

2007 DOE Hydrogen Program Review

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- W.L. Gore:** Will Johnson, Simon Cleghorn

Project Lead: Los Alamos National Lab

May 16, 2007

Project ID #
FCP24

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Overview

Timeline

- New Project for FY07
- 4 year Project Duration

Budget

- Total project funding
 - DOE Cost: \$6,550,000 (4 yrs)
 - Cost Share: \$290,811

Funding for FY07

LANL	\$1000k
Industrial Partners	\$300k
Other National Labs	<u>\$350k</u>
FY07 Total	1650

Barriers

Water management is critical to optimal operation of PEM Fuel Cells

- Energy efficiency
- Power density
- Specific power
- Cost
- Start up and shut down energy
- Freeze Start Operation

Partners

- Direct collaboration with Industry, Universities and other National Labs (see list)
- Interactions with other interested developers
- Project lead: Los Alamos National Lab

Organizations / Partners

- Los Alamos National Lab
 - (Lead: experimental measurements, modeling)
- Sandia National Laboratory (modeling)
- Case Western Reserve University (characterization, modeling)
- W. L. Gore and Associates, Inc. (MEAs)
- SGL Carbon Group (GDLs, MPLs)
- Oak Ridge National Lab (characterization)
- National Institute of Standards and Technology (neutron imaging)

Objectives

- **Develop understanding of water transport in PEM Fuel Cells (non-design-specific)**
- Evaluate structural and surface properties of materials affecting water transport and performance
- Develop (enable) new components and operating methods
- Accurately model water transport within the fuel cell
- Develop a better understanding of the effects of freeze/thaw cycles and operation
- Present and publish results

Approach

- **Develop understanding of water transport**
 - Experimental measurement and testing
 - Characterization
 - Modeling
- Evaluate structural and surface properties of materials affecting water transport and performance
 - Measure/model structural and surface properties of material components
 - Determine how material properties of GDL, MPL, catalyst layers & interfaces affect water transport (and performance)
 - Determine properties change during operation (degradation effects)
- Develop (enable) new components and operating methods
 - Prevent flooding (high power operation)
 - Prevent dehumidification (low RH operation - transportation)
- Develop a better understanding of the effects of freeze/thaw cycles and operation
 - Help guide mitigation strategies.

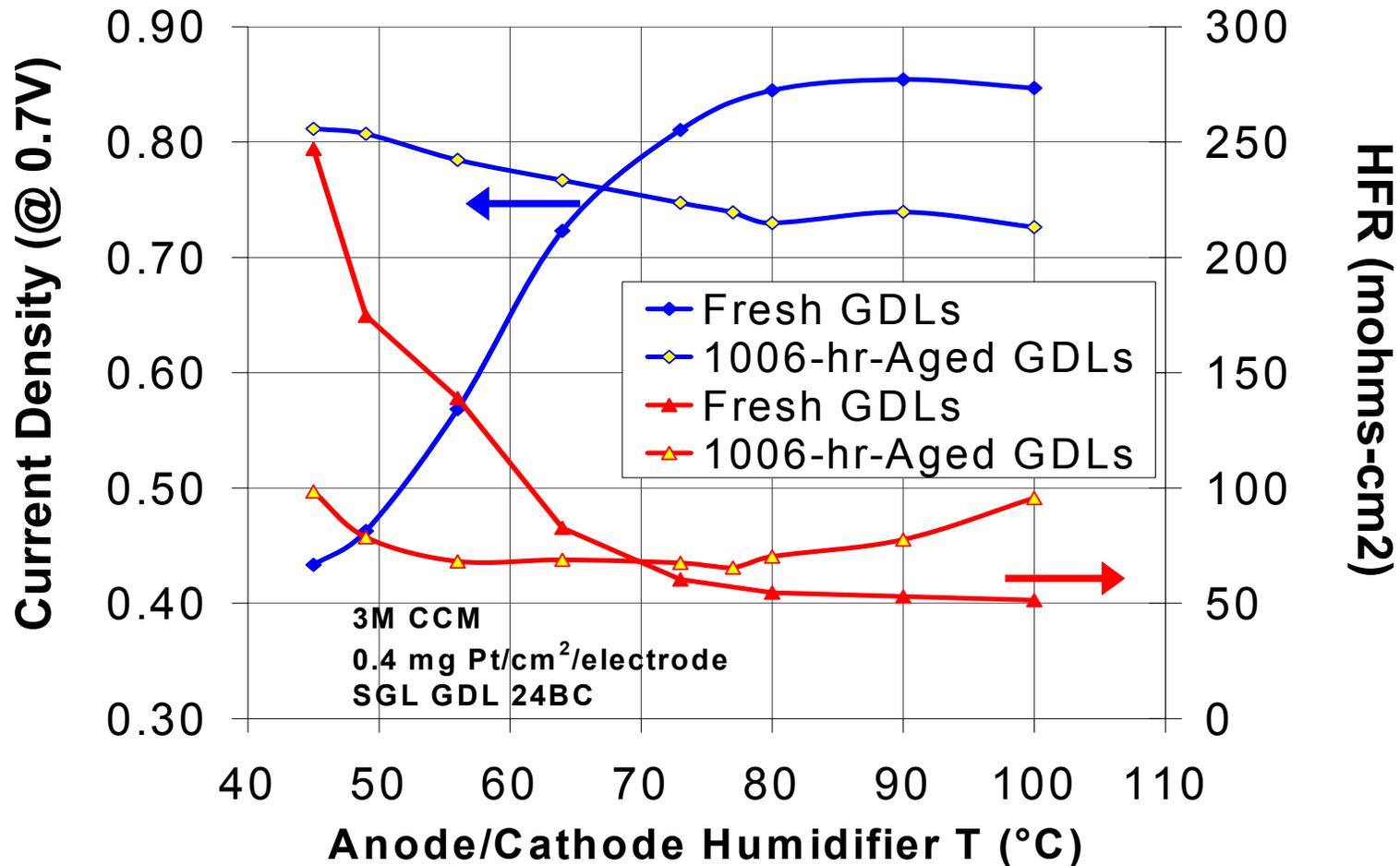
Technical Accomplishments

- Direct water imaging at NIST by Neutrons
 - High resolution (15 μm) cross-section cell design
 - Cross – Section view
 - High resolution
 - Low resolution (150 μm) imaging
 - Imaging of entire 50 cm^2 flowfield area
- GDL Characterization
 - Hydrophobicity characterization
 - Microscopic characterization of hydrophobic coating
 - Elemental compositional characterization
- Modeling of mass transport losses
 - Delineation of mass transport loss from IR, kinetics, etc.
 - Modeling of water-droplet detachment from the GDL/channel interface.

GDL Material Characterization

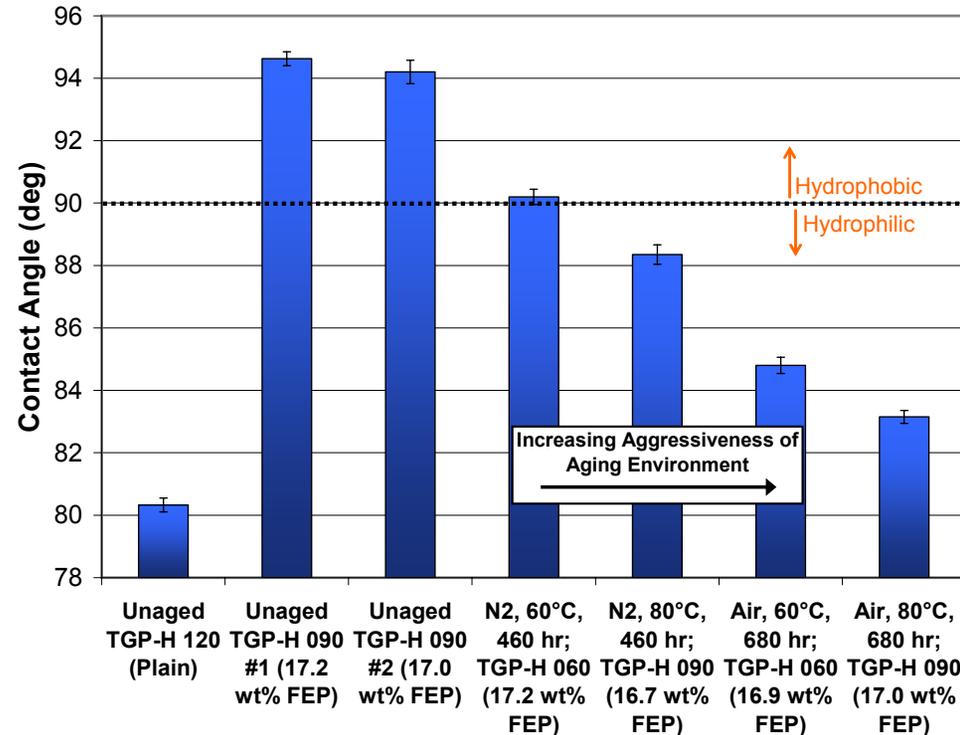
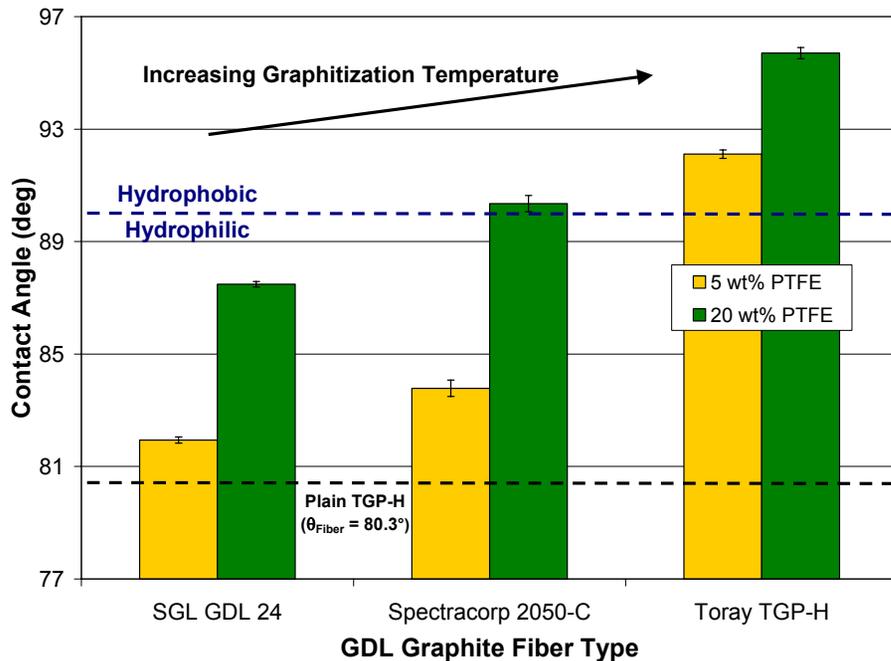
- GDL Characterization
 - Hydrophobicity characterization
 - Contact angle and surface energy measurements
 - Microscopic characterization of hydrophobic coating
 - SEM, TEM
 - Elemental compositional characterization
 - XPS
 - Pore size distribution measurements
 - Water and mercury porosimetry
 - Humidity Fingerprint

Relative Humidity “Fingerprint”



- GDLs after aging show different performance as a function of relative humidity

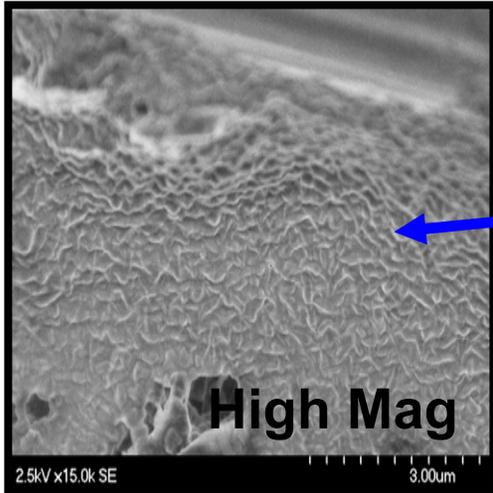
GDL Fiber Chemistry and Contact Angle



- Fiber graphitization can increase single-fiber contact angle $\sim 10^\circ$
- Both graphitization T and PTFE loading can change the liquid-water wetting regime of the GDL substrate.

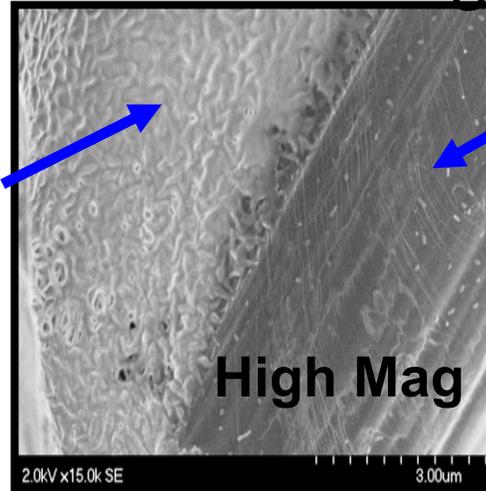
Teflon Distribution on GDLs

Fresh



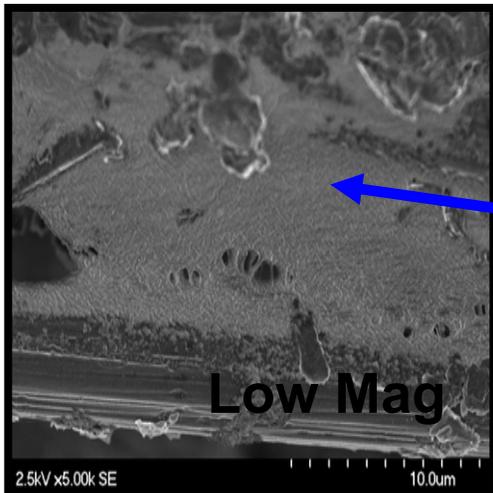
Teflon

After Testing

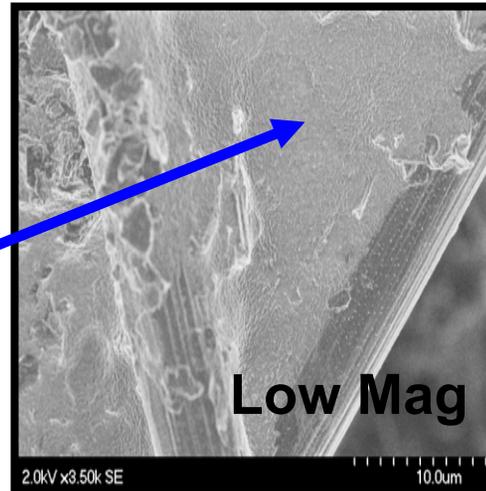


Uncoated fiber

- Carbon fibers not uniformly coated by Teflon
- Teflon 'globules' show less delineation after testing

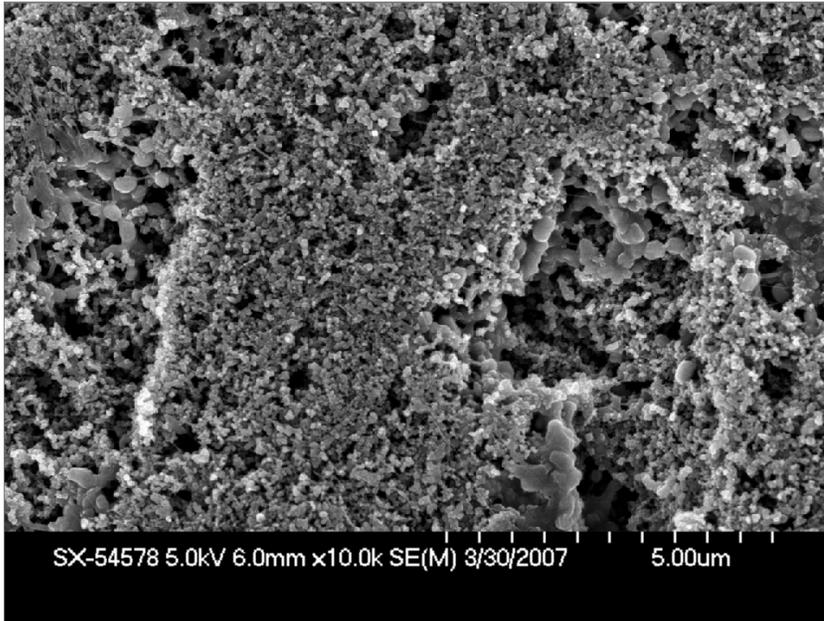


Teflon

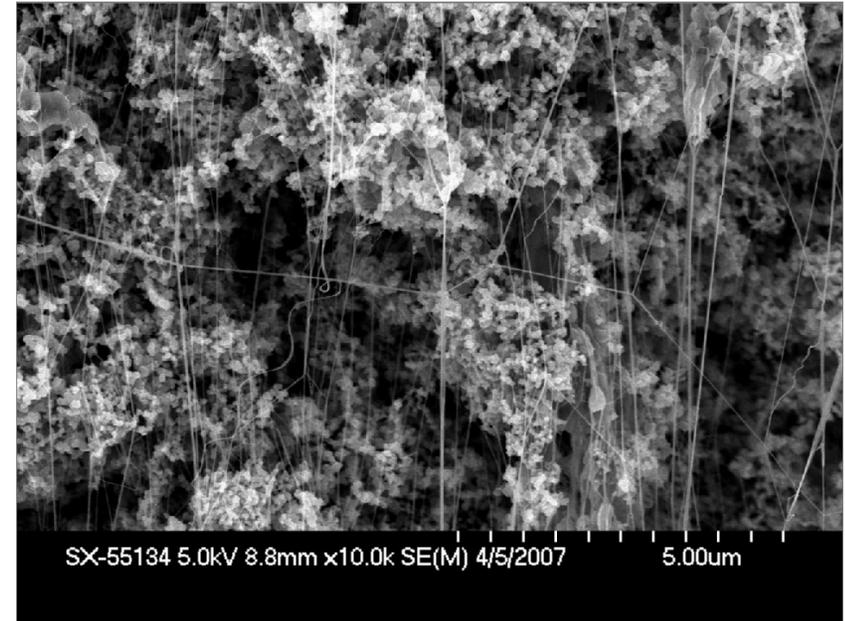


MPL Characterization

Fresh Material

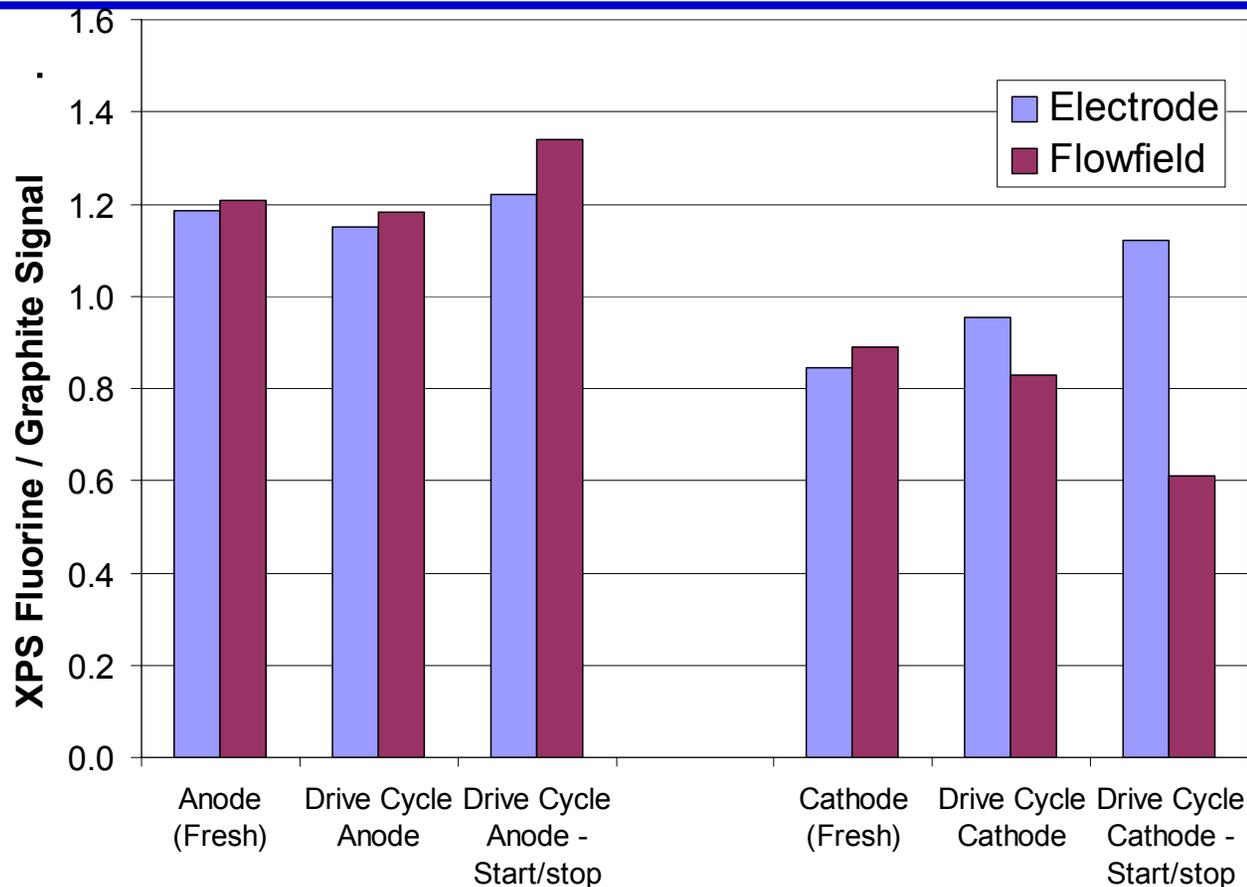


After Drive Cycle Testing



- Fresh Materials
 - Gaps/openings on surface of MPL where Teflon appears to have been non-homogeneously dispersed within the MPL
- After testing:
 - MPL Layer shows higher degree of porosity
 - Surface includes higher degree of long fibers (of Teflon)

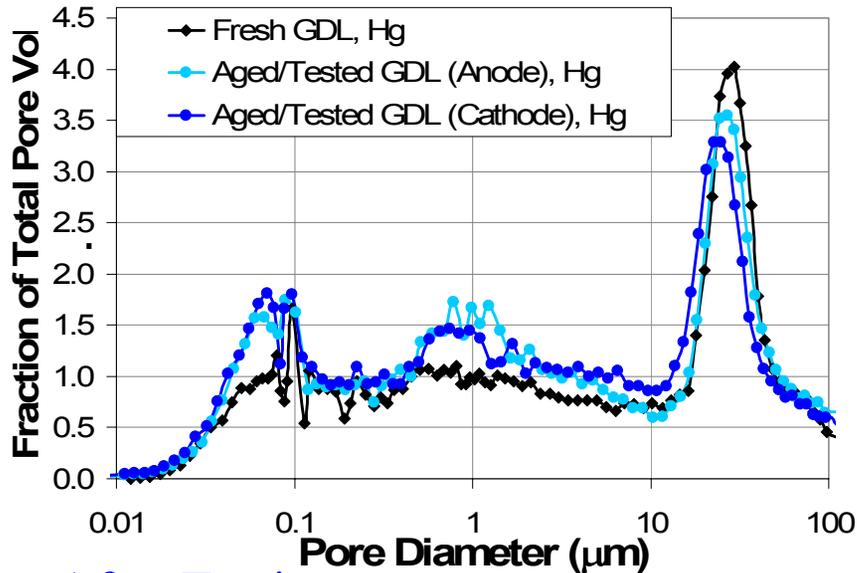
Elemental Compositional Changes of GDL Material



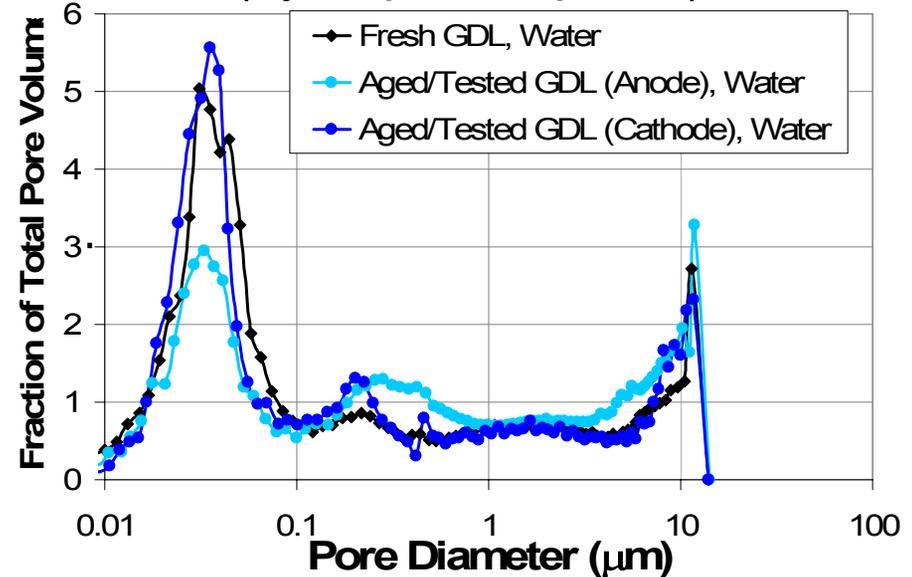
- Anode GDL composition shows little change
- Cathode GDL composition is enriched in fluorine on electrode side
- Cathode GDL composition is depleted in fluorine flowfield side

GDL Porosimetry

Hg Porosimetry of GDL (all pores)



Water Porosimetry of GDL (hydrophobic pores)



After Testing:

- Decrease in large-pore volume (~ 30 to $60 \mu\text{m}$)
- Increase in overall small-pore volume
- Decrease in hydrophobic small-pore volume

Hypothesis

- Large pore volume loss due to irreversible compression
- Small pore volume increase due to loss of carbon from MPL

Modeling of Mass Transport Losses

$$\eta_{tx,elec} = (b^* - b_{ideal}) \cdot \log i_{eff} + \log \left[\frac{(10L_{cath} A_{Pt,elec} i_{0,ideal})^{b_{ideal}}}{(10L_{cath} A_{Pt,elec} i_0^*)^{b^*}} \right]$$

$$\eta_{tx,GDL} = E_{eq}(T, p_{H_2}, p_{O_2}) - V_{iR-free} - \eta_{ORR}^*$$

- Difference in Tafel slope between dry air cathode and humidified O₂ cathode is basis for calculating mass-transport overpotential of electrode.
- After all other overpotentials have been calculated, balance is assigned to cathode GDL.

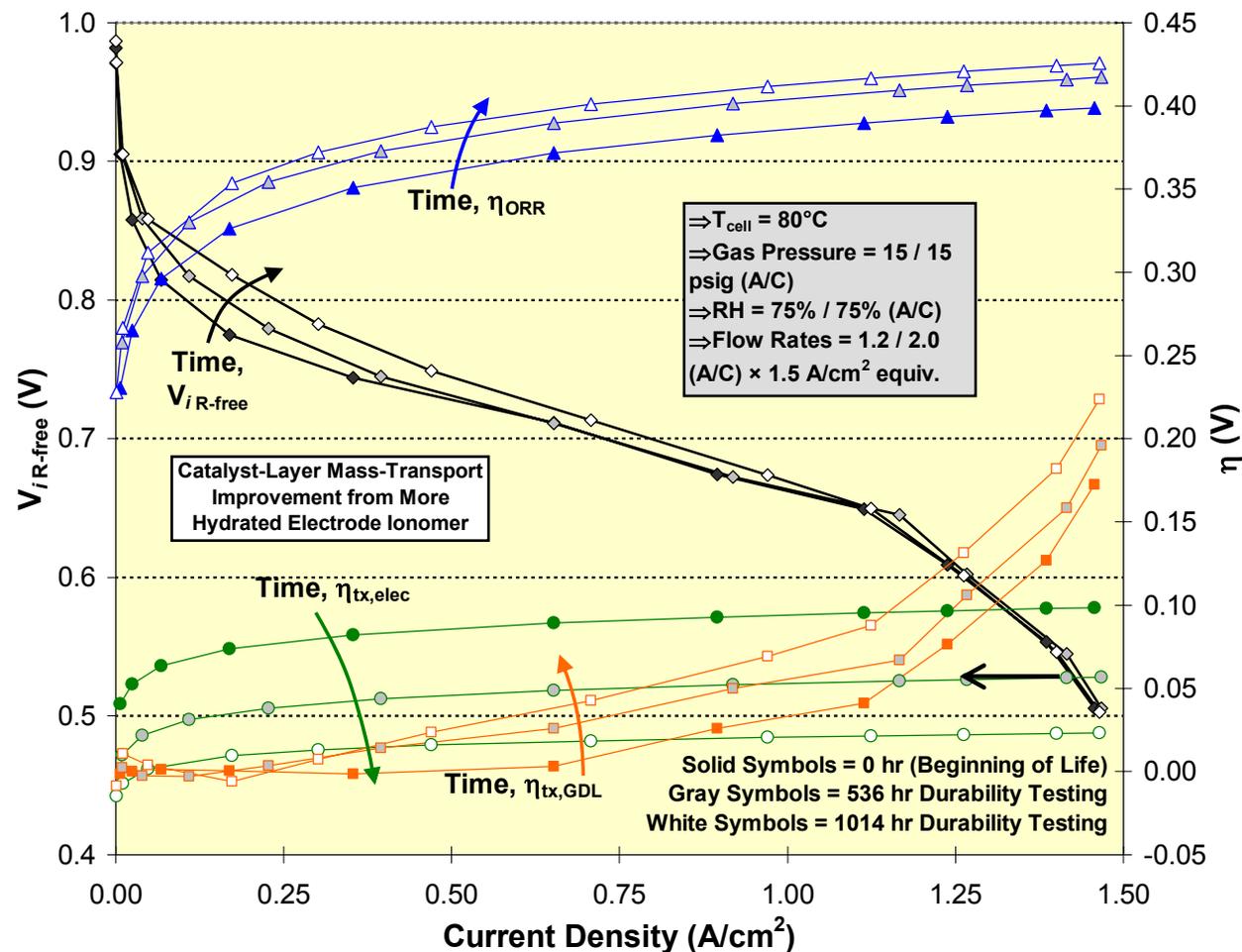
D. Wood, J. Davey, P. Atanassov, and R. Borup, *Electrochemical Society Transactions*, 3 (1), 753-763 (2006).

H.A. Gasteiger *et. al.*, *Handbook of Fuel Cells*, Vol. 3, Part 1, Chapter 46, pp. 593-610, John Wiley & Sons, New York (2003).

M.V. Williams *et. al.*, *J. Electrochem. Soc.*, **152**, A635 (2005).

Durability Performance Modeling

Durability of iR-Corrected Overpotentials



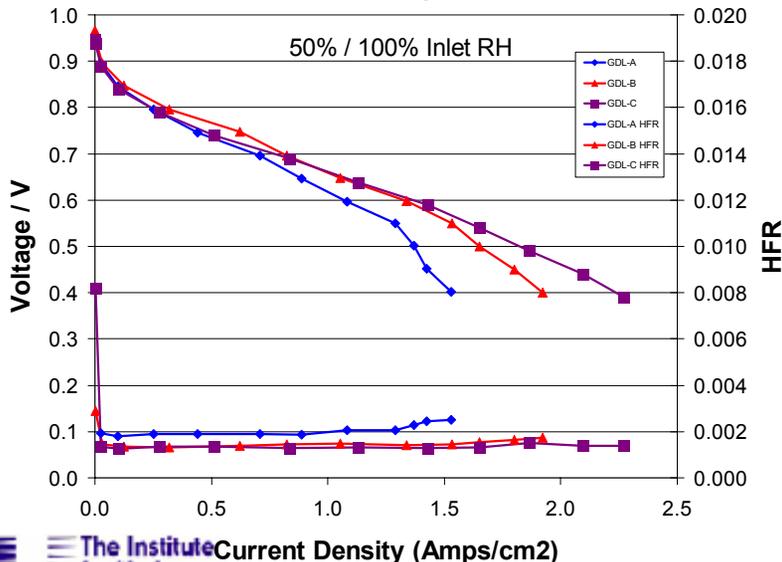
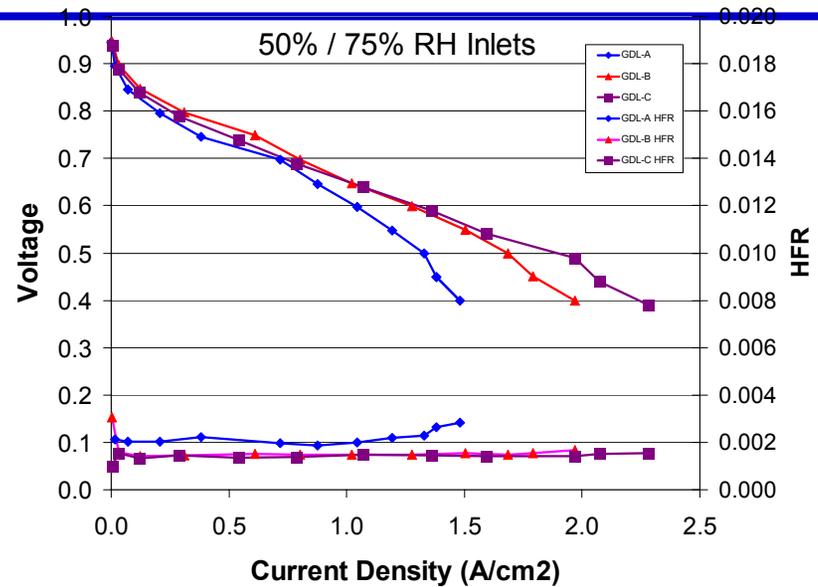
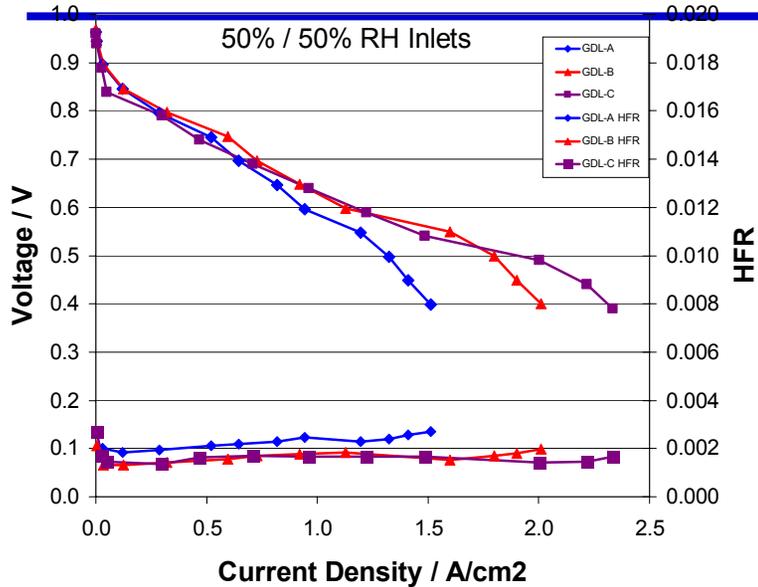
- Modeling of mass-transport losses extrapolated to 'over-potential'
- Definition of components leading to performance degradation
 - Method for analyzing performance losses (iR, ORR, MT)
 - Better understanding of long-term fuel cell test data

Varying GDL Effect (50 cm²)

PROPERTY	UNITS	GDL A	GDL B	GDL C
Name		GDL 24 BC	GDL 24 BC	GDL 24 DC
Thickness	μm	237	231	239
Substrate Loading	% PTFE	5%	5%	20%
MPL Loading	% PTFE	23%	10%	10%
Aerial weight	g/m ²	101.1	105.8	117
Air Permeability	cm ³ /cm ² .s	0.3	0.3	0.7
Specific Resistivity	mΩcm ²	8.4	10.3	11.9

- GDL was varied with identical MEAs to evaluate water transport
- Standard Operating Conditions: 20 psia, 1.1/2.0 stoichs, 80 °C cell temp, 50% anode inlet RH
 - Vary GDL/MPL Teflon loading, cathode inlet RH, current density

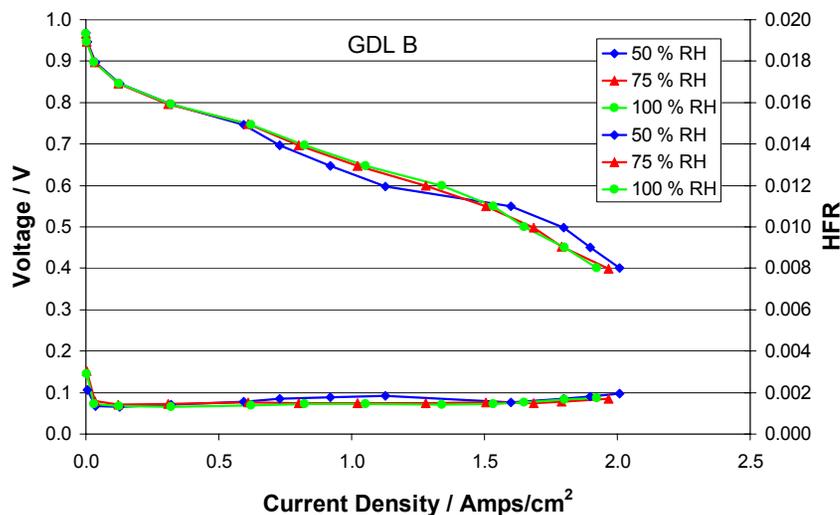
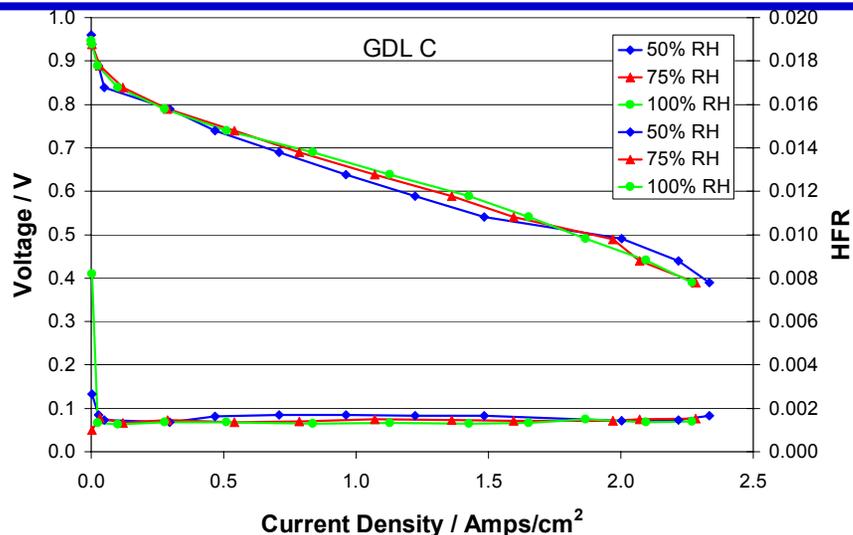
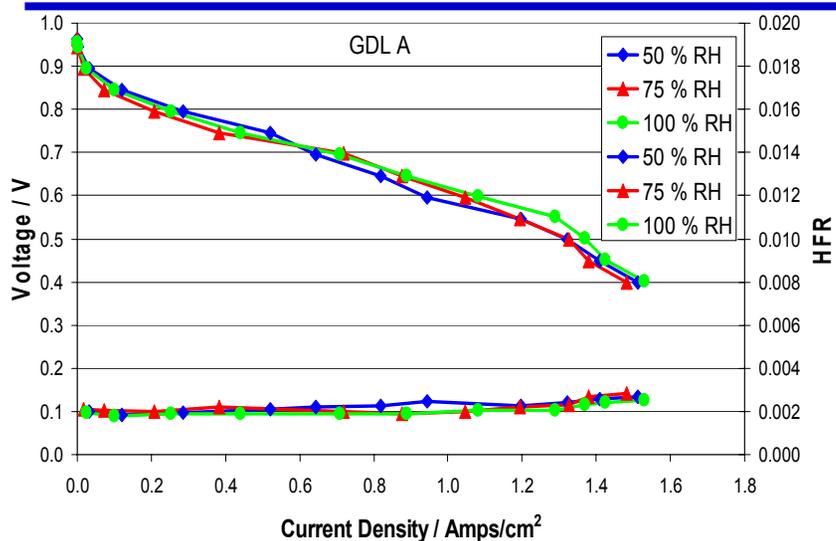
Varying GDL Effect



Low MPL teflon loading shows improved fuel cell performance

Cell Temperature = 80 °C
 Constant Stoich = 1.1 / 2.0
 Pressure = 25 psia
 50 cm² active area
 Anode: 0.2mg/cm² Pt
 Cathode: 0.4mg/cm² Pt
 Anode RH: 50%
 Cathode RH: 50, 75, 100%

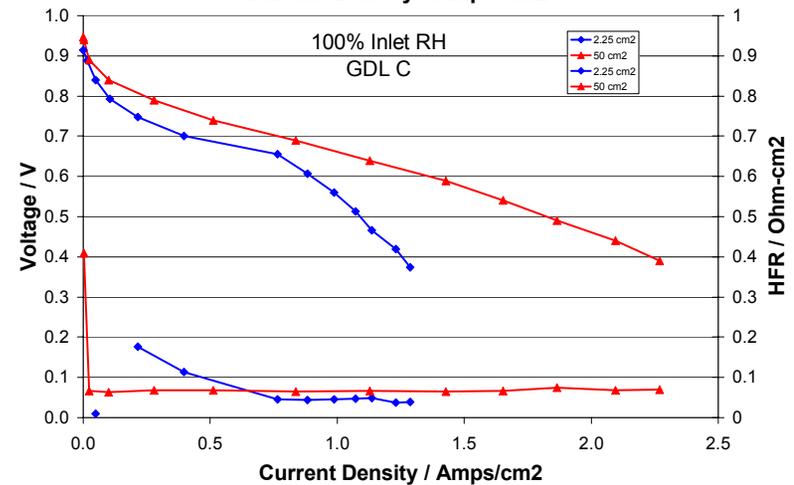
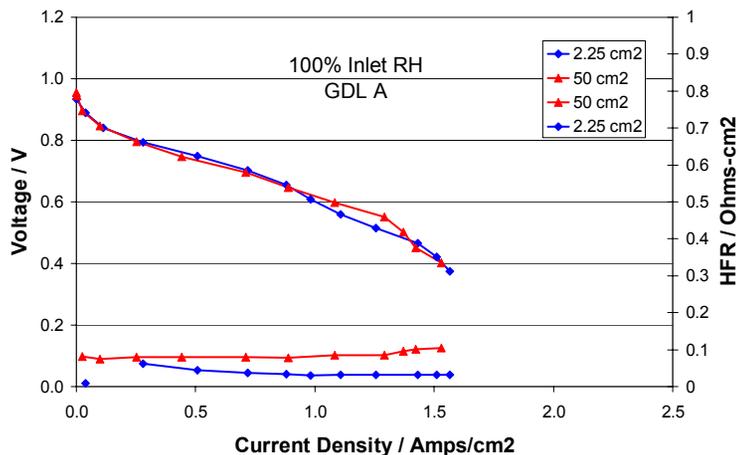
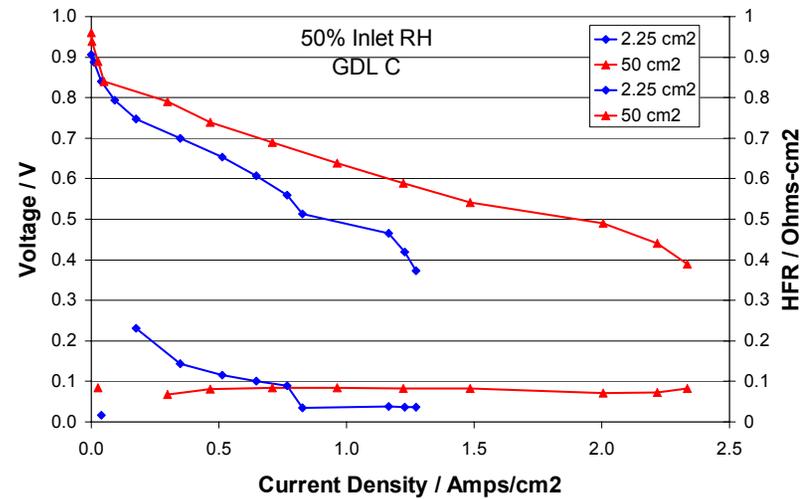
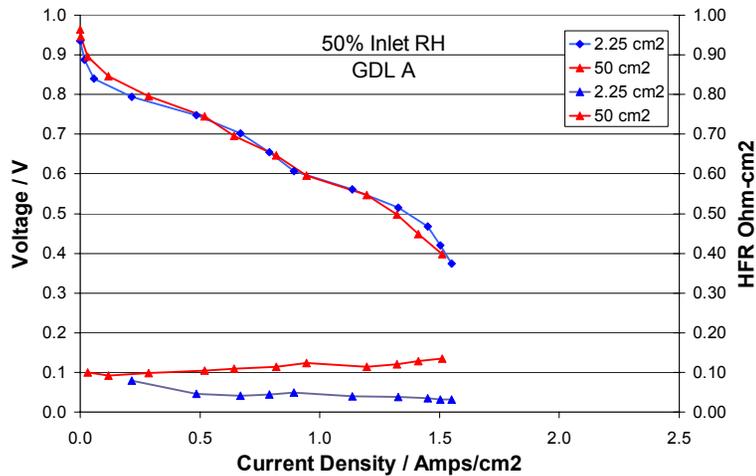
Cathode Inlet RH Effect



- Inlet Cathode RH down to 50% does not affect performance

Cell Temperature = 80 °C
 Constant Stoich = 1.1 / 2.0
 Pressure = 25 psia
 50 cm² active area
 Anode: 0.2mg/cm² Pt
 Cathode: 0.4mg/cm² Pt
 Anode RH: 50%
 Cathode RH: 50, 75, 100%

Comparison of Different Active Area Cells

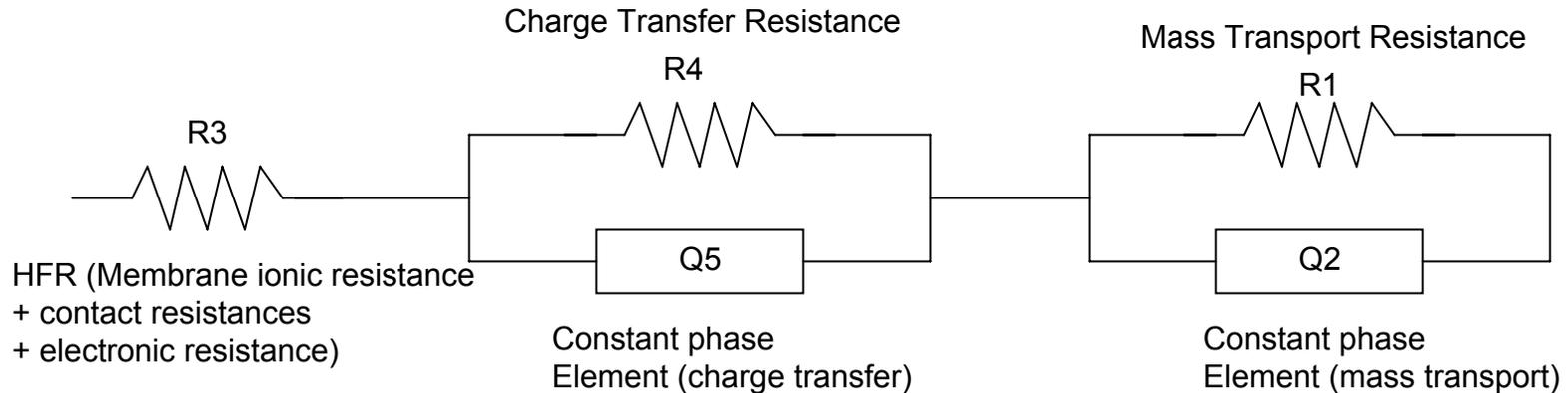


2.25 vs. 50 cm² Performance

- Performance of low area cells can be lower than the 50cm² cells.
- Little pressure drop in these cells, cells may be drier at the current densities.

AC Impedance Evaluation

AC Impedance Model Circuit

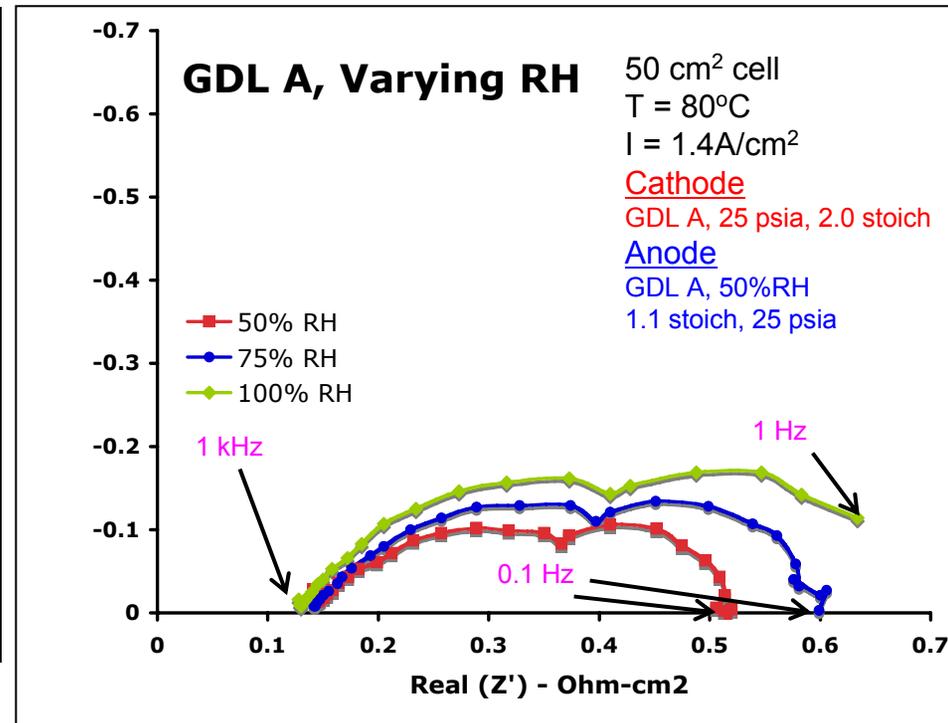
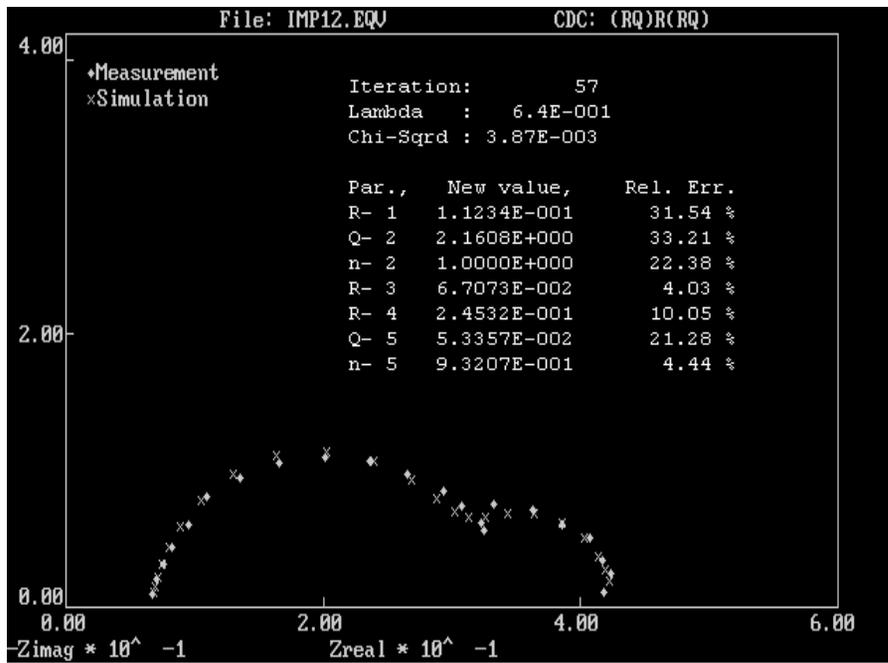


Cell Conditions:
50 cm² cell
T = 80°C

Anode
GDL A
50%RH
25 psia
1.1 stoich

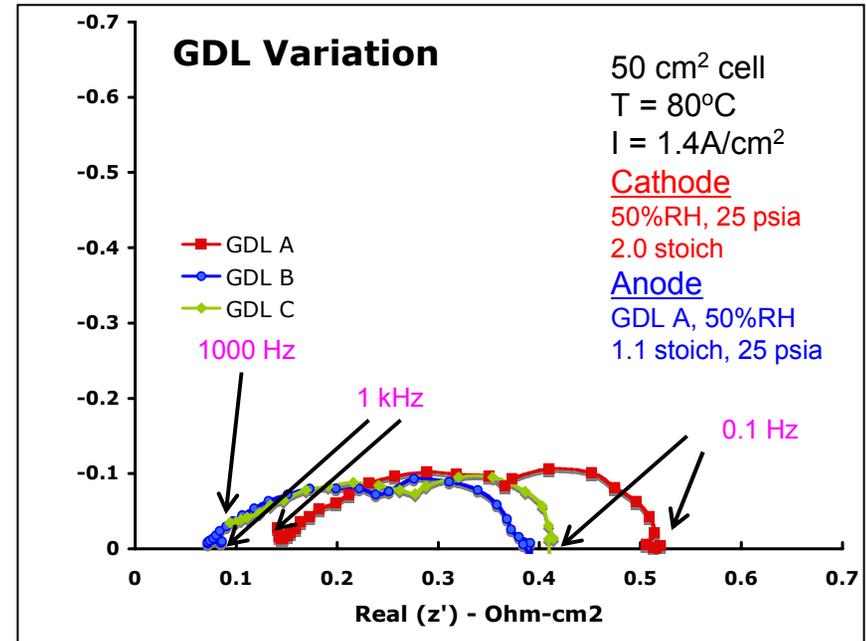
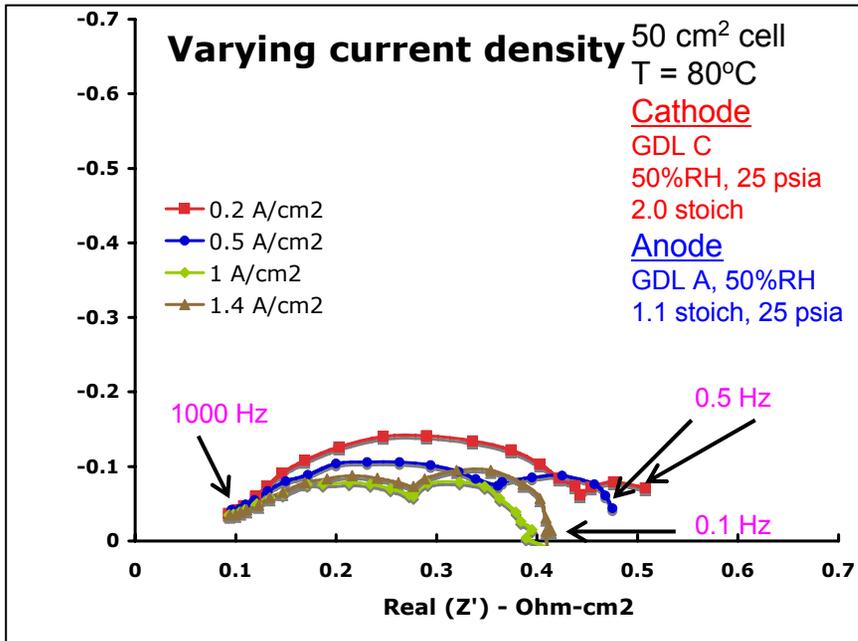
Cathode
GDL C
100%RH
25 psia
2.0 stoich

AC Impedance Evaluation



- High Frequency Resistance (HFR)
 - Decreases with increasing RH
- Charge transfer resistance
 - Decreases with increasing RH
- Mass transfer resistance
 - Increases with increasing RH

AC Impedance Comparison



- **High Frequency Resistance (HFR)**

- Decreases with increasing current
- Greater for GDL with 23% PTFE in MPL

- **Charge transfer resistance**

- Decreases with increasing current
- Greater for GDL with 23% PTFE in MPL

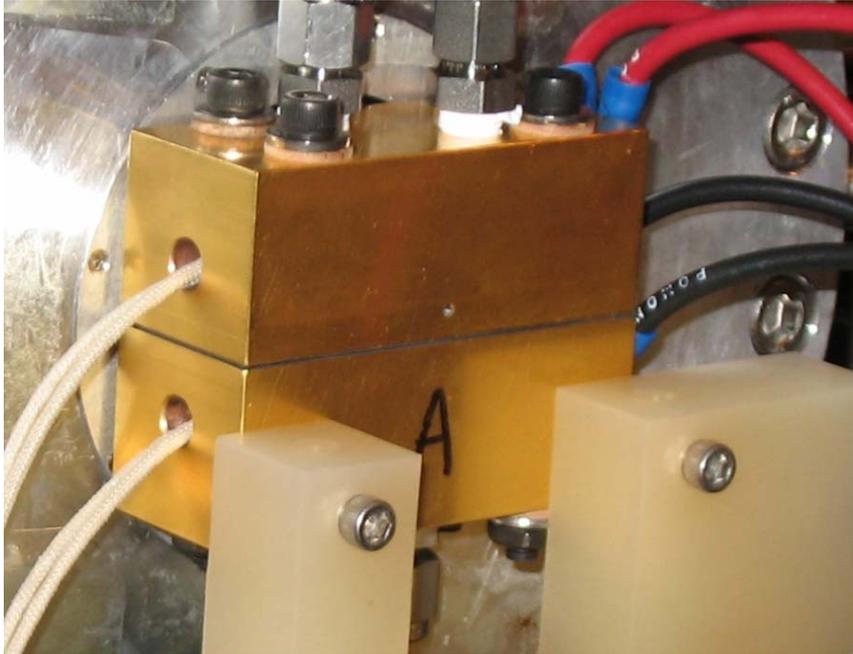
- **Mass transfer resistance**

- Increases with increasing current
- Greater for GDL with 23% PTFE in MPL

Neutron Imaging

Cross-Section Design for High Resolution Imaging

High resolution (15 μm)
cross-section cell



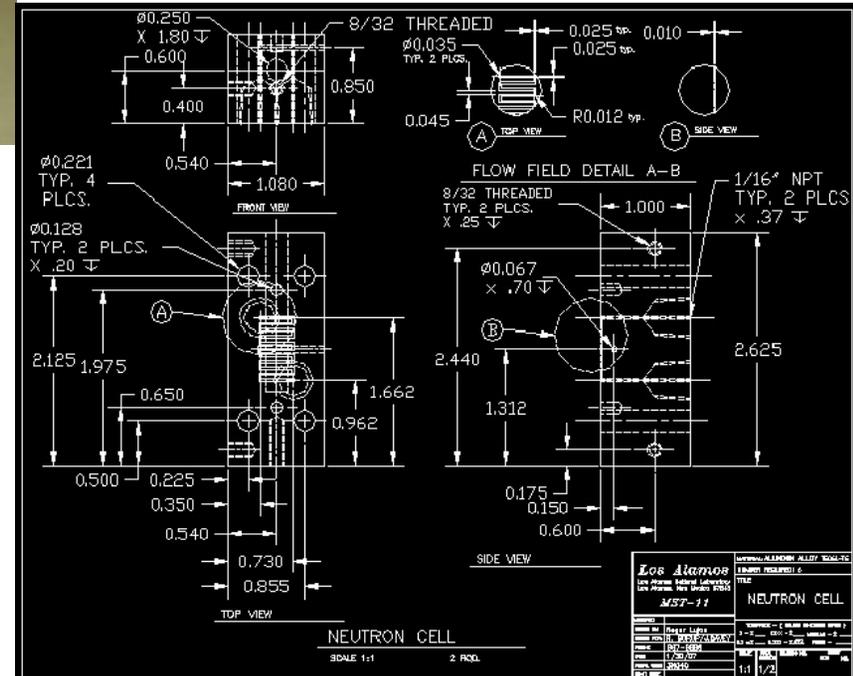
Design Considerations:

- Maximum field of view is 2 cm X 2 cm for the high resolution neutron detector.
 - Limits X dimension to 2 cm.
- Outermost edge to image = 3 cm from the detector for good focus.
 - Detector is 0.5 cm inset of the face plate, \rightarrow 2.5 cm available
- Active area < 1 cm in length
 - Entire cell is < 3 cm from detector

Design:

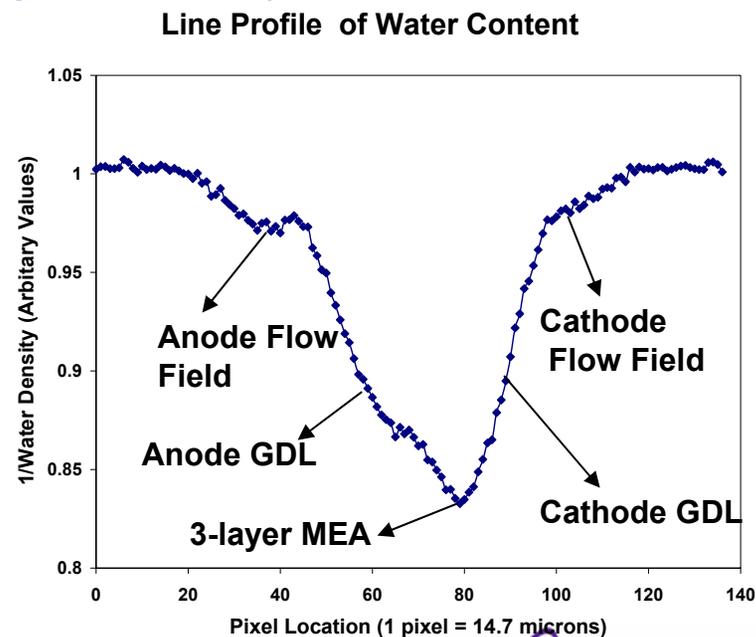
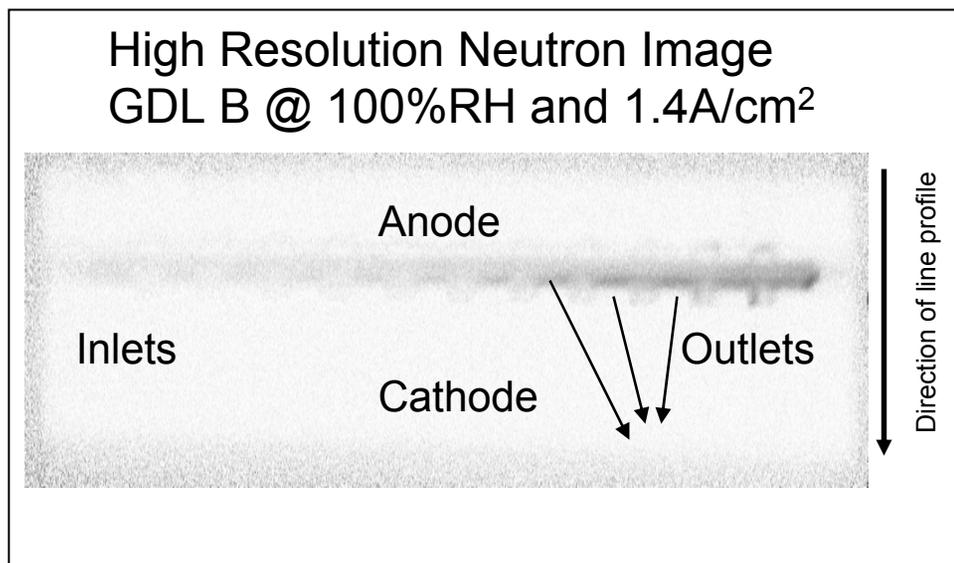
- 2.25 cm² active area
- No hydrocarbon materials
- Metal hardware
 - No plate porosity of hardware for water hold-up
- 1 cm linear water imaging length
- Shallow single serpentine flowfield
 - Attempt to simulate pressure drop of real flowfields

Cross-Section Design for Neutron Imaging



High Resolution (15 μm) Cross-Section Water Imaging of Operating Fuel Cells

- See water in the outer walls of the serpentine flow fields
 - The flow field near the MEA is mostly clear
- Cathode GDL contains more water than anode GDL with more water accumulating toward the outlets
 - Steeper drop in the line profile intensity (darker areas)
- More water in the GDLs above the land than the GDLs above the channel
 - Water created at catalyst layer over land needs to diffuse laterally through GDL and get to the channels before being taken away into the flow fields
- MEA (3 pixels) contains the most water

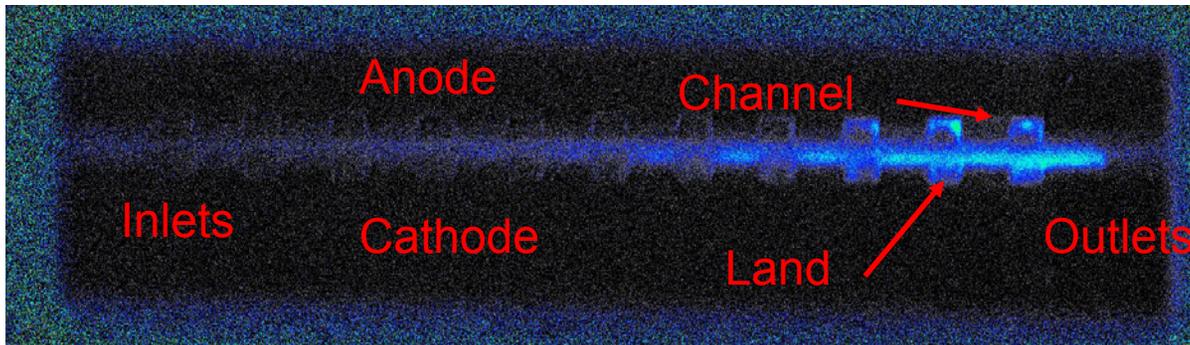


Water Image of MEA Cross-Section

GLD A

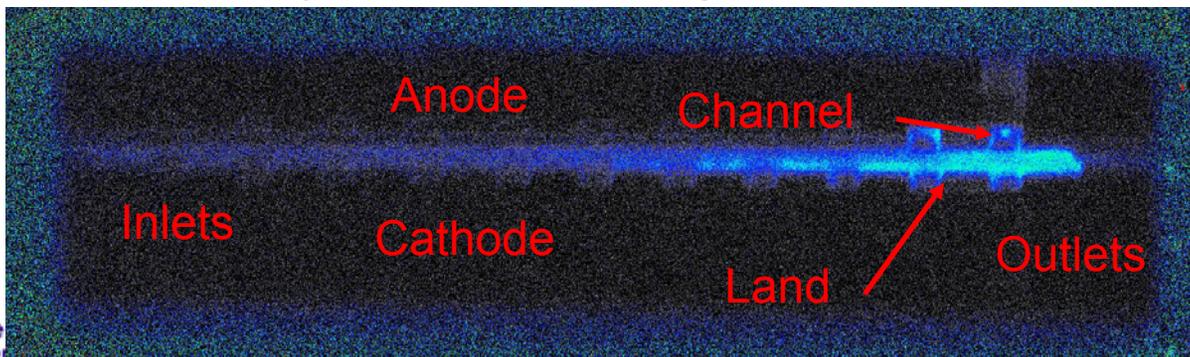
- More water in:
 - Cathode GDL vs the Anode GDL
 - Outlets vs the Inlets
 - GDL above the land vs the GDL above the channel

0.2A/cm²
Cathode
50%RH
2.0 Stoich
Anode
50%RH
1.1 Stoich



- Less water in the flow channels at high current densities
 - Higher flow of gases keeps the channels clear of water
- Significant water content in the anode GDL near outlets
 - Back diffusion of water from cathode to anode is significant (Driven by water concentration gradient)

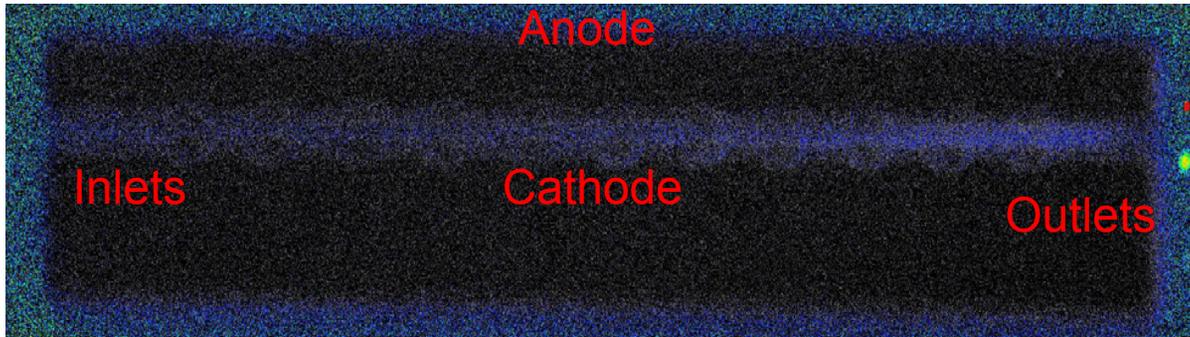
1.4A/cm²
Cathode
100%RH
2.0 Stoich
Anode
50%RH
1.1 Stoich



Water Image of MEA Cross-Section GLD B

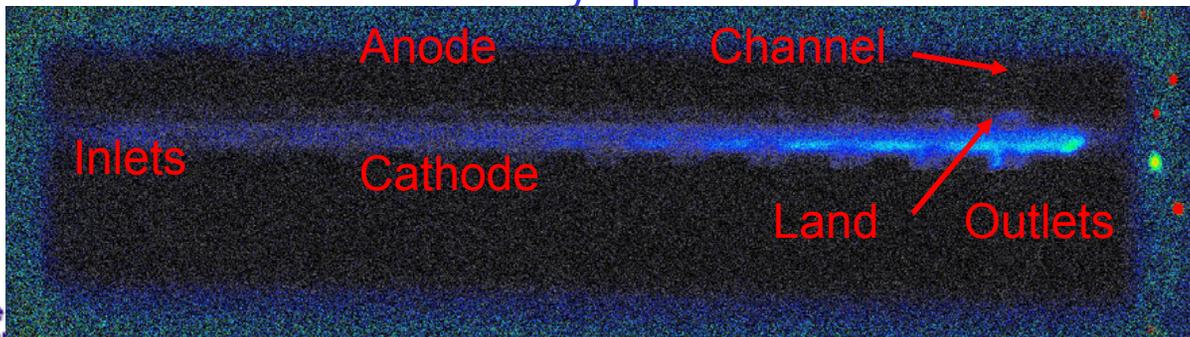
- Less water in GDL B when compared to GDL A
 - Explains better performance of GDL B esp. at higher current densities
 - Teflon content in the microporous layer (10wt% vs 23wt%) has a major influence on water transport
 - Higher teflon content in the MPL leads to flooding

0.2A/cm²
Cathode
50%RH
2.0 Stoich
Anode
50%RH
1.1 Stoich



- High water density at the cathode GDL
 - More near the outlet and over the land area
- Higher water content everywhere when compared to the low RH/low current density operation

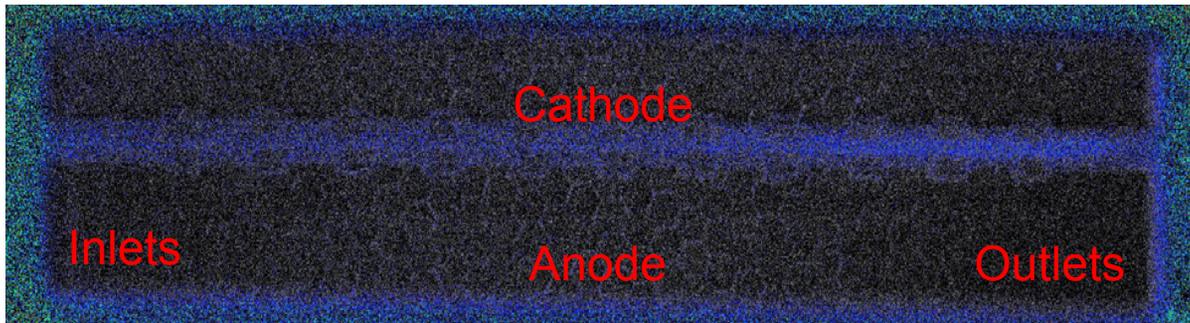
1.4A/cm²
Cathode
100%RH
2.0 Stoich
Anode
50%RH
1.1 Stoich



Water Image of MEA Cross-Section GLD C

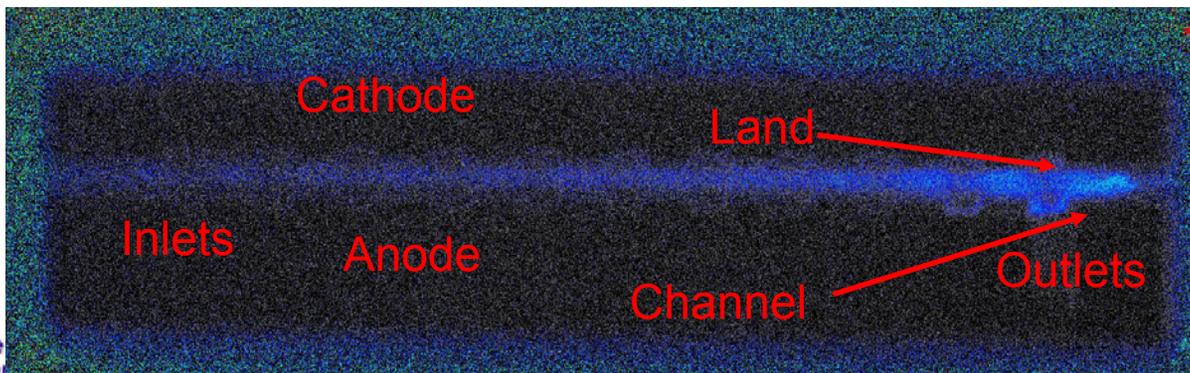
- Similar water in GDL C when compared to GDL B
 - Explains the better performance when compared to GDL A
 - Explains the similar performance of this GDL to GDL B
 - Teflon content in the substrate (5 wt% vs 20 wt%) does not have a major role in determining water content

0.2A/cm²
Cathode
50%RH
2.0 Stoich
Anode
50%RH
1.1 Stoich



- Gravity may play small role in cells with little pressure drop
 - More anode water when anode was at the bottom
 - This water is mainly in the flow filed channels near the outlet (does not affect performance)

1.4A/cm²
Cathode
100%RH
2.0 Stoich
Anode
50%RH
1.1 Stoich

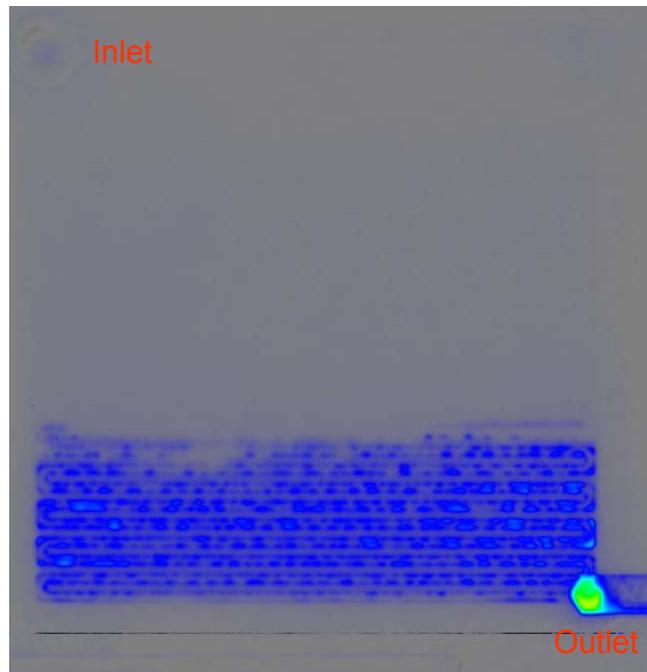


Water Image of Flowfield (50 cm² MEA) GLD A

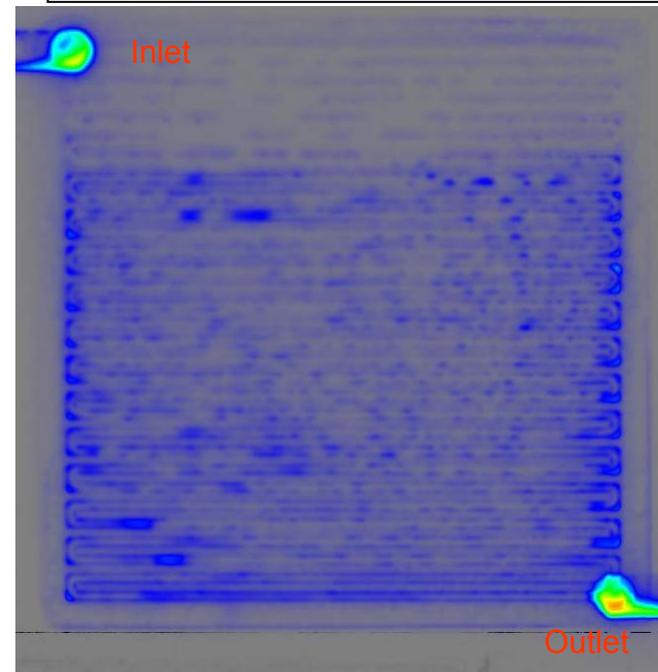
- More water accumulation near the inlet at higher cathode inlet RHs/high current density
- Water accumulates at the turns of the serpentine flow channels
- Low gas flow rate leads to more water in the channels especially near the outlet

Increasing water content →

Water density with respect to dry image
900 image average
1 image/second for 15 minutes



50cm²
Cathode
Air
25psia
2.0 stoich
Anode
H₂
50%RH
25 psia
1.1 stoich



50%RH / 0.2A/cm²

100%RH / 1.4A/cm²

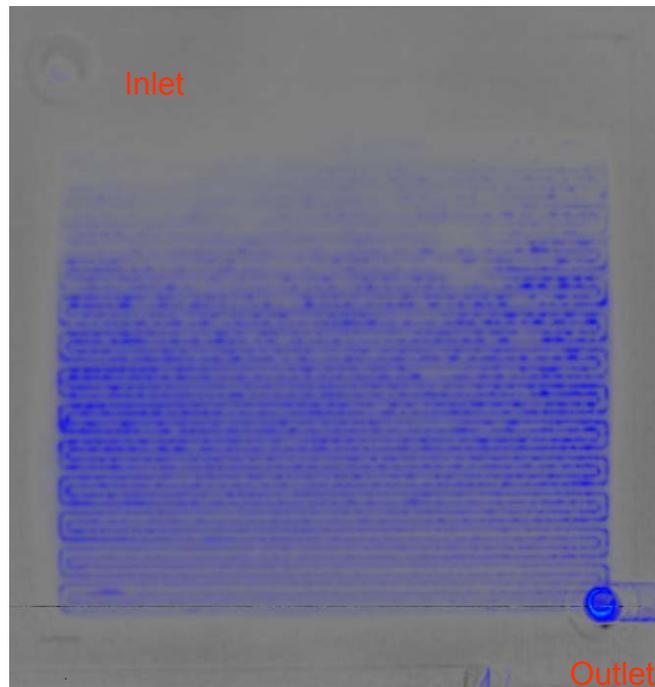
Water Image of Flowfield (50 cm² MEA)

GLD B

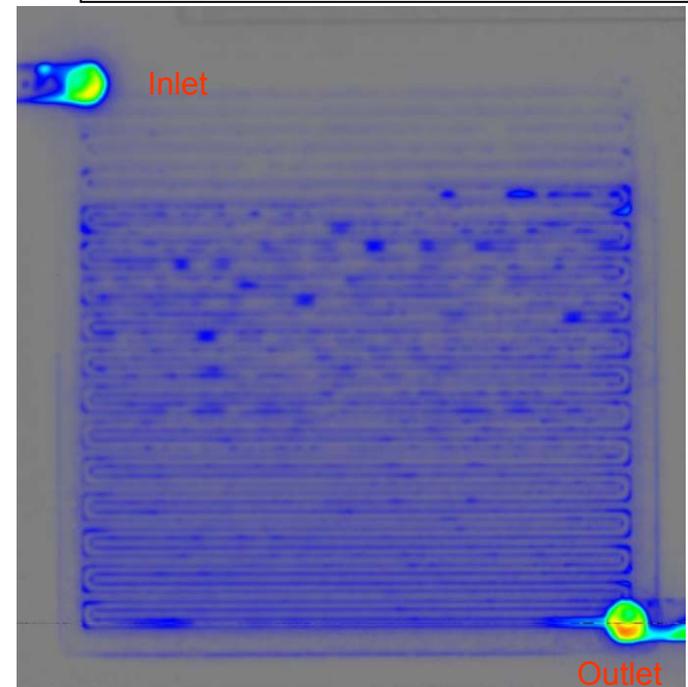
- More water accumulation near the inlet at higher cathode inlet RHs high current density
- Water accumulates at the turns of the serpentine flow channels
- Low gas flow rate leads to more water in the channels especially near the outlet

Increasing water content

Water density with respect to dry image
900 image average
1 image/second for 15 minutes



50cm²
Cathode
Air
25psia
2.0 stoich
Anode
H₂
50%RH
25 psia
1.1 stoich



50%RH/ 0.2A/cm²

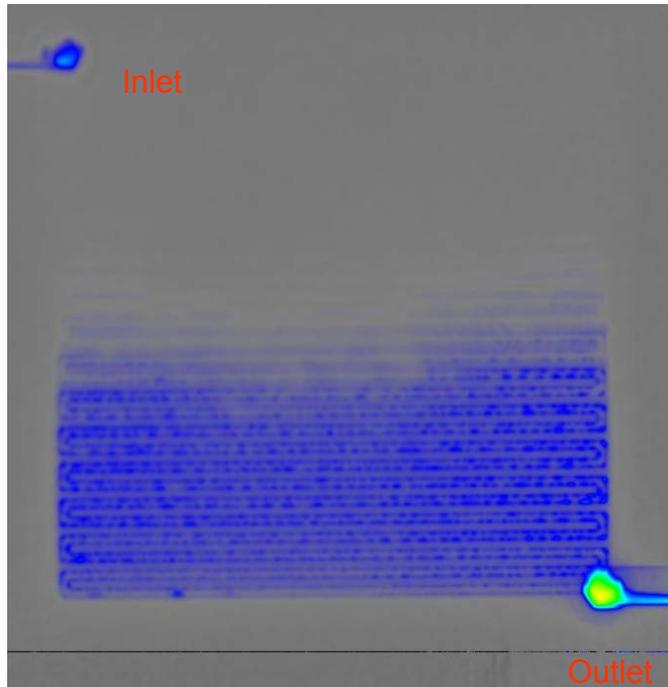
100%RH / 1.4A/cm²

Water Image of Flowfield (50 cm² MEA) GLD C

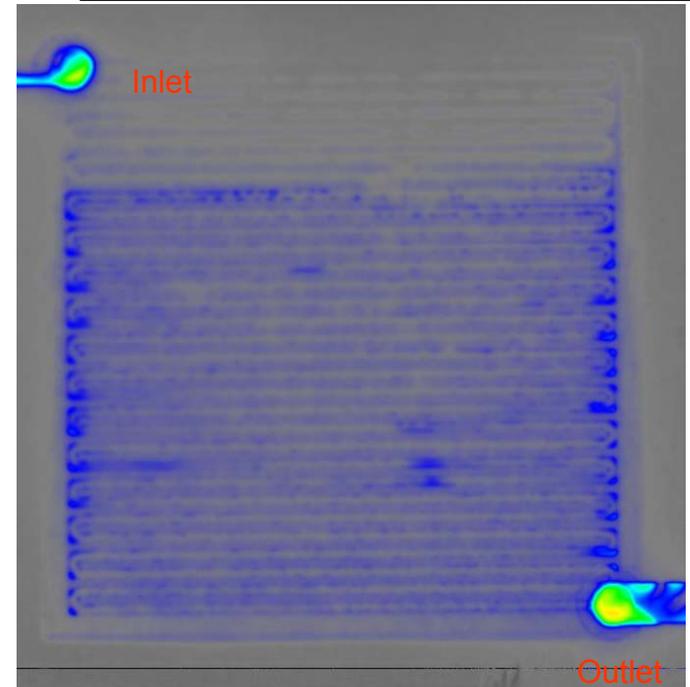
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Increasing water content →

Water density with respect to dry image
900 image average
1 image/second for 15 minutes



50cm²
Cathode
Air
25psia
2.0 stoich
Anode
H₂
50%RH
25 psia
1.1 stoich



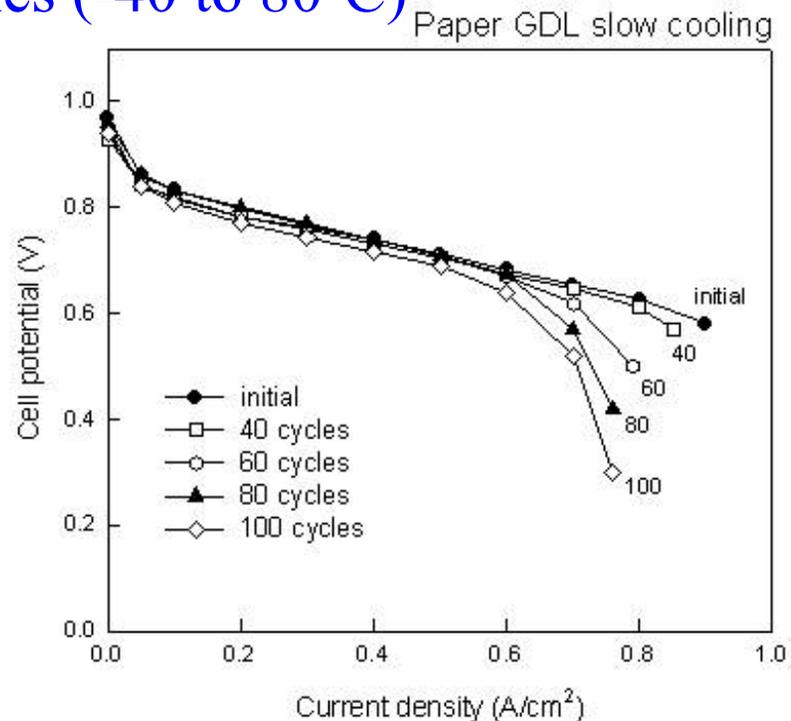
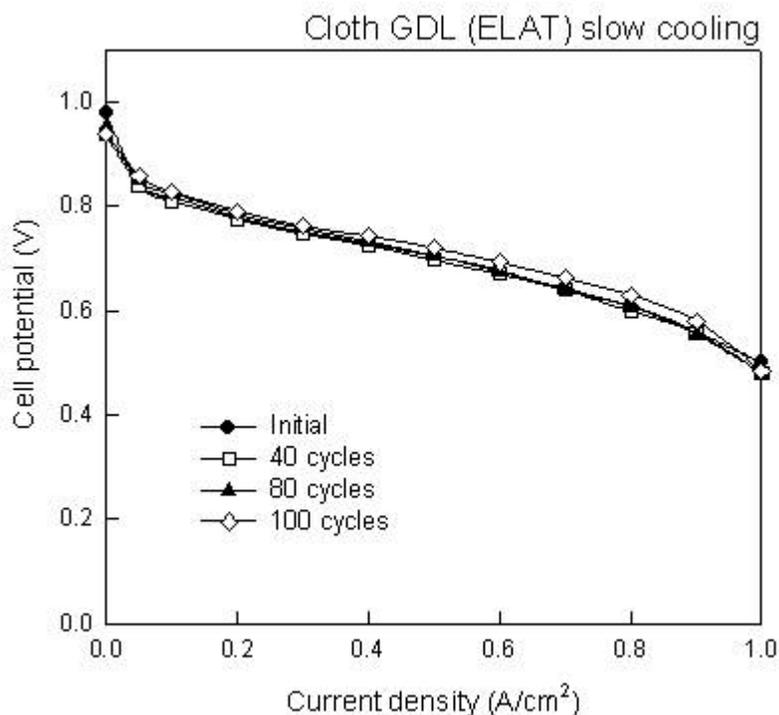
50%RH / 0.2A/cm²

100%RH / 1.4A/cm²

Durability of Cloth and Paper GDLs

Slow Freeze/Thaw Cycles

Freeze Thaw Cycles (-40 to 80°C)



Fully humidified cells: T_{room} to -40°C in 3 hours

No change for cloth backing MEA;

Significant degradation for paper backing MEA after -40-80°C FT cycling

Durability of Paper GDLs

fast Freeze/Thaw Cycles

Freeze Thaw Cycles (-40 to 80°C)

Significant mass transport problem after 80 FT cycles for paper GDL

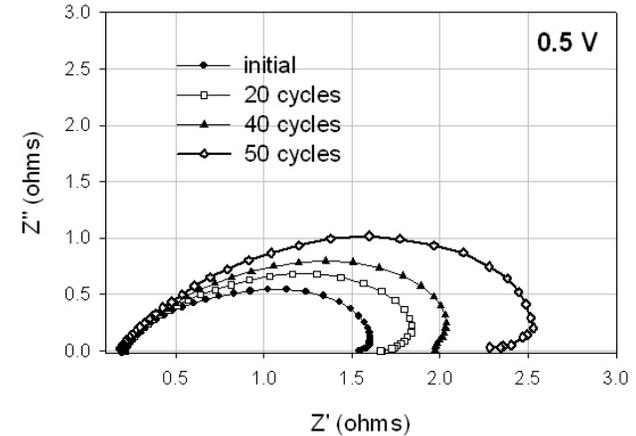
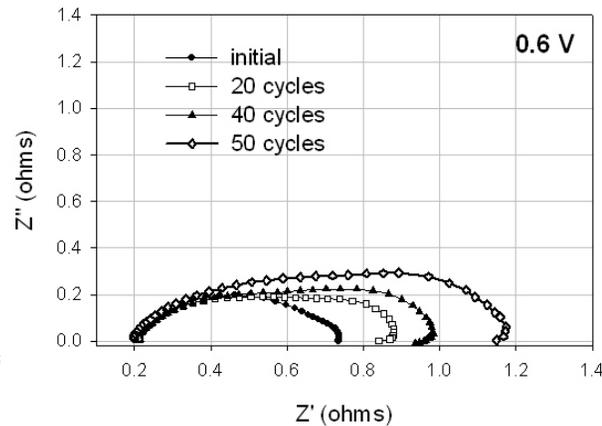
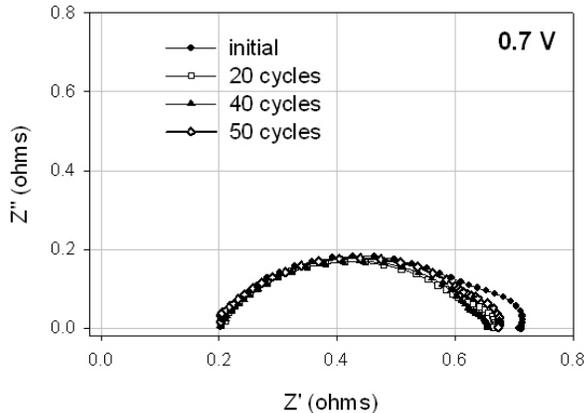
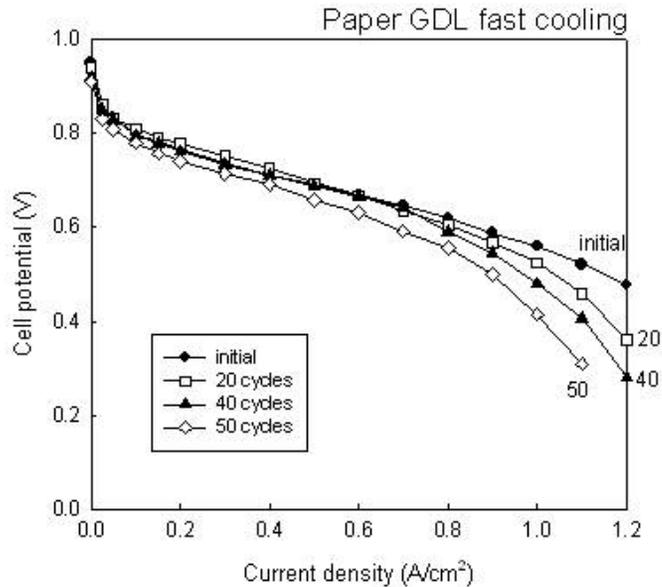
Accelerated testing

Fully humidified cells

Fast cooling rate (30 mins from 80°C to -40°C)

Resistance increases more at lower potentials

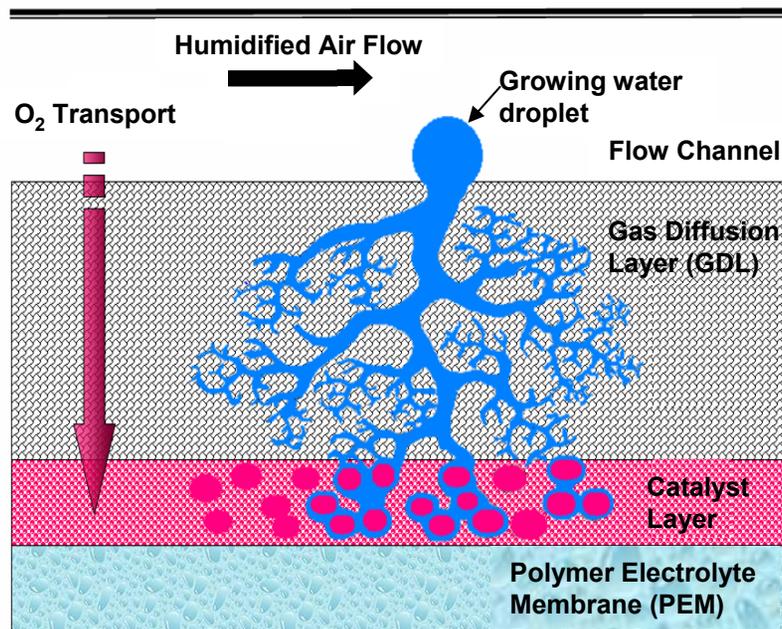
Previous results (confocal microscopy) have shown problems with fiber breakage



Predicting the Onset of Water-Droplet Detachment

Motivation: droplet detachment from GDL/channel interface is a key mechanism for liquid-water removal in PEM fuel cells. Elucidating water-droplet detachment from GDL/channel interface and being able to predict the critical air-flow velocity required to detach droplets can provide useful design and operational guidelines.

Schematic of water-droplet growing and being deformed by flowing air drag at the GDL/flow-channel interface



Ken S. Chen (kschen@sandia.gov)



Simplified Model

**Force balance on water droplet at onset of detachment:
(assumption: pressure drag due to inertial effect dominates)**

Pressure drag exerted on droplet surface = Surface tension acting along contact lines

Solving for the critical air-flow velocity, V_c , yields*:

$$V_c = \left[\frac{H_c}{\rho\mu} \right]^{1/3} \left[\frac{\pi\gamma \sin^2 \theta_s \sin \frac{1}{2}(\theta_a - \theta_r)}{5(\theta_s - \sin \theta_s \cos \theta_s)d} \right]^{2/3}$$

Where:

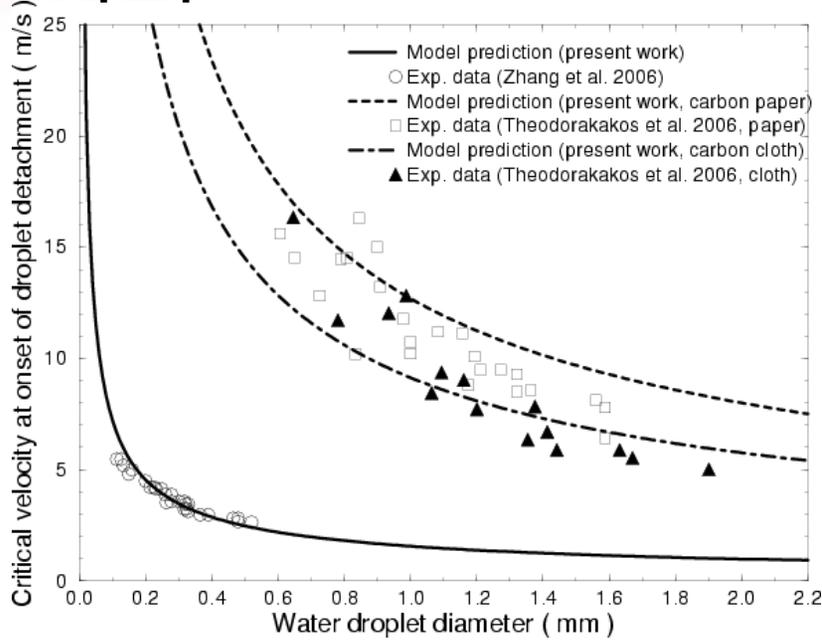
d = water droplet diameter, H_c = channel height,
 ρ = density, μ = viscosity, γ = surface tension,
 θ_s = static contact angle, θ_a = advancing contact angle,
 θ_r = receding contact angle.

- The critical air-flow velocity increases rapidly with decreasing droplet size ($V_c \sim d^{-2/3}$); V_c also increases with raising channel height and decreasing $\rho\mu$.**
- Making GDL surface more hydrophobic (i.e., increasing static contact angle, θ_s) and less rough (i.e., lowering contact-angle hysteresis) reduces critical velocity, V_c .**

*Reference: Chen, K. S., "A simplified model for predicting the critical air-flow velocity at the onset of water-droplet detachment in PEM fuel cells", manuscript in preparation.

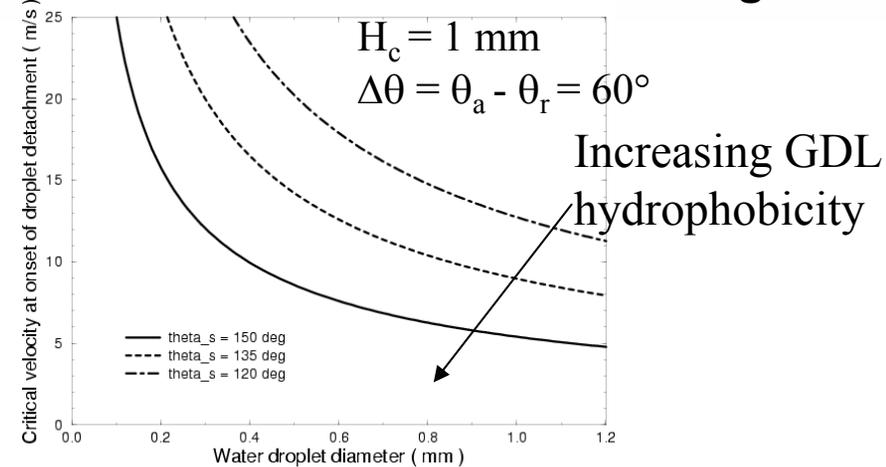
Effects of GDL Properties on Water-Droplet Detachment

Sample prediction and model validation

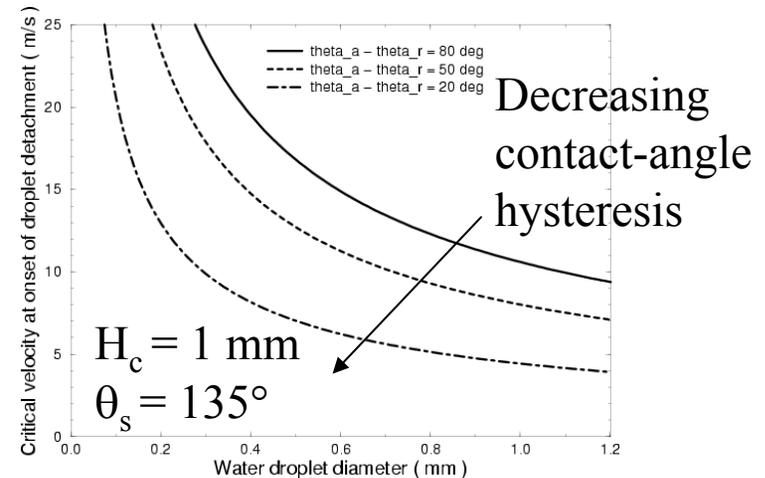


- Model yields good agreement with experimental data.
- More hydrophobic GDL reduces the critical velocity required to detach water droplets.
- Decreasing contact-angle hysteresis (e.g., reducing GDL surface roughness) enhances droplet removal.

Effect of GDL static contact angle



Effect of contact-angle hysteresis



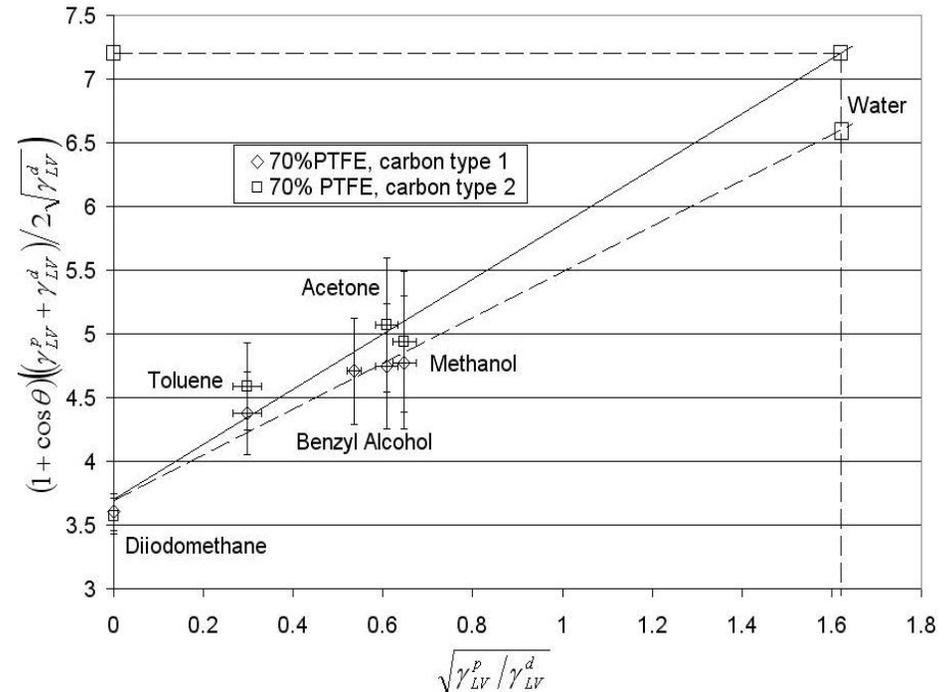
Ken S. Chen (kschen@sandia.gov)



Internal contact angle to water and surface energy of the GDL matrix:

- Technique developed at Case
- Define properties which affect the capillary action in the GDL pores, rather than GDL surfaces.

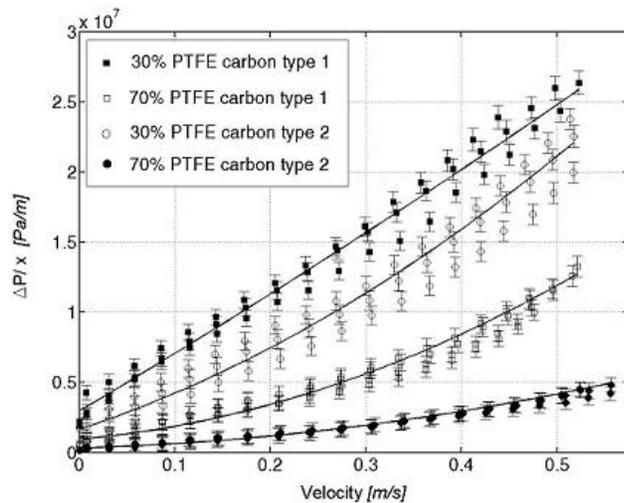
Sample	$\theta_{H_2O} [^\circ]$	γ_{SV}^d	γ_{SV}^p	γ_{SV}
30% PTFE carbon type 1	89 ± 3	13 ± 1	8 ± 2	21 ± 2
70% PTFE carbon type 1	101 ± 3	13.8 ± 0.8	3.1 ± 0.8	17 ± 1
30% PTFE carbon type 2	88 ± 7	14 ± 2	8 ± 3	22 ± 4
70% PTFE carbon type 2	96 ± 7	14 ± 2	4 ± 2	19 ± 3



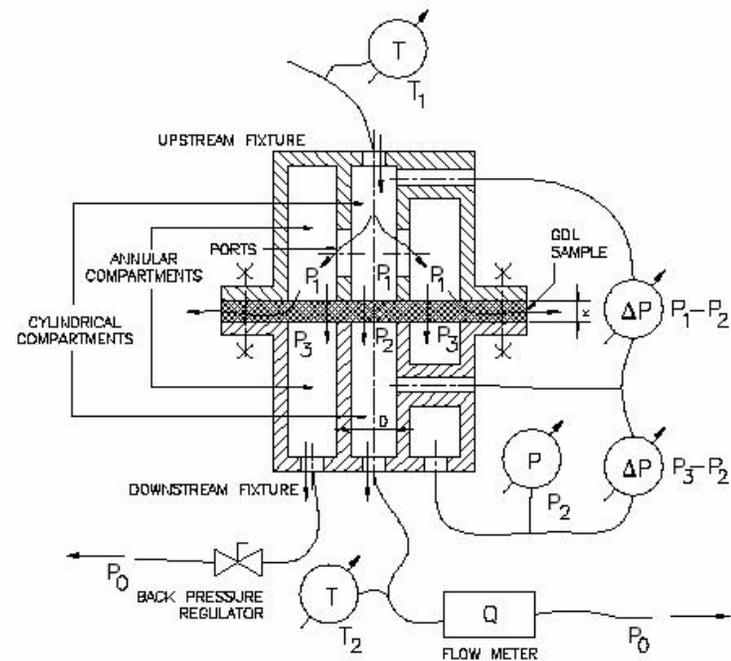
V. Gurau *et al.*, Journal of Power Sources, 160, (2006), 1156-1162

In plane, through-plane, viscous and inertia permeability for micro-porous layers and macro-porous substrates of GDLs

- Developed apparatus and measurement techniques
- Correlate GDL structure with transport properties



Sample	k_{Vz} $\times 10^{-12} [m^2]$	k_{Iz} $\times 10^{-8} [m]$
30% PTFE, carbon type 1	0.118 ± 0.013	34 ± 8
70% PTFE, carbon type 1	0.64 ± 0.05	8.0 ± 0.2
30% PTFE, carbon type 2	0.09 ± 0.02	6.3 ± 0.3
70% PTFE, carbon type 2	4.9 ± 0.7	18.6 ± 0.2

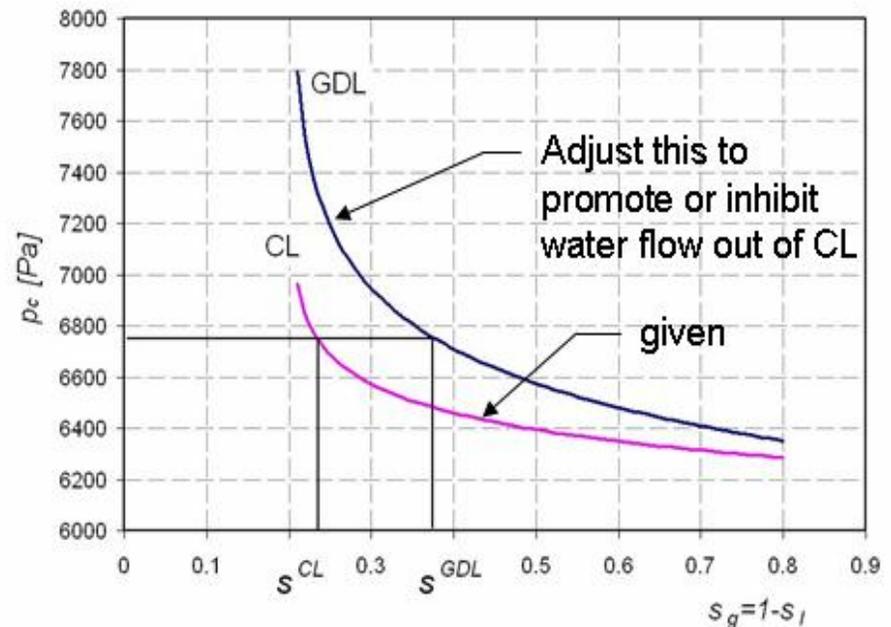


V. Gurau *et al.*, Journal of Power Sources, 165, (2007), 793-802

New method for capillary pressure measurements in GDLs:

Motivation:

- Inherently, catalyst layers lack the flexibility to control the amount of water that resides in their pores during fuel cell operation;
- GDLs may be designed to promote or inhibit water flow out of the catalyst layer pores; to achieve this, one needs to be able to assess the capillary pressure as function of water saturation $P_c(s)$ in the GDL;



New method for capillary pressure measurements in GDLs:

The new method consists in monitoring simultaneously

- the liquid water pressure
- the water saturation in an REV of the GDL sample using neutron imaging (at NIST)

Advantage:

The method is dynamic; no need to wait until saturation equilibrium is achieved in the sample (usually needs more than 24 hours to reach equilibrium for a single point);

Status:

- Apparatus was built at Case
- April 28-29, prove of concept scheduled at NIST using 3M GDL samples (micro-porous layer applied on polyimide films)

Modeling water dynamics and two-phase phenomena in PEMFC (3D)

Developed two-phase model for PEMFC (flow field, GDL, catalyst layer and membrane)

Capture two-phase transport and equilibrium:

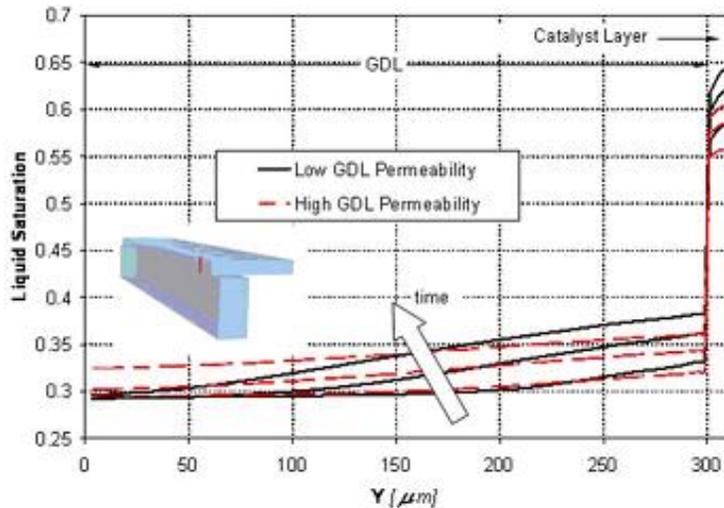
- *within* the fuel cell components (GDL, catalyst layer, membrane)
- *between* the fuel cell components (droplet formation at channel-GDL interface and saturation equilibrium at GDL-catalyst layer interface)

Capture competing mechanisms of water transfer between the catalyst layer pores and the ionomer distributed in the catalyst layer:

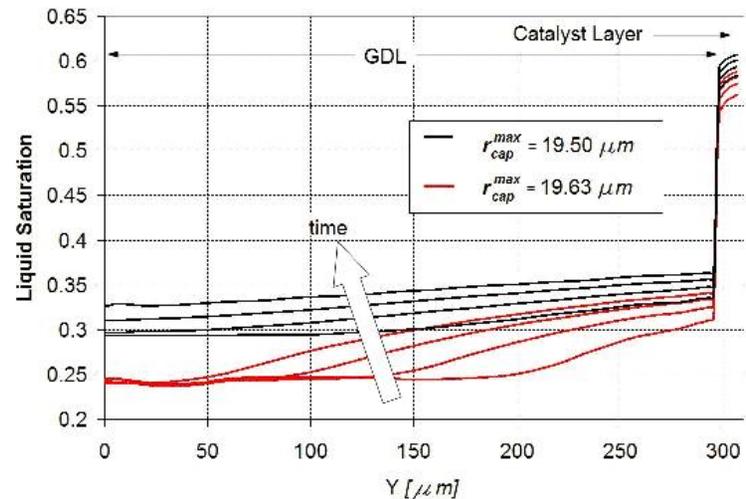
- Sorption/desorption
- Electro-osmotic drag of water out of the ionomer by the secondary current (dominant mechanism, not identified in the past)

Modeling water dynamics and two-phase phenomena in PEMFC (3D) - continued

Study the effect of the GDL structural properties on the amount of water accumulated in the GDL and the catalyst layer during fuel cell operation



Effect of GDL permeability



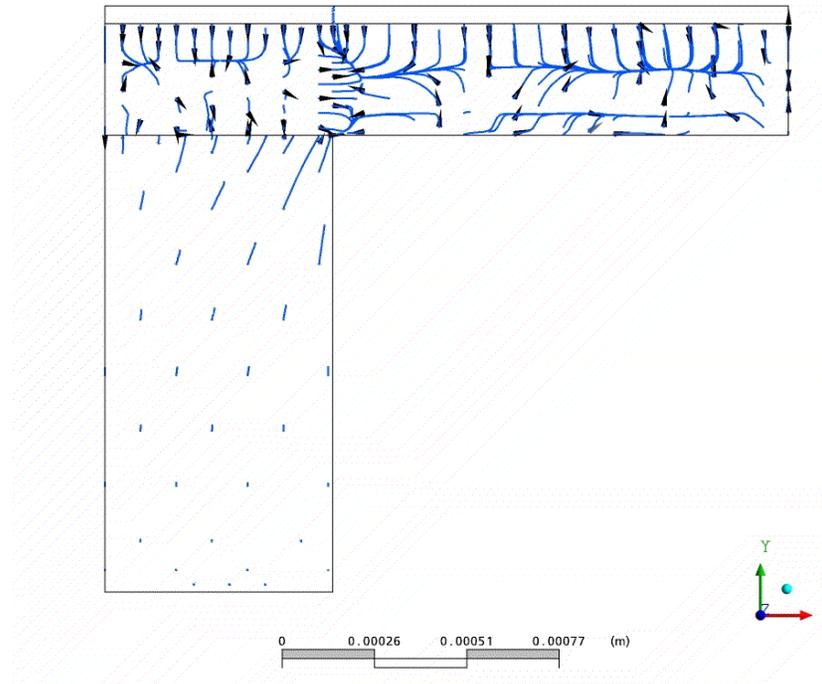
Effect of capillary size in GDL at interface with channel

V. Gurau *et al.*, accepted for publication in *Journal of Fuel Cell Science and Technology*, 2007

Modeling water dynamics and two-phase phenomena in PEMFC (3D) - continued

Study the water dynamics in the fuel cell components

CFX



Dynamics of liquid water stream-lines
in GDL and catalyst layer
(play animation)

Modeling water dynamics and two-phase phenomena in PEMFC (3D) - continued

Study the electro-osmotic drag of water out of the ionomer by the secondary current:

- Phenomenon identified and quantified solely by means of mathematical analysis;

$$S_{\lambda(\text{drag secondary current})} = -n_{\text{drag}} \frac{(a \cdot i_0)_{\text{cathode}}}{F}$$

- Is the dominant mechanism of water transfer between ionomer and catalyst layer pores (sorption/desorption is slow, being diffusion controlled)
- Water content in ionomer (●) may be lower than initially predicted when this phenomenon was ignored
- Additional experimental evidence needed to substantiate it; Demonstrate it with apparatus built at LANL

Modeling water dynamics and two-phase phenomena in PEMFC (3D) - continued

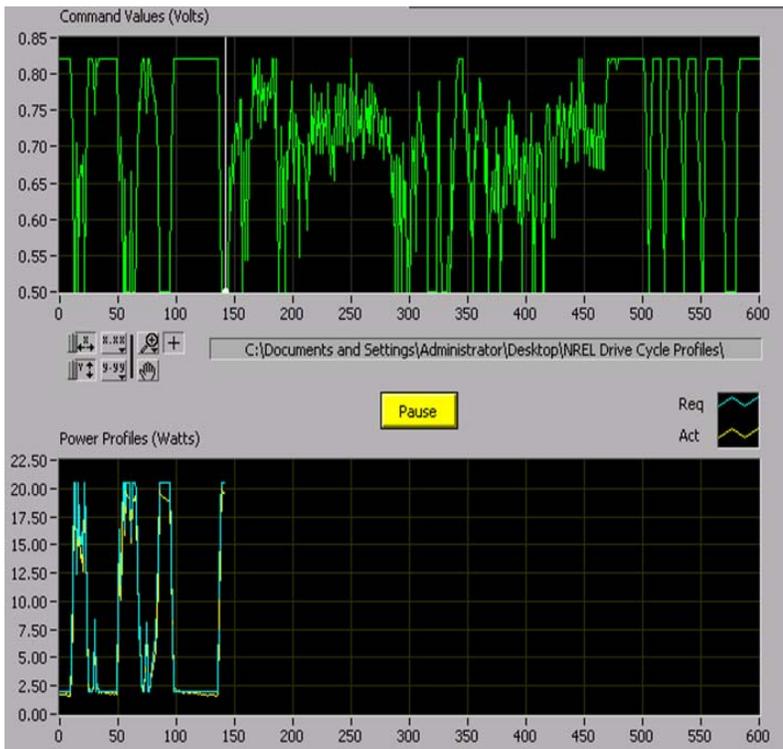
Study two-phase equilibrium at channel-GDL interface

- Equilibrium influences the amount of water contained in GDL
- Currently the model incorporates Tate's law as condition for droplet detachment;
- Needs to implement the more advance model developed at Sandia

RH Transient Tracking Testing

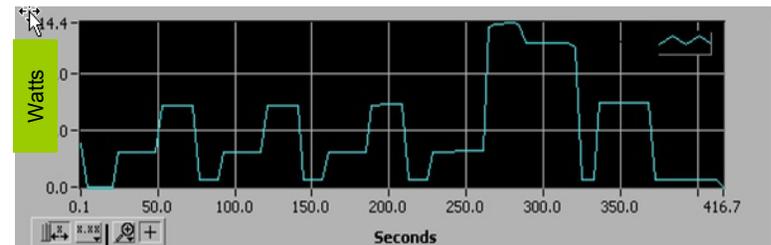
- Realistic drive cycle operation will include:
 - Relative humidity, Temperature, Pressure as a function of power level during drive cycle
 - Shut-down/Start-up

Volts

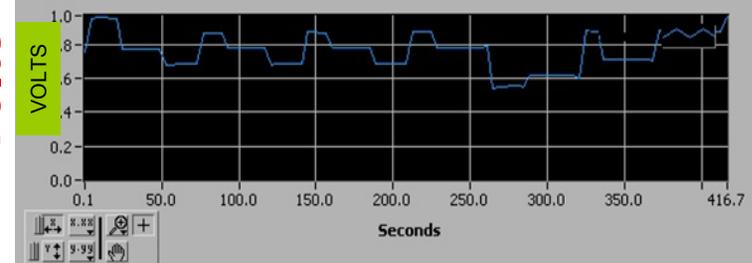


20 min

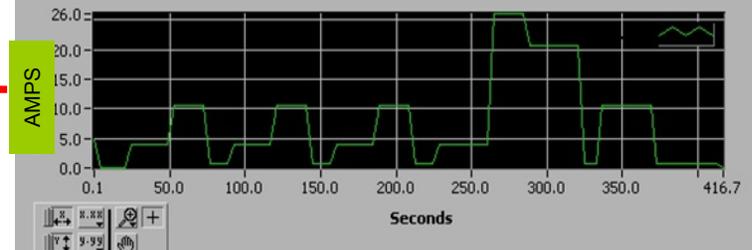
Watts



Volts



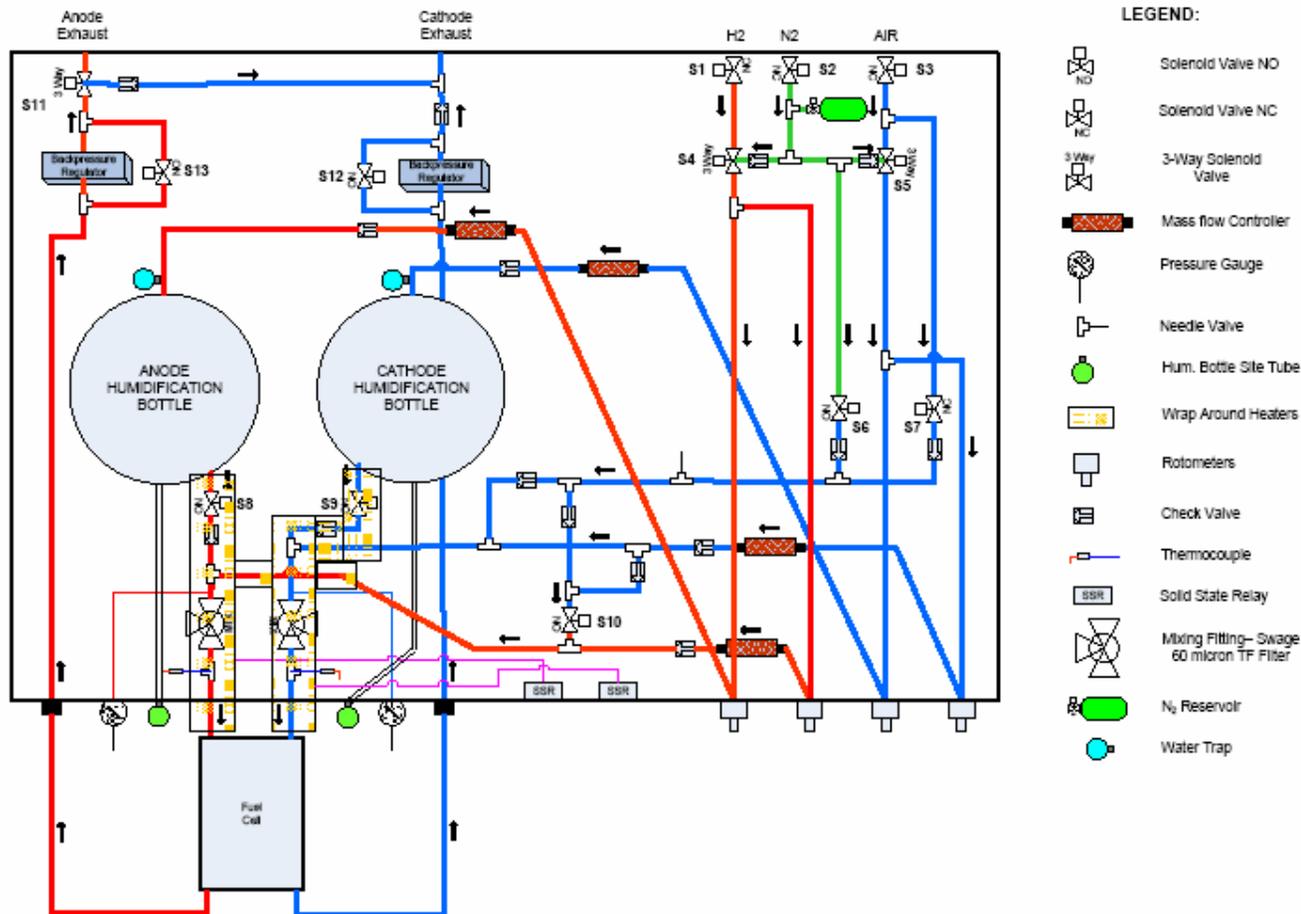
Amps



6 min

RH Transient Tracking Testing

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



Use multiple MFCs for anode/cathode with/without humidification to enable fast inlet humidity transient response

Future Work

- Water Balance Measurements
 - Transient inlet RH measurements
- NIST Neutron Imaging (May 19 – 23)
 - Hydrophillic catalysts
 - Freeze Operation
- Freeze Measurement
 - *in situ* monitoring of ice formation
- Two-phase model development
 - Analyzing flow mal-distribution among PEM fuel cell channels.
 - Sub-model of liquid-water removal due to evaporation at the liquid/gas interface.
 - Develop a multi-dimensional (quasi-3D) model of water transport and removal
 - incorporate sub-models of liquid-water removal via droplet detachment and evaporation

Summary

- Changing mass transport properties during fuel cell operation lead to decreased performance
 - GDL material properties change during aging
 - Mass transport decay correlates to hydrophobicity loss of GDL
 - Fluorine redistributes in GDL during start/stop operation
- Teflon loading in GDL and MPL affects water transport
 - Greater mass transfer resistance for GDL with 23% PTFE in MPL
 - Substrate Teflon content does not have major role in determining water content
- Neutron imaging shows water distribution of flowfield and of MEA cross-section
 - Water build-up in flowfield of both anode and cathode at constant stoichiometric operation
- Freeze/Thaw
 - Significant mass transport problem after 80 FT cycles for paper GDL
- Modeling predicts:
 - More hydrophobic GDL materials reduce the critical velocity required to detach water droplets.
 - Decreasing contact-angle hysteresis (e.g., by reducing GDL surface roughness) enhances droplet removal.