June 8, 2010
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Rangachary Mukundan
(Assistance provided by entire LANL FC team)

2010 U.S. Department of Energy Hydrogen Program and
Vehicle Technologies Program Annual Merit Review
and Peer Evaluation Meeting

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 $600K
    - DOE share: 100%
    - Contractor share: N/A
  - Most DOE-directed effort under the parent task generates proprietary data
  - **FY10 funding:** $600K
- **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
How to Obtain Technical Assistance

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 Working Group 12, 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.
Technically Assisted:

- **Nuvera**
  - Cell Testing
  - Neutron Imaging
- **NIST**
  - Flowfield geometry project
- **NIST**
  - Beam Hardening experiments
  - Hardware
- **University of Michigan**
  - Imaging of Dead-ended anode
- **USFCC**
- **Fuel Cell Technologies**
- **UC Irvine**
- **Fuel Cell Tech Team**
- **IPHE**
  - Cidetec Centro de Tecnologías Electroquímicas
  - CEA-Liten
- **National Network of Digital Schools**
Task:

Performance Testing of Polymer Electrolyte Membrane (PEM) Fuel Cells in Support of the NIST “Metrology for Fuel Cell Manufacturing” Project

Background:

NIST has fabricated reference anode and cathode plates with optimal geometry (i.e., flow field geometry variability < 10 µm) and ten experimental plates with different combinations of intentional geometric perturbations that include sidewall taper, sidewall straightness, channel bottom straightness (variations –in-depth), and variation-in-channel width. These sidewall taper variations range from 0 to 10 degrees, sidewall and bottom straightness (channel variation-in- depth) from 0 to 50 um, and variation in channel width of 0 to 100 um. The straightness perturbation is in the form of a sinusoidal shape with a 2 mm pitch along the base of the channel and 4 mm pitch on its sidewalls.
NIST-Metrology Group

LANL Proposed Plan/Involvement:

1. BOT and EOT diagnostics; electrochemical H₂ Crossover and active surface area measurements (cathode side), polarization curves, and AC-Impedance measurements.

2. Fuel cell testing conditions; 80ºC, pressures TBD, at 100% RH, 50% RH, and 25% RH using both air and oxygen (83.3% H₂ utilization (electrolysis-grade) and 50% utilization for air (oil-less compressor). CV will be run at each RH on the cathode.

3. A baseline polarization curve will be run between each experimental flow field configuration using an agreed upon reference configuration.

4. The outcome of the work performed by LANL will include a compiled report with a documented protocol that includes equipment used, reference cell configuration, a detailed description of the performance evaluation procedure used, all beginning-of-test (BOT) and end-of-test (EOT) data, and all experimental data with a summary of LANL performance conclusions derived from the observations.
Experimental design and testing of NIST’s Flow Fields in Fuel Cells

Identical Hardware and Components:
- 50cm² Hardware (Teledyne CH-50)
- Gas Diffusion Media: SGL 25 BC
- Commercially Available MEA
- Hydrogen(Electrolysis-Grade) and Air(oiless-compressor)

Fuel cell testing conditions:
- 80ºC, 25 psig
- Varying RH: 100%, 50%, and 25%
- H₂/air and oxygen (83.3% H₂ and 50% utilization for air
- CVs at each RH on the cathode
- AC Impedance at 1 A/cm²

Baseline polarization curve between each experimental flow field configuration using a single reference MEA
Preliminary Results:

Electrochemical $H_2$ crossover/Surface Area

CVs are used for quality control as well as to confirm hydrogen cross-over measurements throughout the experiments.

We will be able to compare CVs at the different RHs for each cathode and to ensure the MEA is still usable for other experiments.

$H_2/N_2$ @ 500 sccm
80°C, 25 psig
Scan Rate: 20 mV/s
0.1 – 1.1 V, 3 cycles
Preliminary Results:

**VI Curves/AC Impedance**

**Cathode Plate 1C_VI-Curves**

- Cell Voltage/ Volts
- Current Density/ A*cm\(^{-2}\)

- **100% RH**
- **50% RH**
- **25% RH**

VI curves reflect high current density (increase H\(_2\)O/establish mass transport)

AC Impedance were run at 1 A/cm\(^2\) for each RH, no clear differences observed.
Neutron Imaging Beam Hardening Experiments

multiple active area Fuel Cell hardware

- Neutron Imaging Mu (water calibration constant) is non-linear with water thickness
- Provided hardware and hardware designs to NIST beam line scientists (Hussey and Jacobson) to conduct Beam Hardening Experiments.
- Use fuel cell components to directly measure the beam hardening
- Cuvette of water behind each active area used to calibrate water non-linearity
Dead-Ended Anode (DEA) with no anode recirculation or humidification:

- (University of Michigan - Anna Stefanopoulou and Jason Siegel)
- Measure water profiles and performance of DEA to help U Michigan develop and validate simple control oriented models and simulate the dynamics of large fuel cell stack systems operating with a Dead-Ended Anode (DEA), which do not rely on anode humidification or re-circulation systems. In order to facilitate repeatable transients during
- The single serpentine flow channel used for this testing is not ideal for dead-ended anode operation due to the high likelihood of anode starvation due to liquid water droplets blocking the anode channel at random locations. Frequent purging leads to over-drying of the GDL and MEA, and better represents the operation of flow though a system with dry hydrogen supply.
- Future measurements using higher resolution detector, vertical flowfield and flowfields without the serpentine flow geometry will provide more accurate information for this type of fuel cell stack design
Neutron Imaging of Water Profiles During Dead-Ended Anode Operation

Anode side water thickness
- GORE MEA
- Serpentine Flowfields
- Dead-ended anode with transient purging
- 60C 100% CA RH
- No Anode humidification

Cathode side water thickness
Snapshot of liquid water distribution across GDL showing anode channel flooding.
This task supports the International Partnership for the Hydrogen Economy (IPHE) / European Commission (EC) task in fuel cell testing, safety and quality assurance (FCTESQA)

Status: Protocol comparisons were completed between CEA-Liten (France) and LANL. A test plan was agreed upon and is currently being conducted.

**Protocol Comparison Studies: Fuel Impurities CO and NH₃**

The expansion for FY10 of the working group interaction can be summarized as follows:

1. Establish an acceptable fuel cell test plan with members of the international community that includes but not limited to: operating conditions, polarization curves, contaminant and concentration, time of exposure, and recovery conditions.
2. Test fuel cell using the agreed upon test plan
3. Exchange MEAs and repeat the test plan
4. Disseminate the results
• Yun Wang - Contact

• Investigated the **electrochemical kinetics, oxygen transport and solid water formation within the cathode electrode of polymer electrolyte fuel cells (PEFCs) during cold-start.**
  
  – LANL provided supporting experimental observation using neutron imaging and isothermal cold-start experiment
  – LANL provided ionic conductivity data of Nafion® 117 membranes at subfreezing temperature, to analyze the temperature-dependence of the Ohmic polarization during cold-start.

**Publication:**
Wang, Yun; Mukherjee, Partha P.; Mishler, Jeff; Mukundan, Rangachary; Borup, Rodney L. **Cold start of polymer electrolyte fuel cells: Three-stage startup characterization.** Electrochimica Acta (2010), 55(8), 2636-2644
• **DOE / USCAR Fuel Cell Tech Team**
  – Provided representation to Fuel Cell Tech Team

• **USFCC (U.S. Fuel Cell Council)**
  – Durability working group co-chair (Rod Borup)
  – AST testing
  – Round-robin testing / Impurities working group (Tommy Rockward)
Fuel Cell Technologies

- Developing software for customized impedance protocols, protocol testing for FC Tech Team durability protocol
- RH transient testing capability
Nuvera

- Provided Neutron imaging hardware (designs and fabricated hardware)
  - Cell conditioning for single cell
  - Performance testing at LANL – week visit by Nuvera (Dross, Conti, Beverage)

- Support at NIST
  - Imaging support
  - AC impedance equipment and support
Hi Tommy,

It was such a pleasure to meet you today and to get to see your work. I am very excited about the footage we got. The material was exactly what we wanted and need. You were great on camera, so well spoken for our audience and you've got such energy and enthusiasm for your work.

Thank you so very much for participating in this project and for being such a willing spokesperson. I know you're really busy, so all the more, I truly appreciate the time you took to be with us today!

With my deepest gratitude,

Caroline Hardman
Special Projects Coordinator
National Network of Digital Schools
50404 Calcutta Smith Ferry Rd.
Calcutta, OH 43920
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desk (866) 990-6637 x 1008

Scope: Interview a LANL scientist on the current advancements of Fuel Cell Technologies in a manner that both relates the concept to high school students while also sparking their interest in science.

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Outcome: Completed DVD to be shown as part of CES series (Cutting Edge Science) in US high schools

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LANL Fuel Cell Training/Short Course

LANL MEA Fabrication Process
Hands-on PEM Fuel Cell Testing
Several Demonstrations using Different Analytical Techniques
Multiple Fuel Cell Presentations presented by LANL Scientists

Participants from industries, national labs, and universities

Upcoming 2010 Class Planned for July 20-22
Characterization Methods Available under Technical Assistance

- X-ray Tomography – MicroXCT
  - 3-D imaging of MEAs, catalyst layer delamination
- FTIR (ATR, Transmission, DRIFTS)
  - Surface structure changes, ionomer/membrane functional changes
- XPS – (X-ray Photoelectron Spectroscopy)
  - Carbon corrosion, surface oxidation
- IGC - (Inverse Gas Chromatography)
  - Surface Energies
- Hg and H₂O porosimetry
  - Electrode and GDL pore size distributions and hydrophobic vs. hydrophillic pores
- SEM/ESEM (Environmental Scanning Electron Microscopy)
  - MEA structural and elemental analysis
- TGA/DSC & MS (Thermogravimetric Analysis / Differential Scanning Calorimetry)
  - Component chemical analysis
- Powder XRD (x-ray diffraction)
  - Catalyst particle size distribution, crystallinity
- Laser Ablation ICP-MS
  - Impurities analysis
- Dynamic Vapor Sorption Analysis
  - Water vapor sorption analysis
- Fuel Cell Testing
  - AC Impedance: Mass Transport limitations
Los Alamos

X-ray micro-tomography system
Xradia MicroXCT

GDL
Add-on to ESEM (Environmental Scanning Electron Microscope)
- Leverages other funding
Operates to \( \sim 1 \) torr H2O

**X-Ray Map of GDL**

![X-ray Map of GDL](image)

**SGL area 2 - Counts (30 Frame(s))**

- **Grey**
- **C K**
- **N K**
- **O K**
- **F K**

**SEM**

**X-Ray Spectrometer**
Dynamic Vapor Sorption Analysis
TA Instruments

Water vapor sorption at 60°C

Nafion 212
sPAEN 1.0

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Acknowledgment

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DOE: Fuel Cell Technologies Program
Nancy Garland, Technology Development Manager