# Technical Assistance to Developers

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Tommy Rockward/Adriana Fernandez/ Rod Borup/
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***

Los Alamos

# 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

# NIST-Metrology Group

## 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 $H_2$ 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% $H_2$ 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

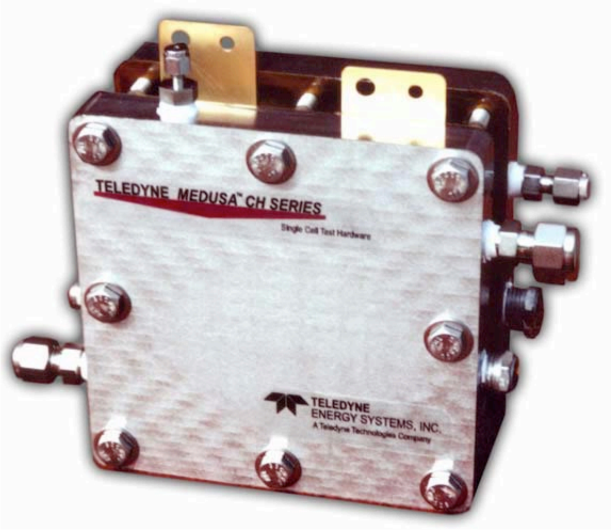
**Identical Hardware and Components:**

- $50 cm^2$ Hardware (Teledyne CH-50)
- Gas Diffusion Media: SGL 25 BC
- Commercially Available MEA
- Hydrogen(Electrolysis-Grade) and Air(oiless-compressor)

**Fuel cell testing conditions:**

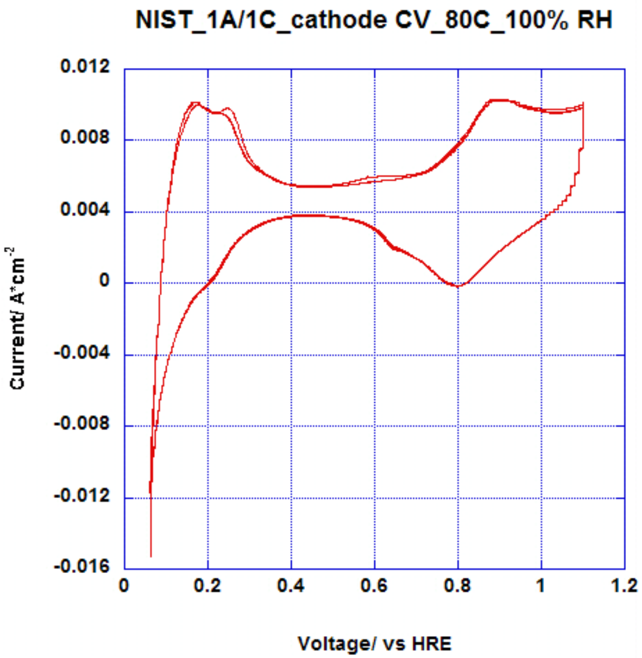
- 80ºC, 25 psig
- Varying RH: 100%, 50% , and 25%
- $H_2$/air and oxygen (83.3% $H_2$ and 50% utilization for air
- CVs at each RH on the cathode
- AC Impedance at 1 $A/cm^2$

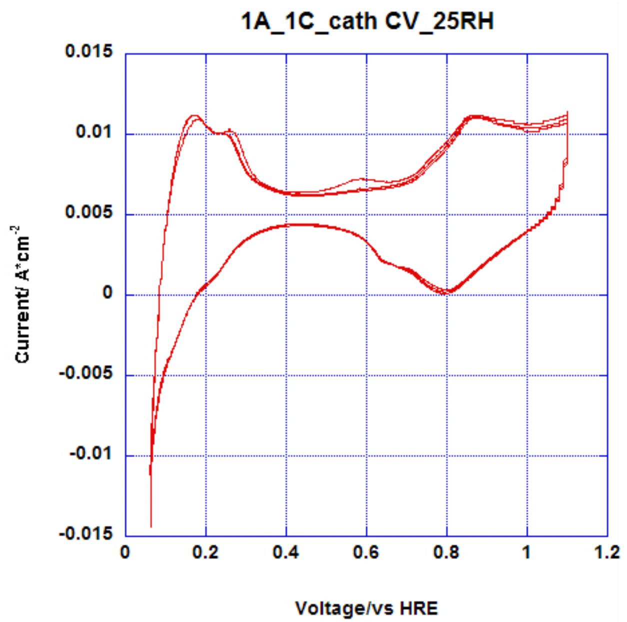
***Baseline polarization curve between each experimental flow field configuration using a single reference MEA***



# Preliminary Results:

## *Electrochemical $H_2$ crossover/Surface Area*



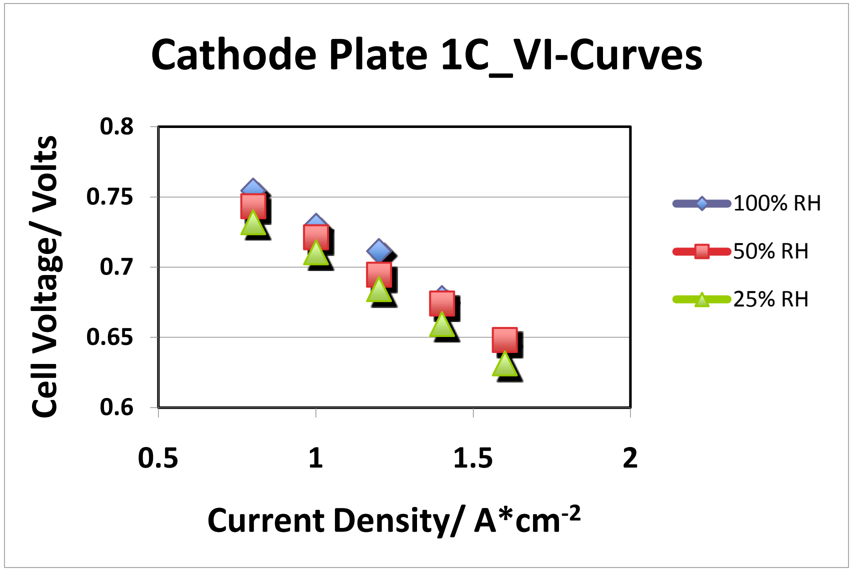



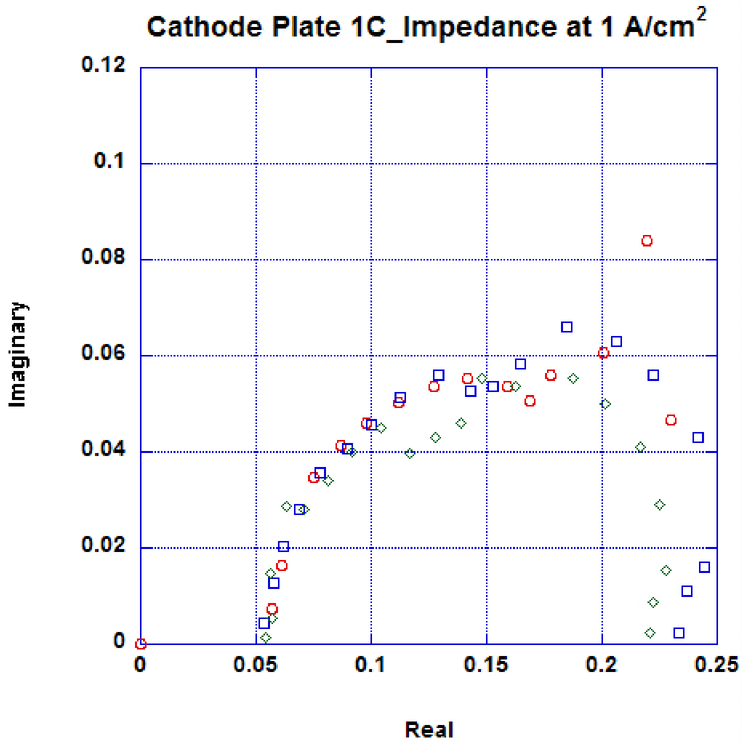

$H_2/N_2$ @ 500 sccm
$80^{O}C$, 25 psig
Scan Rate: 20 mV/s
0.1 – 1.1 V, 3 cycles

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

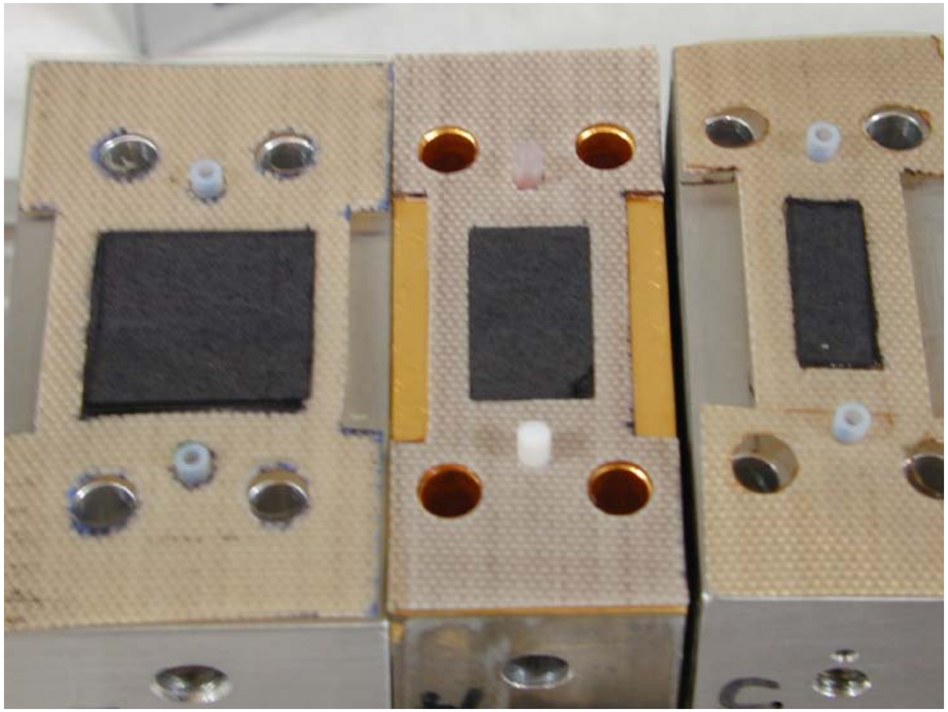
# Preliminary Results:
## *VI Curves/AC Impedance*




VI curves reflect high current density (increase $H_20$/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*



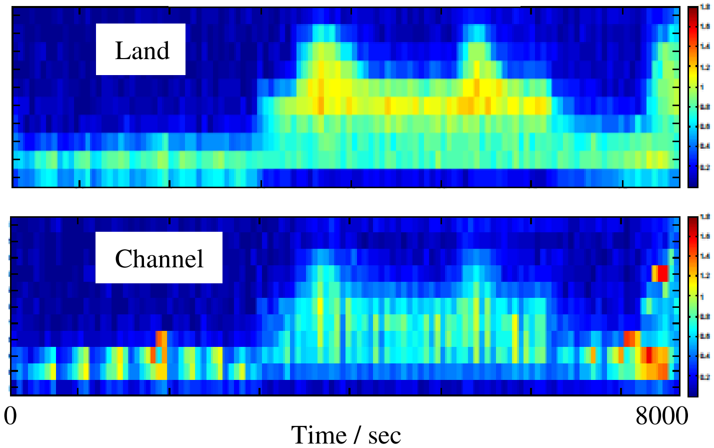
- Neutron Imaging Mu (water calibration constant) is non-linear with water thickness
- Provided hardware and hardware designs to NIST beam line scientists (Hussey and Jacobson) to conduct Beam Hardening Experiments.
- Use fuel cell components to directly measure the beam hardening
- Cuvette of water behind each active area used to calibrate water non-linearity

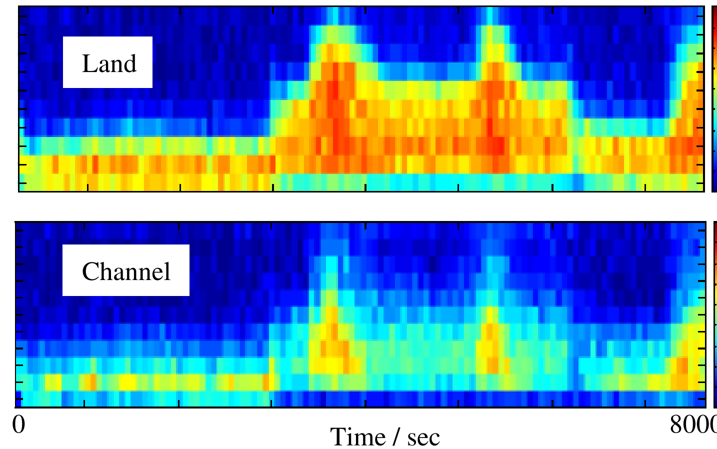
# Neutron Imaging of Water Profiles

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

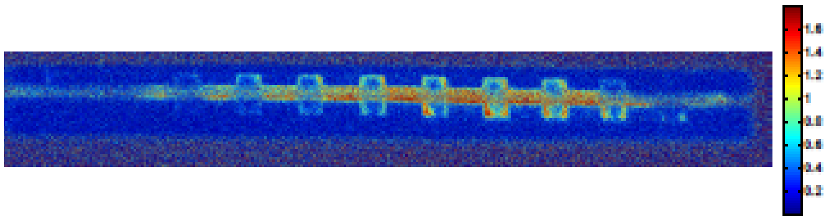
# Neutron Imaging of Water Profiles During Dead-Ended Anode Operation




## 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.

# International Partnership for Hydrogen Economy-IPHE

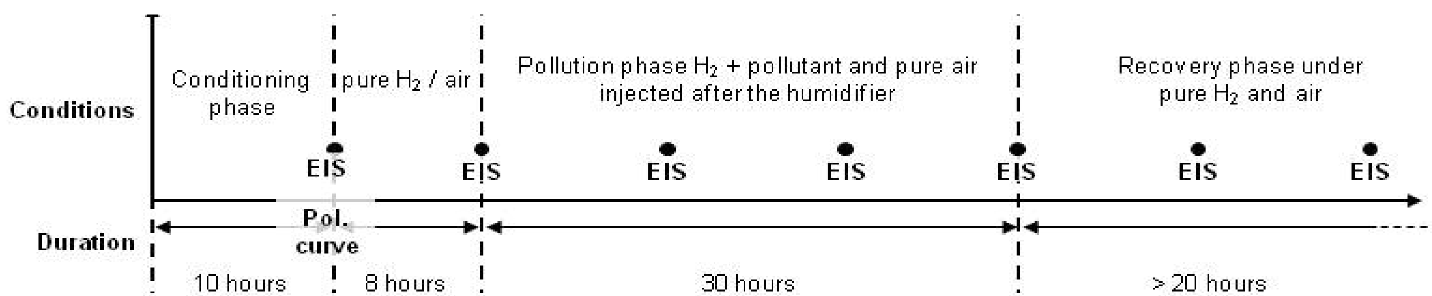
This task supports the International Partnership for the Hydrogen Economy (IPHE) / European Commission (EC) task in
fuel cell testing, safety and quality assurance (FCTES$^{QA}$)

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 $NH_3$**

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



# University of California (Irvine)

- 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

# National Network of Digital Schools

*High School Science Educational Video Series*

**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.

**Testimony:**

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
<http://www.lincolninteractive.com/>http://www.lincolninteractive.com/
mobile (724) 494-2171
desk (866) 990-6637 x 1008

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

# 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***

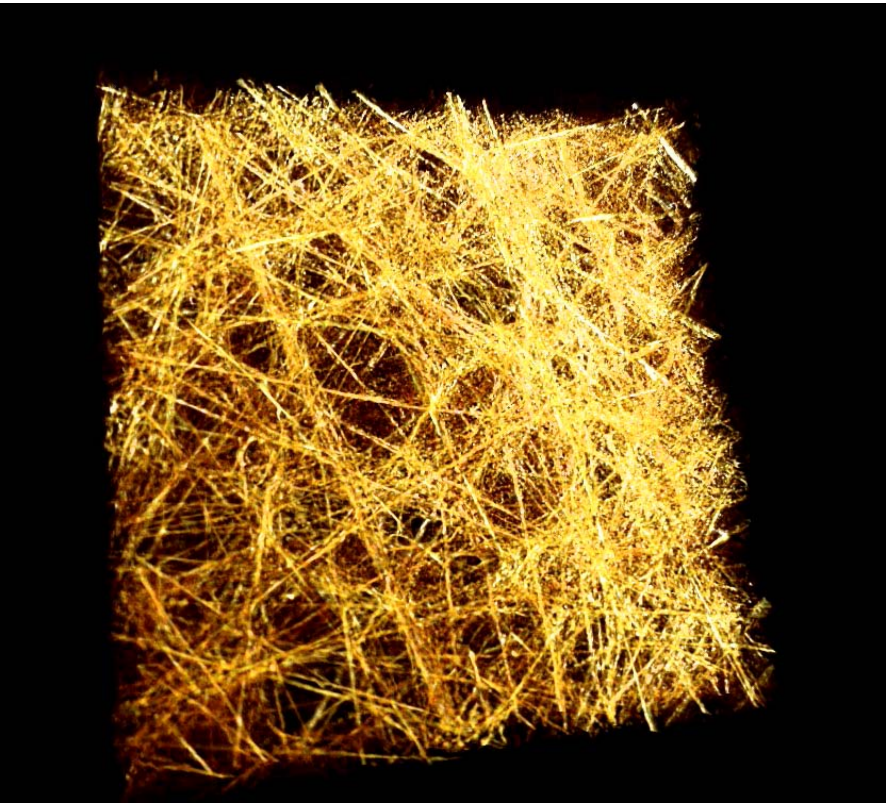
# Characterization Methods Available under Technical Assistance

- 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 $H_2O$ 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, crystallinty
- Laser Ablation ICP-MS
  - Impurities analysis
- Dynamic Vapor Sorption Analysis
  - Water vapor sorption analysis
- Fuel Cell Testing
  - AC Impedance: Mass Transport limitations

# X-ray micro-tomography system

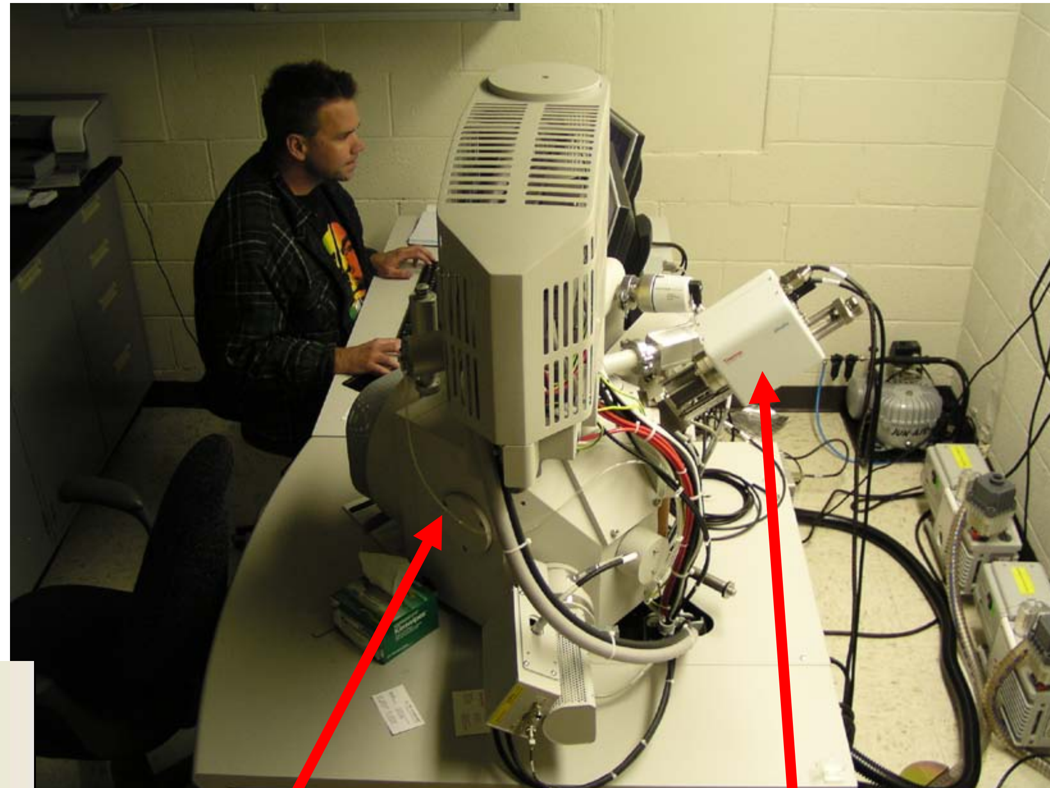
## Xradia MicroXCT

**GDL**




# X-ray Spectrometer
## ThermoScientific

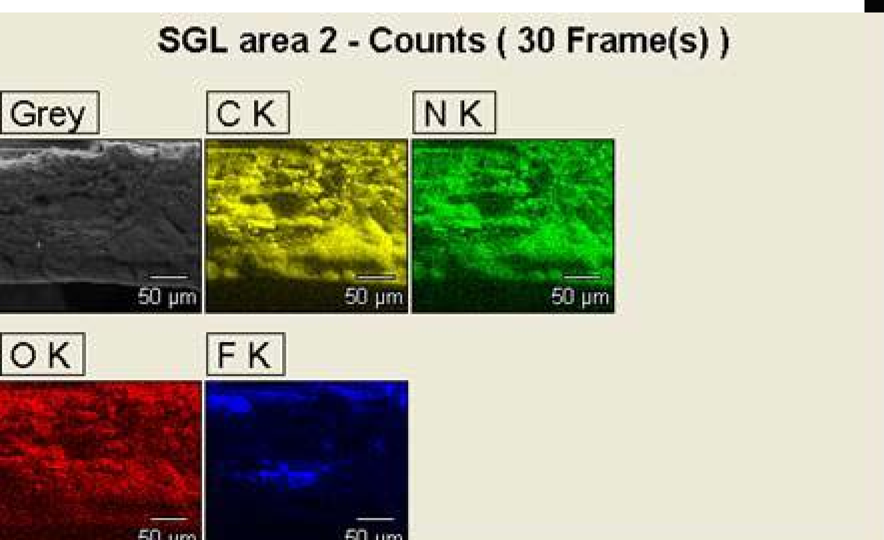
Add-on to ESEM
(Environmental Scanning
Electron Microscope)
- Leverages other funding
Operates to ~ 1 torr $H_2O$



**SEM**

**X-Ray Spectrometer**

## X-Ray Map of GDL



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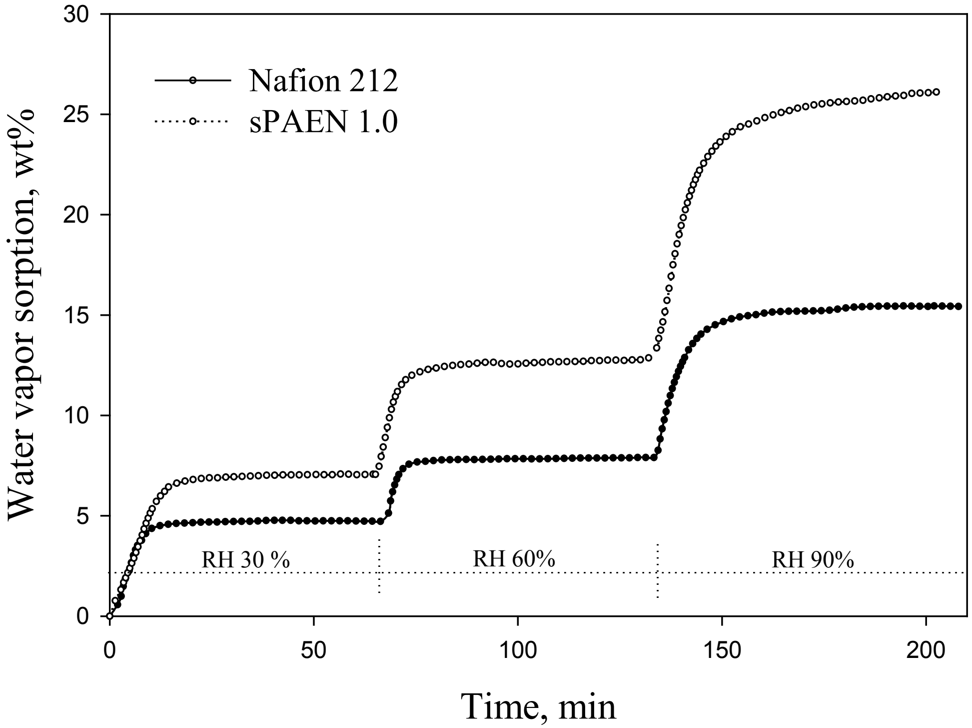
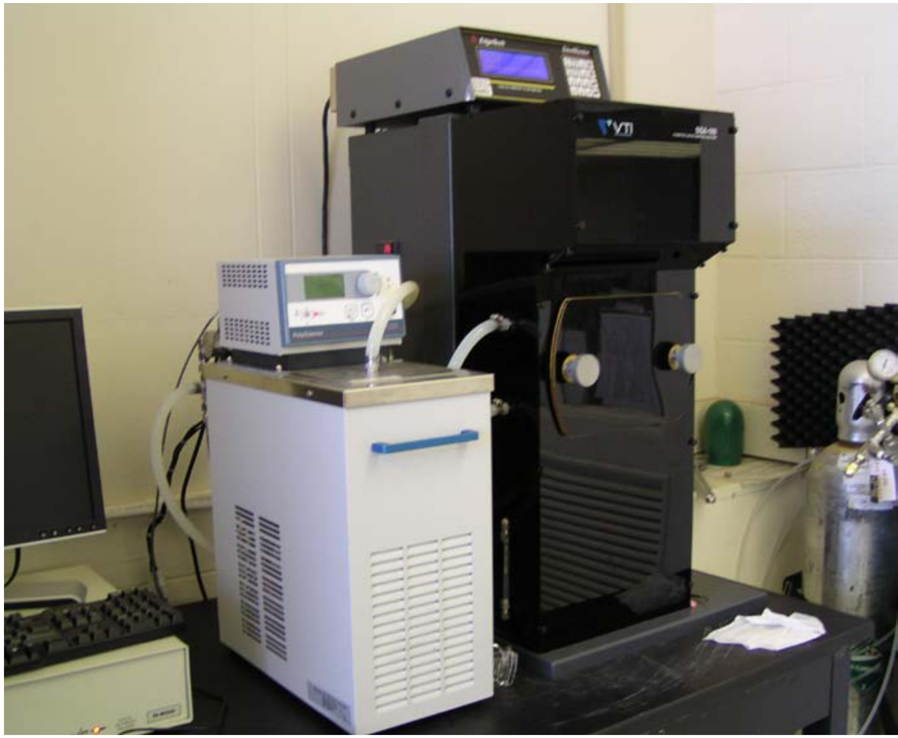
# Dynamic Vapor Sorption Analysis
## TA Instruments

Water vapor sorption at 60°C



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