



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

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May 16, 2007

Project ID
FC15



Overview

Timeline

- Start: April 1, 2006
- End: March 31, 2011
- 20% Complete

Budget

- Total project funding
 - DOE - \$2,500K
 - Contractor - \$625K
- Funding for FY06 \$250K
- Funding for FY07 \$500K

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
- Standardized Characterization Methodologies
 - Conductivity $f(\text{RH}, T, \text{Prep. Procedure})$ [Through- & In-Plane]
 - Characterize mechanical, mass transport and surface properties of membranes
 - Predict durability of membranes and MEAs fabricated from other eleven HT Low RH Membrane Programs
- Provide HTMWG members with standardized tests and methodologies (Short Courses)
- Organize HTMWG bi-annual meetings



Approach

Task 1. Non-Nafion[®] based Poly[perfluorosulfonic acid] - phosphotungstic acid composite membrane and membrane electrode assembly (MEA) fabrication

Task 2. Sulfonated poly(ether ketone ketone) or sulfonated poly(ether ether ketone) - Phosphotungstic Acid Composite Membrane and MEA Fabrication

Task 3. In-Plane Conductivity Measurements

Task 4. Through-Plane Conductivity Measurements

Task 5. Characterize Performance of MEAs

Task 6. Membrane and MEA Durability

Task 7. Meetings and Activities of HTMWG



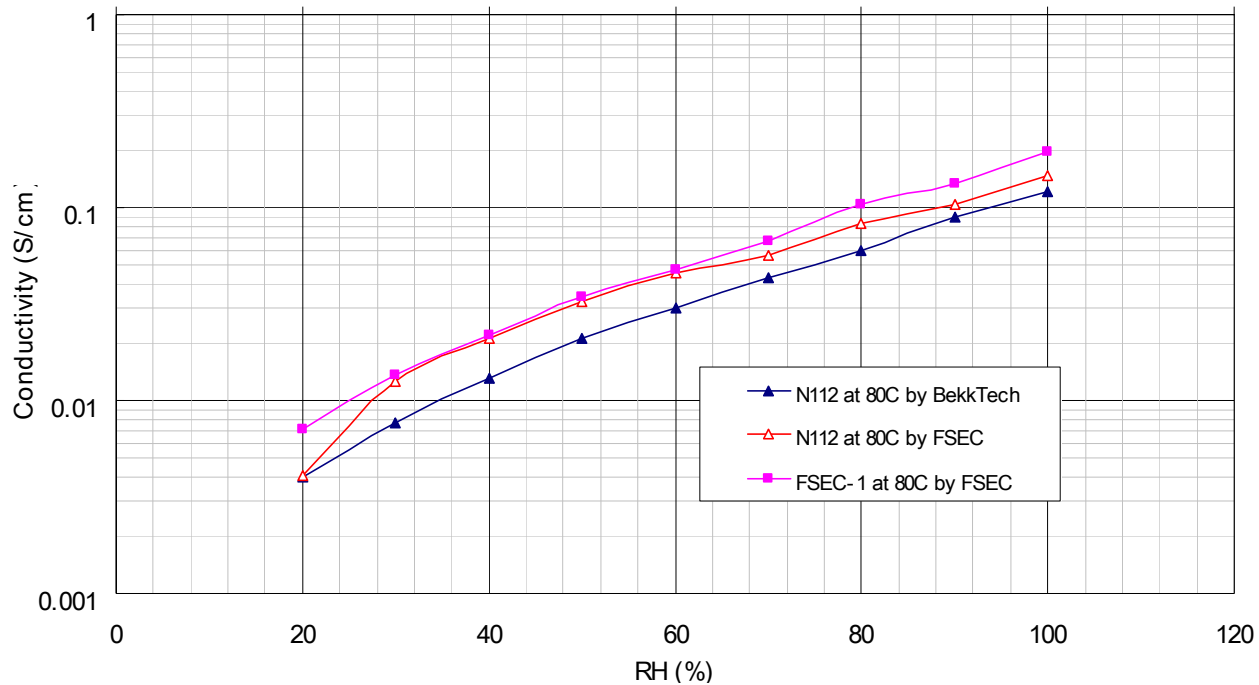
Accomplishments/ Progress/Results

- **Task 1.** PFSA low eq. wt.-Teflon[®]- Phosphotungstic Acid Composite Membrane and MEA Fabrication
 - Fabricated and tested FSEC-1 (solved RH issue for fabrication)
 - Identified and procured alternative low equivalent weight (EW) poly[perfluorosulfonic acid] (PFSA) ionomer
 - Demonstrated process for 1100 EW, started trials on 750 EW



Accomplishments/ Progress/Results

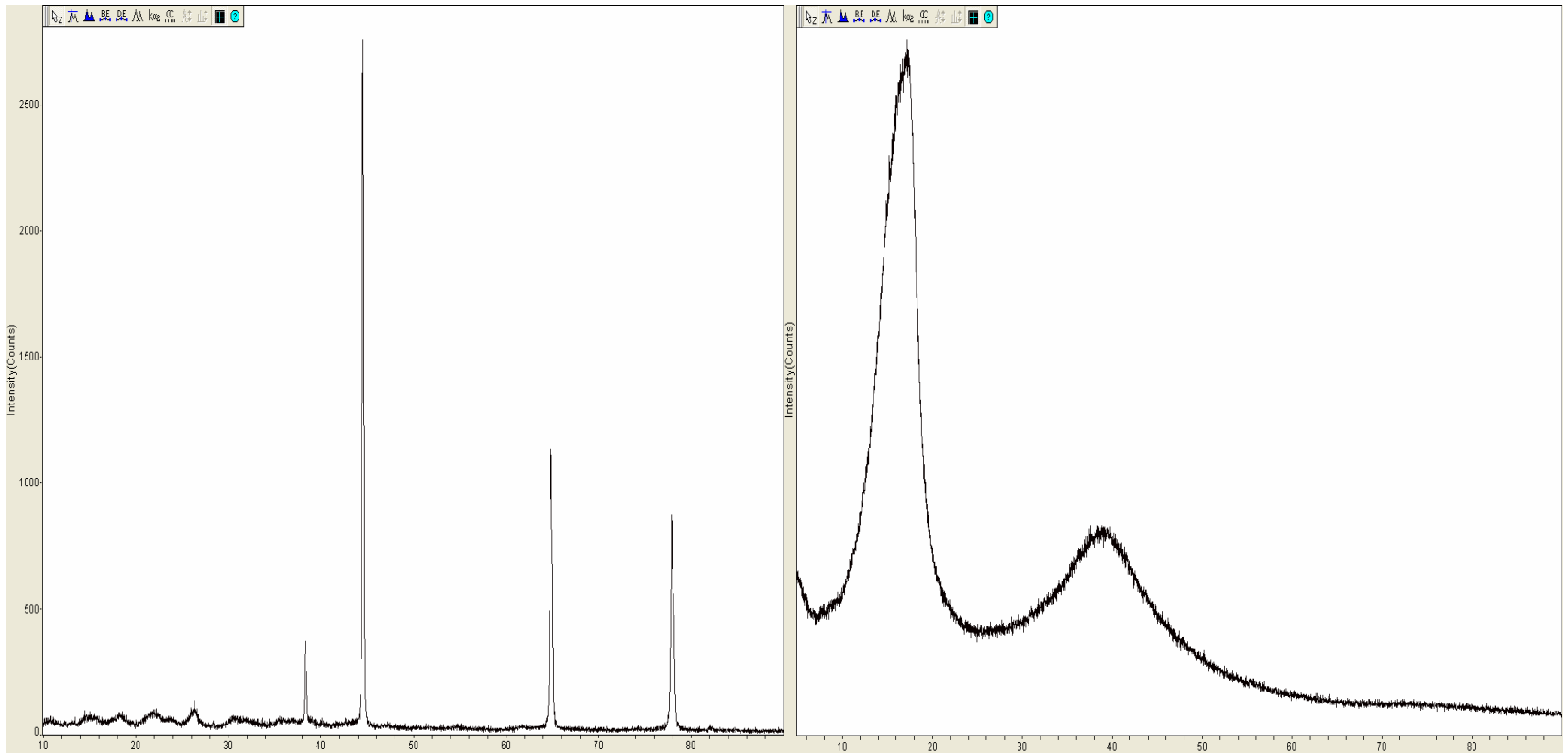
Nafion 112 and FSEC- 1 at 80C



FSEC-1 showed improvement in conductivity compared to Nafion 112



Accomplishments/ Progress/Results



The XRD pattern of FSEC I membrane, left, exhibiting the increased crystalline nature due to incorporation of PTA in the Nafion[®] matrix and Nafion 112, right.



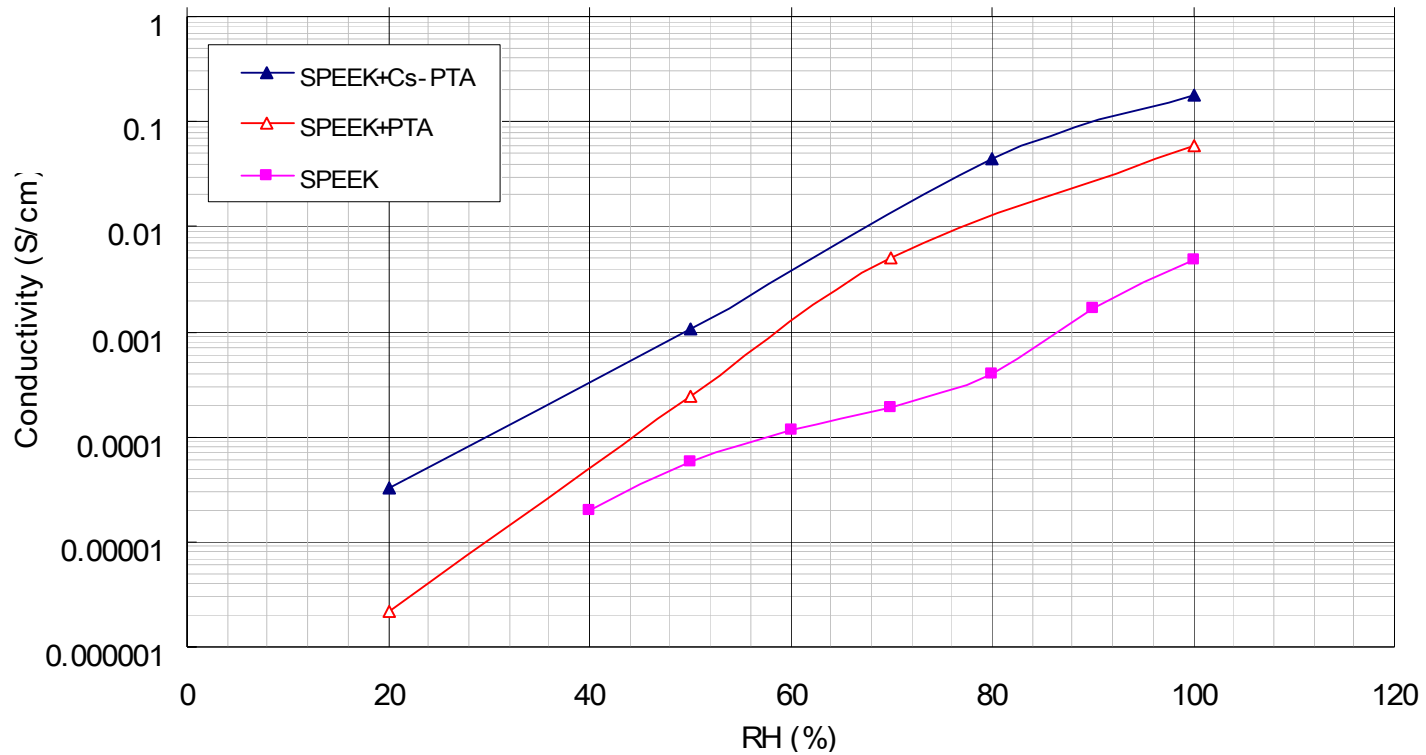
Accomplishments/ Progress/Results

- **Task 2. SPEEK and SPEKK-based PTA Composite Membranes**
 - SPEEK prepared at 30, 60, and 85% degrees of sulfonation
 - SPEKK prepared at 10, 60, and 80% degrees of sulfonation
 - Optimum ranges of sulfonation determined
 - 15% by weight PTA composites fabricated
 - Preliminary σ_{H^+} vs RH data obtained (**completion of Year 1 milestone**)



Accomplishments/ Progress/Results

SPEEK- PTA Composites at 80C





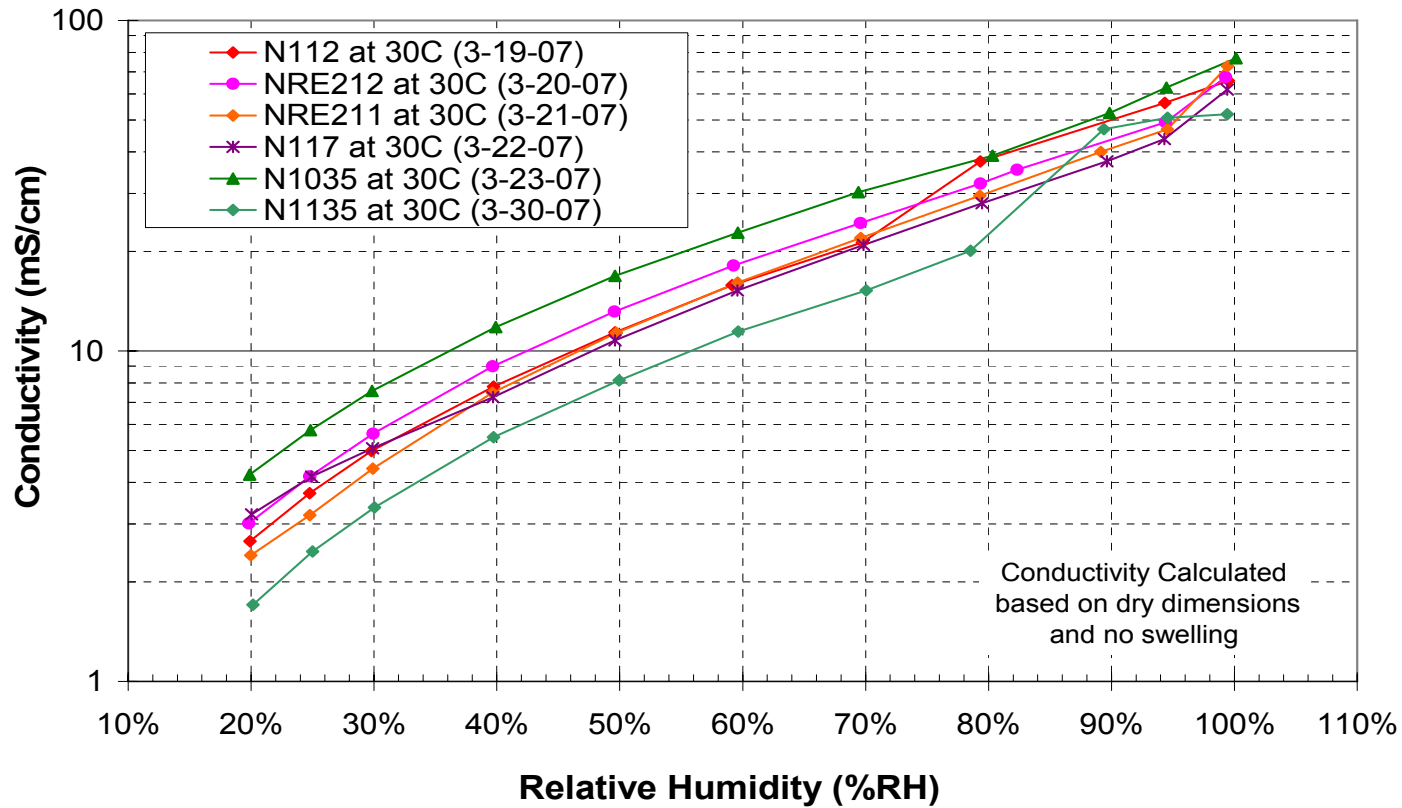
Accomplishments/ Progress/Results

- **Task 3. In-Plane Conductivity Measurement**
 - Established baseline for commercial membranes
 - Nafion[®] NRE-211, NRE-212, N1135, N1035, and N117 samples tested at 30 °C, 80 °C & 120 °C
 - Results compared for FSEC, BekkTech and Scribner



Accomplishments/ Progress/Results

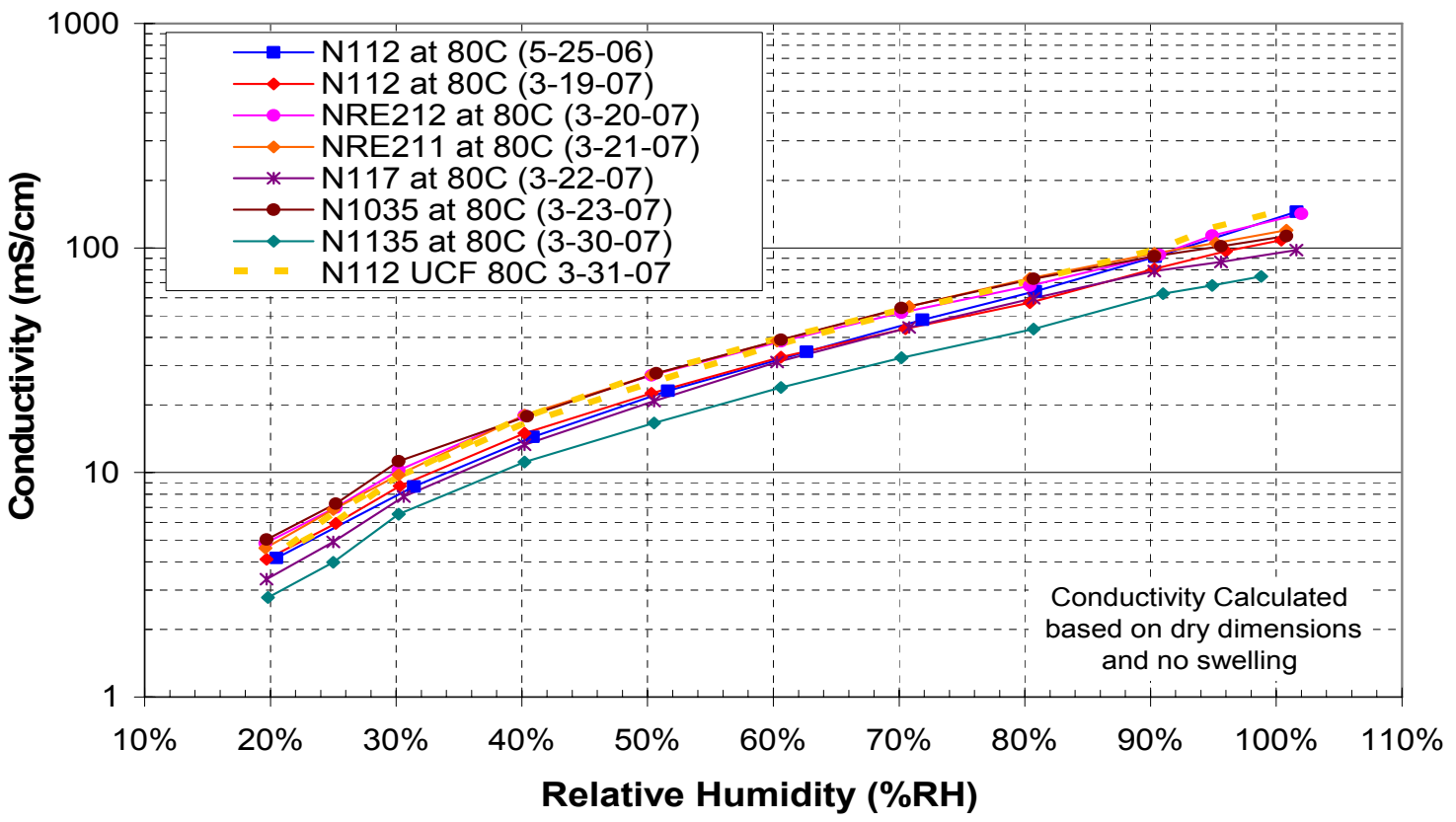
Four Electrode Conductivities of Nafion at 30C





Accomplishments/ Progress/Results

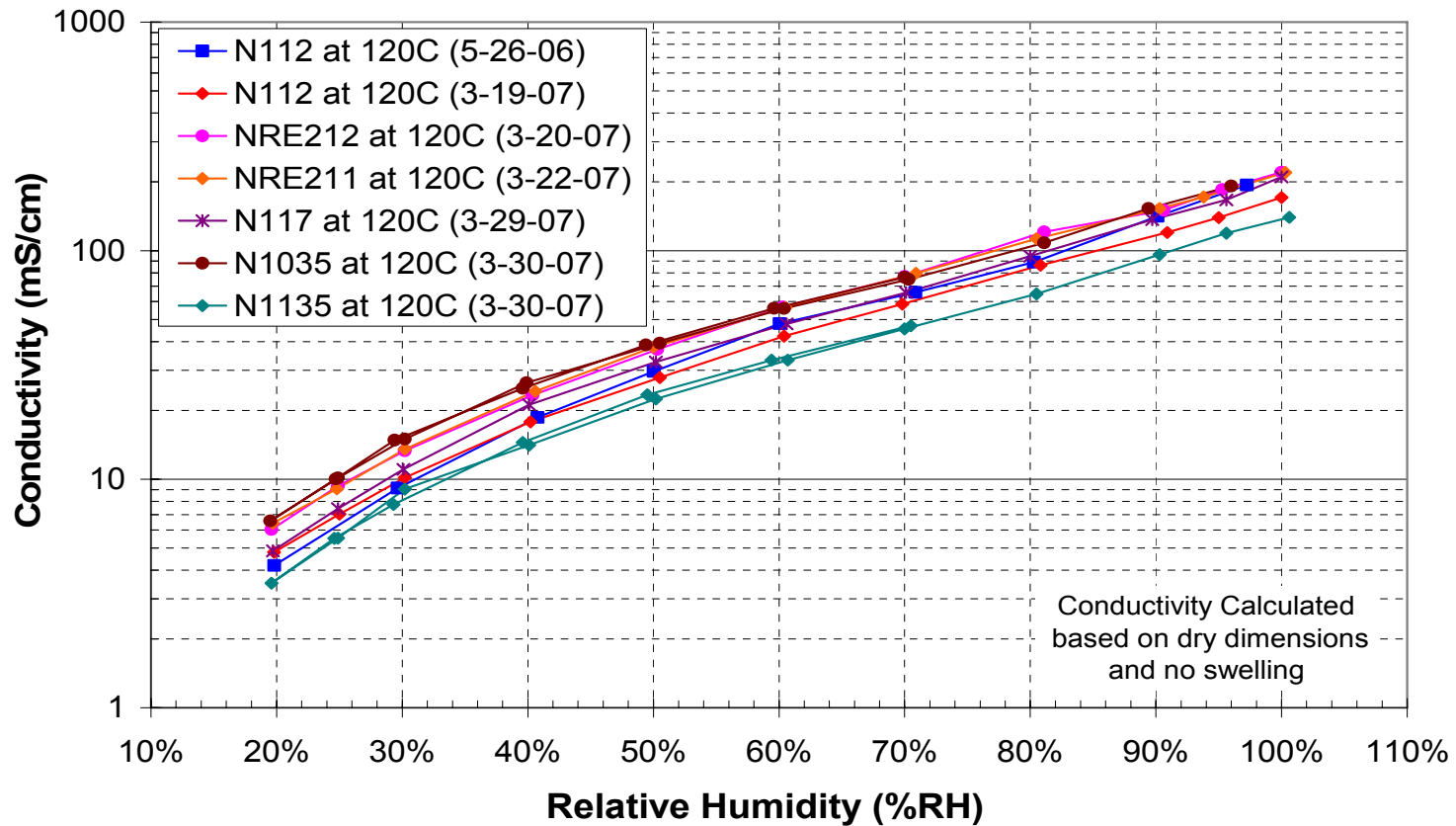
Four Electrode Conductivities of Nafion at 80C





Accomplishments/ Progress/Results

Four Electrode Conductivities of Nafion at 120C





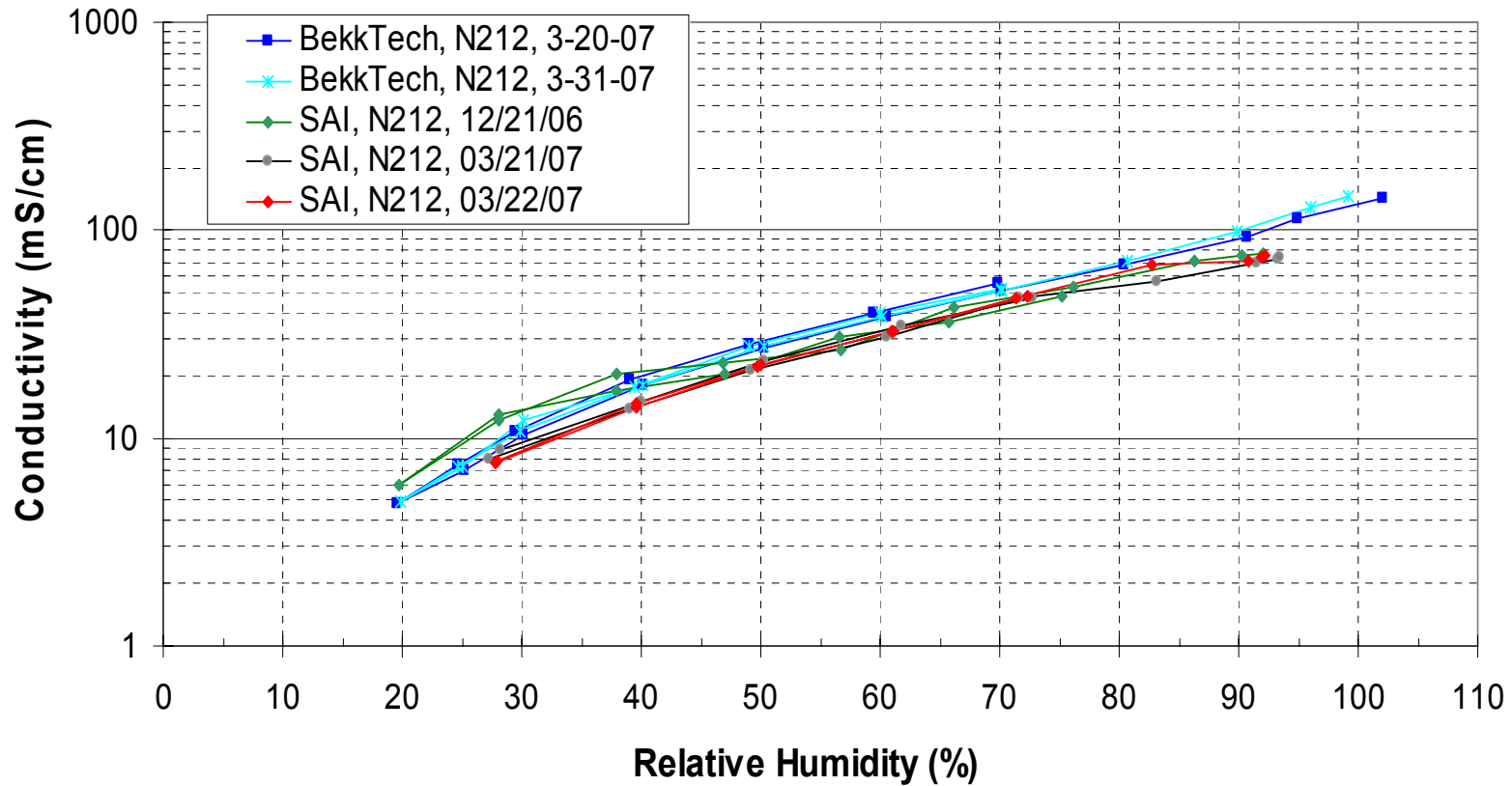
Accomplishments/ Progress/Results

- **Task 4. Through-Plane Conductivity Measurement**
 - Developed & verified performance of primary components of prototype through-thickness membrane conductivity test system, MTS (**Year 1 milestone**).
 - Method allows direct measurement of through-plane conductivity of bare membranes.
 - Developed MTS gas handling system for improved rapid relative humidity cycling at 30 – 120 °C.
 - Developed MTS hardware for safe high temperature, pressurized operation.
 - Gathered and compared Nafion[®] through-plane and in-plane conductivity (literature and BekkTech).
 - Demonstrated feasibility of using Model 850C Compact Fuel Cell Test System (Scribner Assoc. Inc.) for wet-dry gas mixing for rapid dew point and relative humidity cycling.



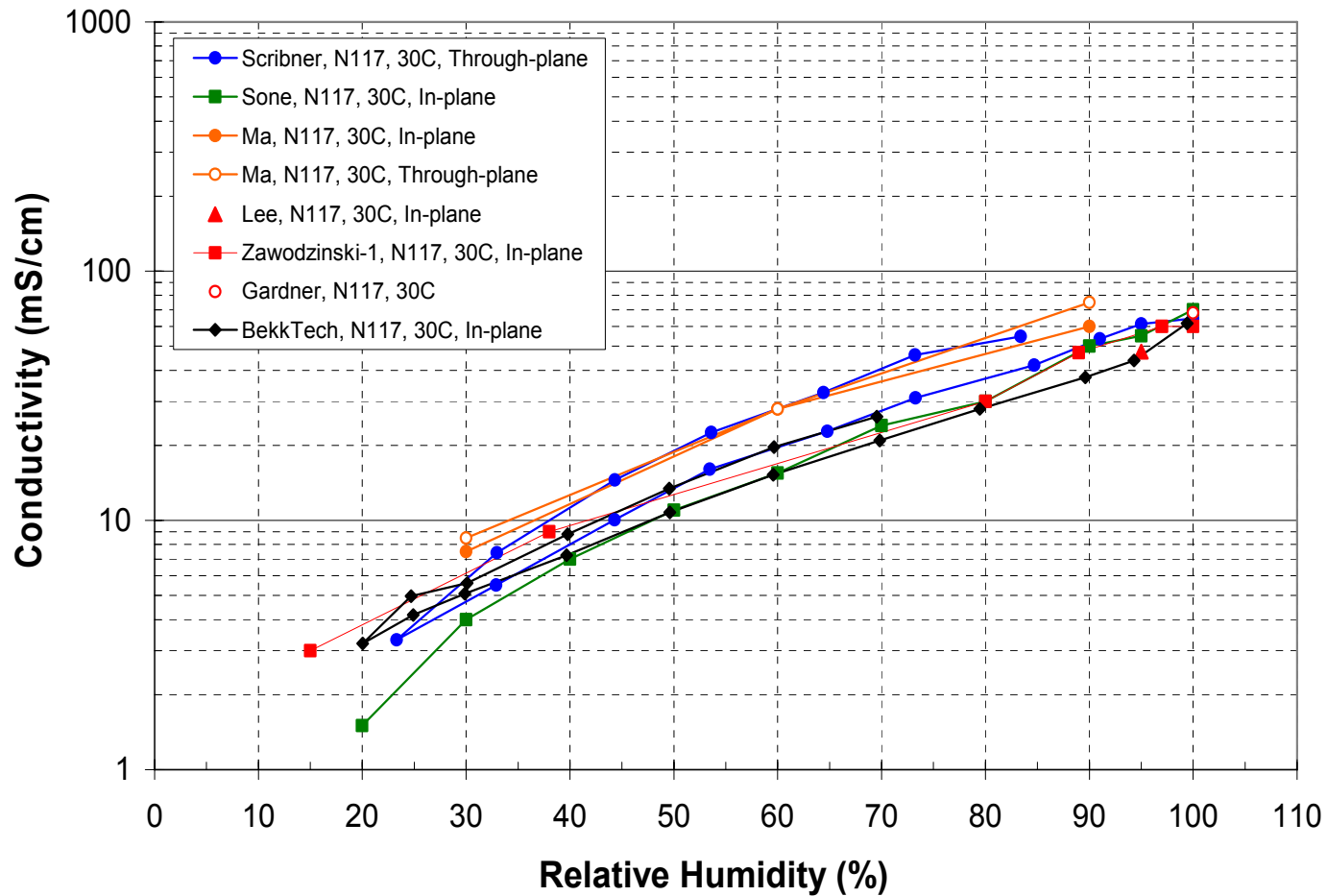
Accomplishments/ Progress/Results

Nafion NRE-212 Conductivity at 80 °C, 100 kPa
In-Plane (BekkTech) and Through-Plane (Scribner)





Summary of Reported Nafion 117 Conductivity at 30 °C





Future Work

- Complete trials on 800 EW material
- Complete analysis of in-plane and through-plane conductivity of commercial membranes (late Sept. 2007)
- Complete characterization of first three membranes from Topic 1 awardees (late Dec. 2007)
- Demonstrate conductivity of 0.07 S/cm, 50% RH, 25 °C (late Dec. 2007)
- Establish MEA test protocol (June 2008)



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- Demonstrated ability to fabricate baseline membrane materials (Nafion[®], SPEEK, and SPEKK). Demonstrated ability to test in-plane conductivity and developed method for through-plane conductivity. Again demonstrated improved conductivity due to PTA addition.



Summary

Tech. Trans. /Collaboration- Formed active partnership with BekkTech and Scribner Associates. Established formal working relationship with the HTMWG

Proposed Future - Fabricate and test new membrane formulations with low EW poly[perfluorosulfonic acid] ionomers, and new formulations of SPEEK,SPEKK. Perform conductivity test on these membranes, as well as the membranes supplied by the Task 1 members. Conductivity testing will be conducted using the new tooling and procedures that are verified in this program.