Component Benchmarking

Subtask Reported:

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

Principal Investigators*

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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

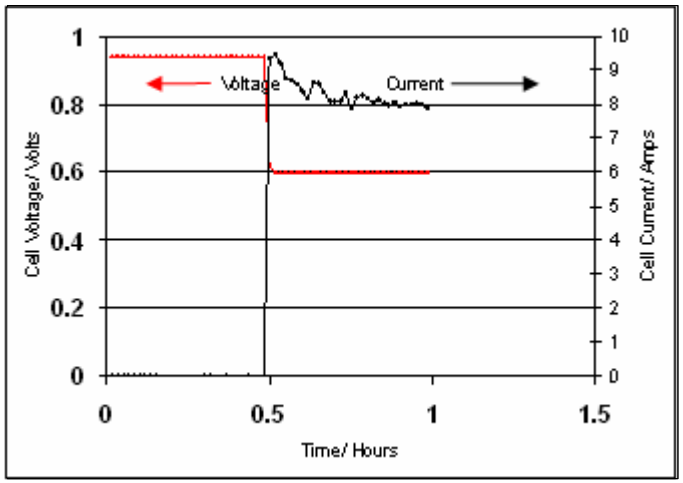
Single Cell Task Force

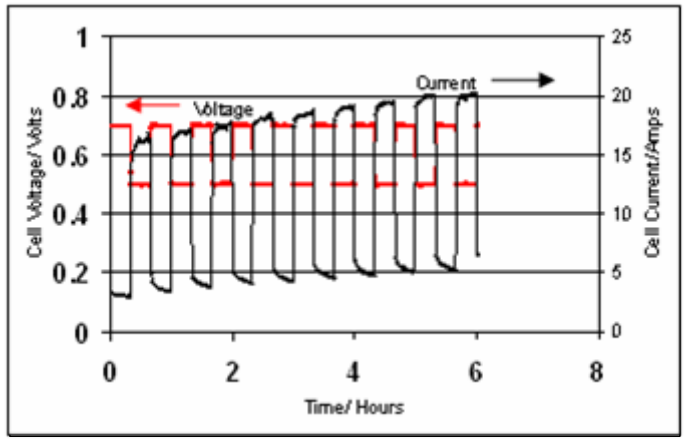
- **Scope**
  - Standardized fuel cell testing protocol to provide a means for comparisons
- **Intent**
  - To enable publishing of test results of a material or component in a consistent, verifiable manner
- **Method**
  - Use of baseline hardware and materials with standard protocol for leak check,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

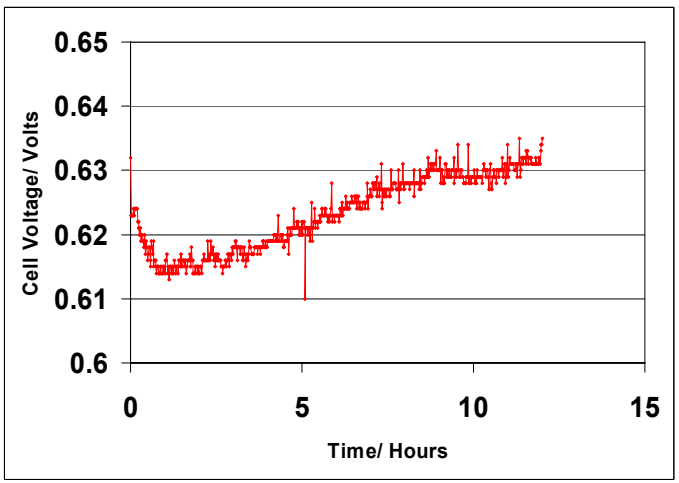
Single Cell Test Protocol

- 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





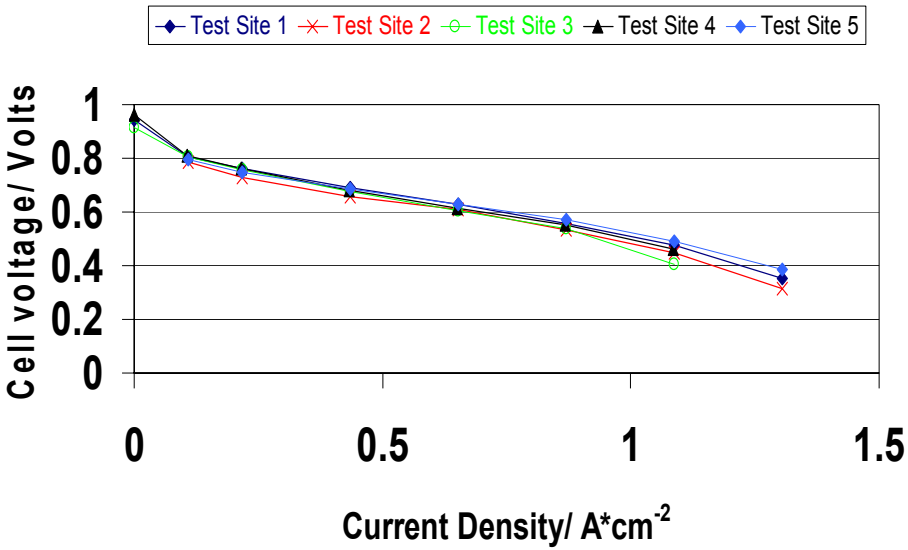


- First Stage
  - Voltage Cycling at 30 minutes per setting (0.94V to 0.60V at 10 Stoich, 10 Amps)
- Second Stage
  - Voltage Cycling for 6 hours at 20 minutes per setting (0.70V to 0.50V)
- Third Stage
  - Constant Current (10A) for 12 Hours

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)

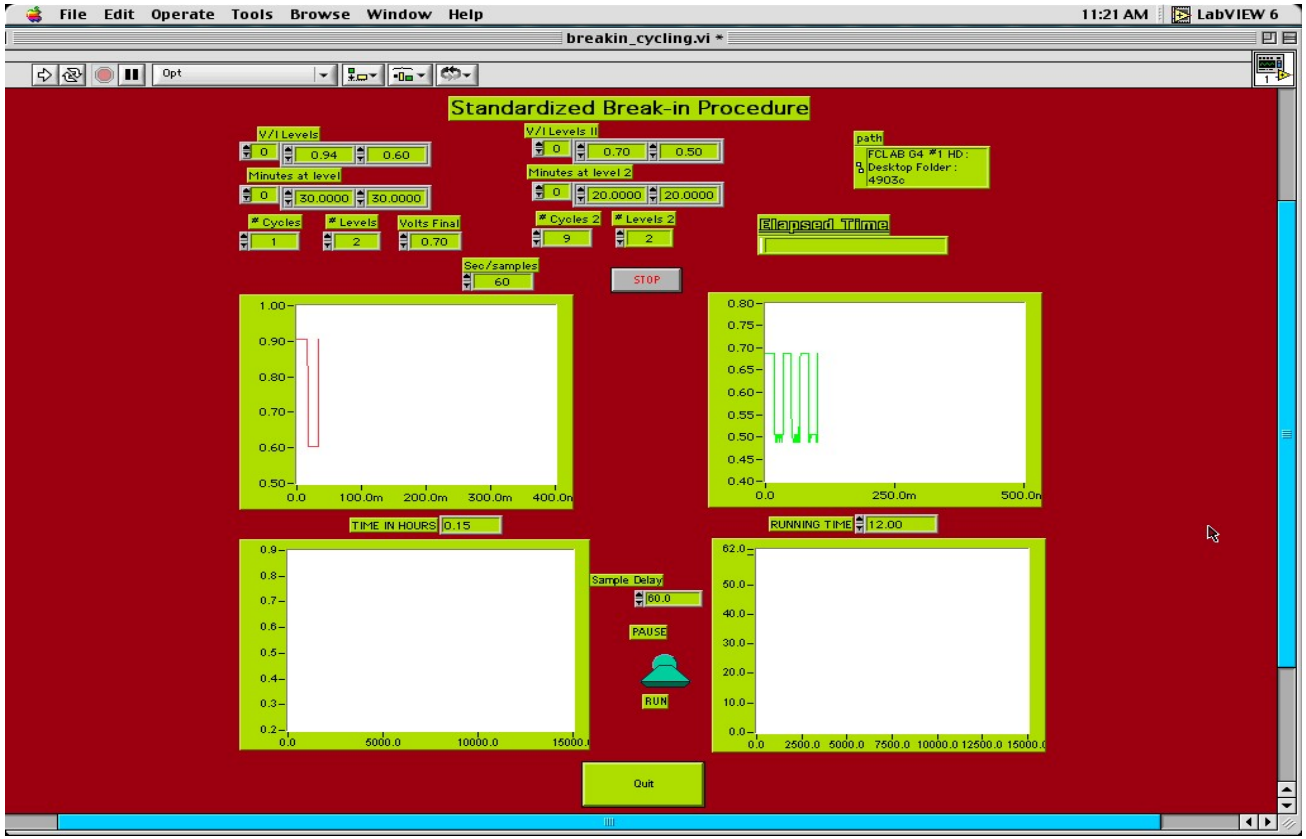
Subsequent Testers: Generate Polarization Curves



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

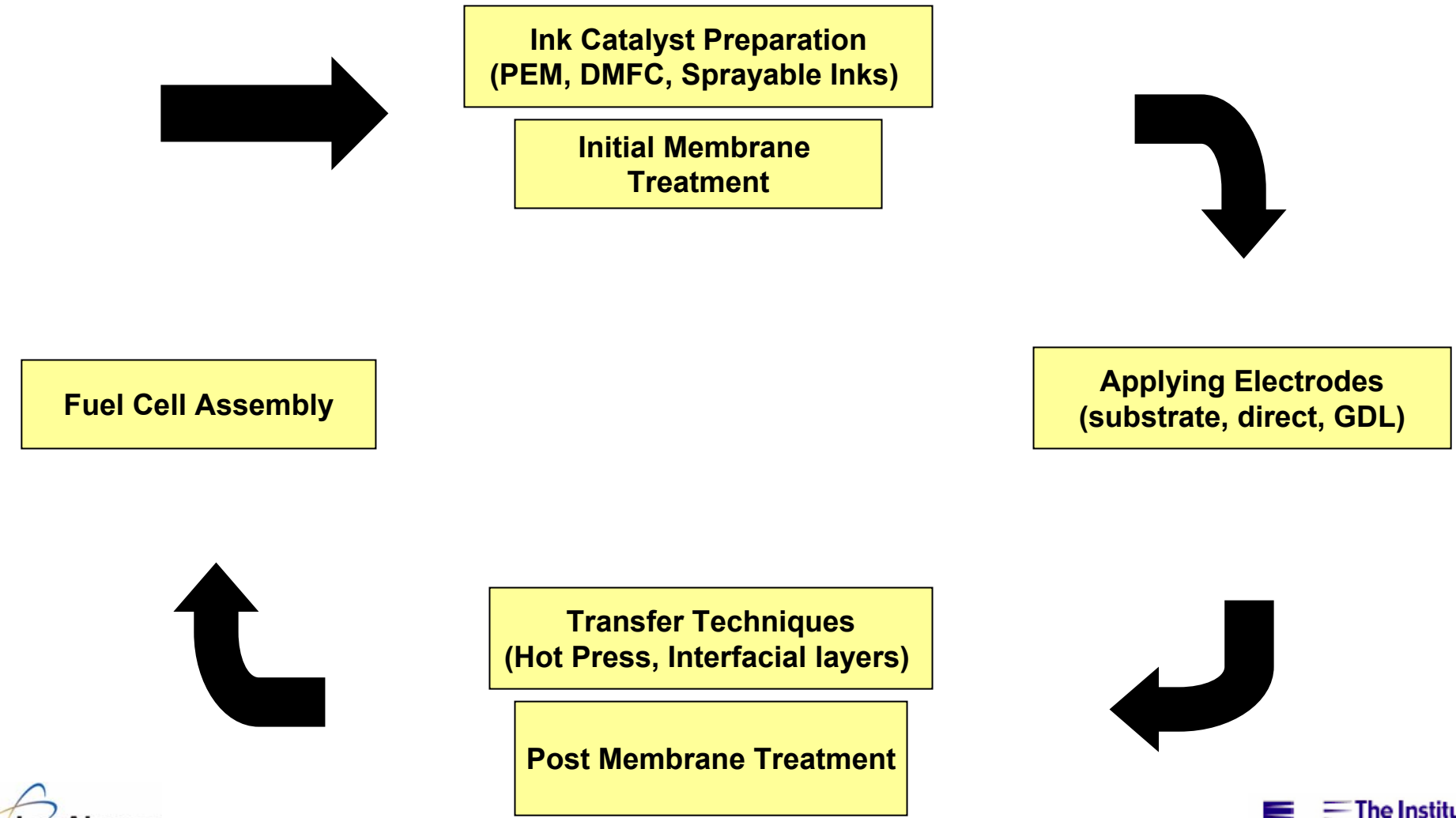


- Qualify cells via leak tests
- Perform initial fuel cell break in
- Conduct post-cell tests

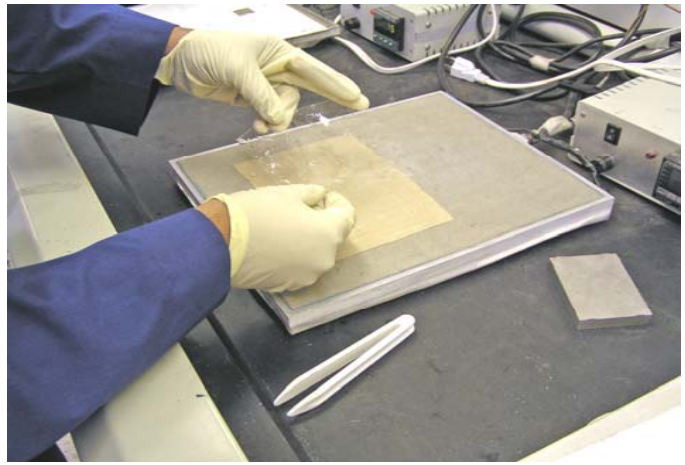
Future Plans

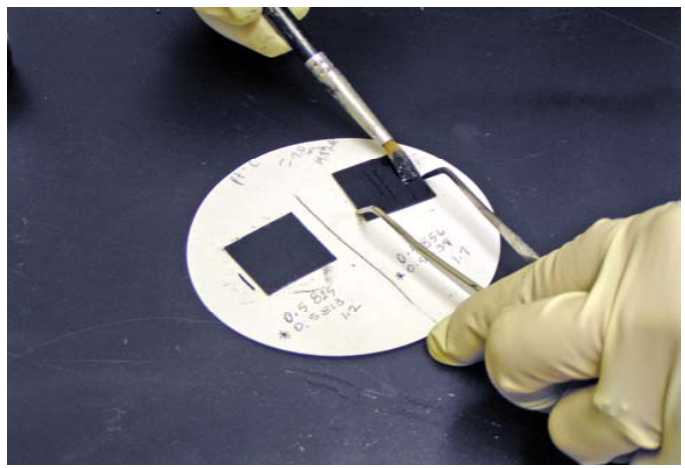
- To utilize lessons learned to improve the existing protocol
- To expand the testing protocol to include longevity and durability testing
- To define a calibration procedure for test stations running these types of measurements

LANL's Fuel Cell MEA Fabrication

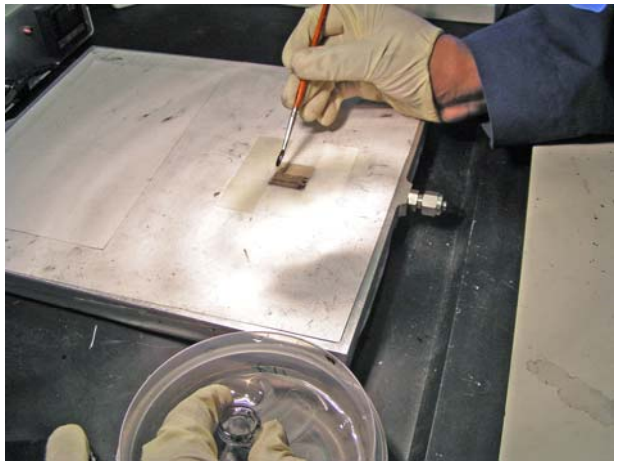


Steps in LANL's MEA Preparation





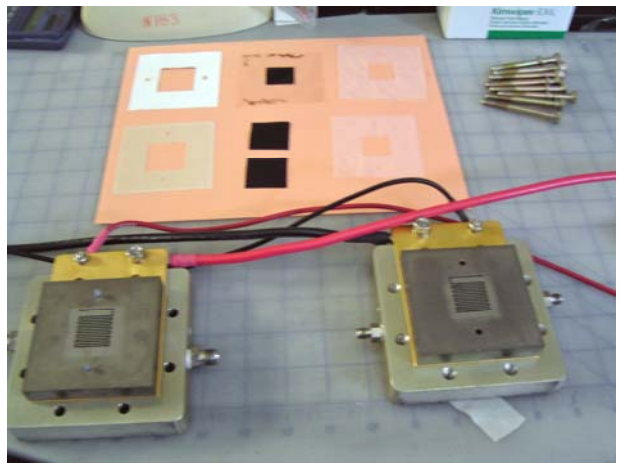
Steps in LANL's MEA Preparation



LANL's Procedure and Protocol*

- Components
  - Various Sizes of Hardware
  - Membrane type: N112, N1135, N115 or N117
  - Pt-Loading: 0.2 mg Pt/cm^2 (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
  - H_2 Leak Test
    - Probe exterior hardware with handheld H_2 detector
    - Dead-end hardware outlet



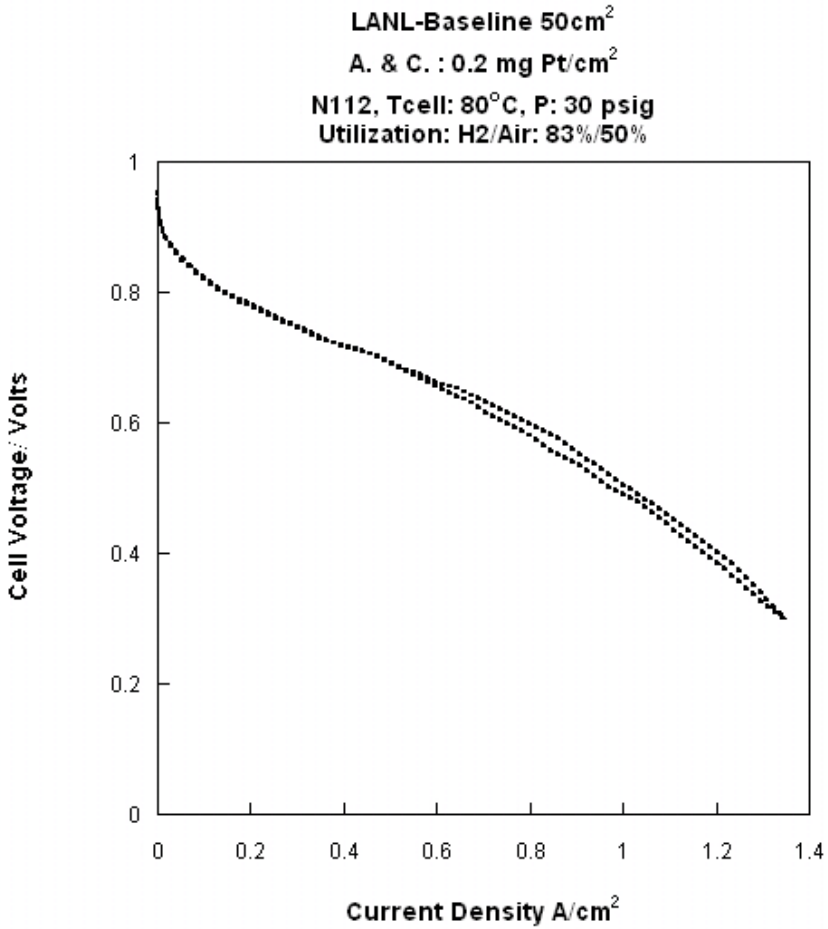
*Variation can be incorporated to suit hardware and/or testing scope

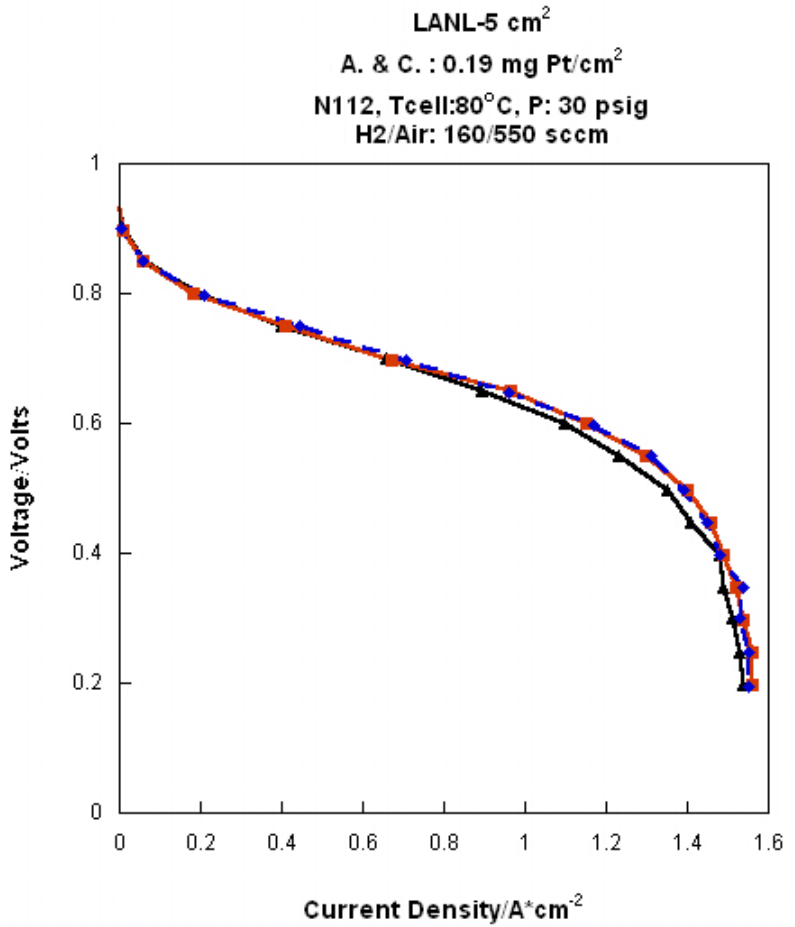
LANL's Procedure and Protocol*

- Initial Operating/Break-in Conditions:
  - $T_{cell/anode/cathode}$: 80/108/80°C
  - $P_{anode/cathode}$: 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

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

LANL's Baseline VIs





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 CO_2 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:

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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*