



Lead Research and Development Activity for DOE's High Temperature, Low Relative Humidity Membrane Program

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June 11, 2008

Project ID #
FC 15



Overview

Timeline

- April 1, 2006
- March 31, 2011
- 40% Complete

Budget

- Total project funding
 - DOE share - \$2,500K
 - Contractor share - \$625K
- Funding received in FY07- \$550K
- Funding for FY08 - \$585K

Barriers

- Barriers addressed
 - D. High Conductivity at Low RH & High T
 - C. High MEA Performance at Low RH & High T
 - A. Membrane and MEA durability
- Targets
 - Conductivity = 0.07 S/cm @ 80% relative humidity (RH) at room temp using alternate material – 3Q Yr 2 milestone
 - Conductivity >0.1 S/cm @ 50% RH at 120 °C – 3Q Yr 3 Go/No Go

Partners

- BekkTech LLC – In-plane conductivity protocols
- Scribner Associates – Through-plane conductivity protocols
- Project management



Objectives

- New polymeric electrolyte/phosphotungstic acid membranes
- Development of standardized characterization methodologies
 - Conductivity f(RH, T, Prep. Procedure) [Through- & In-Plane]
 - Characterize mechanical, mass transport and surface properties of membranes
 - Evaluate fuel cell performance and predict durability of membranes and MEAs fabricated from other eleven HT Low RH Membrane Programs
- Provide HTMWG members with standardized methodologies
- Organize HTMWG biannual meetings



Milestones

Month/Year	Milestone or Go/No-Go Decision
Sept-07	Complete analysis of in-plane and through-plane conductivity of commercial membranes.
Dec-07	Milestone: Complete conductivity characterization of first three membranes from Topic 1 awardees.
Dec-07	Milestone: Demonstrate conductivity = 0.07 S/cm @ 80% relative humidity (RH) at room temp using alternate material
Jun-08	Milestone: Establish MEA test protocol
Sept-08	Milestone: Complete manufacturing of first MEA from working group members
Dec-08	Go/No-Go Decision: Demonstrate conductivity of 0.1 S/cm, 50% RH, 120 °C



Approach

Improve Conductivity:

Task 1. FSEC develops non-Nafion[®] based Poly[perfluorosulfonic acid]-phosphotungstic acid composite membrane and membrane electrode assembly (MEA) fabrication (PFSA-PTA)

Task 2. FSEC develops sulfonated poly(ether ketone ketone) or sulfonated poly(ether ether ketone) - Phosphotungstic Acid Composite Membrane and MEA Fabrication (SPEEK-PTA)

Task 7. Meetings and Activities of HTMWG

Improve FC Performance:

Task 5. Characterize performance of MEAs for Topic 1 members

Task 6. Characterize membrane and MEA durability for Topic 1 members

Standardize Testing

Task 3. In-Plane conductivity measurements by partner

Task 4. Through-Plane conductivity measurements by partner

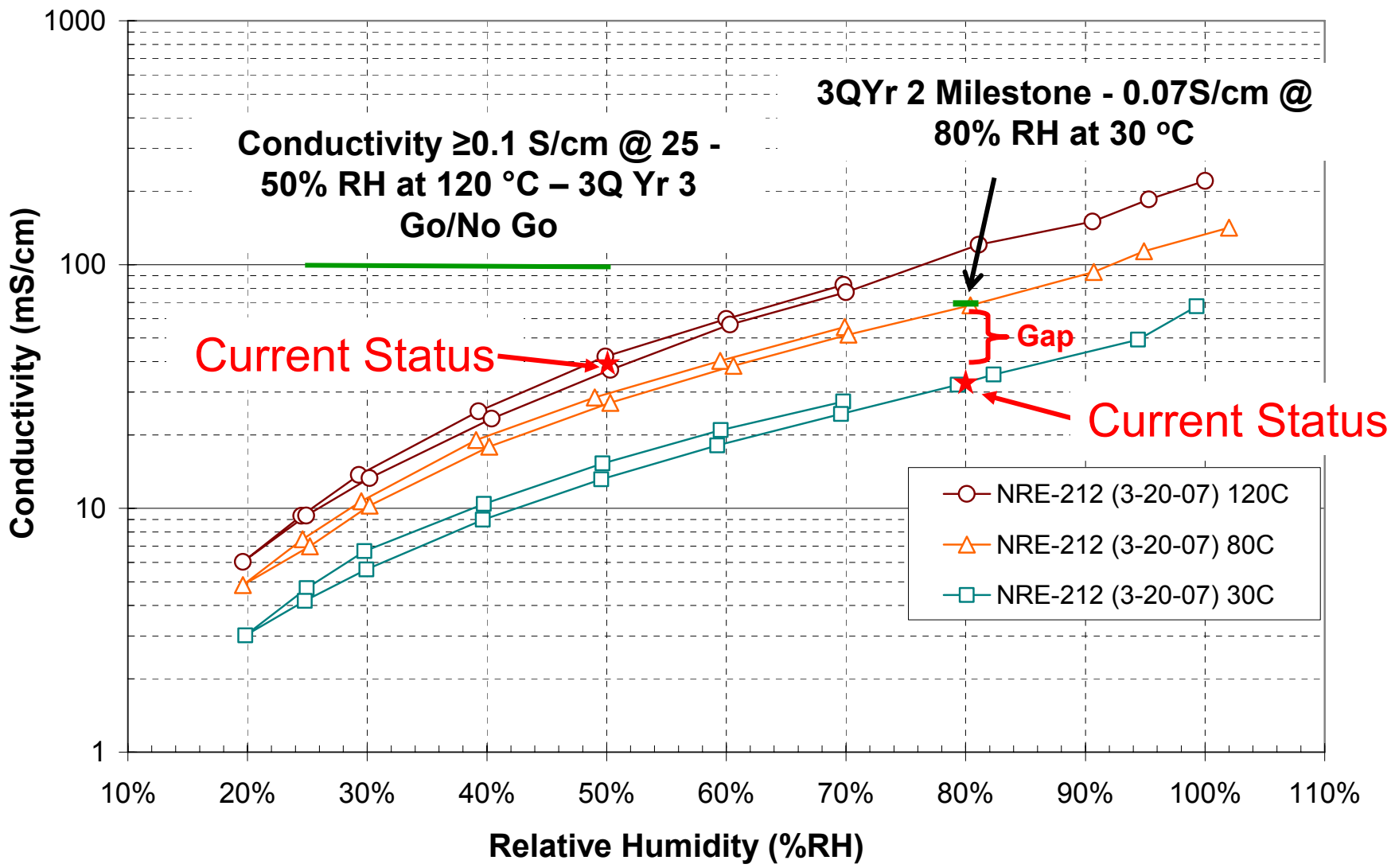


Technical Accomplishments/ Progress/Results

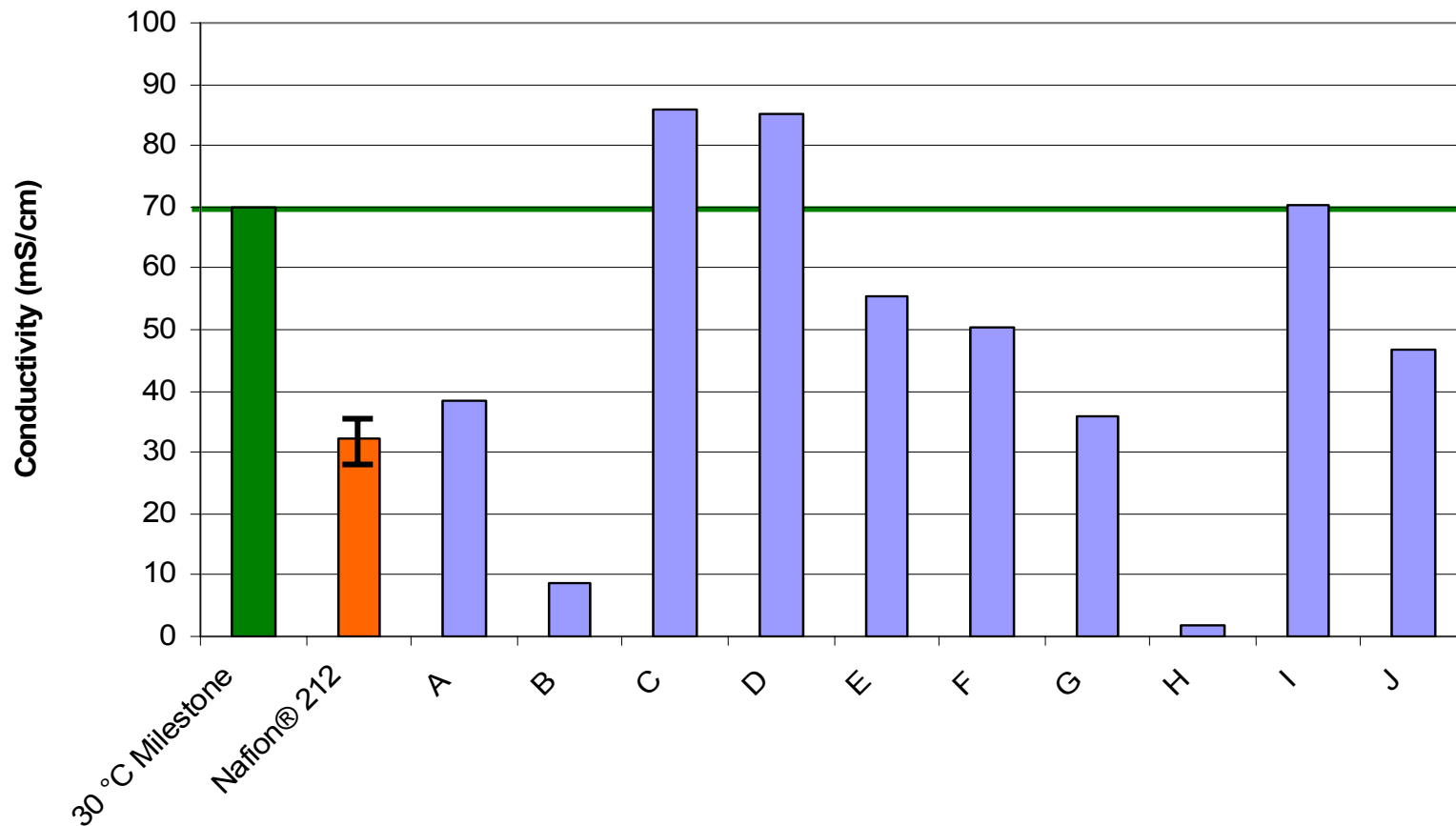
- Conductivity
- Performance
- Durability
 - Chemical
 - Mechanical



In-Plane Conductivity Measurements



30 °C, 80% RH Conductivity Milestone Group 1 Membranes

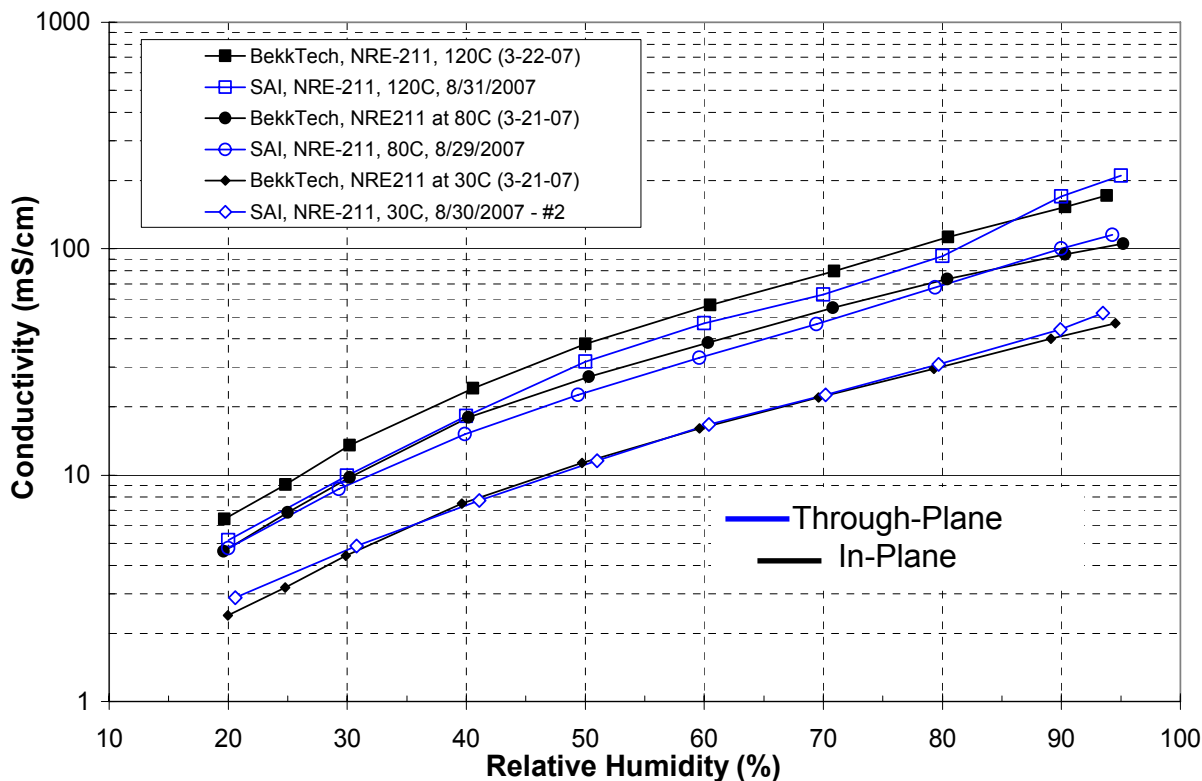


•Samples tested at 30 °C, 80% RH ~100 kPa at BekkTech as of April 24, 2008



In-Plane vs. Through-Plane Conductivity (milestone)

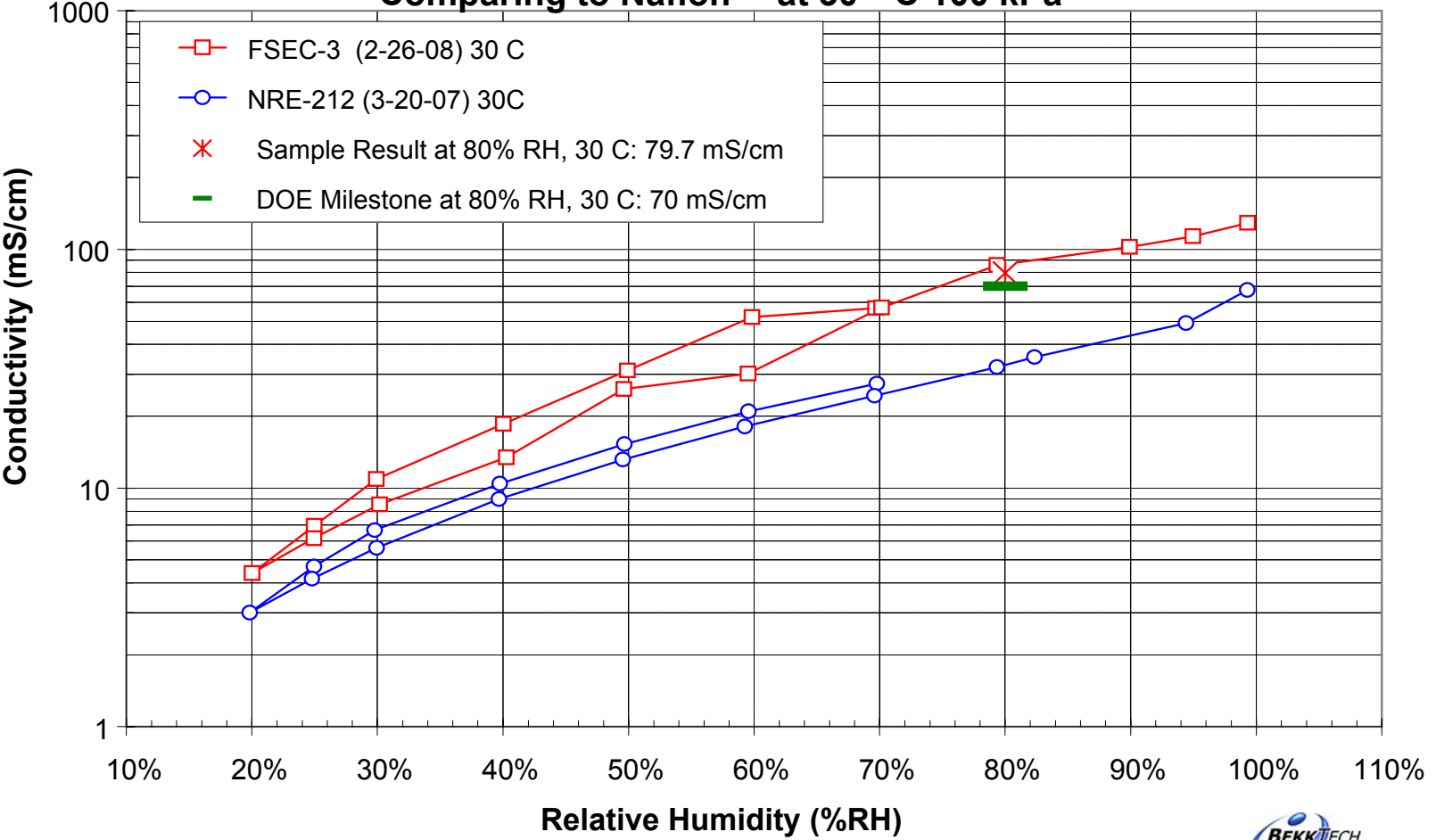
NRE 211 at 30, 80, & 120 °C
Also Tested NRE-212, NE-1135 & N 117





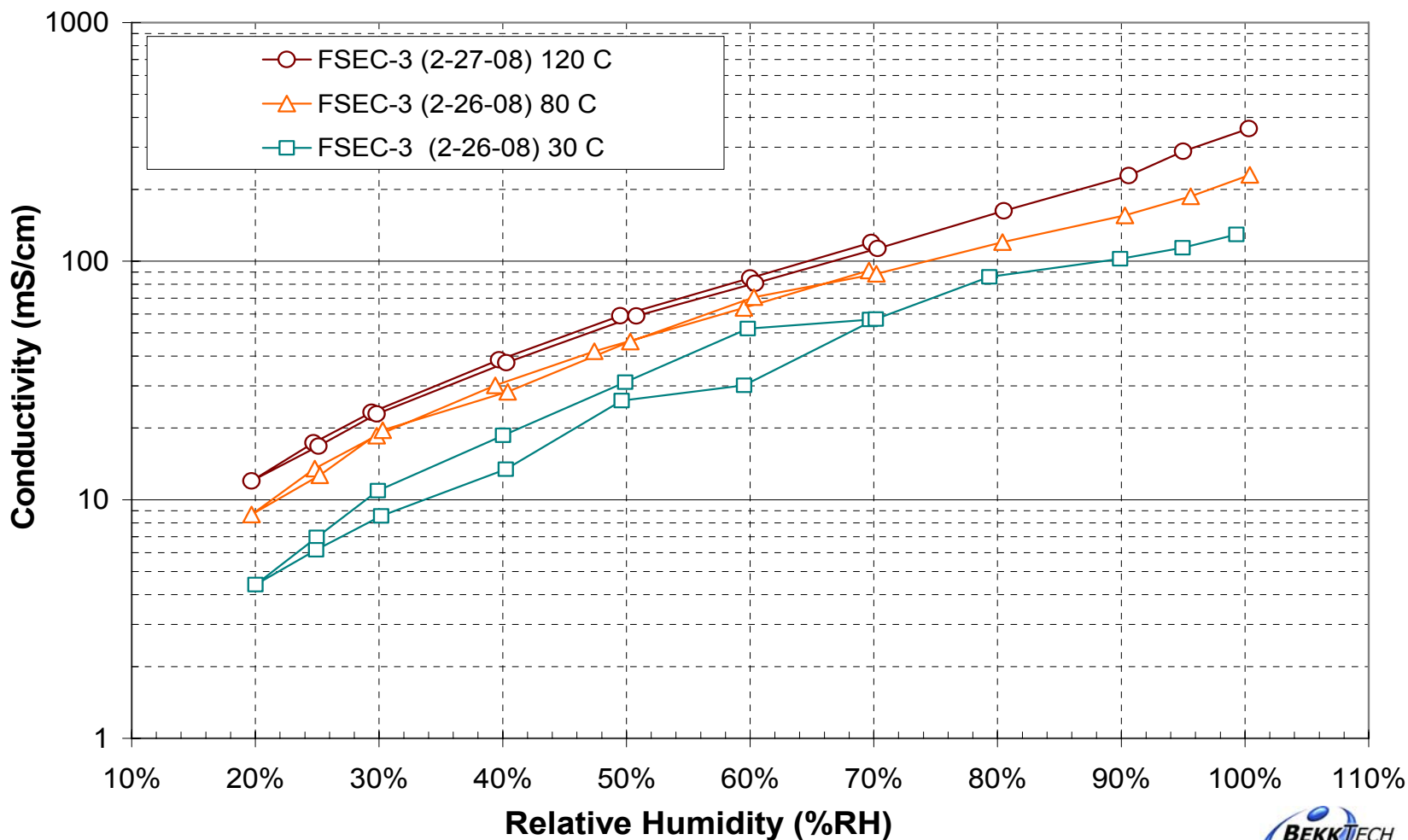
FSEC-3 Meets Conductivity Milestone! (PFSA-PTA)

Comparing to Nafion[®] at 30 ° C 100 kPa



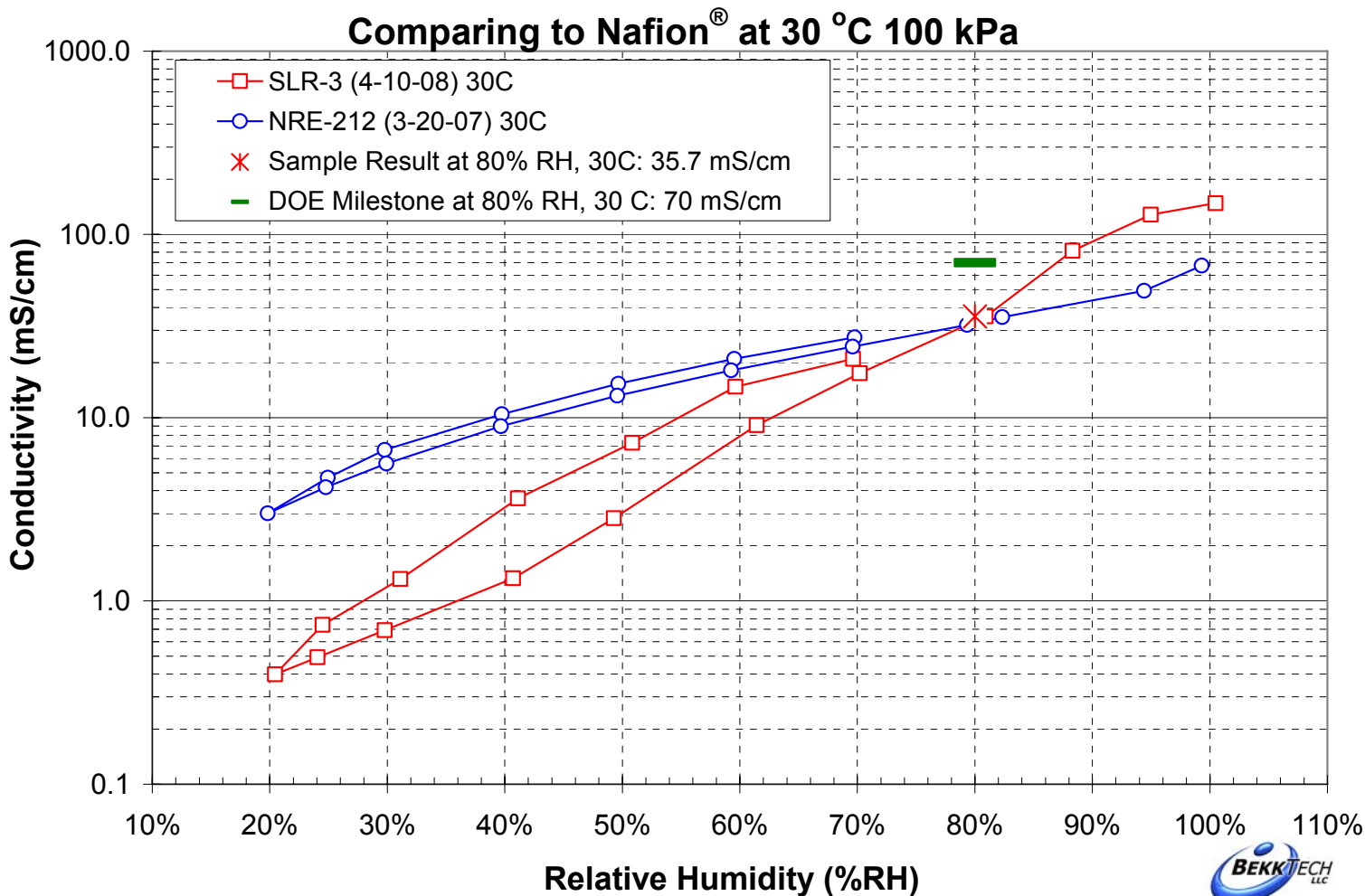


FSEC-3 Tested at 30 °C, 80 °C, 120 °C (PFSA-PTA)





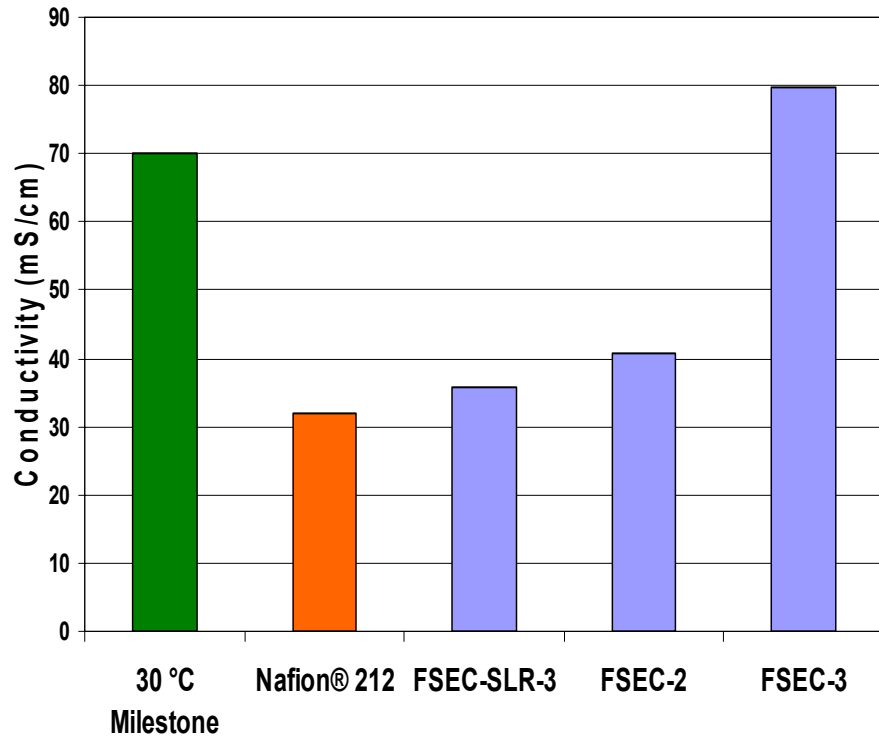
Conductivity of FSEC-SLR3 (SPEEK-PTA)



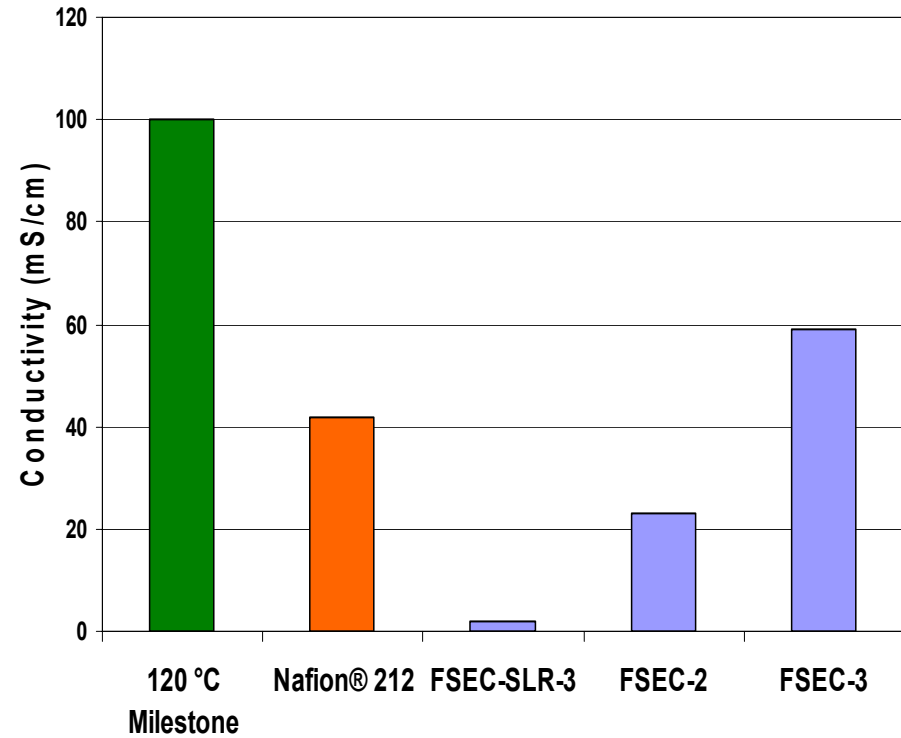


Progress Toward 120 °C Go/No Go

Meet 30 °C, 80% RH Milestone



Status Toward 120 °C, 50% RH Go/No Go





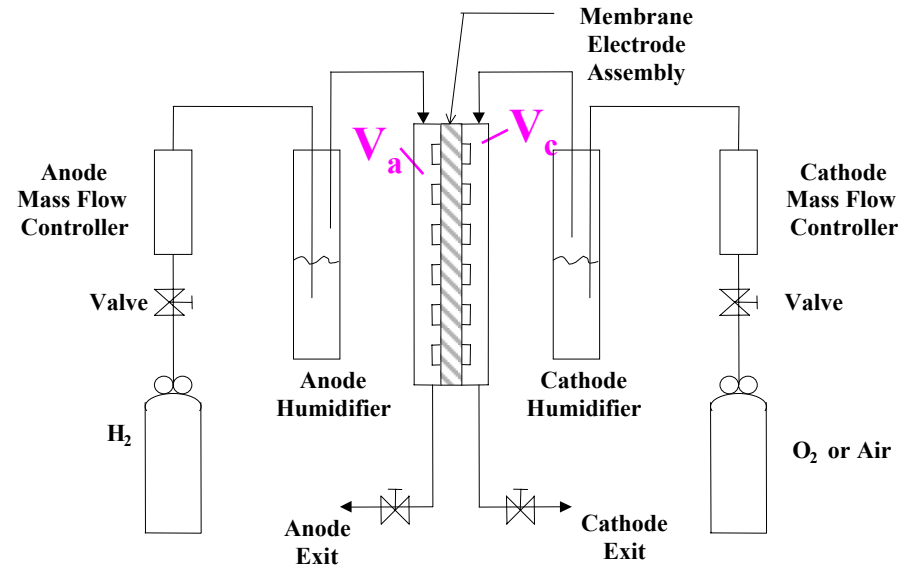
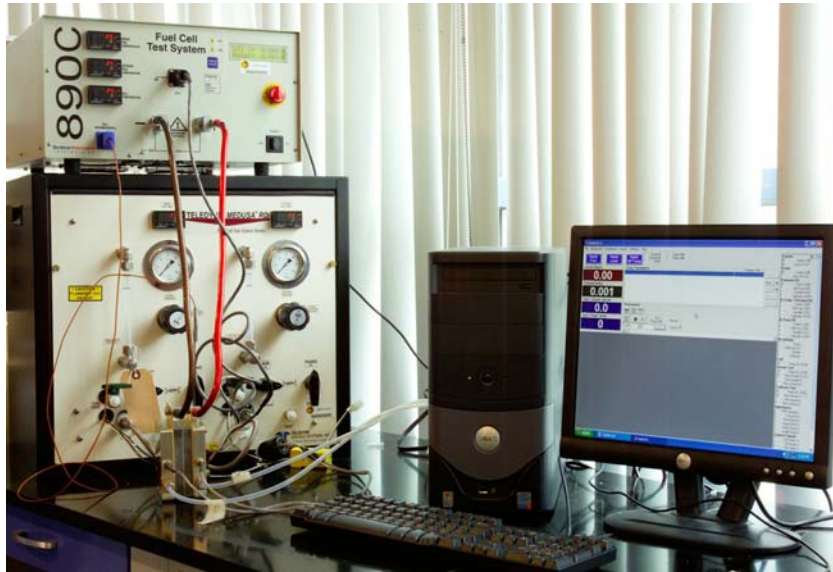
Performance

Jun-08 Milestone: Establish MEA test protocol

**Sept-08 Milestone: Complete manufacturing of first
MEA from working group members**



MEA Test Apparatus

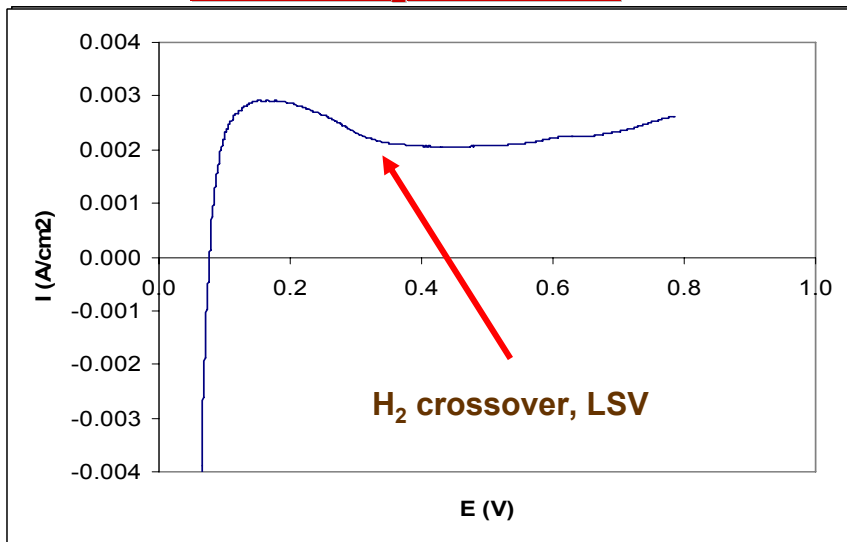


T_{cell}	$T_{\text{cathode humidifer}}$	R. H. Inlet Cathode	P_{total}	Inlet $P_{\text{H}_2\text{O}}$	Inlet P_{O_2} in Air
$^{\circ}\text{C}$	$^{\circ}\text{C}$	%	kPa	kPa	kPa
80	73	75	101	35	13.86
100	90	70	101	70	6.51
100	65	25	150	25	26.25
120	90	35	101	70	6.51
120	82	25	150	51	20.79
120	100	50	150	101	10.29

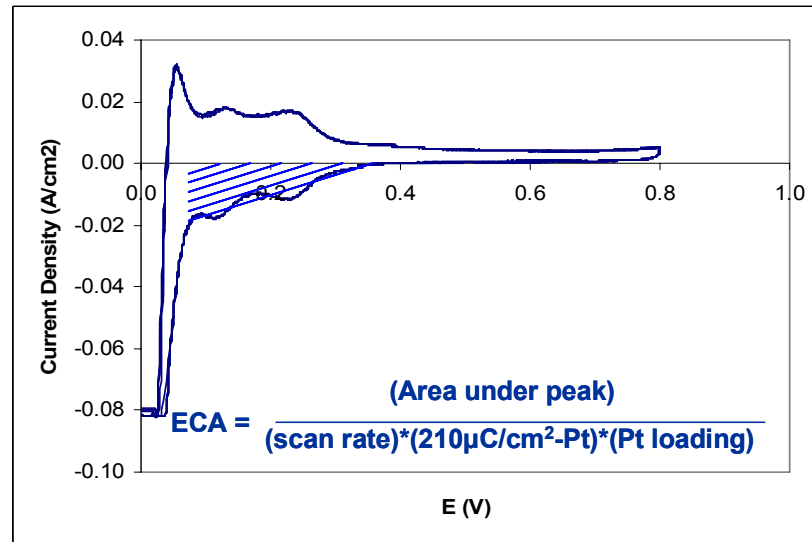


Electrochemical Testing

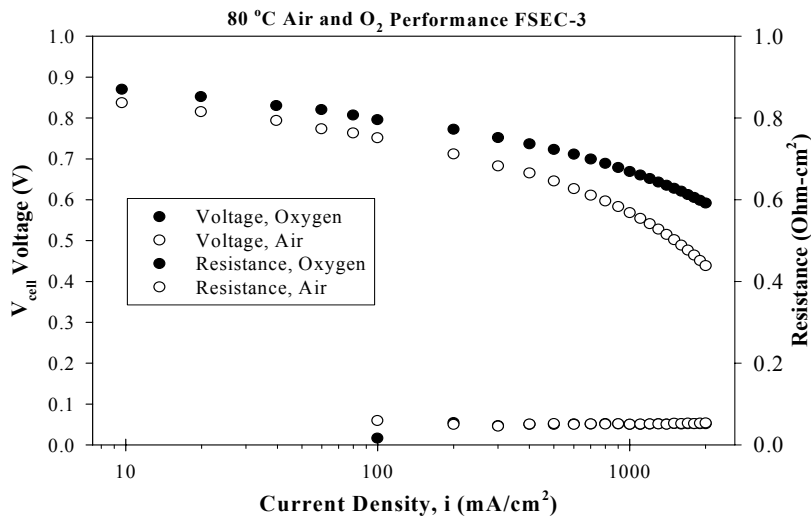
LSV → H₂ Crossover



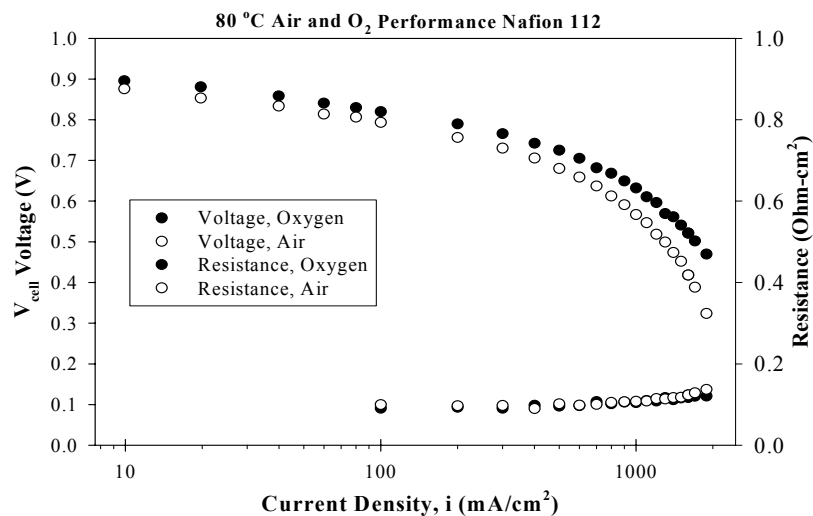
CV → ECA



Cell Performance with FSEC-3



Cell Performance with Nafion® 112



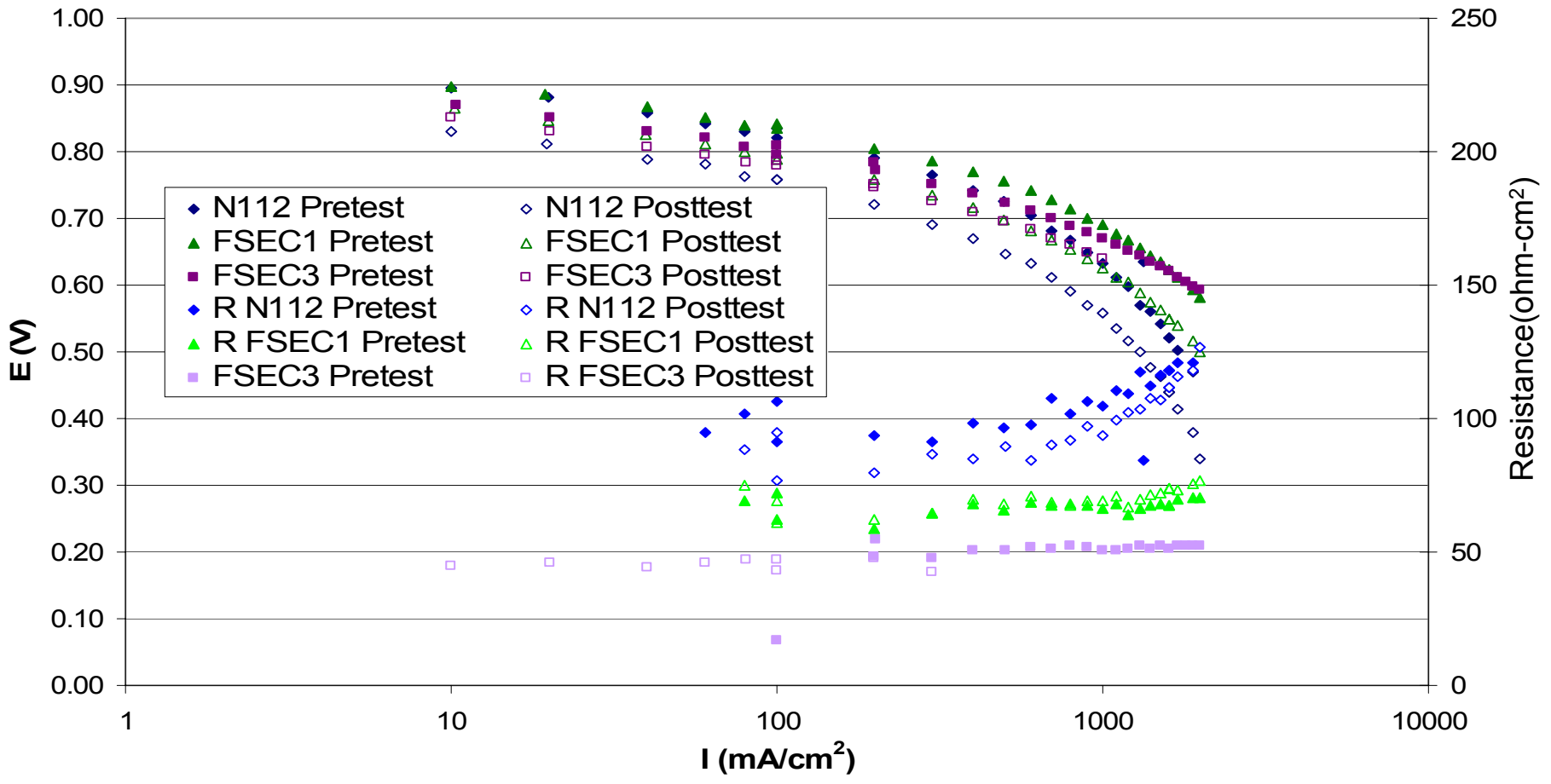


In Situ Investigation of MEA Degradation

- MEAs investigated:
 - Nafion[®] and FSEC-1 and FSEC-3
- Tested MEAs under different degradation conditions:
 - 90 °C; 35% RH; OCV; 100 hr
- Degradation evaluated in several ways:
 - Electrochemical Pre- and Post-testing
 - H₂ crossover, ECA, polarization, resistance
 - Material testing before and after degradation test
 - mechanical strength, materials science
 - During the test
 - fluoride emission rate, voltage monitored



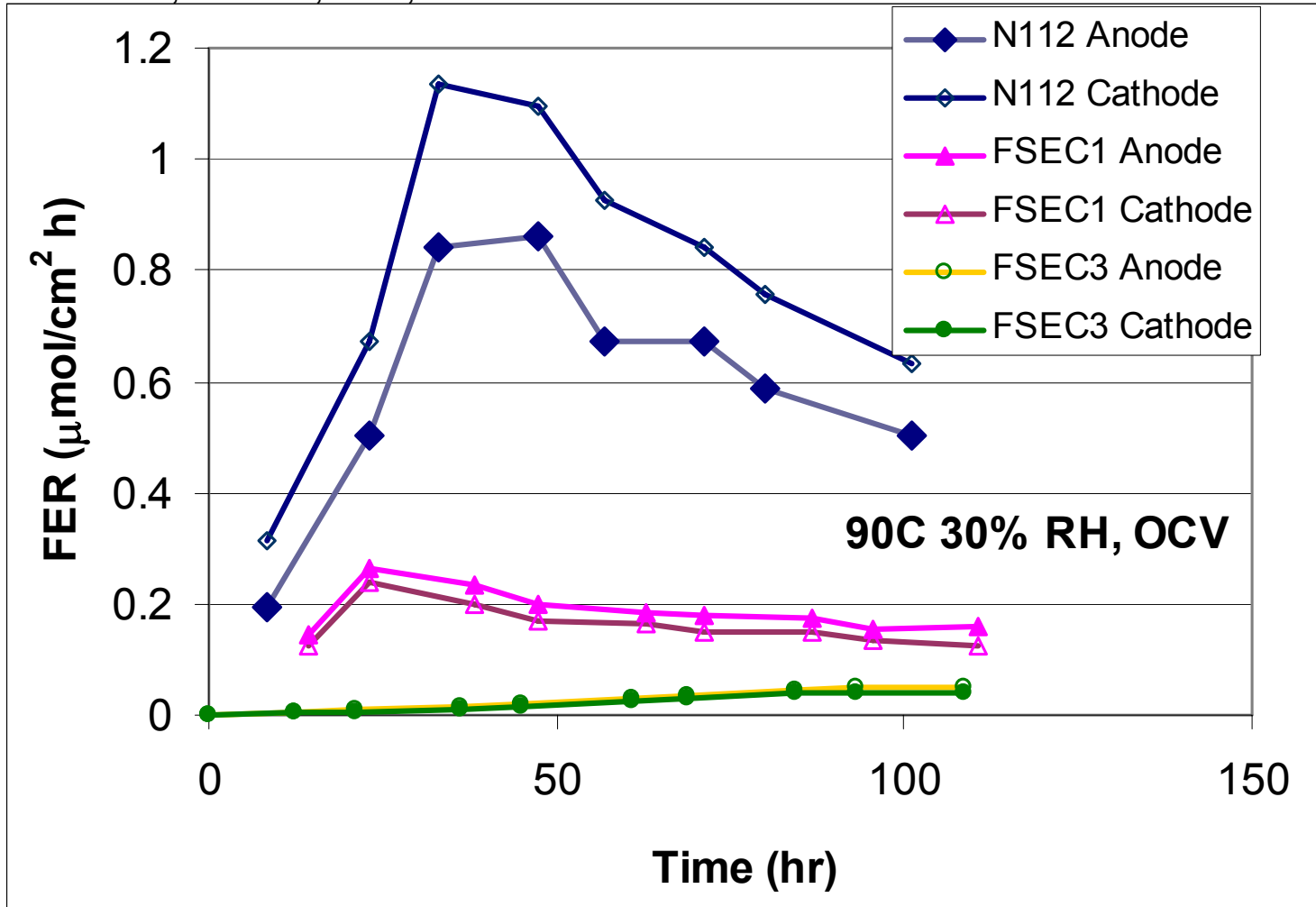
Membrane Durability





Fluoride Emission Rate

90 °C; 35% RH; OCV; 100 hr



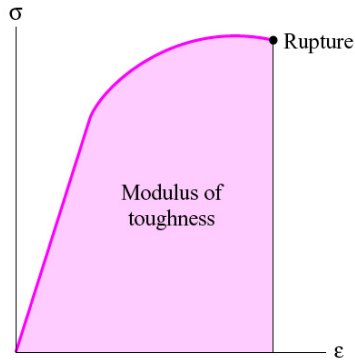


Rationale for the Investigation of Membrane/MEA Mechanical Degradation

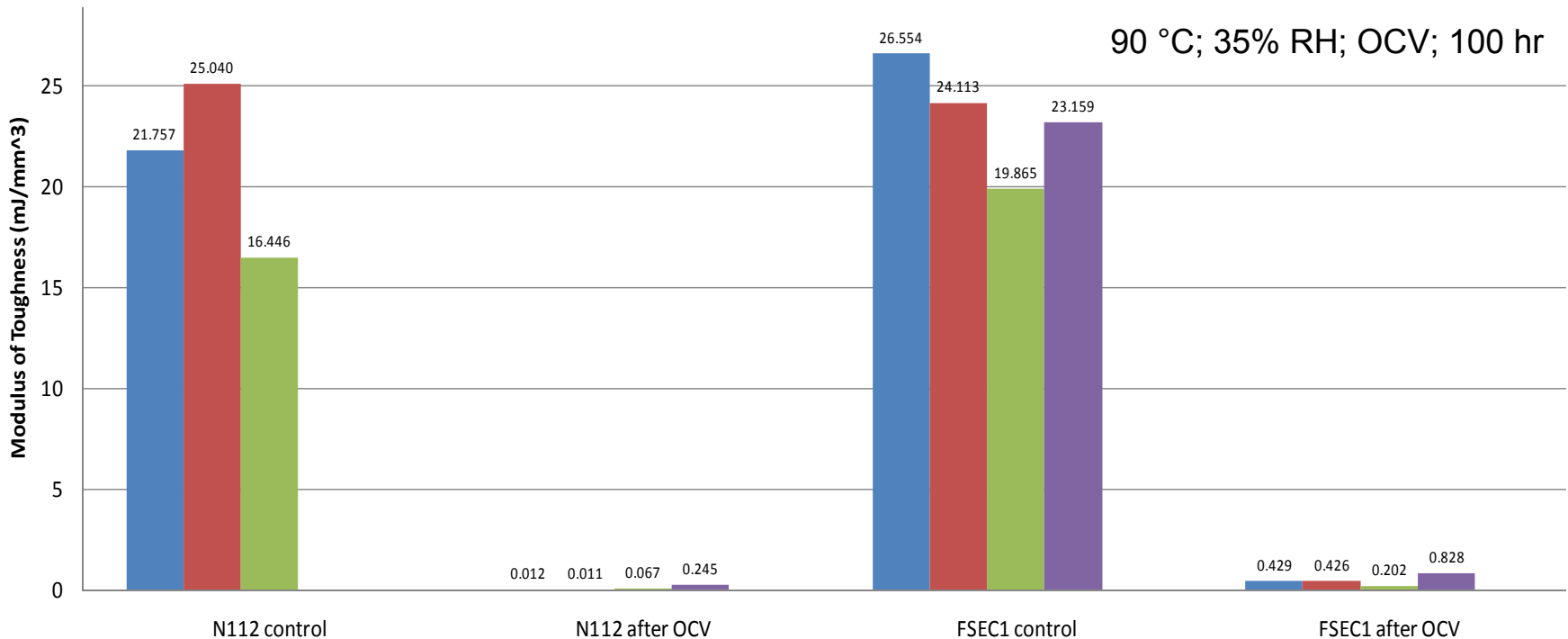
- Mechanical properties degradation: phenomena and relevance
 - The beginning-of-life (BOL) mechanical properties of membranes are adequate, typically
 - Mechanical properties rapidly decay as a result of accumulated chemical (e.g., load cycling + OCV) and mechanical effects (e.g., RH cycling)
 - Fracture of mechanically weakened membrane can be the life-limiting failure mode for PEM devices
- It is important to
 - quantify the membrane mechanical robustness while optimizing other properties of high temperature membrane
 - further understand the underlying mechanisms that are responsible for the mechanical decay



Membrane/MEA mechanical degradation: modulus of toughness



Modulus of toughness = Energy per unit volume necessary to rupture the material, Joule/m³ or milli-Joule/mm³





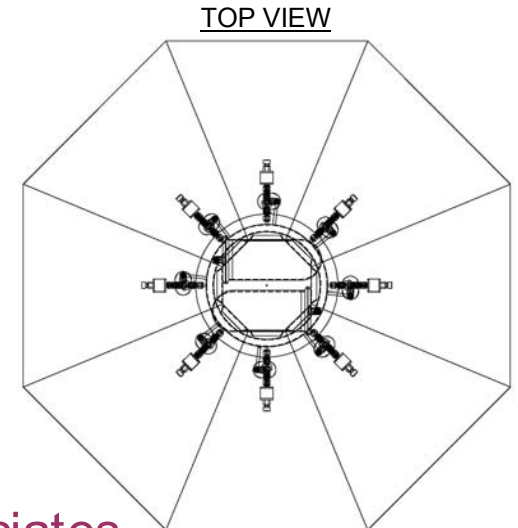
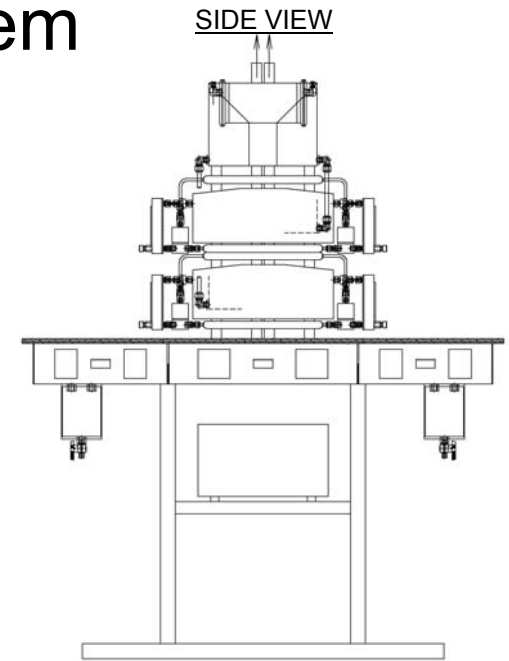
Future Work

- Complete characterization of HTMWG membranes
- Establish MEA test protocol (milestone)
- Manufacture first MEA from HTMWG membrane (milestone)
- Demonstrate conductivity of 0.1 S/cm, 50% RH, 120 °C (Go/No Go)



8-Cell MEA Durability Test System

- Simultaneous, independent operation of 8 cells
- Fully automatic – 24/7 operation
- Common RH system
- Adjustable cell temperature and reactant flow
- Individual cell diagnostics
- Manual over ride
- Individual cell replacement





Summary

- **Relevance** - A new membrane material for PEM Fuel Cells with sufficiently improved conductivity at high temperature (120 °C) and low RH is required for the transportation F/C market. A new method for measuring membrane conductivities with sufficient accuracy and reliability is required for DOE program decisions.
- **Approach** - Develop and demonstrate new materials for membranes, and define and apply new tools and procedures for membrane conductivity testing.
- **Tech. Accomplishments /Progress**
 - FSEC-3 exceeds conductivity goal, demonstrating conductivity >0.07 mS/cm at 80% RH and 30 °C.
 - Manufactured MEAs from Nafion[®] and FSEC membranes
 - Performance and durability testing of Nafion[®] and FSEC MEAs
 - Much reduced FER with FSEC membranes
 - Provided independent conductivity measurements for HTMWG members
- **Collaborations**
 - Active partnership with BekkTech LLC and Scribner Associates
 - Working closely with HTMWG members to provide accurate data under standardized conditions
 - Provided protocol to HTMWG members
 - Demonstrated Agreement between through-plane and in-plane conductivity measurements