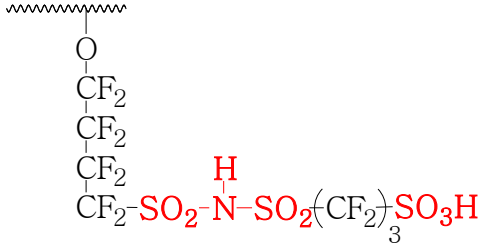
- 3M does not disclose manufacturing cost for any product.
- Lower EW ionomers will always be more expensive than higher EWs due to the higher cost of the functional monomer.
- The bissulfonyl fluoride is similar in cost to the 3M monomer.
- Material cost expected to be the major contributor to both PFSA and PFIA at production volumes.

| $CF_2=CF_2$ | << | $CF_2=CFO-(CF_2)_4-SO_2F$ | ~ | $FO_2S-(CF_2)_3-SO_2F$ |
|---|---|---|---|---|
| $ | | $$$ | | $$$ |




Traditional PFSA

$CF_2=CFO-(CF_2)_4-SO_2F$




PFIA

½ $CF_2=CFO-(CF_2)_4-SO_2F$
½ $FO_2S-(CF_2)_3-SO_2F$

# Summary

*Data for single membrane construction shown each column*

| Characteristic | Units | 2017 & 2020 Targets | 725 EW (20um) | 725EW-S (14um) | PFIA (20um) | MS#4 PFIA-S (14 um) | Associated Milestone |
|---|---|---|---|---|---|---|---|
| Maximum oxygen cross-over | mA / $cm^2$ | 2 | <0.5 | <0.5 | <0.5 | <0.5 | #8 |
| Maximum hydrogen cross-over | mA / $cm^2$ | 2 | 1.5±0.1 | 1.1±0.3 | 0.9±0.3 | 1.1±0.2 | #8 |
| Area specific proton resistance at: | | | | | | | |
| 120°C and water partial pressure of 40 kPa | Ohm $cm^2$ | 0.02 | 0.068[a] | [b] | 0.042[a] | [b] | #8 |
| 80°C and water partial pressure of 25 kPa | Ohm $cm^2$ | 0.02 | 0.027 | 0.047 | 0.020 | 0.027 | #1, #8 |
| 30°C and water partial pressures up to 4 kPa | Ohm $cm^2$ | 0.03 | | | 0.02 | | #8 |
| -20°C | Ohm $cm^2$ | 0.2 | | | 0.1 | | #8 |
| | | | | | | | |
| Minimum electrical resistance | Ohm $cm^2$ | 1,000 | 16,000[c] | 5,600[c] | 6,900[c] | 5,700[c] | #8 |
| | | | | | | | |
| Cost | $ / $m^2$ | 20 | n/a | n/a | n/a | n/a | #8 |
| Durability | | | | | | | |
| Mechanical | Cycles with <10 sccm crossover hours | 20,000 | 8,300 | >20,000 | 12,000 | >23,000 | #4,#7,#8 |
| | | | | | | | |
| Chemical | hrs | >500 | 47 | 894 | - | 742 | #4,#7,#8 |

a. Calculated from through plane bulk measurements
b. In-cell test method under development
c. Data provided by GM

# Future Work

- Remainder of FY2015
  - Task 1, 2, & 3
    - Additional lab batches of PFICE 3 or 4 ionomer (Q3 & Q4 2015)
    - Second pilot scale batch of PFIA ionomer (Q3 2015)
    - Continued nanofiber development (ongoing)
  - Milestone #7 (June ‘15)
    - Initiate durability testing (Q3, 2015)
    - Complete performance testing (Q3, 2015)
  - Milestone #8 Go/No Go (Sept. ‘15)
    - Initiate durability testing on 10um pilot scale PFIA with support and additive (Q3 2015)
    - Fabricate supported membrane made with PFICE 3 or 4 (Q4 2015)
    - Select PFIA or PFICE based membrane to meet milestone targets(Q4 2015)
- FY2016
  - Milestone #9 (Dec. ‘15)
    - Fabricate sufficient quantities of membrane for stack testing (Q1 2016)
  - Milestone #10 (March ‘16)
    - Task 5; Fabricate MEAs and Initiate stack testing (Q2 2016)

# Technical Back-up Slides

# Full Milestone Table

| MS ID | Full Milestone | Date |
|---|---|---|
| 1 | 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 <50% RH. | January 9, 2014 |
| 2 | Identify one or more polymer systems for further development in a nanofiber support that provides a membrane with x-y swelling of < 5% after boiling in water. | April 8, 2014 |
| 3 | Develop electrospinning conditions for one or more 3M ionomers that provides fiber diameter of <1 micron. | May 22, 2014 |
| 4 - Go/No-Go | 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 95C, 50%RH, 150 kPa inlet pressure, and 0.4 mg/cm2 total pgm catalyst loading). | October 16, 2014 |
| 5 | Prepare at least one additional MASC polymer. Demonstrate conductivity of 0.1 S/cm or higher at 80°C and <40% RH. Evaluate in a supported membrane in Fuel Cell and ex situ tests. | March 6th, 2015 |
| 6 | Prepare dense electrospun films with and without surface treatment of the support polymer with a maximum void fraction of <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. | April 3rd, 2015 - ongoing |
| 7 | Prepare an ionomer formulation (ionomer, stabilizing additive) with optimum performance and durability that provides >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. | July 1, 2015 |
| 8 - Go/No-Go | 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. | October 1, 2015 |
| 9 | 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) | January 1, 2016 |
| 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. | April 1, 2016 |
| 11 | Begin post mortem analysis of MEAs to determine failure mode. | July 1, 2016 |
| 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. | October 1, 2016 |

# Nanofiber Fabrication Task 2.1

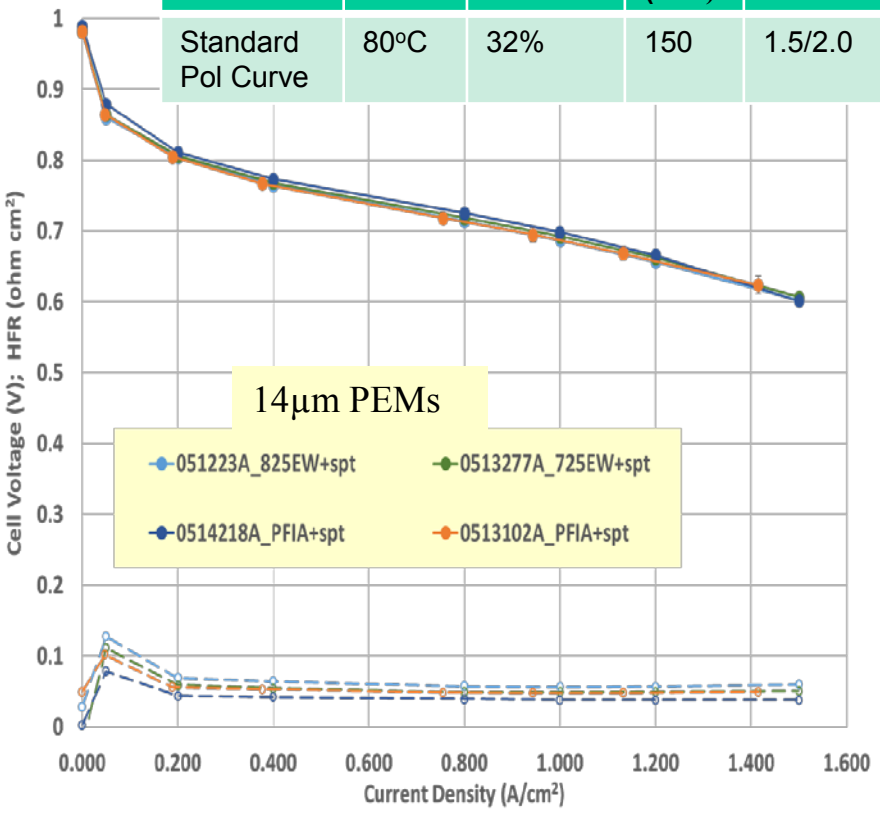
| *Nanofiber Samples* | | | | | |
|---|---|---|---|---|---|
| Coded Sample | Form | Coded polymer | Coded Source | Basis weight (g/m2) | Objective |
| Q1 and Q2 samples | | | | | |
| S1 | roll | B1 | P1 | 4.3 | Control |
| S2 | roll | B2 | P1 | 3.2 | Improved tear strength |
| S3 | roll | B2 | P1 | 4.3 | Improved tear strength |
| S4 | test patch | FC3 | L2 | n/a | Electrospining feasibility |
| S5 | test patch | FC4 | L2 | n/a | Electrospining feasibility |
| S6 | test patch | FC5 | L2 | n/a | Electrospining feasibility |
| S7 | test patch | FC6 | L2 | n/a | Electrospining feasibility |
| S8 | roll | HC3 | P1 | 4.3 | Modulus study |
| S9 | roll | FC1 | P1 | 4.3 | Modulus study |
| S10 | roll | FC1 | P1 | 3.2 | Modulus study |
| S11 | sheet | FC3 | L1 | 5 | Improved tear strength |
| S12 | sheet | FC3 | L1 | 5 | Improved tear strength |
| S13 | sheet | HC2 | V | 5.7 | Modulus study |
| S14 | sheet | HC2 | V | 14.2 | Modulus study |
| Q3 samples | | | | | |
| S15 | roll | FC1 | P1 | 4.3 | New polymer |
| S16 | roll | FC1 | P1 | 3.2 | New polymer |
| S17 | sheet | H4 | L3 | 4.0 | New polymer |
| S18 | sheet | FC3 | P1 | 4.1 | New polymer |
| S19 | sheet | FC4 | P1 | 4.2 | New polymer |
| S20 | sheet | B1 | P1 | 4.4 | New process |
| Q4 Samples | | | | | |
| S21 | sheet | FC1 | P2 | 5.02 | MD/TD (alternate supplier) |
| S22 | sheet | HC1 | P2 | 4.33 | MD/TD (alternate supplier) |
| Q5 Samples | | | | | |
| S23 | roll | ePTFE-1 | P3 | 2.25 | ePTFE Comparison |
| S24 | roll | ePTFE-2 | P3 | 5.66 | ePTFE Comparison |
| S25 | roll | ePTFE-3 | P3 | 6.33 | ePTFE Comparison |
| S26 | roll | FC1 | P1 | 4.4 | MD/TD experiment |
| S27 | roll | FC1 | P1 | 4.28 | MD/TD experiment |
| S28 | roll | FC1 | P1 | 4.36 | MD/TD experiment |
| S29 | roll | FC1 | P1 | 4.39 | MD/TD experiment |
| S30 | roll | ePTFE-4 | P3 | 5.40 | ePTFE Comparison |

| | | | |
|---|---|---|---|
| Polymer Codes | HC = Hydrocarbon | Source Codes | L = Lab |
| | FC = Fluorocarbon | | P = Pilot or production line |
| | B = Blend | | V = Vanderbilit |

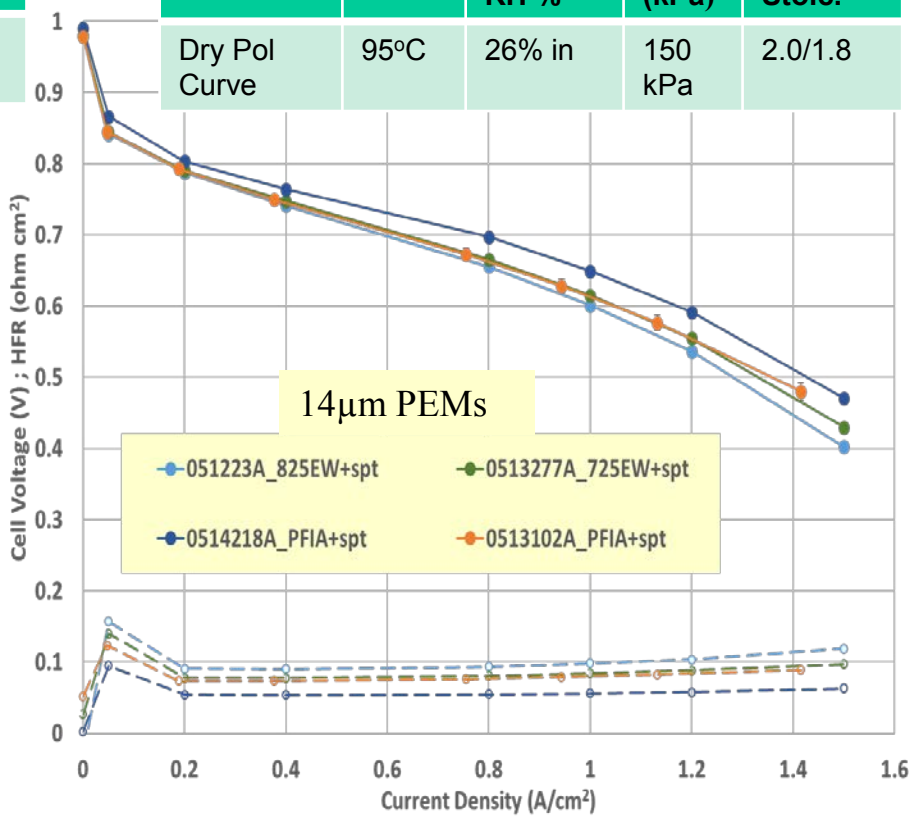
- Rolls of electrospun nanofibers are typically 100 meters long by 25 cm wide
- Sheet samples are typically 10cmx 10cm up to 22cm x 28 cm

# Fuel Cell Performance

| Protocol | Cell T | An / Ca RH % | P (kPa) | H2/Air Stoic. |
|---|---|---|---|---|
| Standard Pol Curve | 80°C | 32% | 150 | 1.5/2.0 |

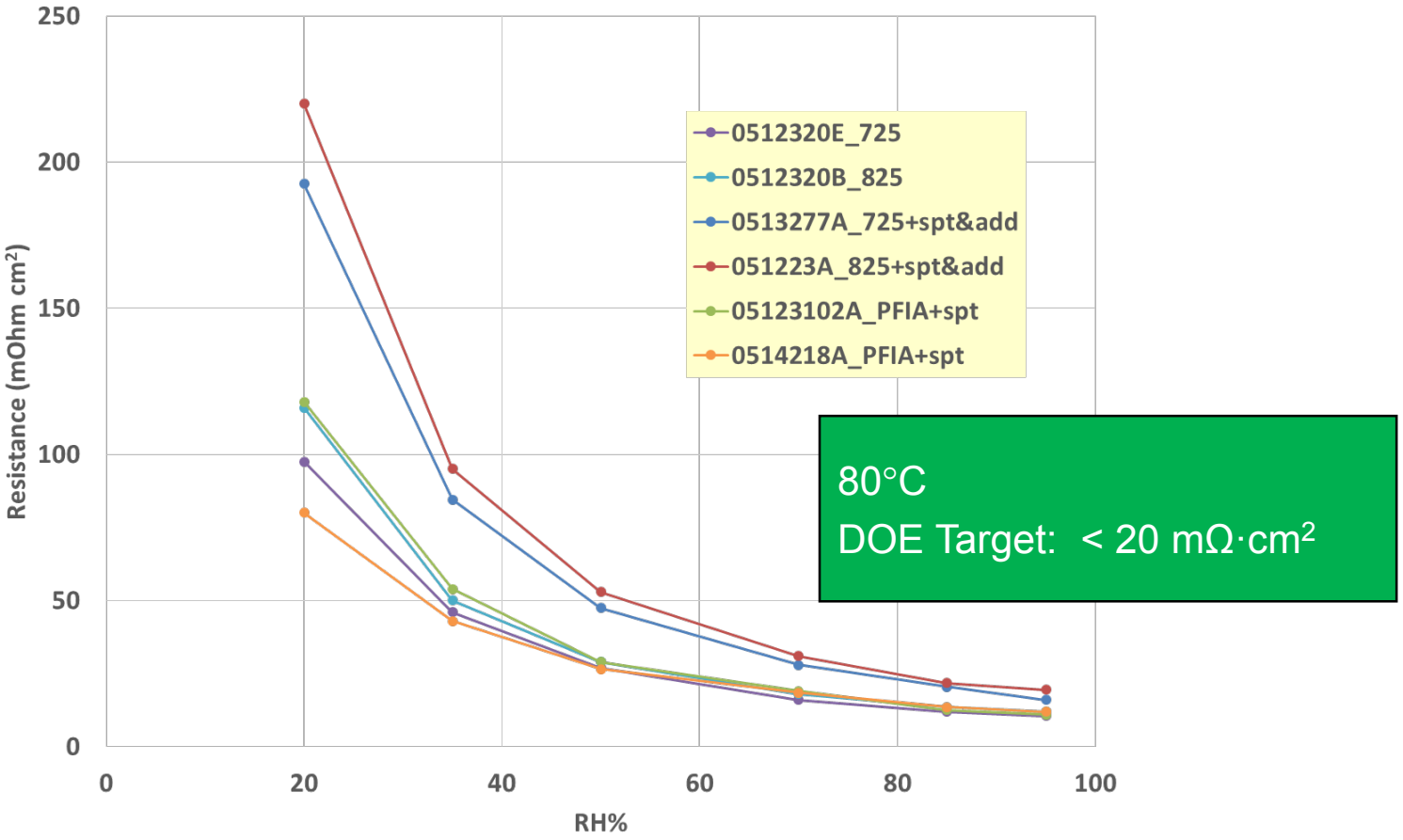



| Protocol | Cell T | An / Ca RH % | P (kPa) | H2/Air Stoic. |
|---|---|---|---|---|
| Dry Pol Curve | 95°C | 26% in | 150 kPa | 2.0/1.8 |



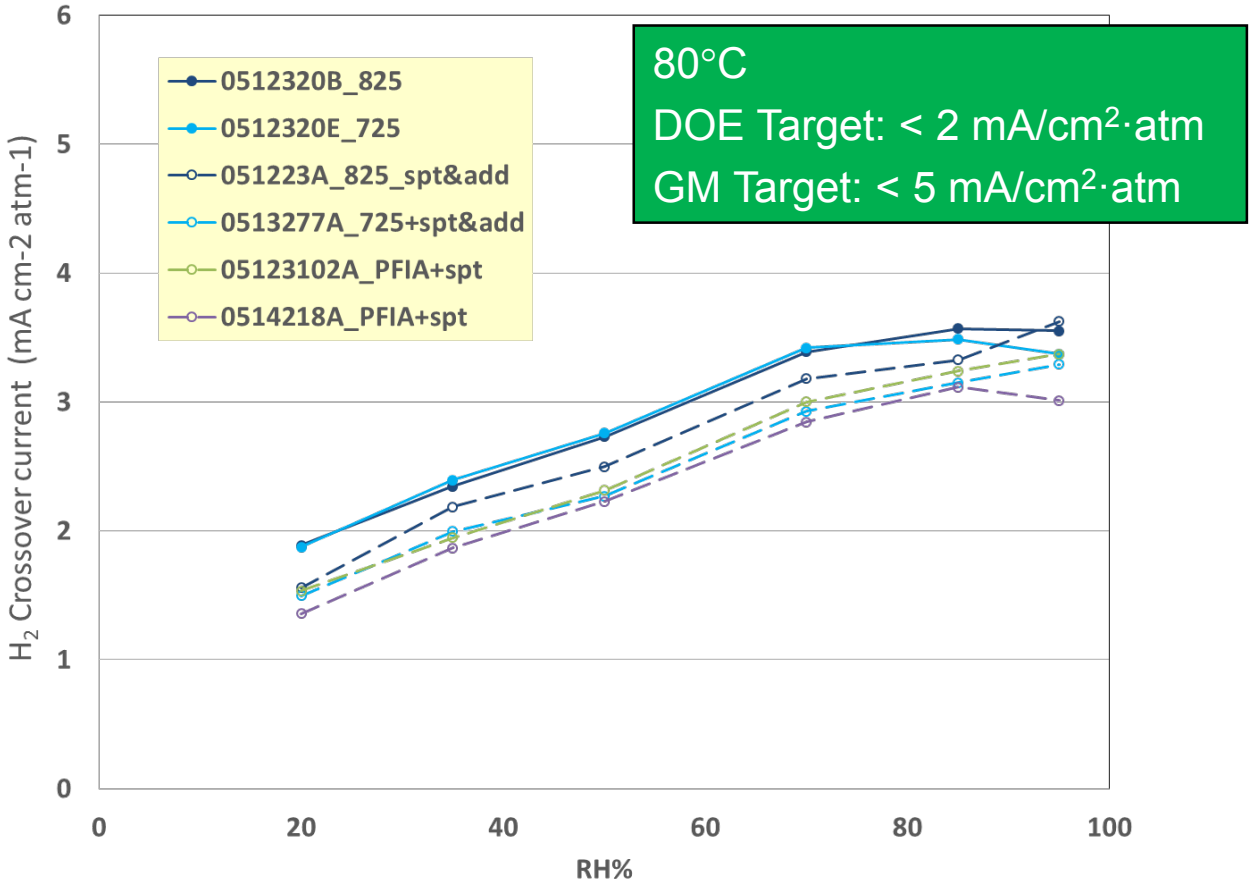

- At standard, relatively wet, conditions all supported PEMs show similar performance and HFR
- At dry conditions supported PFIA PEMs show superior performance and lower HFR
- Latest scaled-up lot of supported PFIA PEMs has better performance at dry conditions than the earlier lot

# Proton Transport Resistance




- ASR of supported 14µm PFSA PEMs is lower than that of non-supported 20µm PEMs
- Supported PFIA PEMs have lower ASR than supported PFSA PEMs
- Latest scaled-up lot of supported PFIA PEMs have lower ASR at dry conditions than the earlier lot

# Hydrogen Crossover




- H2 Crossover of supported 14µm PEMs is lower than non-supported 20µm PEMs
- 825EW PFSA supported PEM has slightly higher crossover than 725EW PFSA & PFIA PEMs

# *Plasma Treatment of PFSA and PPSU Mats*

Contact angle with water was determined to quantify the effect of oxygen plasma.

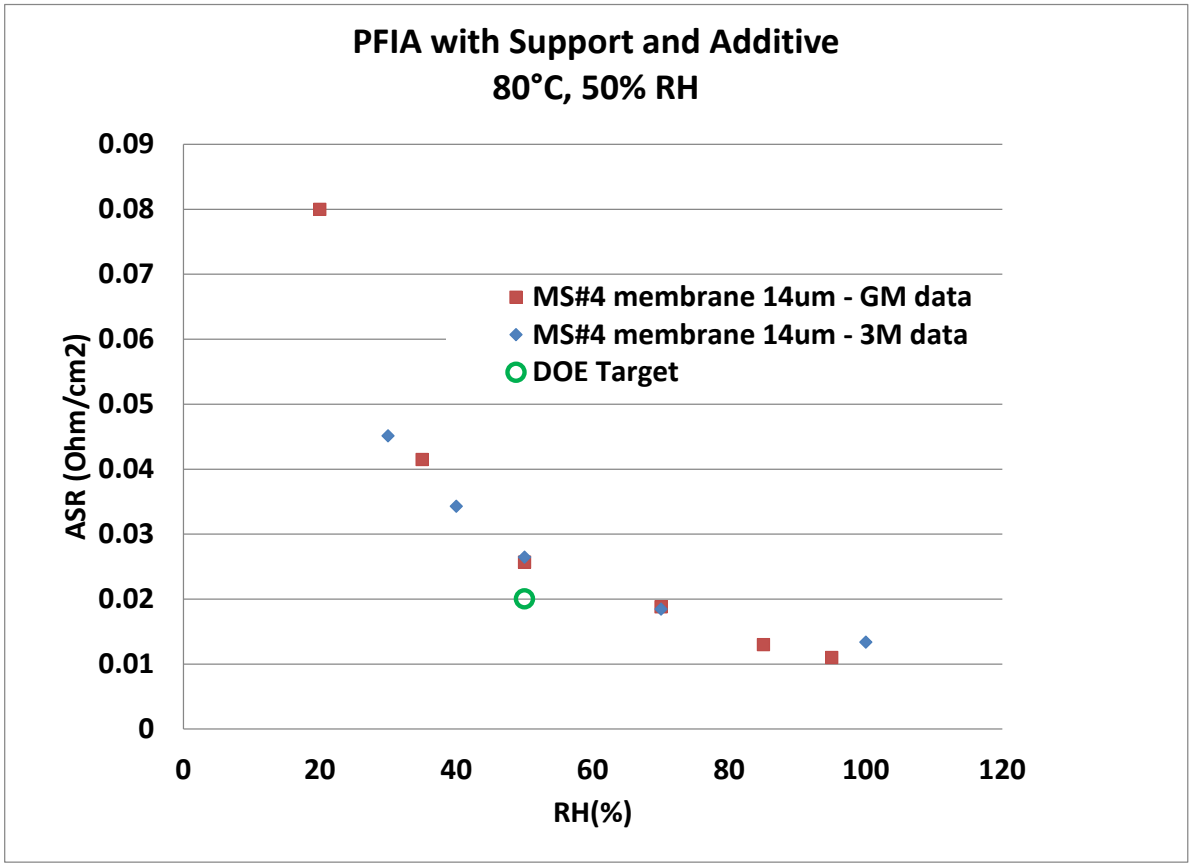
| eSpun Mat Type | Plasma RF 100 W | | | |
|---|---|---|---|---|
| | 0 sec | 30 sec | 120 sec | 300 sec |
| Raw PPSU mat | 131° | 0° | 0° | 0° |
| Raw PFSA mat | 136° | 118° | 119° | 0° |
| Annealed PFSA mat | 136° | 146° | 138° | 145° |

- PPSU becomes hydrophilic after 30 sec exposure.
- Raw PFSA 825EW shows hydrophilicity after 300 sec exposure, while annealed PFSA mat (partially welded/fused fibers) shows no hydrophilicity even after 300 sec.

**Optimum plasma exposure time: 30-120 sec.**

# Milestone #4

In-cell area specific resistance measurements as a function of relative humidity.




Accomplishments:

- Very good agreement between 3M and GM in-cell measurements
- Membrane resistance of about 27 mOhm*cm2 does not meet the DOE target of 20 mOhm*cm2