

Component Benchmarking

Subtask Reported:

*Single Cell Testing Second Round Update and
Technically-assisted Industrial and University
Partners*

Principal Investigators*

Tommy Rockward

Los Alamos National Laboratory

May 2006

* Entire Los Alamos Fuel Cell Research Team

LA-UR-06-2957

Project # FCP 9

Overview

- Timeline
 - Start: 10/03
 - End: ongoing
 - % complete: N/A
- Budget
 - FY05 funding: \$250K
 - FY06 funding: \$350K
 - DOE share: 100%
 - Contractor share: N/A
- Barriers
 - A. Durability
 - B. Cost
 - C. Electrode performance
- **Partners/Collaborators**
 - Next slide

Technically-Assisted Collaborators/Partners

- USFCC
- Working Group 12 Doc: ISO 14687
Hydrogen Quality Standard
- Donaldson
- OSRAM/Sylvania
- Brookhaven National Laboratory
- University of New Mexico
- NASA
- University of Illinois
- Oak Ridge National Laboratory
- Augustine Scientific
- Porous Materials Inc.
- Surface Measurement Systems
- Gore
- FreedomCAR (GM, Ford, and
Daimler-Chrysler)
- University of Texas
- Air Force Research Lab
- NRC – Canada (Ottawa)

Approach

- Component Benchmarking (standardized testing resulting in confidence in MEA and component performance is essential to overcoming Fuel Cell Barriers)
 - USFCC single cell test protocol
 - Durability protocol development
- Technical Assistance to Developers (sharing technical assistance to developers)
 - Most DOE-directed effort under the parent task generates proprietary data

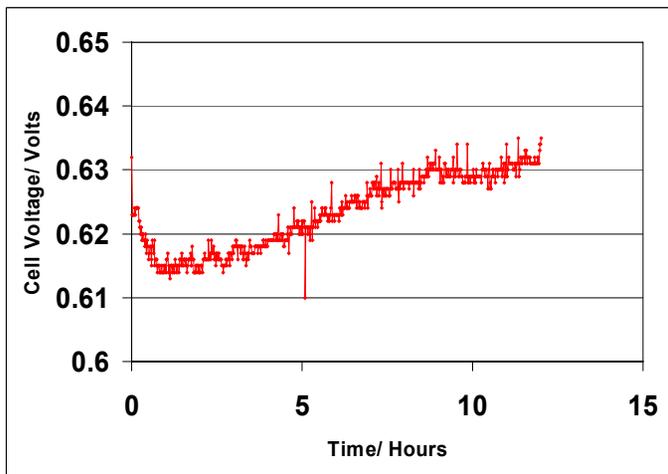
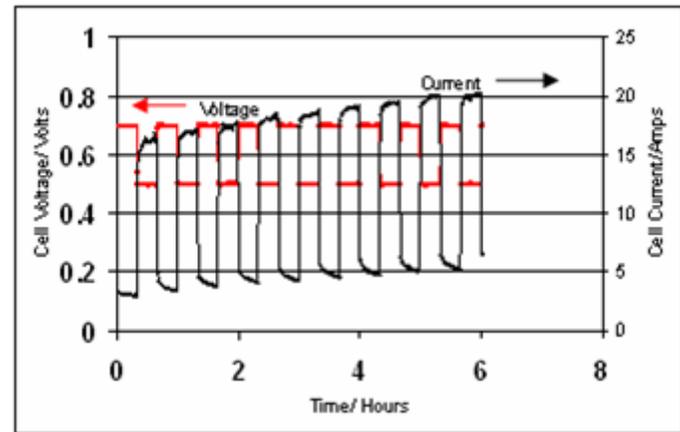
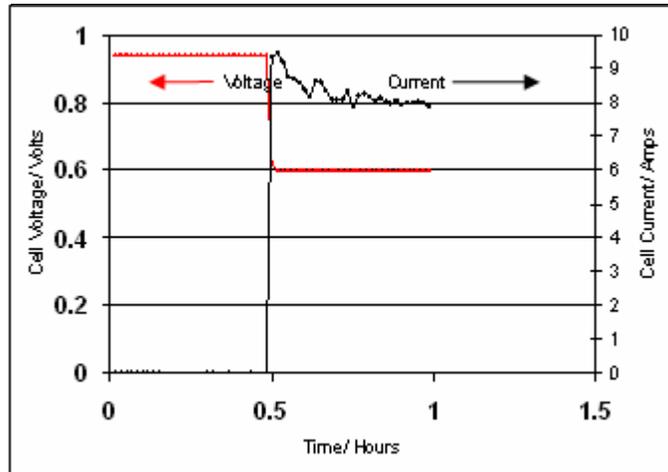
Single Cell Task Force

- **Scope**
 - Standardized fuel cell testing protocol to provide a means for comparisons
- **Intent**
 - To enable publishing of test results of a material or component in a consistent, verifiable manner
- **Method**
 - Use of baseline hardware and materials with standard protocol for leak check, break-in, conditioning and polarization curves
- **Amendments to Protocol**
 - Initial break-in polarization curve performed at higher temperature (80°C)
 - Agreed upon data format
 - Increased number of testing cells to five

Single Cell Test Protocol

- Cell Assembly
- Pressure Testing
 - Verify proper hardware sealing
 - Determine a gross crossover leak rate
 - Electrochemically determine hydrogen crossover
- Initial Break-In
- Conditioning
- Polarization Curve

Break-In Stages

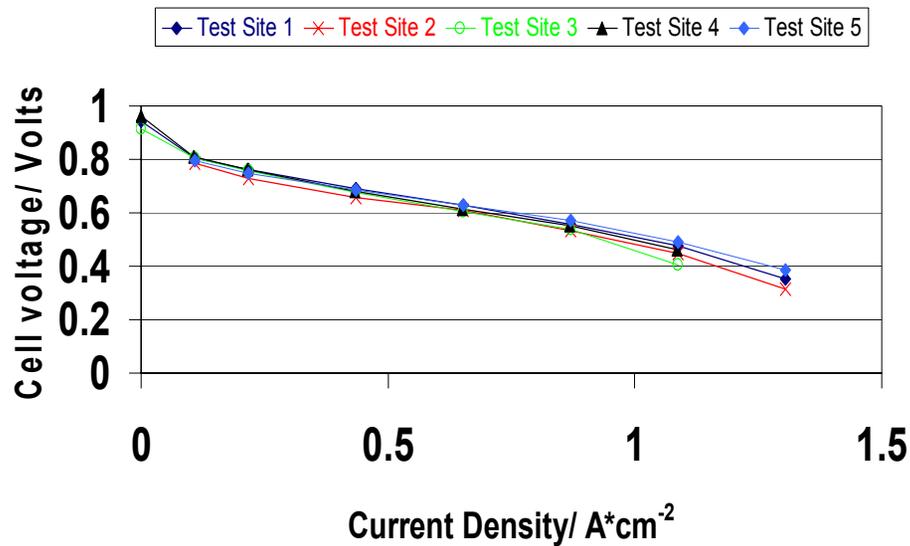


- First Stage
 - Voltage Cycling at 30 minutes per setting (0.94V to 0.60V at 10 Stoich, 10 Amps)
- Second Stage
 - Voltage Cycling for 6 hours at 20 minutes per setting (0.70V to 0.50V)
- Third Stage
 - Constant Current (10A) for 12 Hours

Conditioning Stage

- Necessary to re-humidify the fuel cell prior to running a test
- 20A Load for 4 hours
- Considered complete when voltage is equilibrated(2-3 mV)

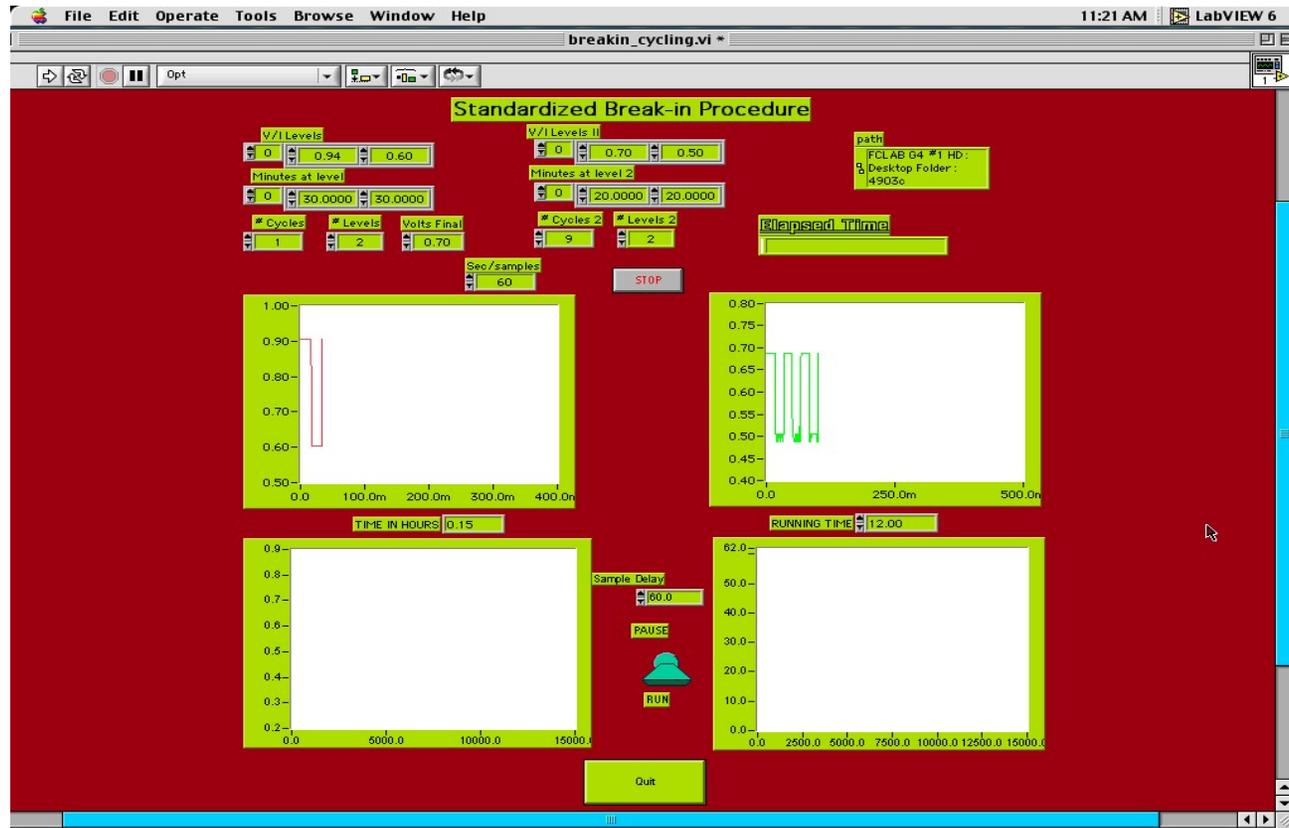
Subsequent Testers: Generate Polarization Curves



Steps	Current (Amps)	H ₂ Flow (SLPM)	Air Flow (SLPM)
0(< 1 m)	0	0.042	0.166
1	5	0.042	0.166
2	10	0.084	0.332
3	20	0.167	0.663
4	30	0.251	0.995
5	40	0.334	1.327
6	50	0.418	1.658
7	60	0.501	1.990

- Protocol I (60oC, Ambient pressure)
- Protocol II (80oC, 25 PSIG)

LANL's Role: Second Round-Robin Testing



- Qualify cells via leak tests
- Perform initial fuel cell break in
- Conduct post-cell tests

Future Plans

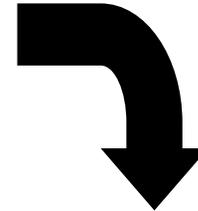
- To utilize lessons learned to improve the existing protocol
- To expand the testing protocol to include longevity and durability testing
- To define a calibration procedure for test stations running these types of measurements

LANL's Fuel Cell MEA Fabrication



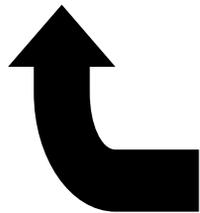
**Ink Catalyst Preparation
(PEM, DMFC, Sprayable Inks)**

**Initial Membrane
Treatment**



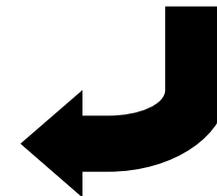
**Applying Electrodes
(substrate, direct, GDL)**

Fuel Cell Assembly

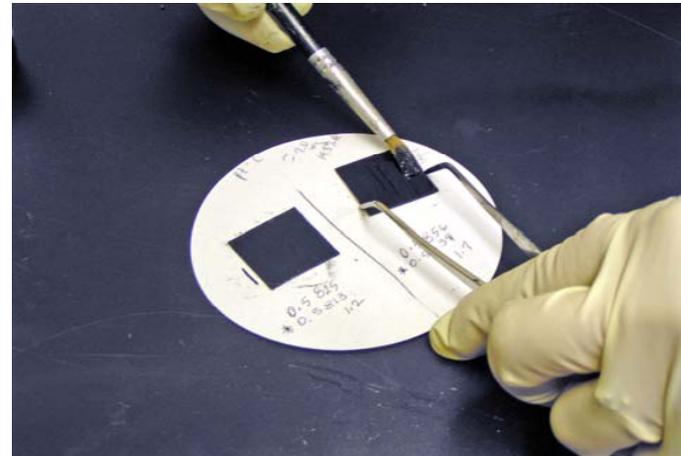
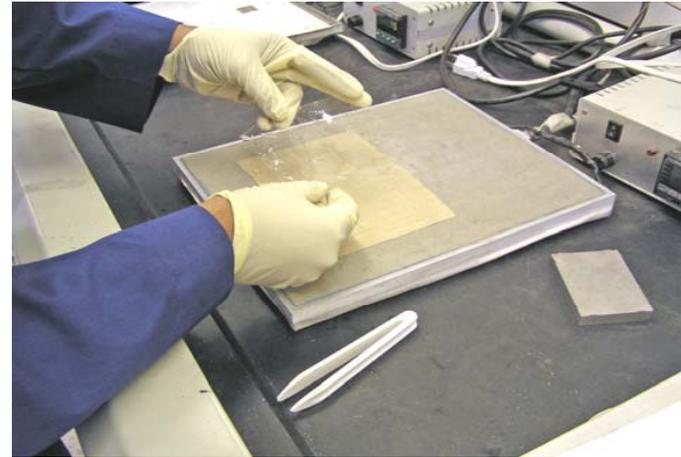


**Transfer Techniques
(Hot Press, Interfacial layers)**

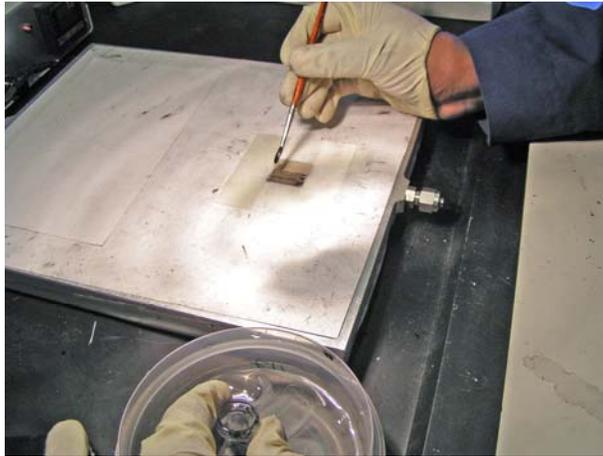
Post Membrane Treatment



Steps in LANL's MEA Preparation

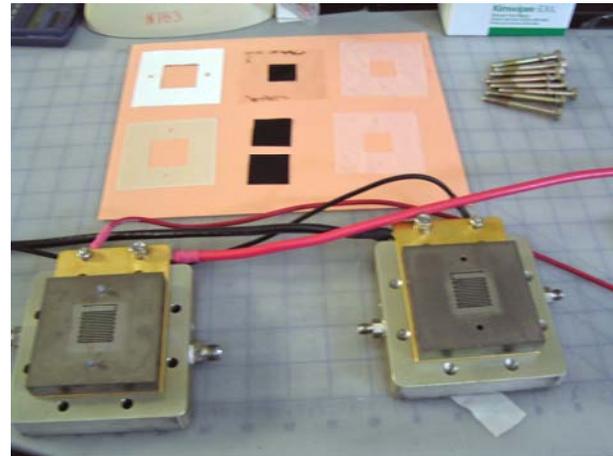


Steps in LANL's MEA Preparation



LANL's Procedure and Protocol*

- Components
 - Various Sizes of Hardware
 - Membrane type: N112, N1135, N115 or N117
 - Pt-Loading: 0.2 mg Pt/cm² (20% Pt/C each electrode)
 - Verified by substrate calculation and XRF measuring
 - Profilometry for coating uniformity
 - Backings: 1-sided/2-sided ELAT
 - Orientation is fuel cell size dependent (water mgmt.)
 - Sealing Materials
 - Silicon gaskets & teflon masks
- Cell Assembly
 - Five layer configuration and gaskets
 - Torque
 - Star-like pattern 25 in-lbs increments to ~90 in-lbs
 - H₂ Leak Test
 - Probe exterior hardware with handheld H₂ detector
 - Dead-end hardware outlet



*Variation can be incorporated to suit hardware and/or testing scope

LANL's Procedure and Protocol*

- Initial Operating/Break-in Conditions:
 - $T_{\text{cell/anode/cathode}}$: 80/108/80°C
 - $P_{\text{anode/cathode}}$: 30/30 psig
 - H_2/Air Flowrate: size dependent
 - Break-in Procedure:
 - Constant Current or Voltage
 - Current or Voltage Pulsing
 - Measure and Record as a function of time
 - Measure and Record High Frequency Resistance vs. time
- Core Testing: (Before, During & After)
 - Gas diffusion contact angle measurements (Sessile drop Method)
 - Cyclic Voltammetry
 - Anode/cathode
 - H_2 cross-over
 - H_2 Pump Experiments
 - VIR (H_2 and air Flowtracking)
 - MKS Flow controller calibration (corrected for temperature & pressure)
 - Constant Voltage or Current tracking

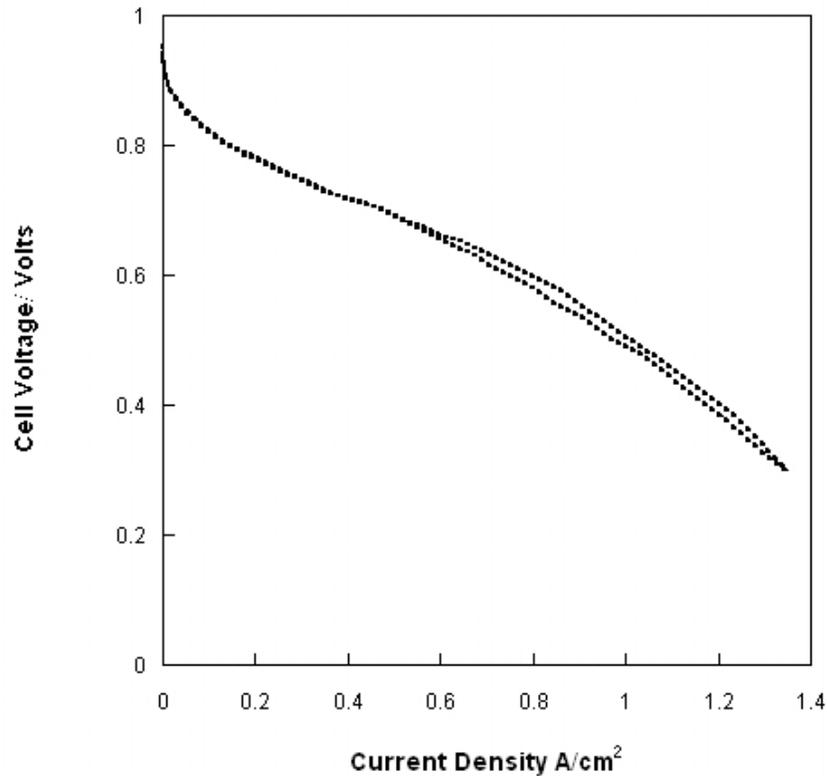
LANL's Baseline VIs

LANL-Baseline 50cm²

A. & C. : 0.2 mg Pt/cm²

N112, Tcell: 80°C, P: 30 psig

Utilization: H₂/Air: 83%/50%

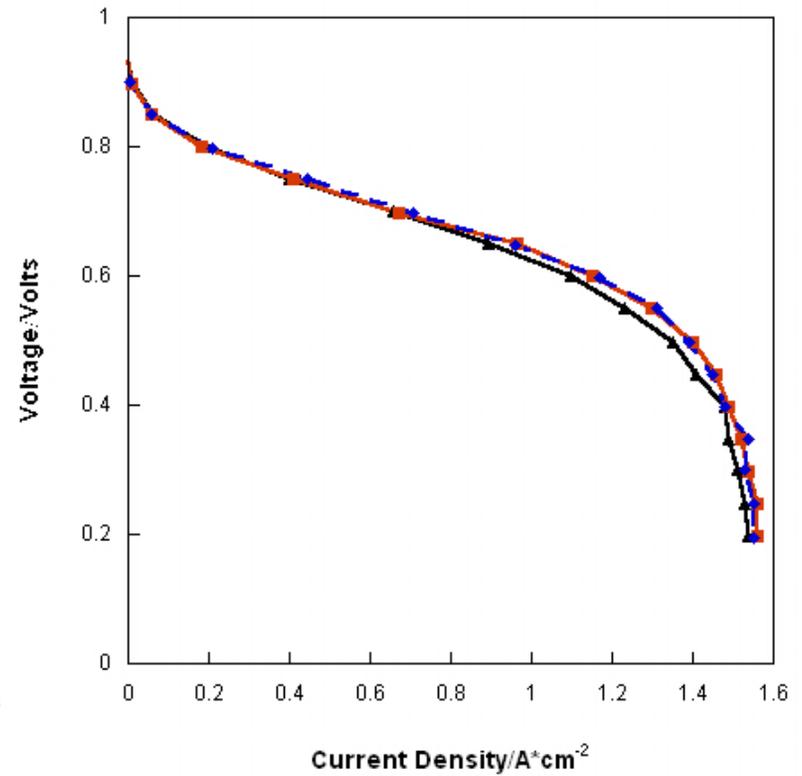


LANL-5 cm²

A. & C. : 0.19 mg Pt/cm²

N112, Tcell: 80°C, P: 30 psig

H₂/Air: 160/550 sccm



LANL Testing Facility/Equipment

Testing Facility:

- Over sq. ft of Lab space
- A Class 100/10000 cleanroom
- 6 Fume hoods
- 2 Hydrogen electrolyzers distributed to every lab
- 2 Pd-membrane Hydrogen Purifiers
- 2 Central oil-free air supply systems distributed to every lab
- 2 Centralized de-ionized water systems distributed to every lab
- 2 CNC numerical controls
- 2 Modular fuel processors
- 2 Reformers (Diesel and Gasoline)
- 4 Hot presses for preparing MEAs

Fuel Cell Testing Equipment:

- 33 Single-cell FC test stands
 - PEFC and DMFC compatible
 - Stoichiometric Flow tracking
 - Drive-Cycle and/or potential cycling
 - Capable of custom gas mixing
 - Multiple hardware sizes
 - Computer automated
- 1 LANL designed Segmented cell and in-house developed software
- 2 Fuel cell stack test stands (upgradeable 20kW load bank)
- 2 Fuel Processing Test stands (capable of chemical flows equivalent to 50kW)
- Freeze-Thaw Environmental Chamber

LANL Materials Testing Facility/Equipment

In-house characterization capabilities for analyzing material:

- X-ray: (Fluorescence, Diffraction, and energy dispersive spectroscopy)
- Thermogravimetric, Differential Thermal, and Evolved Gas Analysis
- Differential Scanning Calorimetry
- Atomic Force, Scanning Electron, and Optical Microscopy
- High pressure/high temperature adsorption/desorption measurements
- Dielectric microscopy
- Water Analysis
 - F⁻, ion selective probes, and ICP-MS

Gas Analysis Capabilities:

- 2 GC/Mass Spec
- 2 Mass Spectrometer
- 11 Gas Chromatographs
 - Including flame ionization, thermal conductivity detectors and Helium photo-ionization
- BET/chemisorption
- Multiple Non-dispersive Infrared CO and CO₂ analyzers
- 1 Density meter
- 1 Parr pressure reactor
- 1 Mercury porosimeter
- FTIR
- 5 paramagnetic gas analyzers
- FID

Technical Assistance to Developers

- Due to the proprietary nature of the data collected, limited data is available for public disclosure. LANL contributions may likely be found in DOE presentations of collaborators (see slide 3 **Technically-Assisted Collaborators/Partners**). For further information contact:

Ken Stroh, Ph.D., Group Leader
Deputy Director, Institute for Hydrogen and Fuel Cell Research
Program Manager for Fuel Cells & Transportation
PO Box 1663, Mail Stop D429
Los Alamos National Laboratory
Los Alamos, NM 87545
(505) 667-6832 MST-11 Group Office
(505) 667-7933
stroh@lanl.gov

Future Work

- Remainder of FY 06
 - Second round of round-robin testing for USFCC single cell test protocol
 - Contribute to durability protocols
 - Continue assistance to developers at DOE discretion
- FY 07
 - Complete testing for USFCC single cell test protocol
 - Contribute to durability protocols
 - Continue assistance to developers at DOE discretion

Project Summary

Goals

- Provide assistance to fuel cell community in establishing standardized testing protocols (i.e.. single cell and durability)
- Provide technical assistance to developers to accelerate fuel cell commercialization

Achievements

- Completed first round of USFCC single cell protocol testing and initiated second round
- Participated in durability and accelerated test protocol establishment
- Provide assistance to developers

Critical Assumptions and Issues

- Standardized Testing
 - Need input from industry and other researchers
 - Specialized/calibrated equipment may be required for testing.
 - Protocols need to be accepted and widely implemented to assure value.
 - We are participating in working groups and protocol development in order to promote accepted standards and procedures.

Response to Reviewers Comments

- “Need to bring in more analytical resources to support durability testing.”
 - We have significant analytical tools (see slides 18 and 19 for example) and have used these extensively for durability testing (see DOE presentation FC 28, PEM Fuel Cell Durability, LANL).
- “Through USFCC, become the advocate of standard method for durability and accelerated test protocols.”
 - We have been a significant contributor to a USFCC durability test protocol, and our involvement is continuing.

Publications and Presentations

- David Lane (W.L. Gore and Associates), Eric Teather (DuPont Fuel Cells), Tommy Rockward (Los Alamos National Laboratory - LANL), Francisco Uribe (LANL), Dawn McNeil, (Teledyne Energy Systems), Ross Bailey (Greenlight Power Technologies), Michael Pien (ElectroChem, Inc.), "Establishing a Standardized Single Cell Testing Procedure through Industry Participation, Consensus and Experimentation," Proc. 2004 Fuel Cell Seminar
- Davey et al., Overview of Fuel Cell Membrane Electrode Assemblies (MEAs) at Los Alamos National Laboratory (LANL), 1st Symposium on MEA Manufacturing, Dayton 2005
- *Proprietary letter reports to DOE and developer on DOE-directed Technical Assistance to Developers under parent task*