

# Component Benchmarking

Subtask Reported:

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

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(contributions were made from entire LANL FC Team)

Los Alamos National Laboratory

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LA-UR-08-2562

PROJECT ID: FCP4

This presentation does not contain any proprietary or confidential information

# Overview

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- **Timeline**
  - Start: 10/03
  - End: ongoing
  - % complete: N/A
- **Budget**
  - “Technical Assistance to Developers” funded at \$570K/y
    - DOE share: 100%
    - Contractor share: N/A
  - Most DOE-directed effort under the parent task generates proprietary data
  - **FY08 funding: \$570K/y**
- **Barriers**
  - ... is essential to overcoming Fuel Cell Barriers
  - Sharing technical assistance to developers
  - A. Durability
  - B. Cost
  - C. Electrode performance
- **Partners/Collaborators**
  - Full list Available

# Technical Assistance to Developers / Component Benchmarking

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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.

**In addition, LANL scientists interacted with several of the ‘solicitation winners’ outside and/or beyond any proposed collaborations.**

# Visitors

(Fuel Cell visitors to MPA-11/LANL)

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- General Motors
- Univ. of New Mexico
- Cabot Fuel Cells
- Fuel Cell Technologies
- Oak Ridge National Lab
- Argonne National Lab
- Brookhaven National Lab
- Sandia National Lab
- (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

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

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- National Research Council of Canada
- NREL (National Renewable Energy Lab)
- University of Illinois (Urbana Champaign)
- University of New Mexico
- RPI (Rensselaer Polytechnic Institute)
- Case Western Reserve
- Virginia Polytech & State University
- University of California (Riverside)
- University of California – Santa Barbara
- Palmetto FC Technologies
- Ca Fuel Cell Partnership
- DANA Corp.
- Smart Chemistry Corp.

# Technically-Assisted Collaborators/Partners

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- USFCC
  - Single Cell Task Force
  - Durability Task Force
- Working Group 12 Doc: ISO 14687 Hydrogen Quality Standard
- Cabot Fuel Cells
- W.L. Gore
- SGL Carbon
- BASF
- CIDETEC - Centro de Tecnologías Electroquímicas
- U Conn
- Palmetto FC Technologies
- Ca Fuel Cell Partnership
- DANA Corp.
- Smart Chemistry Corp.
- FreedomCAR (GM, Ford, and Daimler-Chrysler) - FC/SC&S/Prod/H2Store/EC Store
- 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
- University of California – Santa Barbara
- Fuel Cell Technologies
- General Motors
- NREL (National Renewable Energy Lab)
- Virginia Polytech and State University
- NIST (National Institute of Science and Technology)

# Interaction with Nuvera

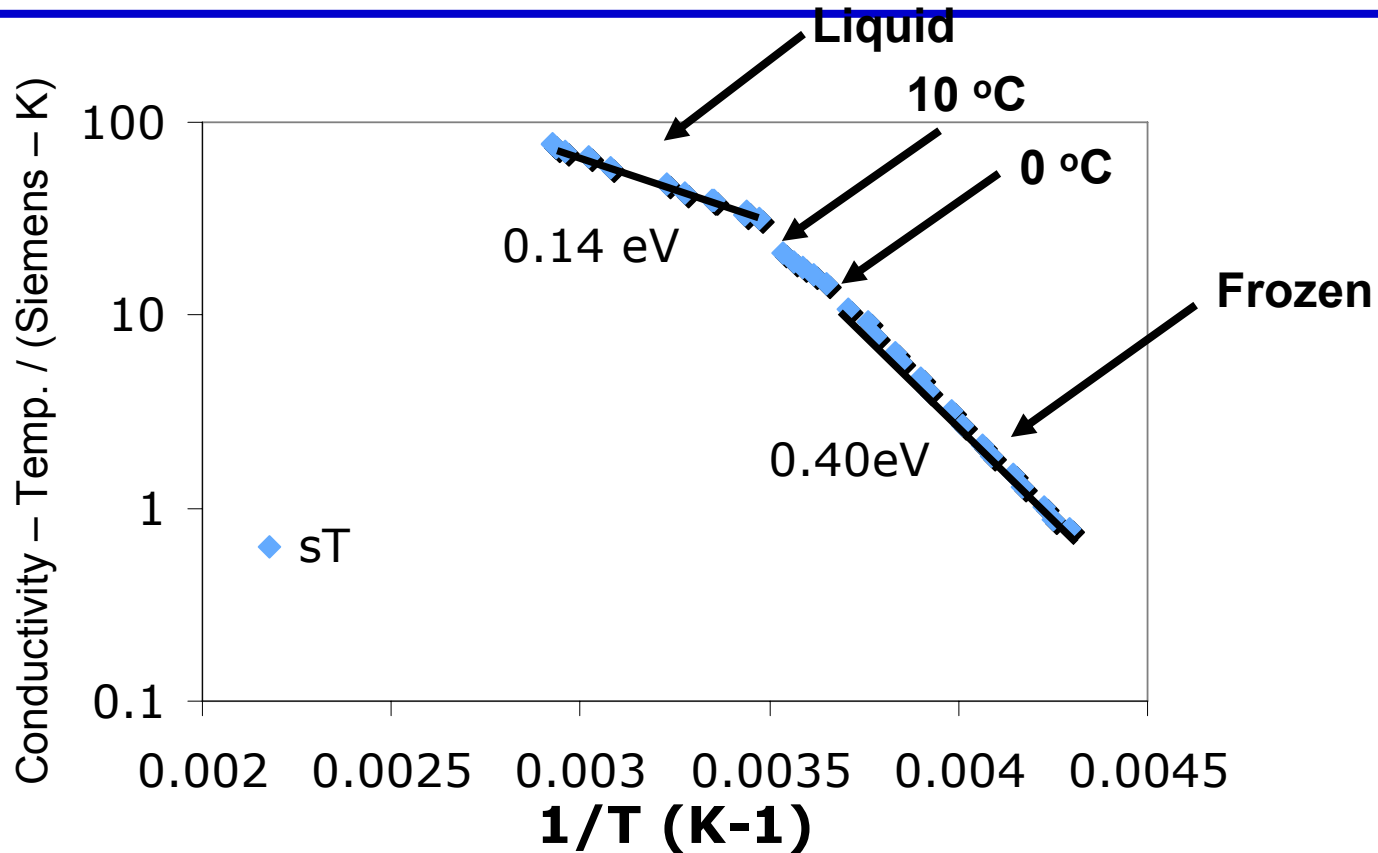
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Nuvera Fuel Cells: (Subfreezing Start/Stop Protocol for an Advanced Metallic Open-Flowfield Fuel Cell Stack) James Cross

(DOE funded Water Transport Project:

- Measure membrane conductivity at low temperature including freezing
- Non-commercial MEAs used in Nuvera

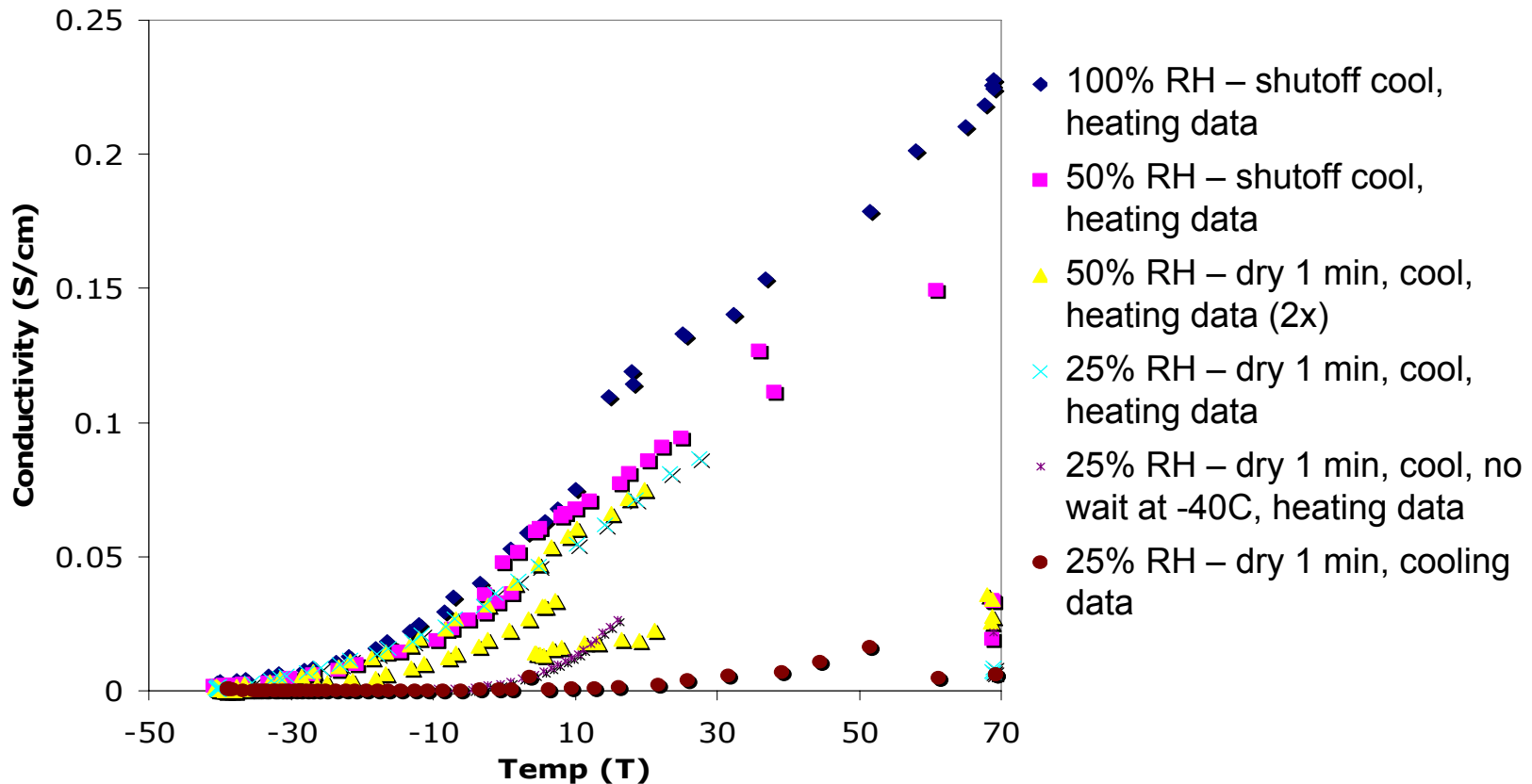
# Activation Energy Difference Frozen and Liquid



- Conductivity has a higher activation energy for proton transport < 0 °C
- Transition region between 0 and 10 °C
- Lower activation energy > 10 °C (Liquid water)



# Conductivity with Temperature



# Interactions with Cabot Fuel Cells

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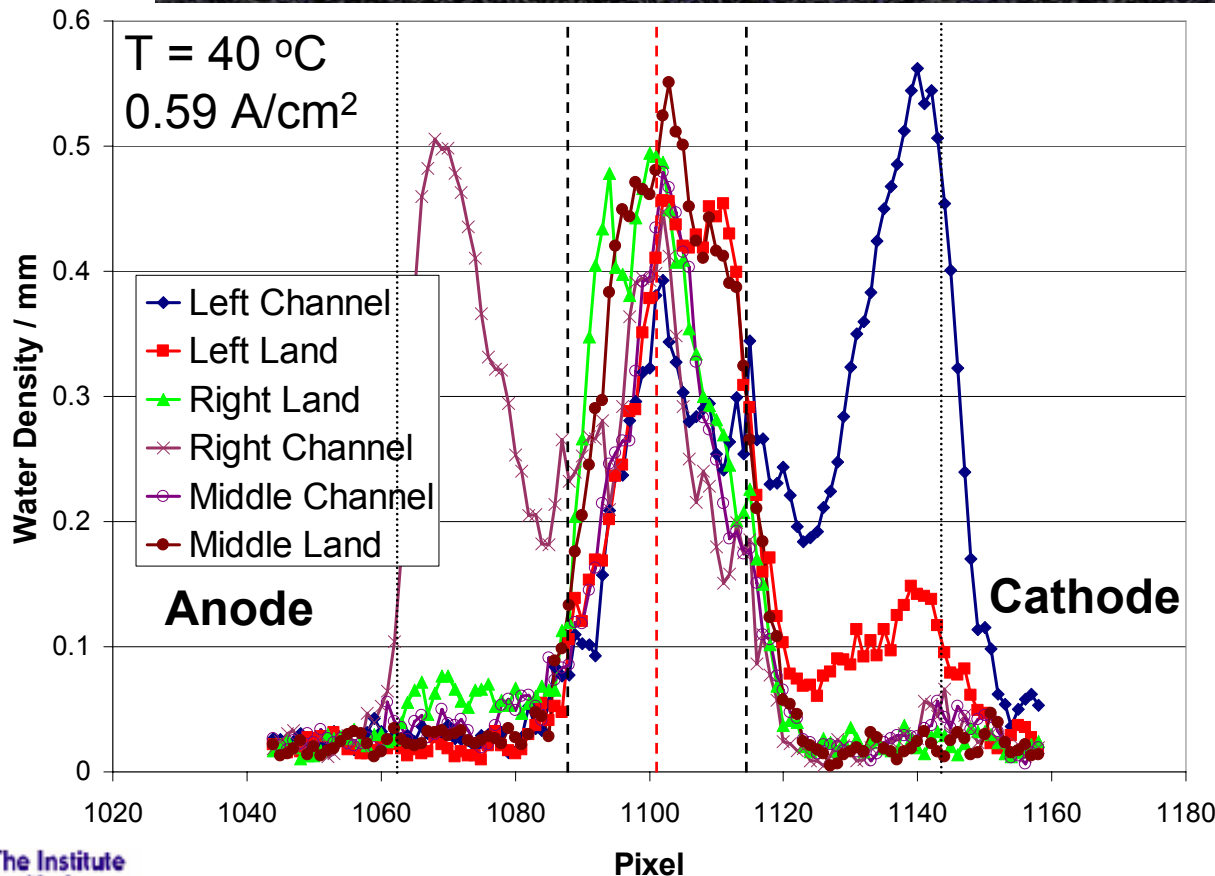
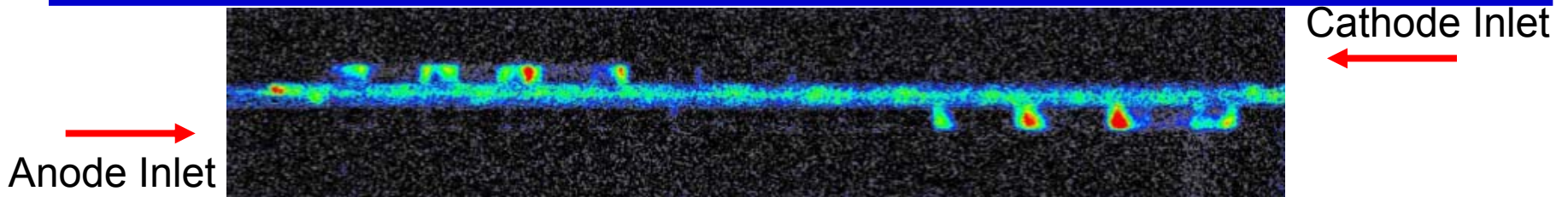
- Cabot Catalysts
  - Accelerated durability on alloy catalysts
  - Carbon corrosion measurements on modified carbon supports
    - (Carbon corrosion resistant supports)
  - Neutron imaging of hydrophilic cathode catalysts
    - (Operation at a lower RH )

# Interactions with 3M (with Water Transport Project)

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- 3M
  - Neutron imaging of NSTF (nano-structured thin film)  
High resolution cross-sections (2.25 cm<sup>2</sup>)
  - Low resolution (50 cm<sup>2</sup>)

# 3M NSTF HR Neutron Imaging



- Counter Current Gas Flow
- Large difference in water accumulation at low temperatures as current increases
- More water in middle of MEA
- Less total water in 3M materials than others

# Accelerated Durability Protocols

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## DOE

### *Cathode catalyst stability:*

Test #1. Voltage cycling: 0.7 and 0.9V (H<sub>2</sub>/N<sub>2</sub>)

### *Cathode carbon corrosion:*

Test #1. Potential hold at 1.2V

### *Membrane durability:*

Test #1. MEA Chemical Cycle (Steady state OCV) - F release or equivalent for non-fluorine membranes

Test #2. Membrane Mechanical Cycle  
Cycle RH 0 to 90°C dewpoint

## USFCC

### *Cathode catalyst stability:*

Test #1. Voltage cycling: 0.6 and 0.96V (H<sub>2</sub>/air)

Test #2. Voltage cycling: 0.6 and 1.2V (H<sub>2</sub>/N<sub>2</sub>)

### *Cathode carbon corrosion:*

Test #1. Potential hold at 1.2V

Test #2. Potential hold at 1.5V

### *Membrane durability:*

Test #1. Fenton's reagent test

Test #2. Combined chemical and mechanical stability test - RH cycling followed by load cycling

Test #3. Steady state Open Circuit Voltage (OCV) test

Test #4?. RH cycling (mechanical stability)??

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

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## Objectives:

- Collaborate with the EU in the frame of the FCTES<sup>QA</sup> Program for formulating standard Fuel Cell Testing protocols.
- LANL participates in the reviewing, assessment and experimental evaluation of the protocols.

## Activities:

1. Began testing in-house FC Protocol round robins.
2. Will participate in hydrogen impurities follow-on project.

# Participated in the FCTES<sup>QA</sup> Workshop

(Honolulu, Hawaii, Nov. 13, 2007)

(With the attendance of representatives of 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).

# Evaluating Standard Procedures

## -Protocol Comparison Tests

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- a) LANL prepared and tested three 50 cm<sup>2</sup> fuel cell using LANL's MEA fabrication.
- b) LANL used three different protocols (EU, JARI, USFCC/LANL). Korea and China to be tested after protocols are received.
- c) Comparisons of each are made.

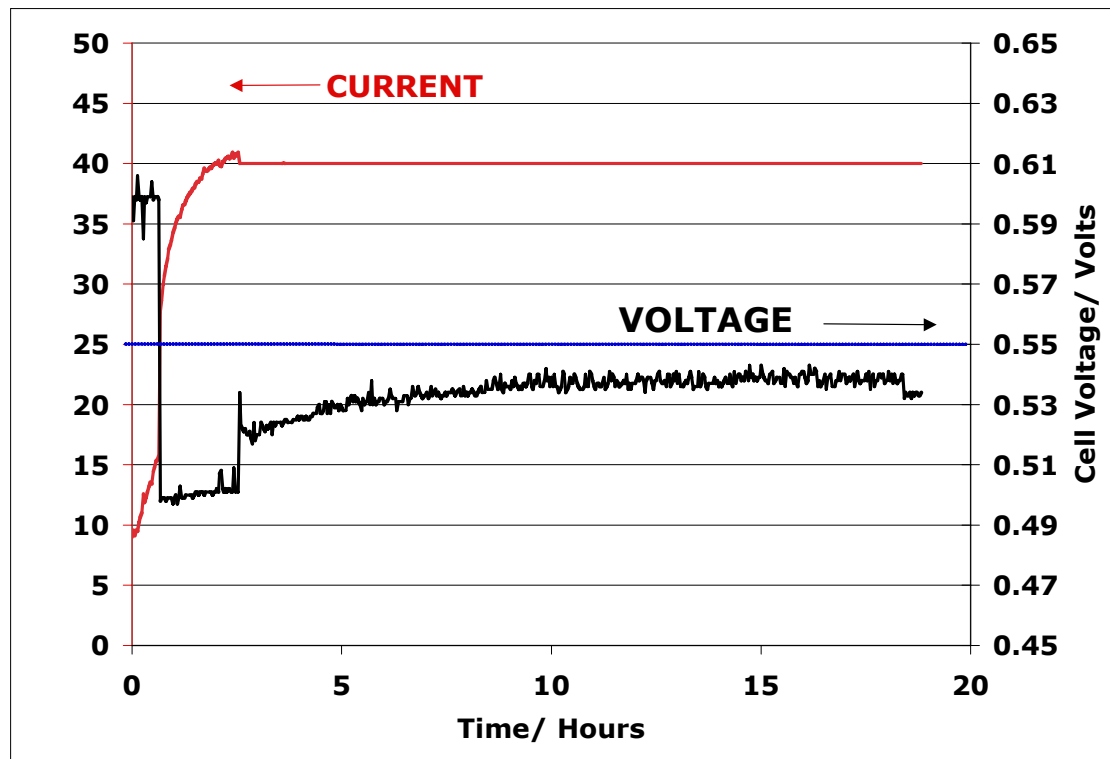


# Protocol Comparison Tests

## -using EU, JARI, and USA's Protocols

Galvanostatic mode at (T, P)  
Increase current by 100 mA/cm<sup>2</sup>  
Cell voltage > 500 mV until stable

Operating Conditions:  
80°C, Fully humidified,  
25 Psig (sea-level)  
H<sub>2</sub>/Air: 696/1740 sccm  
(fixed)



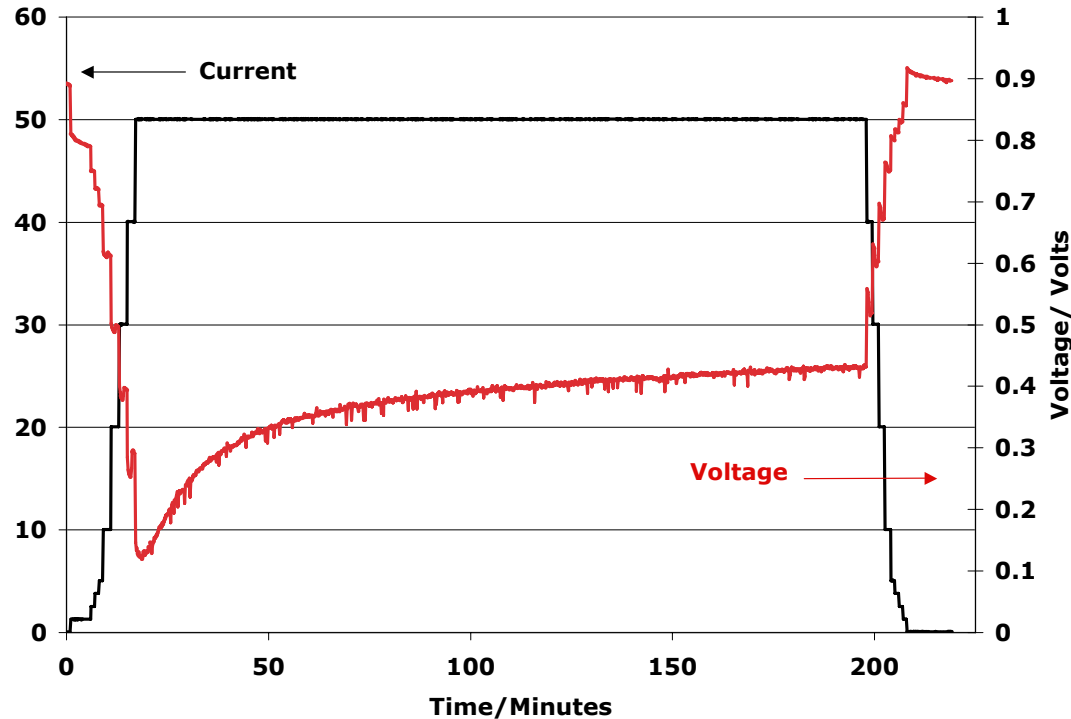
European Union (EU)

# Protocol Comparison Tests

## -using EU, JARI, and USA's Protocols

The graph shows incremental current increases up until 50 Amps (3 hrs). Flow tracking began after 20 A.

**Operating Conditions:**  
80°C, Fully humidified,  
25 Psig (sea-level)  
H<sub>2</sub>/Air: 1.4/2.5 stoich  
above



JARI

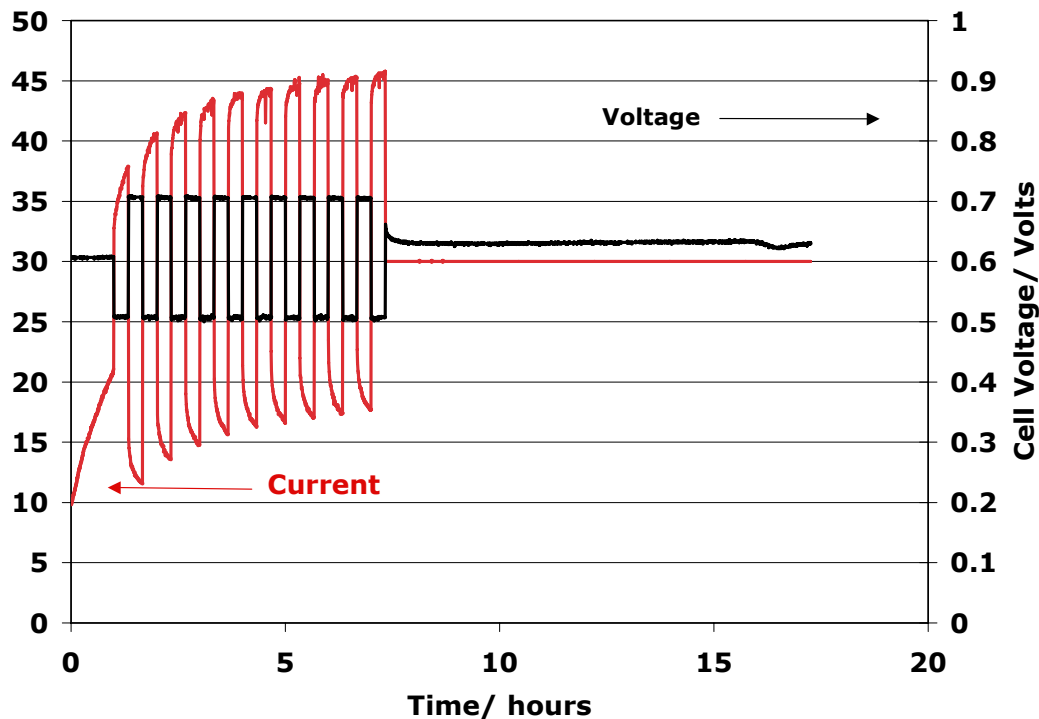
(Japanese Automotive Research Inst.)

# Protocol Comparison Tests

## -using EU, JARI, and USA's Protocols

**0.6 V for 1 hour**  
**Cycled between 0.5 and 0.7 V**  
**(20 min each for 9 cycles)**  
**30 A(cc) overnight**

**Operating Conditions:**  
**80°C, Fully humidified,**  
**25 Psig (sea-level)**  
**H<sub>2</sub>/Air: 696/1740 sccm**  
**(fixed)**



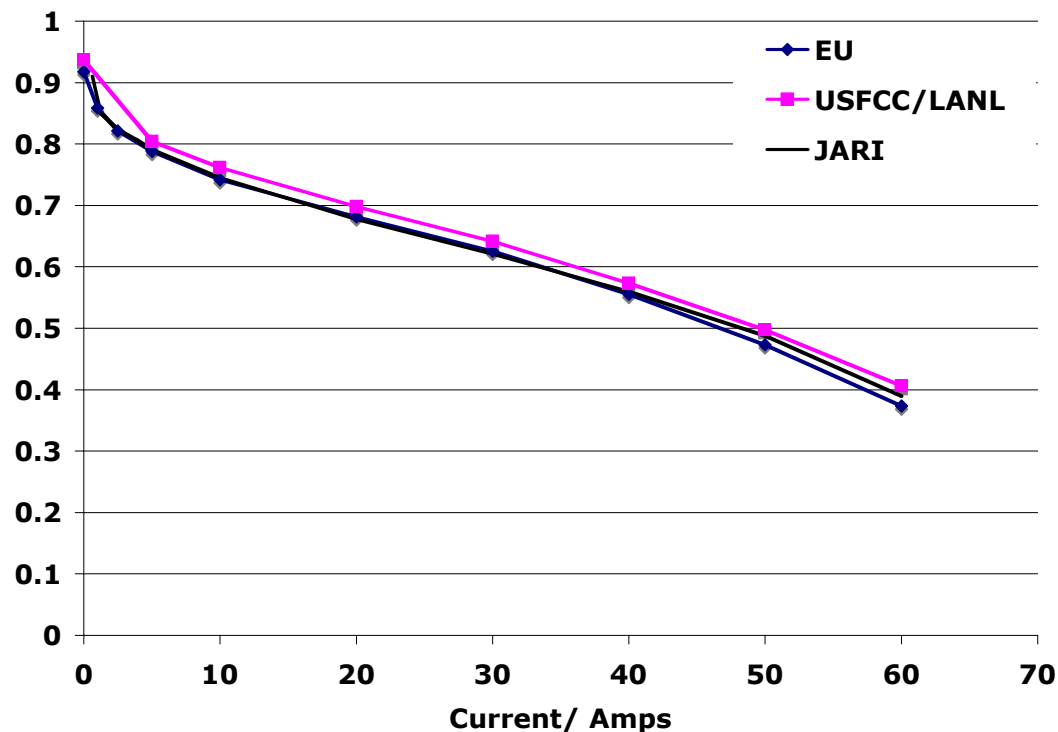
USFCC\_LANL

# Protocol Comparison Tests

## -using EU, JARI, and USA's Protocols

N112, 50cm<sup>2</sup>  
A/C: 0.2/0.2 mg Pt/cm<sup>2</sup>

Operating Conditions:  
80°C, Fully humidified,  
25 Psig (sea-level)  
H<sub>2</sub>/Air: 1.4/2.5 stoich  
tracked ≥ 20A

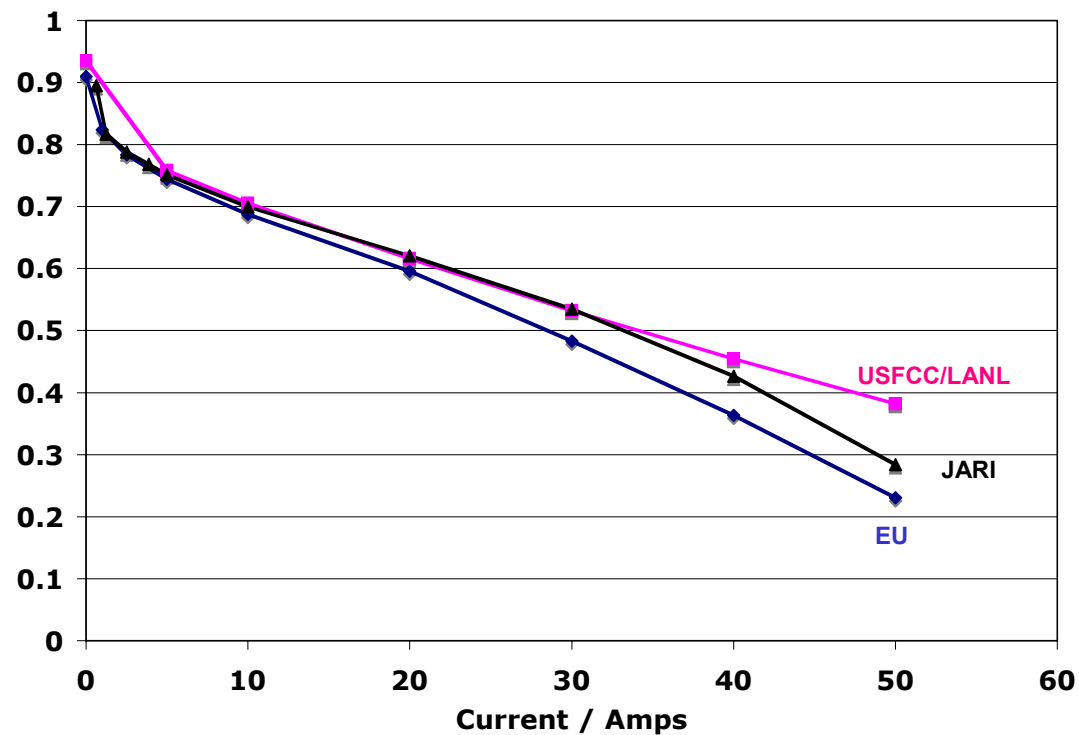


# Protocol Comparison Tests

## -using EU, JARI, and USA's Protocols

N112, 50cm<sup>2</sup>  
A/C: 0.2/0.2 mg Pt/cm<sup>2</sup>

Operating Conditions:  
60°C, Fully humidified,  
Ambient (sea-level)  
H<sub>2</sub>/Air: 1.4/2.5 stoich  
tracked ≥ 20A

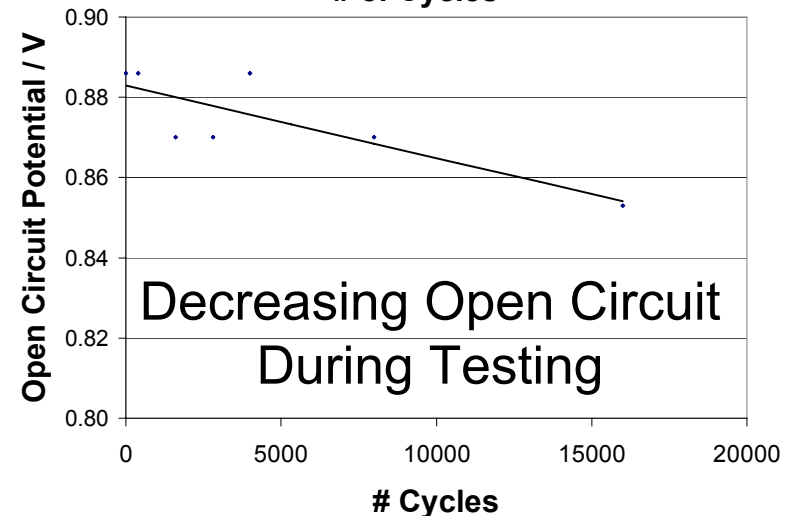
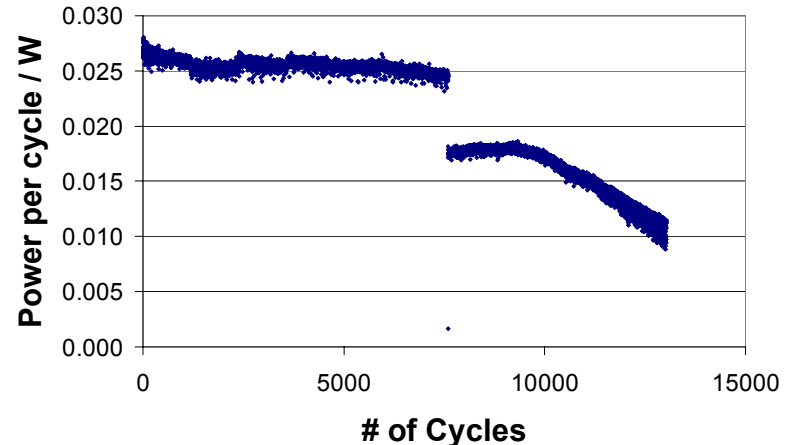


# Comparison of Accelerated Testing Protocols

## Test #1. Voltage cycling: 0.6 and 0.96V (H<sub>2</sub>/air)

Counter/reference electrode gas	Hydrogen
Working electrode gas	Air
Voltage Range	0.6 to 0.96 V
Scan Rate	Potential stepped
Potential Hold	30 sec
Total cycle time	60 sec
P <sub>counter/reference</sub>	270 kPa (2.7 bara or 39.7psia)
Flow hydrogen	Equivalent to St=1.2 at 0.6V BOL
P <sub>working</sub>	270 kPa
Flow air	Equivalent to St=2.0 air flow at 0.6V BOL
RH <sub>Air</sub>	80 °C (100%)
RH <sub>Hydrogen</sub>	80 °C (100%)
Temp <sub>cell</sub>	80 °C

Can't reach 0.96V with thin membranes  
(Test starts with Initial OCP 0.90V)



- Not going to be consistent catalyst test

# *LANL Fuel Cell Training Class*

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*November 2007*

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

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

**10 Participants from industry, national labs, and universities**

# Hydrogen Safety

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**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 H<sub>2</sub> inflow
- Two sensors are installed in every room for redundancy & coverage
- Sensors installed at ceiling level where accumulation is most severe
- H<sub>2</sub> 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)