

# Technical Assistance to Developers

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### Los Alamos National Laboratory

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Project ID: FC052



# **Overview**

- Timeline
  - Start: 10/03
  - End: ongoing
  - % complete: N/A

- Budget
  - Technical Assistance to Developers" funded at \$570K
    - DOE share: 100%
    - Contractor share: N/A
  - Much DOE-directed effort under the parent task generates proprietary data
  - FY11 funding: \$570K

- Barriers
  - Sharing technical assistance to developers
  - A. Durability
  - B. Cost
  - C. Electrode performance
- Partners/Collaborators
  - Full list Available



# **Relevance/Objective**

### How to Obtain Technical Assistance:

This task supports Los Alamos technical assistance to fuel-cell component and system developers <u>as directed by the DOE</u>. Assistance can be made available through DOE Program Contact, Nancy Garland.

### **Objective**

This task is expected to include testing of materials and participation in the further development and validation of a single cell test protocols with the U.S. Fuel Cell Council. This task also covers technical assistance to Working Group 12, the U.S. Council for Automotive Research (USCAR) and the USCAR/DOE Fuel Cell Technology Team. This assistance includes making technical experts available to the Tech Team as questions arise, focused single cell testing to support the development of targets and test protocols, and regular participation in working and review meetings.

## **Technically Assisted/Collaborators:**

- Fuel Cell Tech Team
- DOE Durability Working Group: Develop various new material durability Testing protocols
  - Working group participants include:
    - LANL: P. Zelenay, E. Brosha, M. Wilson, R. Borup, R. Mukundan
    - External: S. Kocha, M. Perry, G. James, S. Wessel, P. Atanassov, D. Meyers
- Treadstone Conghua "CH" Wang Metal Bipolar Plates
- Hydrogen Engineering Center of Excellence---Diborane Experiments
- INVAR Neutron Imaging Holder
- PNNL Catalyst support measurements
- Ballard Power Systems Characterization



## Durability Working Group Activities: AST Protocol Development

- 1) RDE/RRDE measurements of Performance and Durability of Non-PGM ORR Electrocatalysts
- 2) ORR Electrocatalysts Performance & Durability Measurements in Half Cell with Liquid Electrolyte under RDE/RRDE Conditions
- 3) Protocols for Evaluating Alternative Electrocatalyst Supports for PEMFC
- 4) Start/Stop Protocols for Durability Life Testing



### Approach to develop ASTs:

- Solicit and incorporate involvement/participation from the industry, university and national lab experts
- Stress protocols must:
  - target anticipated component failure modes
  - be quick and effective
  - be easy to reproduce in any laboratory
- Example: Preliminary catalyst screening using rotating ring disk electrode technique (RRDE)
  - saves time for material comparison
  - failure mode detection
  - experimental conditions easily controlled
- Fuel cell testing limited to only the most promising catalysts



#### **Experimental Apparatus**





Teflon<sup>®</sup> Ring Disk

Oxygen reduction reaction (ORR)

> Ring current  $H_2O_2 \rightarrow O_2 + 2 H^+ + 2 e^-$



Rotating Ring Disk Electrode Technique (RRDE) allows:

- Electrochemically generated species to be monitoring as they past the ring by laminar flow
- Investigating reactive mechanisms
- Commercially available equipment

Disk current  $O_2 + n H^+ + n e^- \rightarrow x H_2O + y H_2O_2$ 



### **Technical Accomplishments/Highlights** Our approach to develop ASTs cont'd:

Pyrolyzed Fe/polyaniline catalyst  $(O_2 - top, no O_2 - bottom)$ 

Pyrolyzed Fe/cyanamide catalyst  $(no O_2)$ 





Stress test should include potentials more cathodic than the reversible surface red-ox system, where surface Fe is weaker bound Stress test should be performed in an environment similar to that in a fuel cell  $(H_2SO_4)$ 

Performance Comparison of Nafion<sup>®</sup> and Aquivion<sup>®</sup> Evaluation of short-side chain ionomer (Solvay Solexis)



Ionomer	LSC PFSA (Nafion <sup>TM</sup> )	SCC PFSA (Aquivion <sup>TM</sup> )
EW	1000	830
Density (g/cm <sup>3</sup> )	1.97	2.06
Conductivity (S/cm)	101	147
Water uptake (wt.%)	19	30



### Performance Comparison of Nafion<sup>®</sup> and Aquivion<sup>®</sup>

#### **Experimental Details**

MEA fabrication, testing conditions, and materials used were identical except for the cathode ionomer: Nafion and Aquivion.

Testing conditions: 80°C, 30 psig, 100% RH, H<sub>2</sub>/Air : 200/500 sccm fixed

```
Catalyst Ink– 5:2 catalyst to ionomer (weight)
20 wt% Pt/C
Catalyst loading – 0.2 \text{ mg}_{Pt}/\text{cm}^2 (1.0
\text{mg}_{catalyst}/\text{cm}^2)
Hot press condition with Nafion 212 Na<sup>+</sup>
membrane, 2 steps (300, 700 pound) hot
press under 210 °C for 3 min on each step
Boiling in 0.5 M H<sub>2</sub>SO<sub>4</sub> solution and D.I. water
```

#### **Experimental Results**





## Scanning XRF

- X-ray spectroscopic chemical analysis at 30 micron intervals
- Polycapillary optics provide 2000 times increase in signal over previous systems
  - Detection limits improved



#### Uses

- Determine coating uniformity
- Impurity uptake
- Measurement Localized catalyst loss
- Optical inspection of MEAs



### Analysis for MEA Uniformity

Scanning XRF using two Pt peaks at different energy provide complementary information:

•L lines show total Pt distribution (both sides of MEA)

•M lines show only one side of MEA



### **Invar Hi-Res NI Fuel Cell Holder**

#### **Prior Generation**





- LANL designed, built, and provided a new improved high resolution fuel cell holder for neutron imaging at NIST. New holder provided to NIST to make available for all users.
- Previous HDPE holders exhibited movement in response to changes of temperature-particularly problematic for freeze start studies between -20 and 80 °C.
- New holder made with Invar, also known as FeNi36 (64FeNi in the US), is a nickel steel alloy notable for its uniquely low coefficient of thermal expansion--  $\alpha$  of about 1.2 × 10<sup>-6</sup> K<sup>-1</sup> (1.2 ppm/°C).
- New holder design allows for horizontal or vertical mounting, is robust and rigid, and with its short arm extensions provides room for covering the cell with insulation.

**Invar Holder** 

#### Invar and Fuel Cell



### Hydrogen Engineering Storage Center of Excellence

**Request:** Study the impact (if any) of Diborane as an impurity in the anode feedstream of a 50  $cm^2$  operating fuel cell.



0.2

-0.005

-0.01

0.4

0.6

Voltage vs HRE

1.2

1

1.4

1.6

Status: Tests are on-going Testing multiple inlet concentrations

### **Support of DOE Catalyst Support Project**

**Pacific Northwest National Laboratory** 

(Vilayanur V Viswanathan)

-0.01

Request: Test two 25 cm<sup>2</sup> MEAs at 80°C, 100% RH, with 3 stoichs at anode and cathode using air and oxygen. Measure AC Impedance and Cyclic Voltammetry

![](_page_14_Figure_4.jpeg)

Voltage vs HRE

![](_page_14_Figure_5.jpeg)

# Summary

- Numerous collaborators have been assisted with other assistance in progress.
- Protocols are in the early stage of development, work is on-going, protocols to be vetted through DOE Durability Working Group members.
- State-of-Art characterization equipment used to support Fuel Cell developers and DOE funded projects
- Novel equipment manufactured and is available for use.

# Acknowledgement

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