
Component Benchmarking

Subtask Reported:

USFCC Durability Protocols *and Technically-assisted Industrial and University Partners*

Principal Investigators*

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* Entire Los Alamos Fuel Cell Research Team

LA-UR-07-2812

Project ID#: FCP1

This presentation does not contain any proprietary, confidential or otherwise restricted information

Overview

-
- Timeline
 - Start: 10/03
 - End: ongoing
 - % complete: N/A
 - Budget
 - “Technical Assistance to Developers” funded at **\$350K/y**
 - DOE share: 100%
 - Contractor share: N/A
 - Most DOE-directed effort under the parent task generates proprietary data
 - FY07 funding: **\$570K/y**
 - Barriers
 - Understanding the durability of materials and components is essential to overcoming Fuel Cell Barriers
 - Sharing technical assistance to developers
 - A. Durability
 - B. Cost
 - C. Electrode performance
 - **Partners/Collaborators**
 - Full list Available

Objectives

This task supports Los Alamos technical assistance to fuel-cell component and system developers as directed by the DOE. 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 the U.S. Council for Automotive Research (USCAR) and the USCAR/DOE Freedom Cooperative Automotive Research (FreedomCAR) 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.

Approach: Visitors List



DOE Hydrogen Program

(Fuel Cell visitors to MPA-11/LANL June '06 – March '07)

- General Motors
- DaimlerChrysler
- Cabot Fuel Cells
- Osram Sylvania
- Electro-Science laboratories Inc
- Toyobo Co. Ltd Research Center
- Fuel Cell Technologies
- AES Corporation
- ATK GASL
- Oak Ridge National Lab
- Argonne National Lab
- Brookhaven National Lab
- Sandia National Lab
- NIST (National Institute of Science and Technology)
- (NRL) Naval Research Laboratory
- Norwegian University of Science and Technology
- SINTEF Materials & Chemistry (Norway)
- Korea Research Institute of Chemical Technology
- Japan National Institute of Advanced Industrial Science & Technology (FC-Cubic)
- Japan Minister of Economy, Trade and Industry
- Japan New Energy Development Organization

Visitors Cont'd



(Fuel Cell visitors to MPA-11/LANL June '06 – March '07)

DOE Hydrogen Program

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- National Research Council of Canada
 - NREL (National Renewable Energy Lab)
 - OGAs
 - University of Illinois (Urbana Champaign)
 - University of New Mexico
 - RPI (Rensselaer Polytechnic Institute)
 - Case Western Reserve
 - University of Texas at Austin
 - Virginia Polytech & State University
 - Dongshin University (Korea)
 - University of California (Riverside)
 - Hawaii Natural Energy Institute
 - U.S. Senator Pete Dominici
 - John Angell, U.S. Senate Finance Committee
 - DOE Deputy Secretary Clay Sell
 - New Mexico State Energy & Environment Advisor

Approach: Technically-Assisted Collaborators/Partners



DOE Hydrogen Program

- USFCC
 - Single Cell Task Force
 - Durability Task Force
- Working Group 12 Doc: ISO 14687 Hydrogen Quality Standard
- OSRAM/Sylvania
- Cabot Fuel Cells
- Dongshin University (Korea)
- Clemson University
- University of Quebec
- Augustine Scientific
- Porous Materials Inc.
- W.L. Gore
- SGL Carbon
- NEDO/AIST/LANL MOU & Workshop
- BASF
- CIDETEC - Centro de Tecnologías Electroquímicas
- U Conn
- FreedomCAR (GM, Ford, and Daimler-Chrysler) - FC/SC&S/Prod/H2Store/EC Store
- University of Texas (Austin)
- Brookhaven National Laboratory
- University of New Mexico
- Oak Ridge National Laboratory
- Argonne National Laboratory
- Sandia National Lab
- University of Illinois Urbana-Champaign
- University of California – Riverside
- SINTEF NTNU Norway
- Fuel Cell Technologies
- General Motors
- NREL (National Renewable Energy Lab)
- Virginia Polytech and State University
- University of North Dakota
- National Research Council, Canada
- NIST (National Institute of Science and Technology)

Invited Presentations

(21)

The 2nd FC-Cubic Workshop, Tokyo, Japan Feb 6, 2007 (**Borup**)
2nd Annual Fuel Cells Durability & Performance, Dec 8 2006 (**Borup**)
Advances in Materials for Proton Exchange Membrane Fuel Cell Systems
2007, Asilomar California, Feb 18 – 21, 2007 (**Borup & Pivovar**)
2nd Symposium on MEA Manufacturing, Dayton Ohio, August 2006 (**Davey**)
The 2nd FC-Cubic Workshop, Tokyo, Japan Feb 6, 2007 (**Garzon**)
Advances in Materials for Proton Exchange Membrane Fuel Cell Systems
2007, Asilomar California, Feb 18 – 21, 2007 (**Kim & Pivovar**)
9th Annual International Symposium, Small Fuel Cells, 2007, Miami, March
7-9, 2007 (**Kim**)
3M Company, St. Paul, Minnesota, January 5, 2007. (**Pivovar**)
Polymer Science Department, University of Southern Mississippi,
Hattiesburg, Mississippi, January 31, 2007. (**Pivovar**)
Alkaline Membrane Fuel Cell Workshop, Phoenix, Arizona, December 11,
2006. (**Pivovar**)

Invited Presentations

2nd Symposium on MEA Manufacturing, Dayton Ohio, August 2006

(Rockward)

Implementation Team, Western Governor's Association Policy Resolution 06-02, March 2007. **(Stroh)**

University of Rochester, Rochester, NY, July 27, 2006. **(Stroh)**

Department of Chemistry, University of Quebec, Montreal, Canada. April 2006. **(Uribe)**

Institut National de la Recherche Scientifique, Varennes, Quebec. Canada. April 2006. **(Uribe)**

3M Company, St. Paul, Minnesota, June 7, 2006. **(Zelenay - 2)**

Department of Chemical Engineering, New Mexico State University, Las Cruces, New Mexico, April 28, 2006. **(Zelenay)**

The Electrochemical Society, Local Twin Cities Section, Minneapolis, Minnesota, June 7, 2006. **(Zelenay)**

2006 Taiwan Small Fuel Cells Symposium, Longtan, Taiwan, June 27-29, 2006. **(Zelenay)**

3rd International Hydrogen and Fuel Cell Expo, Tokyo, Japan, Feb. 7-9, 2007. **(Zelenay)**

LANL Collaboration with the European Union (EU) -Fuel Cell Testing, Safety & Quality Assurance Program



Objectives:

- Collaborate with the EU in the frame of the FCTES^{QA} Program for formulating standard Fuel Cell Testing protocols.
- LANL participates in the reviewing, assessment and experimental evaluation of the protocols.

Activities:

1. Revision of FC Testing Protocols.
2. Participation in the FCTES^{QA} 1st Meeting (Venice, Italy, Sept. 21-12, 2006)
3. Participation in the FCTES^{QA} Workshop (Honolulu, Hawaii, Nov. 13, 2006)
4. Future participation in FC Testing Protocols round robins.
5. Will participate in hydrogen impurities follow-on project.

Results:

1. Revision of FCTES^{QA} Testing Protocols

LANL workers reviewed the FCTES^{QA} document “PEFC: Test Procedures, Version 1.0 (June 06)”.

- Full version reviewed (129 pages).
- Document’s corrections and comments were submitted.
- Recommendations on specifics about testing were proposed.

2. FCTES^{QA} 1st Plenary Meeting (Venice, Italy, Sept. 21-12, 2006)

- a) Attended to the Plenary Sessions on:
Stationary Power, PEFC, MCFC and SOFC .
- b) Active participation on specific discussions of various aspects of PEFC test modules. A module is characterized by the study of the effect of one main input parameter on one output.
- c) LANL extensive expertise in developing and testing similar protocols(*) in the USA was acknowledged in this workshop.
- d) LANL defined its potential collaboration on protocol evaluation round robins.

(*) Collaboration with the USFCC

3. FCTES^{QA} Workshop

(Honolulu, Hawaii, Nov. 13, 2006)

(along with the EU, USA, Japan, Korea and China)



- a) Workshop included three general subjects: Testing procedures for Stationary Applications; Revised FCTESTNET testing procedures on PEFC; and Fuel Quality issues.
- b) Presentation and discussion of PEFC session was centered on the following aspects of the testing protocols: objectives, technical aspects, approach, activities, and work plan.
- c) The activities includes the round robin testing plan and the contributions of the participating organizations.

LANL offered involvement in two round robins:

- i) Evaluation of the a FCTESTNET single PEFC protocol on 4 equivalent cells provided by EU. Same cells will also be tested by other organizations.
- ii) Evaluation of the standard test procedures of: EU, USA, Japan, Korea and China. (Each protocol will be evaluated with a home made single PEFC. The corresponding deliverable will be 4 polarization curves).

4. Working Group 12- Round Robin Tests

- a) LANL prepared and tested a 50 cm² fuel cell using LANL's MEA fabrication.
- b) The fuel cell hardware is being used in a round robin testing plan between several sites. These test are geared towards providing assistance and/or lab testing verification.
- c) LANL also provided testing guidance for the subsequent testers.

THE RESULTS OF THESE TESTS ARE KEPT CONFIDENTIAL UNTIL ALL SITES HAVE COMPLETED TESTING, THE TEST IS ON-GOING.

5. FreedomCAR Durability Protocols

DOE CELL COMPONENT DURABILITY TEST PROCEDURES AND METRICS FOR PEM FUEL CELLS

(Electrocatalysts, Supports, Membranes, and Membrane Electrode Assemblies)

- Electrocatalyst Cycle and Metrics
- Catalyst Support Cycle and Metrics
- MEA Chemical Cycle and Metrics
- Membrane Mechanical Cycle and Metrics

- **LANL review and comment of Durability Protocols**
 - Set to perform #1, #2, #3, #4
 - Performed versions of #1, #2

6. USFCC Durability Protocols

DOE Hydrogen Program

The Durability Task Force of the Materials and Component Working Group is developing standard accelerated stress test (AST) protocols to be used as a common baseline for the fuel cell industry. The intent of this is to:

1. Provide a common set of non-proprietary conditions to enable comparison of test data between companies.
2. Provide relevant testing conditions for those component developers who do not have extensive fuel cell experience.

6. USFCC Durability Protocols

DOE Hydrogen Program

Proposed protocols:

Cathode catalyst stability:

Test #1. Voltage cycling between 0.6 and 0.96V (H₂/air)

Test #2. Voltage cycling between 0.6 and 1.2V (H₂/N₂)

Cathode carbon corrosion:

Test #1. Potential hold at 1.2V

Test #2. Potential hold at 1.5V

Membrane durability:

Test #1: Fentons reagent test

Test #2: Steady state Open Circuit Voltage (OCV) test

Test #3: RH cycling (mechanical stability)

Test #4: Combined chemical and mechanical stability test

Durability Round-Robin:

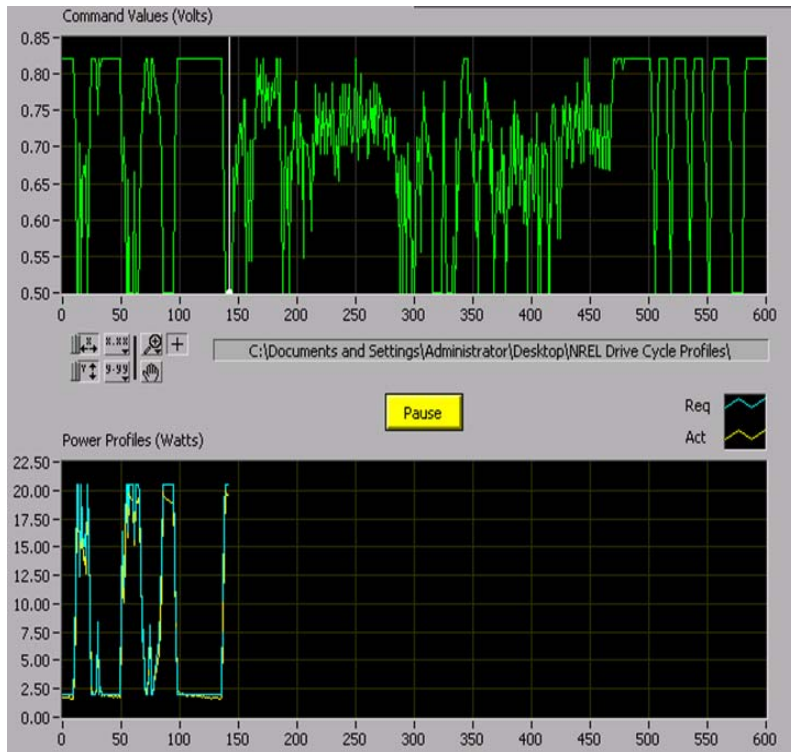
- LANL participated in the reviewing and development of the protocols.
- Committed to Participate in USFCC Accelerated Stress Testing (AST) Protocol Development Round Robin

6. Catalyst Stability Protocols

<p>Catalyst stability AST #1: Step change in voltage from 0.6V to 0.96V with air on cathode</p>	<p>Catalyst stability AST #2: Linear sweep in voltage from 0.6V to 1.2V with N₂ on cathode</p>
<p>Representative of fuel cell operation over the expected voltage ranges (e.g., OCV to 1.5A/cm²) Longer test, may be useful for an estimation of catalyst durability Does not include V spikes which may occur due to start-up/shutdown operations (system dependent)</p>	<p>This is an accelerated test useful for screening catalyst stability. This test may not be suitable for catalyst on very corrosion susceptible carbon supports, where significant corrosion may occur, confounding results. Includes V spikes that may occur due start-up/shutdown operations without potential control (system dependent). Useful for acceleration whether those spikes occur in system or not.</p>

6. Fuel Cell Drive Cycle Protocol Development

US06



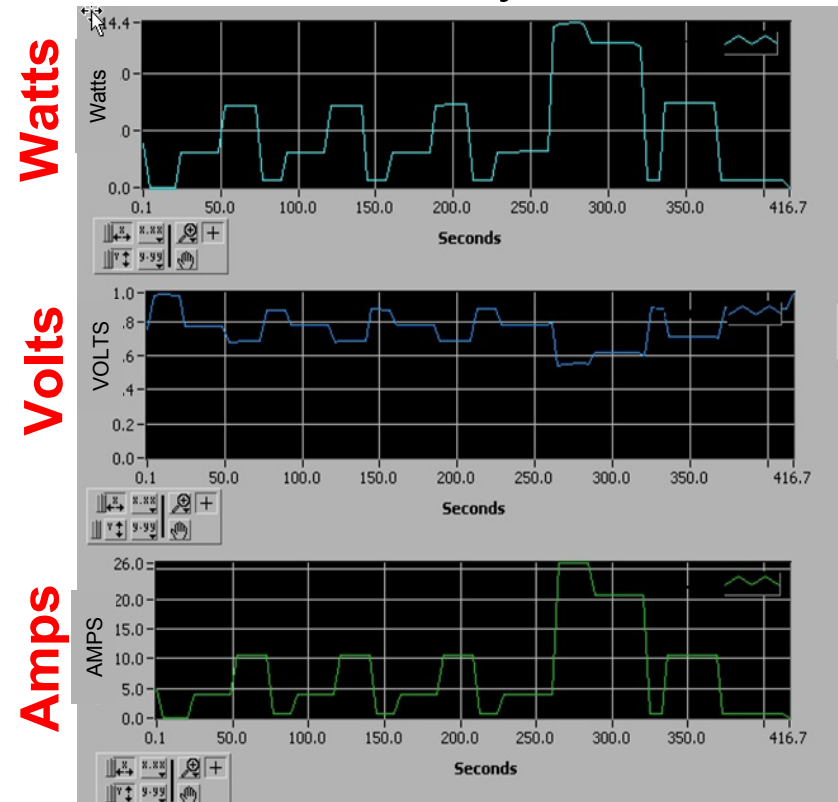
Volts

Watts

20 min

- 'Worst-case' Drive cycle depending upon Fuel Cell / battery hybridization

DOE Durability Protocol



Watts

Volts

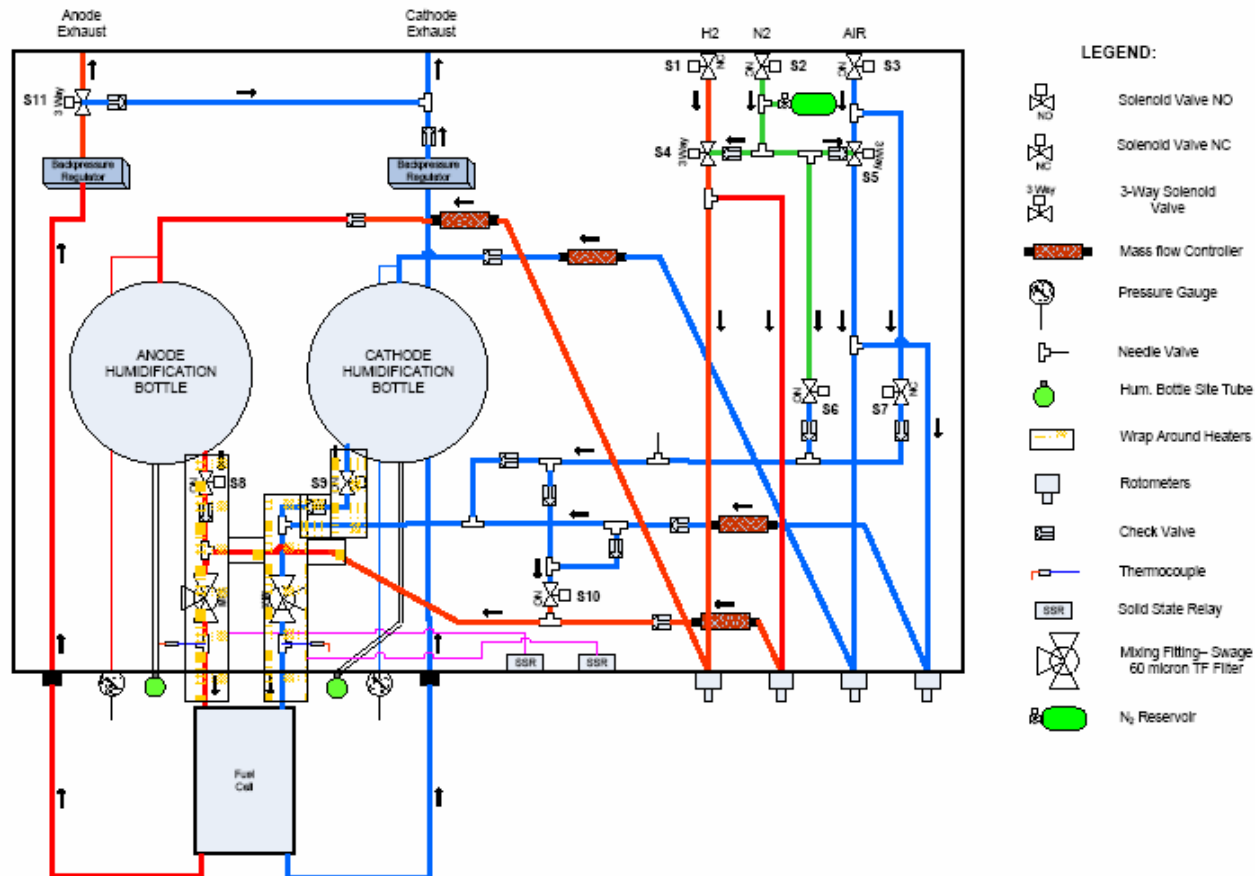
Amps

6 min

- Drive cycle defined by DOE, slower potential cycling – requires batteries providing transients

7. Transient Humidity Tracking

FUEL CELL TEST STATION
WITH GAS FLOW HUMIDIFICATION TRACKING
AND STOP/START SIMULATION



8. Proprietary Catalyst Evaluation

1. Baseline Pt supported on high surface area carbon support
2. Pt/MCB catalyst - Pt supported on modified carbon black - for improved durability and operation at lower humidification levels
3. Pt alloy catalyst - Pt alloy catalysts for Pt content reduction and improved durability towards alloy sintering during cycling
4. Pt supported on corrosion resistant carbon black for improved carbon corrosion
5. Pt alloy supported on corrosion resistant carbon support (combination of the alloy from 3 and corrosion resistant support as in 4)

LANL Fuel Cell Training Class

DOE Hydrogen Program

**LANL MEA Fabrication Process
Hands-on PEM Fuel Cell Testing**

**Several Demonstrations using Different Analytical Techniques
Multiple Fuel Cell Presentations presented by LANL Scientists**

Coming August 2007, Registration deadline June

**Contact: Tommy Rockward
trock@lanl.gov
505-667-9587**

Training Class Details



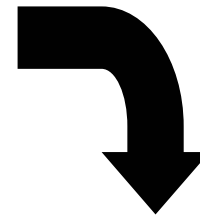
DOE Hydrogen Program

-hands-on



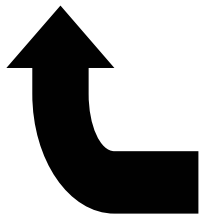
**Ink Catalyst Preparation
(PEM, DMFC, Sprayable Inks)**

**Initial Membrane
Treatment**



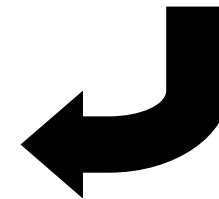
**Applying Electrodes
(substrate, direct, GDL)**

**Fuel Cell Assembly &
Testing**



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

Post Membrane Treatment



Training Class Details

-Presentations and Material Analysis

- **The Decal Method**
- **AC Impedance**
- **Gas Analysis**
- **Analytical X-Ray Techniques**
- **Electrochemistry**
- **Hydrogen Safety in the Lab**

Collaborator's Remarks

- “Your facility’s capability and your personal expertise in the fuel cell field are an invaluable resource for the federal government and industry. It is clear that your past, present, and future work coupled with the freedom to disseminate research information (in most cases) afforded through the federal government will clearly help this country and the industry realize the commercialization of PEM Fuel Cells for automobiles. The key to commercialization of this technology in the automobile industry is open communication, which appears to be understood by only a few...”
- “...worked side-by-side with our staff demonstrating best practices for fuel cell testing while educating us on many important issues including gas purity, ac impedance, flow measurements, and hydrogen safety to name just a few...took us through step-by-step procedures for fuel cell start-up and shut-down and answered in detail all of our questions related to the rationales behind each of these procedures...also offered explicit suggestions on how we might improve our measurements and calibration capabilities.”

Hydrogen Safety

Our approach, developed over >40 years of safe hydrogen handling, to deal with this hazard is:

- In labs with hydrogen supply from cylinder banks or from a hydrogen generator, hydrogen sensors have been installed and are interlocked with the hydrogen gas supply to block further H₂ inflow
- Two sensors are installed in every room for redundancy & coverage
- Sensors installed at ceiling level where accumulation is most severe
- H₂ sets off the alarm at 10% of Lower Flammability Limit (LFL)
- In rooms that use only bottled hydrogen, only a single cylinder is in the room at any given time and bottle sizes are limited to ensure being safely below the LFL of the confined space even with complete release of a full cylinder

Work has been reviewed and approved through Los Alamos National Laboratory's formal safety programs:

- Hazard Control Plan (HCP) - Hazard based safety review
- Integrated Work Document (IWD) - Task based safety review
- Integrated Safety Management (ISM)

Technical Assistance to Developers may be Available

Contact Person:

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