FC-PAD

Fuel Cell – Performance and Durability

FC137 – Electrode Layer Integration

Thrust Coordinator: Shyam S. Kocha

Wednesday, June 8th, 2:15 pm

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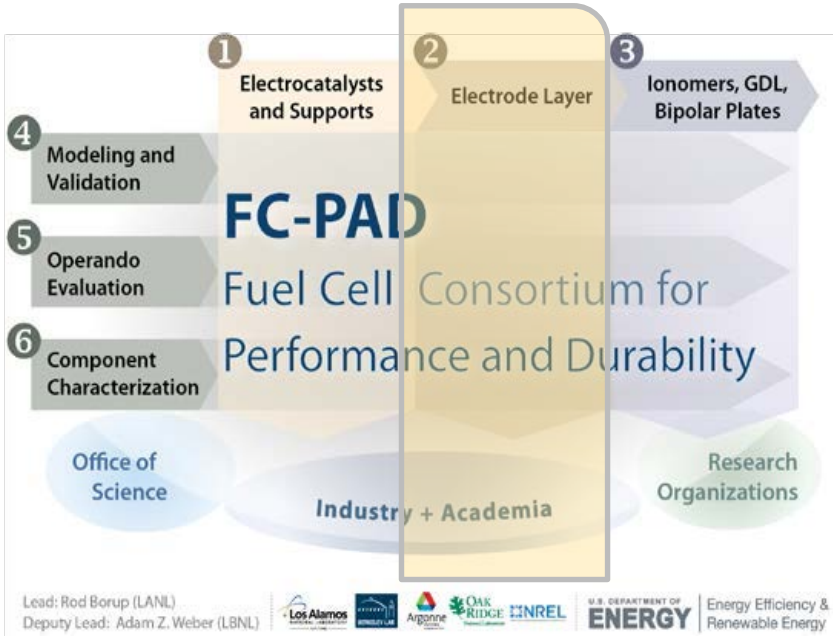
FC-PAD Overview & Electrode Layer Integration



Electrode Layer Integration involves contributions from all three Thrust Areas & cross-cutting competencies

Component Characterization

Operando Evaluation

Modeling & Validation

Ionomer, GDL, Electrocatalyst & Support

Cathode **Electrode Layers**

Membrane

Anode

FC-PAD Electrode Layer Participants

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Yu Seung Kim

OAK RIDGE National Laboratory

Karren More
David Cullen

NREL NATIONAL RENEWABLE ENERGY LABORATORY

Shyam Kocha
KC Neyerlin
Jason Christ
Jason Zack

Electrocatalyst Sources

Commercial & Lab

Umicore
IRD
TKK
NECC

NREL
ANL

Membrane Electrode Assembly Sources

Industry

GM
IRD

University

USC

National Labs

NREL
LANL

FUEL CELL FCPAD PERFORMANCE AND DURABILITY

Overview

Timeline

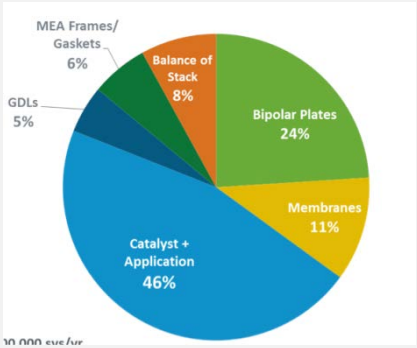
Project start date: 10/1/2015
Project end date: 09/30/2020

Partners

- IRD, New Mexico, USA
- Umicore, Germany
- NECC, Japan
- GM, USA
- Tanaka Kikinzoku Kogyo (TKK), Japan
- Partners to be added by DOE DE-FOA-0001412

Barriers

- The electrocatalyst remains a challenge for reducing the **cost** to meet system cost targets



- **Catalyst Ink formulation** is still a black art
- The **catalyst layer** is not fully under-stood and **is key in lowering costs** by meeting rated power. Rated power@ low Pt loadings reveals unexpected losses
- **Durability** targets have not been met

Relevance - Objectives

The primary objective of this consortium is to advance performance and durability of polymer electrolyte membrane fuel cells (PEMFCs).

- Improvements in component stability and durability
- Improvements in cell performance due to optimized transport
- Development of new diagnostics, characterization tools, and models
- Develop new capabilities (such as advanced diagnostic tools or models) to aid developers, advance knowledge of component properties, and develop advanced structures, strategies, and methods to achieve these objectives
- As a resource to DOE and industrial developers, the consortium will provide technical capabilities to future projects focusing on performance and durability of PEMFCs

Expected Outcome

PEMFC MEAs and components that demonstrate world-class performance and durability, meeting and exceeding the consortium 2020 targets. The major durability targets include 5000 hours of operation under simulated vehicle power cycling and shut-down/start-up cycling with < 10% loss in rated power. In terms of performance, the key targets are meeting efficiency, power, startup time and energy, and related metrics within the cost and durability constraints, specifically developing MEAs with SOA catalysts that demonstrate performance > 1W/cm^2 with Pt loading < 0.125 mg/cm^2.

Approach

Approach & Overview of Thrust Specific Objectives

1. Identify sources for SOA electrocatalysts that meet or exceed the DOE mass activity targets of 440 mA/mg_{Pt}
2. Integrate SOA electrocatalysts that meet or exceed the DOE mass activity targets of 440 mA/mg_{Pt} and optimize the catalyst layer to attain the DOE peak power density requirements of $1W/cm^2$ & 0.125 g_{Pt}/kW while simultaneously meeting durability targets.
3. Identify the source(s) of the unanticipated substantial performance losses observed at loading below 0.1 mg_{Pt}/cm^2 using existing and novel diagnostic techniques. Ascertain the proportion of losses that can be attributed to transport limitations and kinetics.
4. Mitigate the losses due to transport limitations in the catalyst layer by developing/fabricating new electrode layer structures that, for e.g., have two phases for proton transport and explore alternative ionomers and pore morphology. Model novel electrode designs and diagnostics.

MEA Materials Specifications, Selection and Optimization

Electrocatalysts

Commercial Sources

- IRD:
  - IRD CAT0023, 55wt% PtCo/C ✓
- Umicore:
  - Elyst: Pt50 0550; 45.9wt% Pt, 5.5 nm XRD ✓
  - Elyst P30 0670; 27.5 wt% Pt; 3 wt% Co, 4.2 nm XRD ✓
- NEChemcat:
  - PtCo/NE-GM ✓
  - Core-shell Pt ML/Pd/NE-H ✓
- TKK:
  - TEC10E50E, Pt/HSC, 47.5wt% Pt, 2.5 nm XRD ✓
- 3M NSTF ✓

National Lab Sources

- NREL ETFECS ✓
- ANL Frame ✓

Automotive Source

- General Motors
  - Proprietary ✓

Academic Source

- USC Pt/ACCC ✓

Ionomers

Commercial Sources

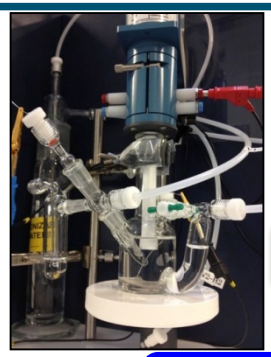
- Ion Power ✓
  - Nafion-D2020
- 3M
  - Proprietary ✓

Fabrication Techniques

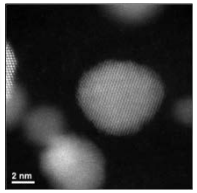
- Slot Die
- Spray Coating
- Sputtering
  - Ionomer Free
- Electrospinning
- Stratification
- Carbon Dilution

Materials under examination by FC-PAD at this time.

MEA Evaluation & Optimization Process



TEM



Electrocatalyst

RDE

XRD

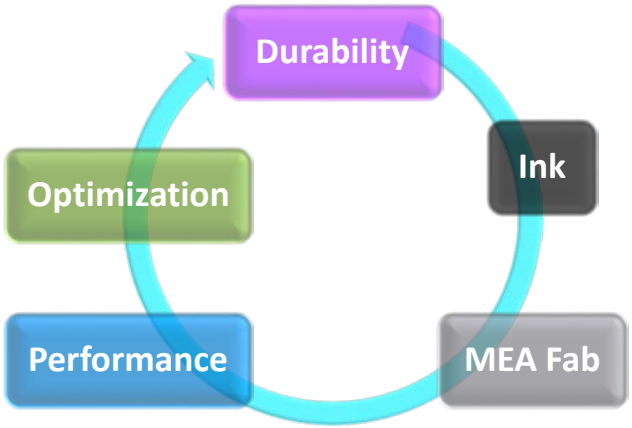
Modeling

- Diagnostics
- Performance
- Microscopy
- Durability

- Optimization
- Special diagnostics
- Durability

Catalysts

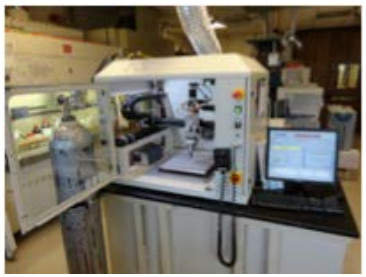
Sonication



GO

NOGO

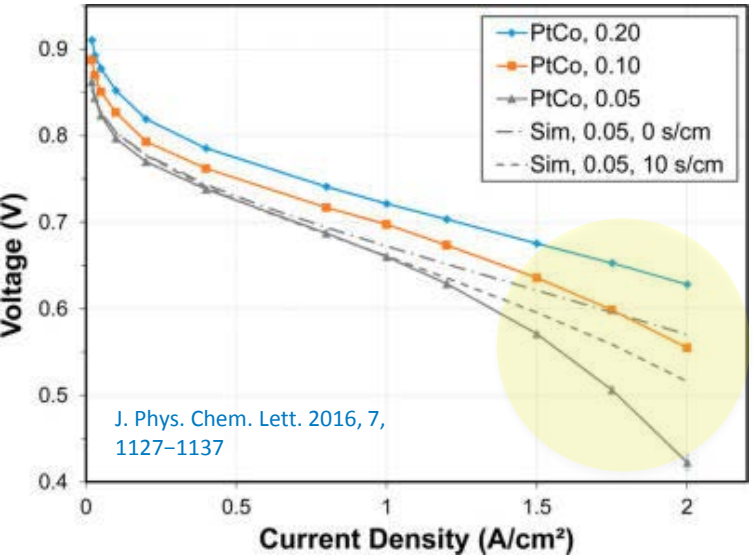
Performance ← ORR ← ECA

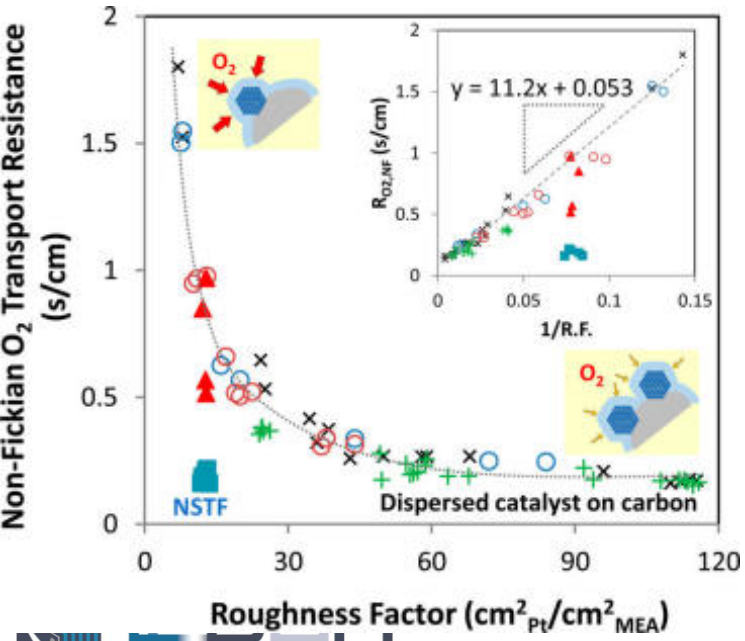


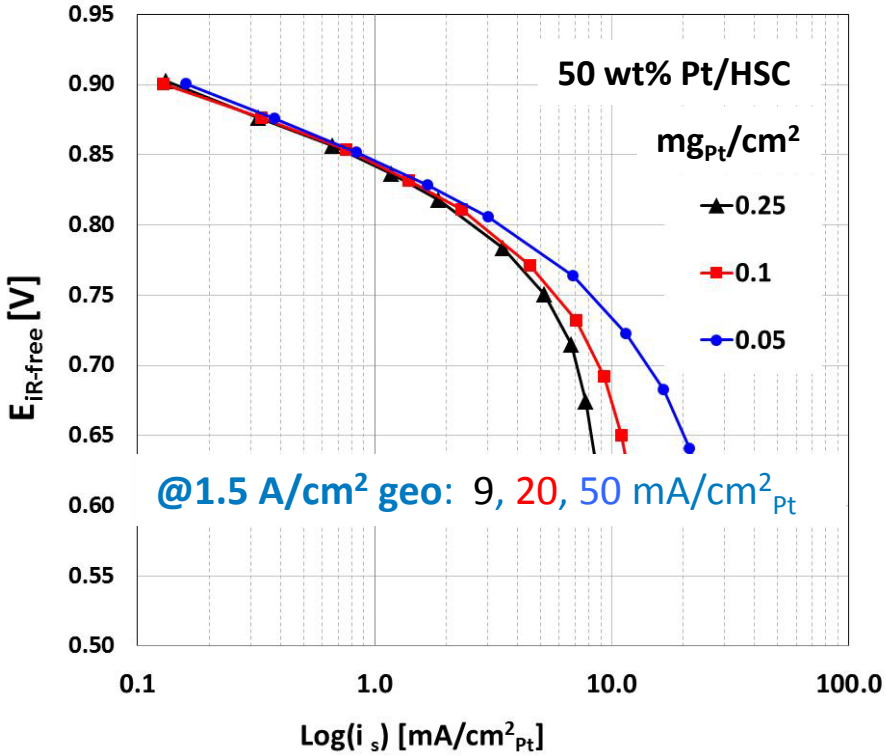
Catalyst Ink MEA-Fab

FUEL CELL FCPAD PERFORMANCE AND DURABILITY

Relevance-Objective: Impact of Low Pt Loadings



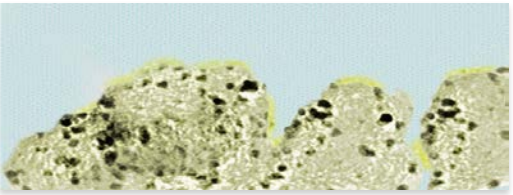




At low loadings the current density per catalyst site is higher. Purported increase in so termed local Pt resistance ($R_{O2,local}$).

Ionomer adsorption/blocking and thickness as well as low ECA are possible causes of losses observed in performance at low loadings

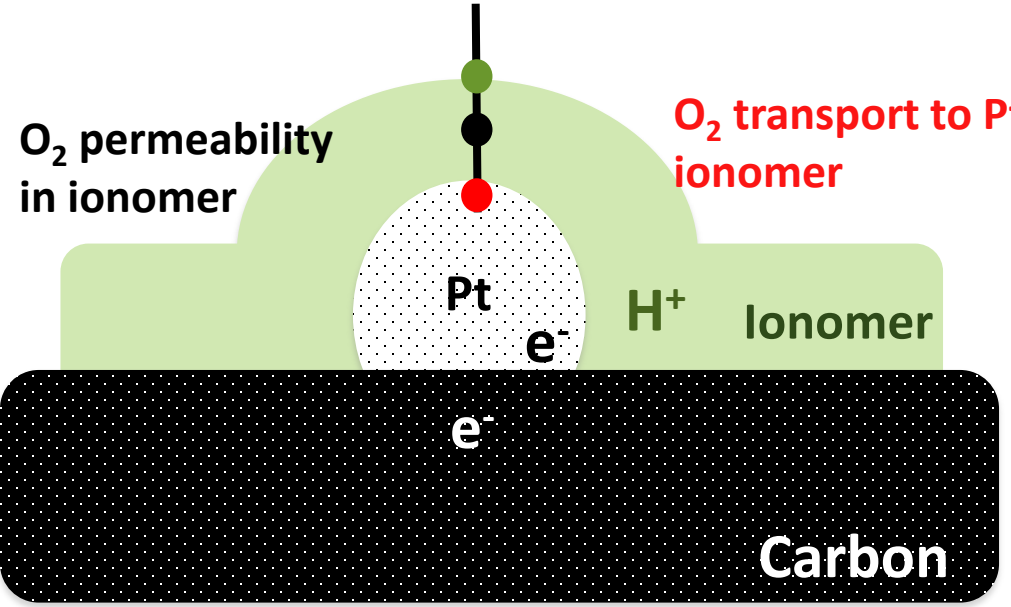
Sources of Additional Losses : RO_2



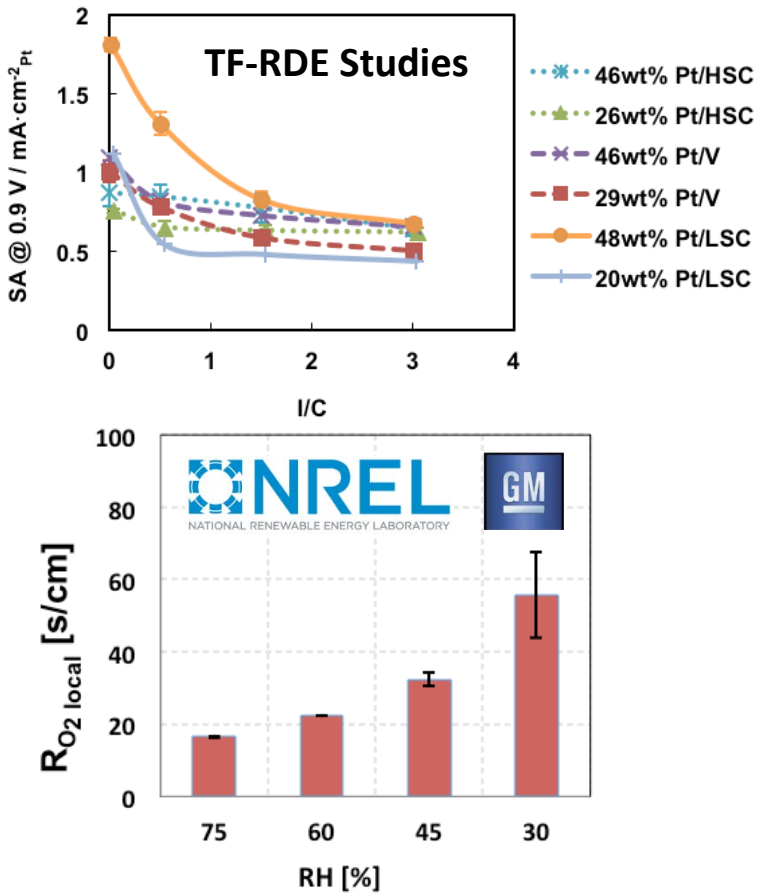
Modified TEM
of Pt/C w ionomer

O_2 dissolution into ionomer

Interfacial transport resistance



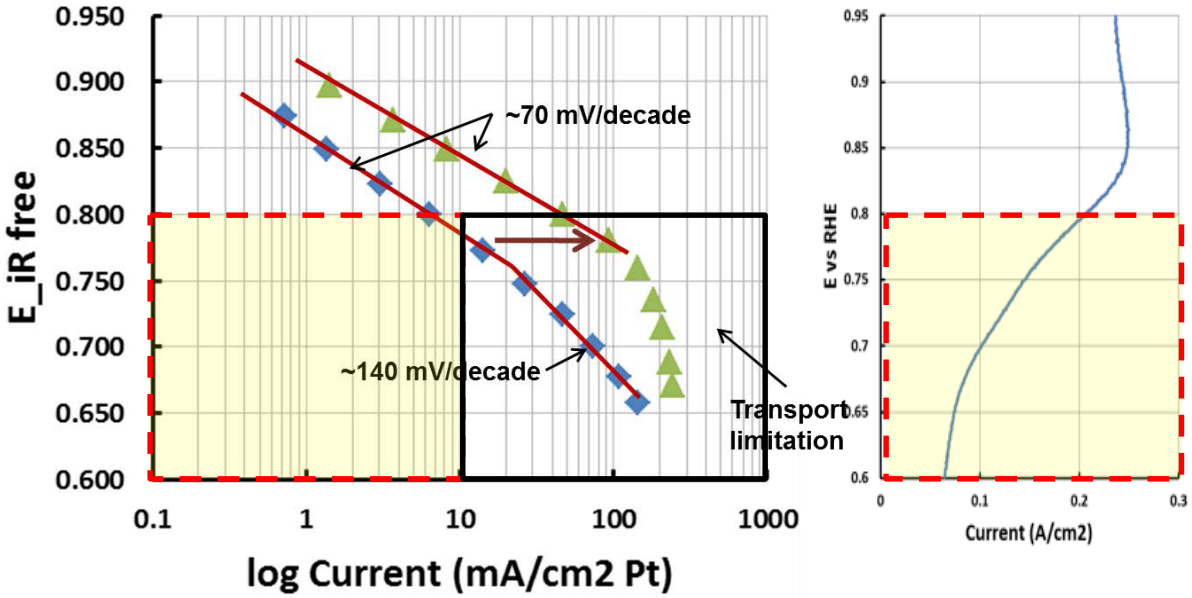
Ionomer blocking/poisoning



Multiple sources of losses have been hypothesized. Some insights into the effect of ionomer and RH have been recently identified.

Relevance-Objective: The Role of Kinetics?

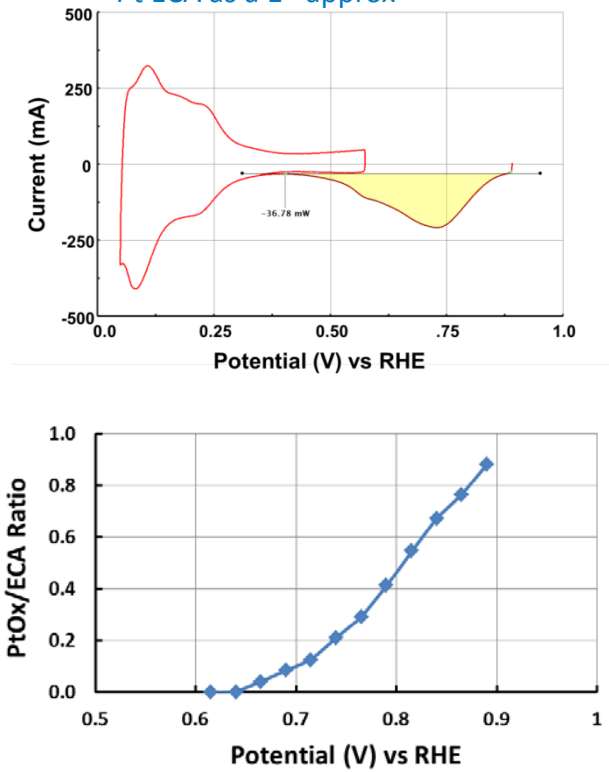
0.045 mg/cm^2, 150 kPa, 80^{o}C, 100% RH



Potential range of interest for oxide dependent kinetic measurements

Current range affected by $R_{O2,local}$

PtOx Measurements normalized to Pt ECA as a 1st approx

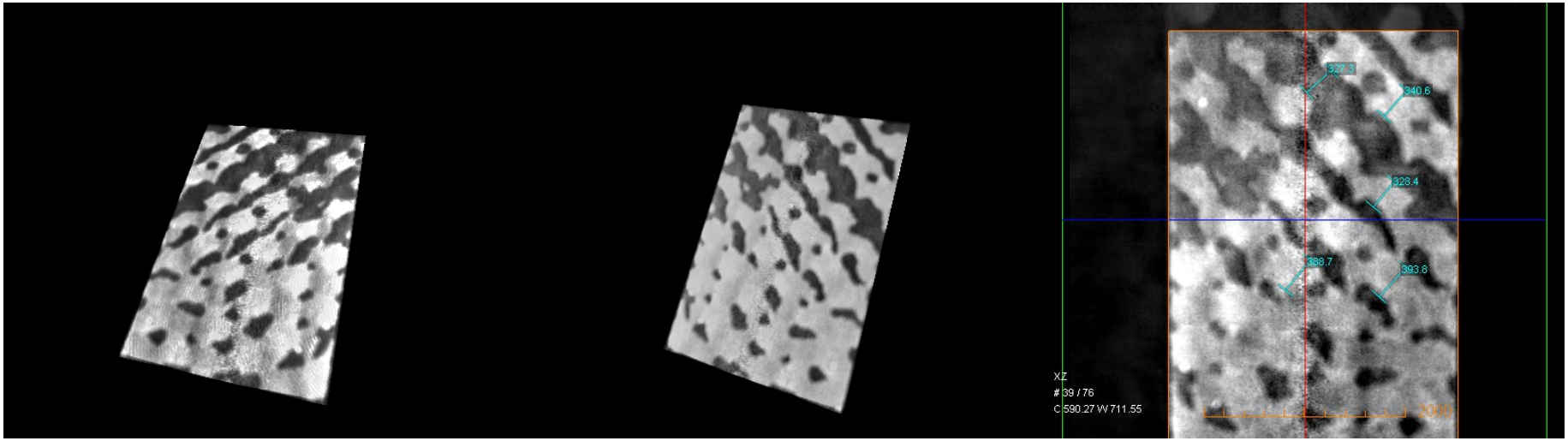


Subramanian, N. P., et al. *Journal of The Electrochemical Society* 159.5 (2012): B531-B540.

Kinetic losses, if any, need to be accounted for, prior to attributing the residual losses to transport related phenomena.

Relevance-Objective: Electrode Design

Stratified Electrode Structures for Improved High Current Performance

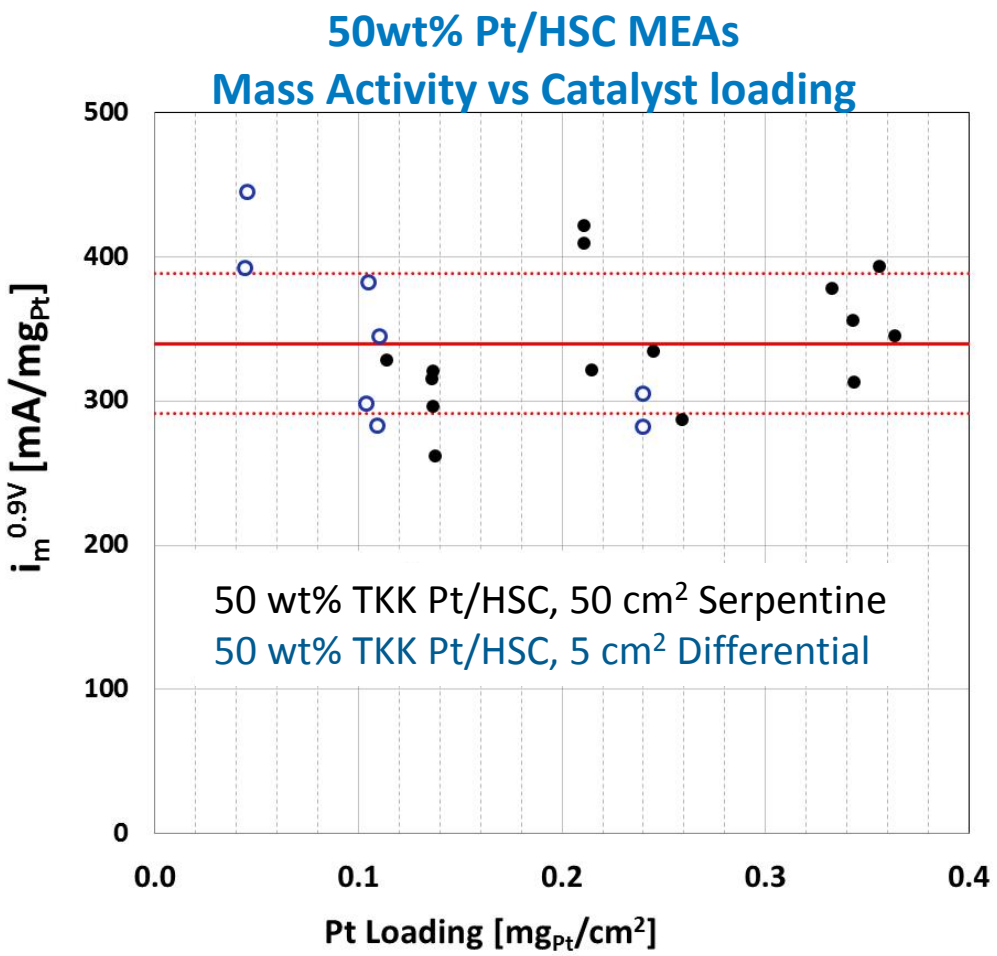


Sample width is 2.8 mm (entire width visible, X-ray tomography taken at low magnification to capture the features, ~0.3 to 0.4 mm in size

Irregular catalyst layer thickness can lead to enhanced gas and water transport in and out of the catalyst layer, respectively. The stratified structure is expected to have the same performance in the kinetic region where the performance is controlled by the overall Pt loading. However at high current densities, the thinner sections of the stratified catalyst layers should allow for better mass transport properties.

Accomplishments

Baseline ORR Activity

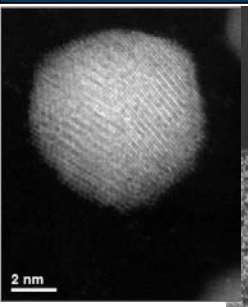


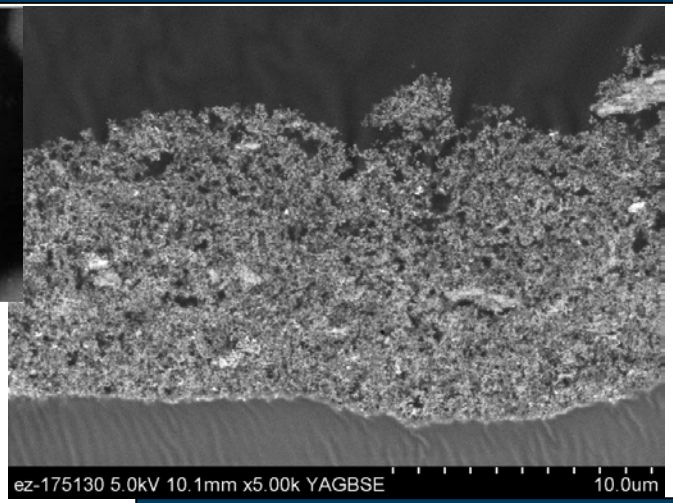
Hardware
Active Area 5, 50 cm^2
Triple Serpentine FF
Spray Coated CCMs
Operating Conditions
0.90 V
80^{o}C
100 kPa PO_2
Stoic~9.0
100 %RH
Protocol
Anodic Sweep
5 mins/point
# of Samples 20

Baseline TKK TEC10E50E Pt/C at various loadings conducted using FC-PAD protocols and operating conditions as well as hardware.

Electrocatalysts and Electrode Layers

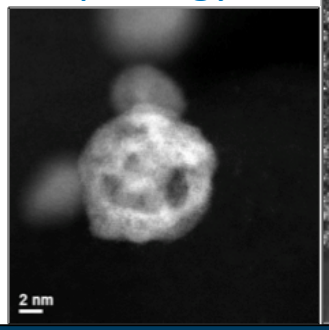
Umicore PtCo/C Median Pt-Co particle size of ~3.7nm – FCPAD Fabricated MEA

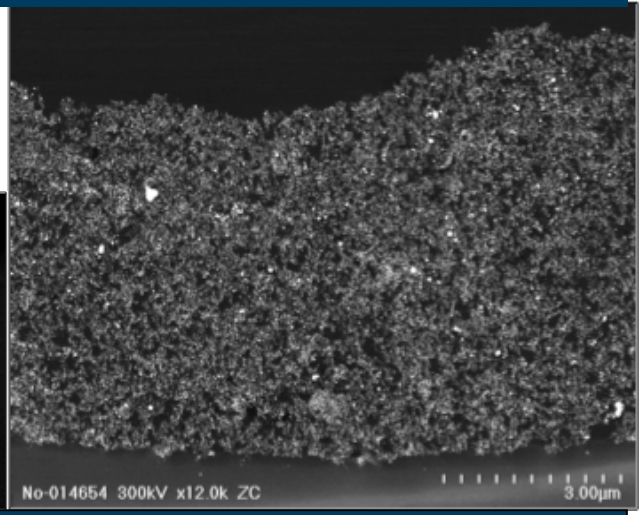




IRD Fabricated PtCo/C CCM
Median Pt-Co particle size of ~5–6nm

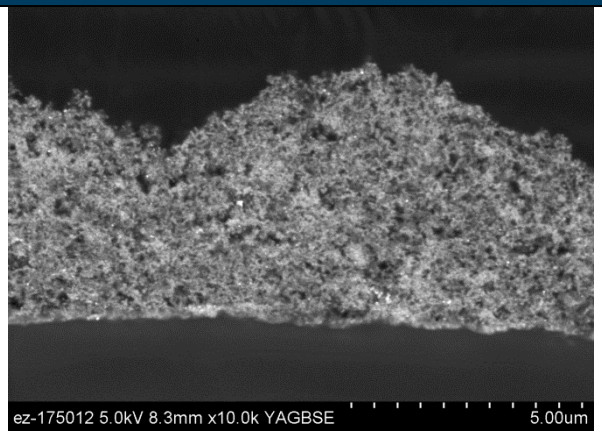
"spongy" Pt_3Co morphology

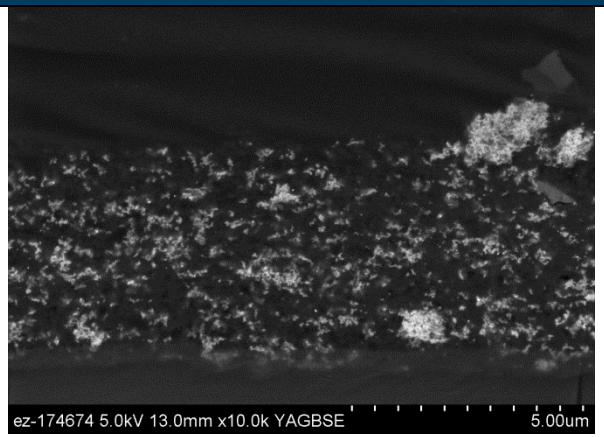




TKK Pt/HSC – FCPAD Fabricated MEA

Undiluted-0.2 mg_{Pt}/cm^2 Carbon diluted 0.05 mg_{Pt}/cm^2

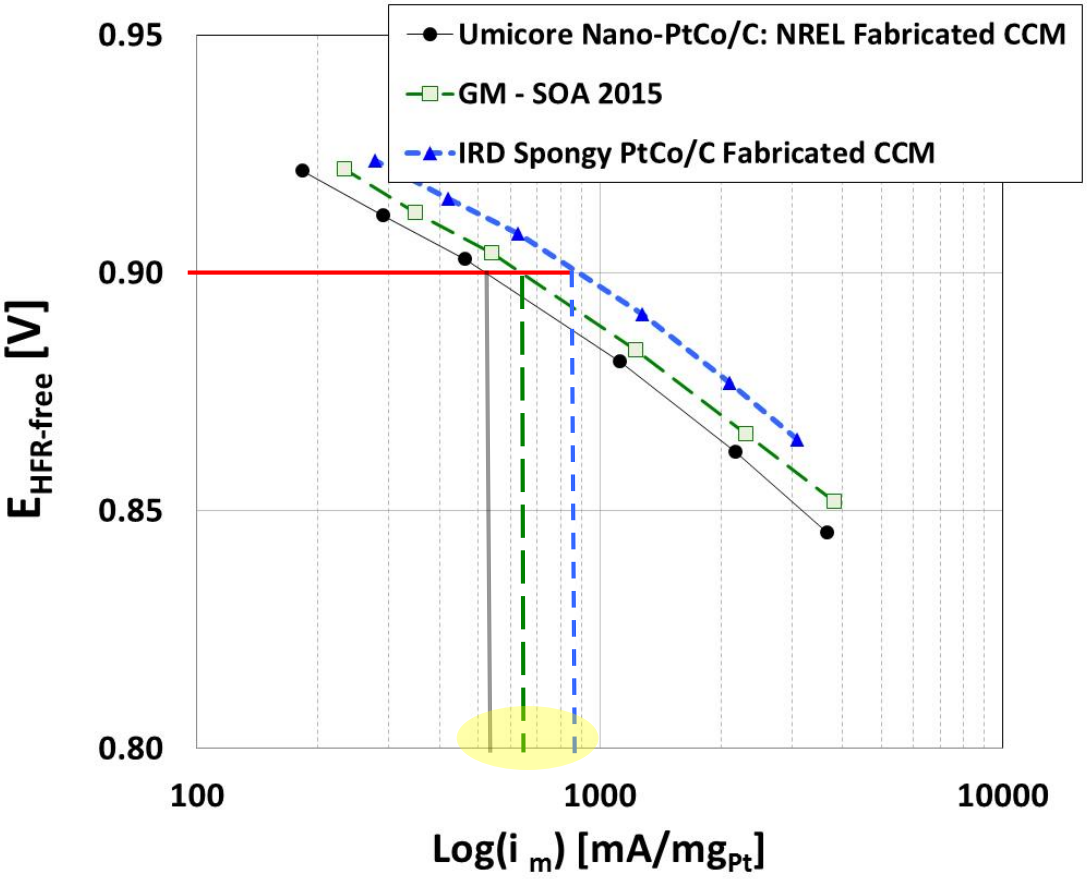




FUEL CELL FCPAD PERFORMANCE AND DURABILITY

MEA: ORR Activity of SOA Catalysts

ORR MA Tafel Plots; H_2, O_2 150 kPa, 80°C, 100% RH, S=2/9



ORR Activity @ 0.90 V

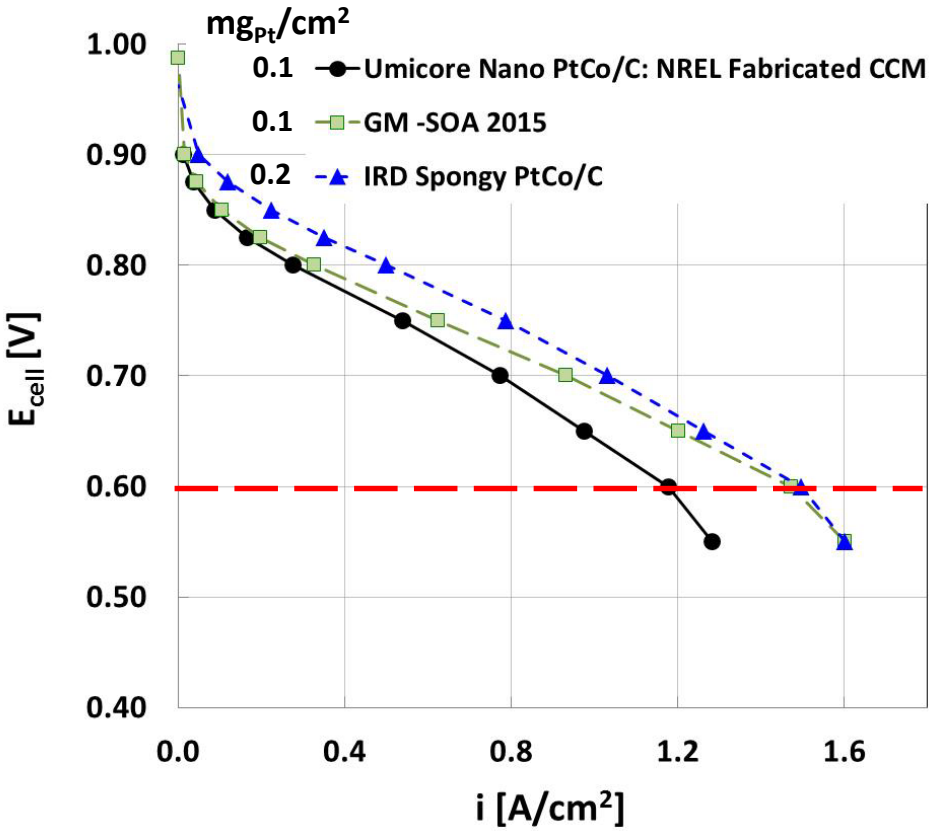
	MA	SA	ECA
Umicore	514 ±40	1406 ±135	37 ±2
GM	620 ±60	1440 ±130	43 ±1
IRD	820 ±20	2000 ±6	41 ±1

MA = mA/mg_{Pt} ; SA = $\mu A/cm^2_{Pt}$ ECA = m^2/g_{Pt}

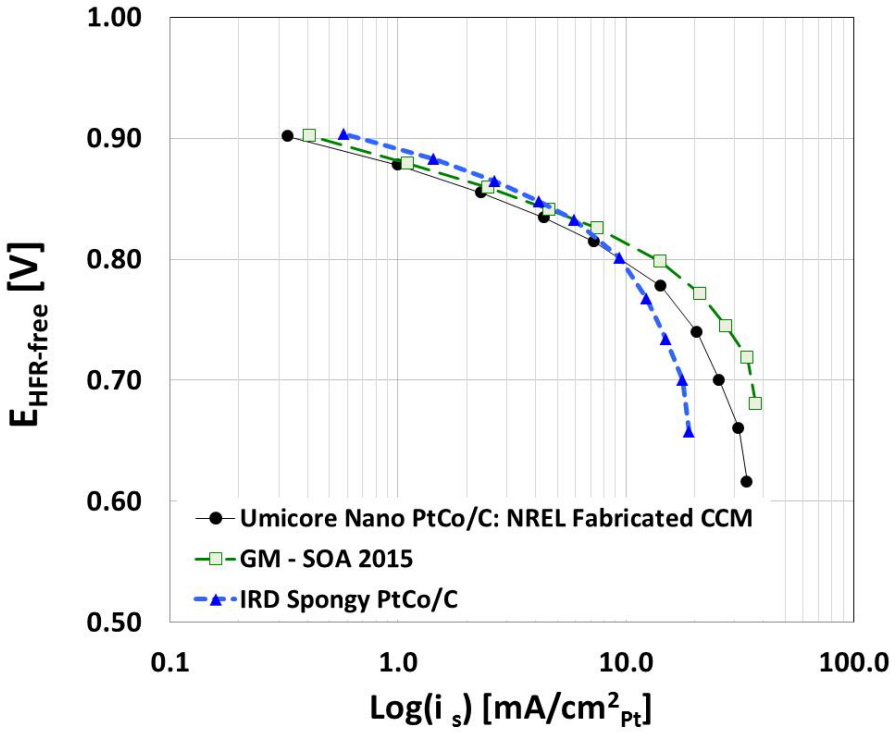
ORR mass activity of all three SOA catalysts/MEAs >440 mA/mg_{Pt}.

MEA: H_2-Air Performance of SOA Catalysts

Wet H_2-Air I-V Curves, 80°C, 150 kPa, 100% RH



E_{cell} vs. I (A/cm^2_{geo})

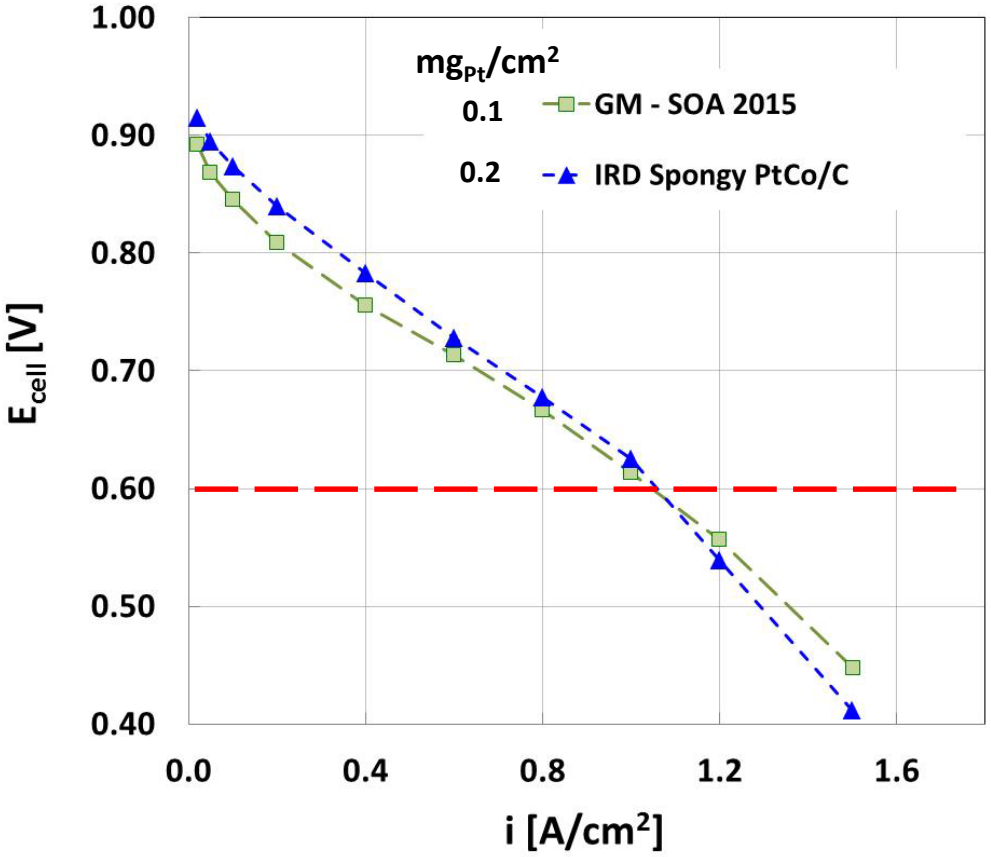


$E_{HFR\text{-}free}$ vs. I (mA/cm^2_{Pt})

At E_{cell} = 0.60V, the three MEAs have current densities ~ 1.2–1.42 A/cm²

MEA: H_2-Air Performance of SOA Catalysts

Dry H_2-Air I-V Curves, 80°C, 150 kPa, 42% RH



We note that the Target performance of 1 W/cm² at rated power does not define *RH or T, but is limited by having to meet the* $Q/\Delta T \leq 1.45$ *constraint*.

At 0.60V, the MEAs have current densities ~ 1 A/cm²

MEA: Performance Summary vs. Targets

US DOE Targets

	A:C (mg/cm^2_{Pt})	MA @ 0.9V (mA/mg_{Pt})	I@0.60 V (A/cm^2)	P@0.6V (W/cm^2)	g_{Pt}/kW_r
SOA DOE	0.05:0.20	~300	–	–	0.25
2020 DOE Target	0.025:0.1	>440	~1.67	1.0	0.125

Anode loading [mg/cm^2_{Pt}]
0.05 0.025

H_2/Air 150 kPa, 100% RH 80°C

	MA (mA/mg_{Pt})	I A/cm^2	P W/cm^2	SOA g_{Pt}/kW_r	2020 g_{Pt}/kW_r
Umicore	514	1.18	0.71	0.21	0.18
GM	620	1.47	0.88	0.17	0.14
IRD	820	1.50	0.90	0.29	0.26

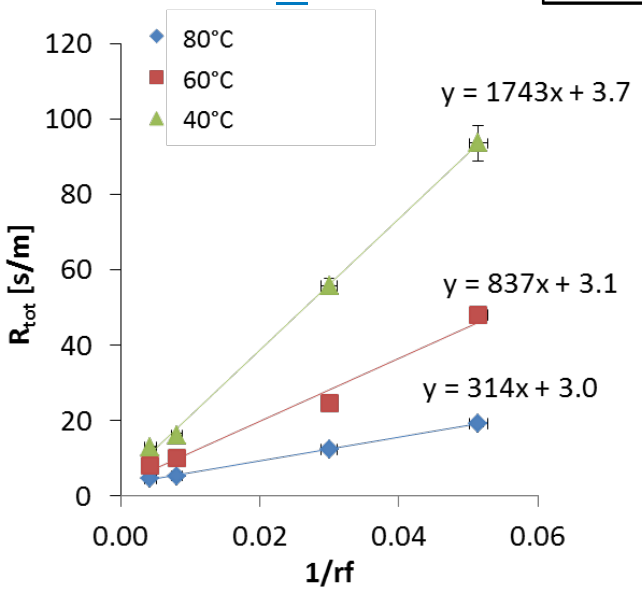
DOE ORR activity (0.90V) targets have been met; rated power still unmet.

HCD Diagnostics – Elucidation of $R_{O2,local}$

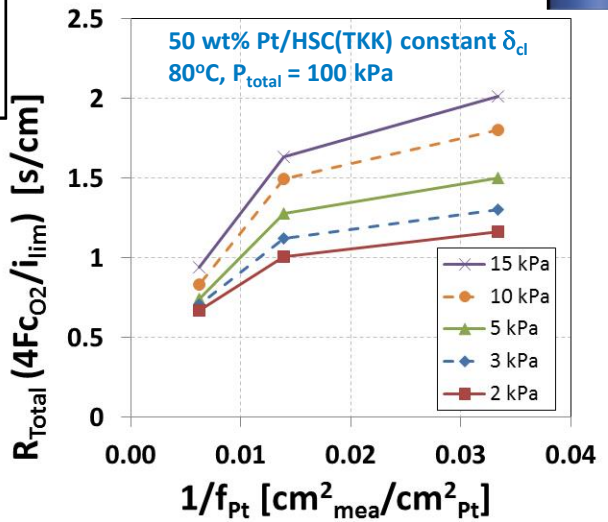
$$R_{tot} \approx n * R_{GDL} + R_{cf} + \frac{1}{rf} * R_{cl,A_{Pt}}$$

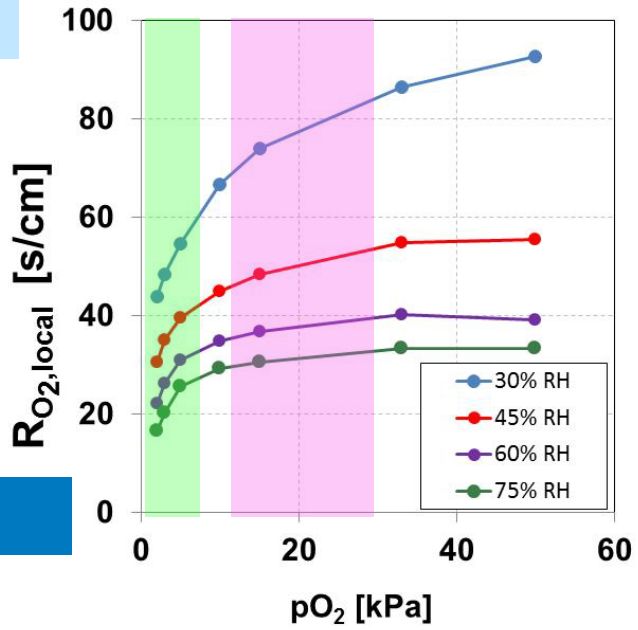
Greszler, et al. *Journal of The Electrochemical Society* 159.12(2012): F831-F840.

LBNL H_2 pump



- $R_{O2,local}$ increases with decreasing RH
- $R_{O2,local}$ increases with increasing pO_2 (i.e. water production)



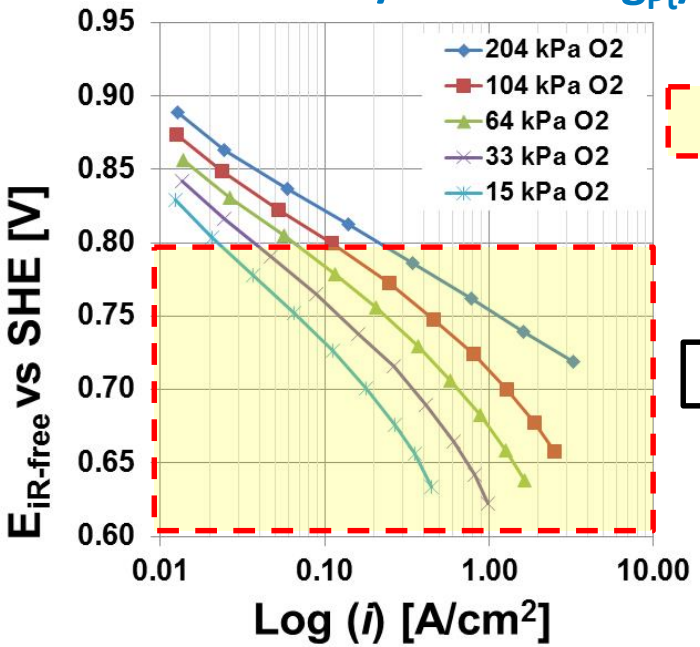


- Impact of temperature is significant (3-17 s/cm from 80-40°C)
- Hydrogen resistances ~1/10th those measured with oxygen at comparable conditions
- Suggests importance of O_2 kinetics & water production

Ionomer effects can be separated from ORR kinetics

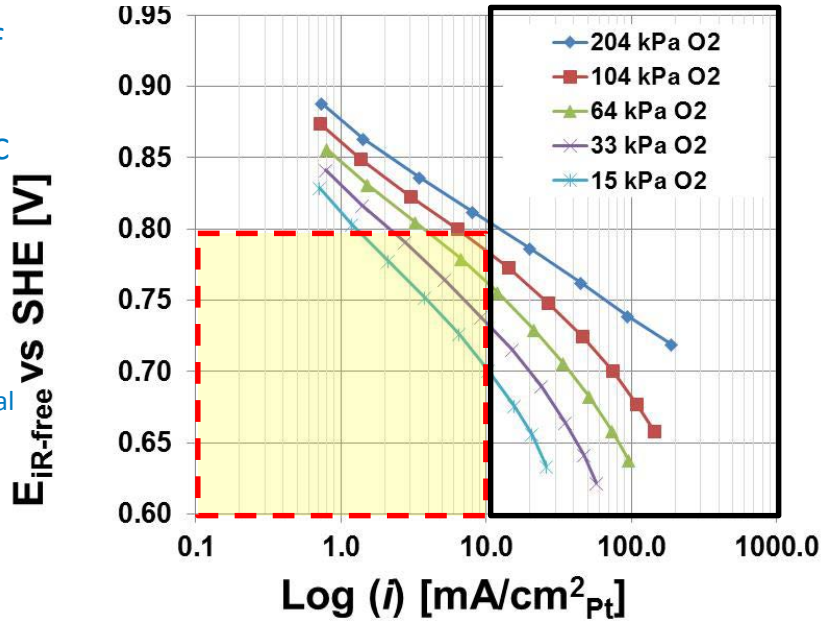
Oxide Dependent Pt Kinetics

Pt/Vu 0.05 mg_{Pt}/cm^2, H_2/O_2 80°C, 100% RH, 150kPa



Potential range of interest for oxide dependent kinetic measurements

Current range affected by $R_{O2,local}$



Requires vacuum system to lower reactant pressure/potential to acquire data in region of interest

Becomes even more difficult to access prior to onset of $R_{O2,local}$ as mass activity increases

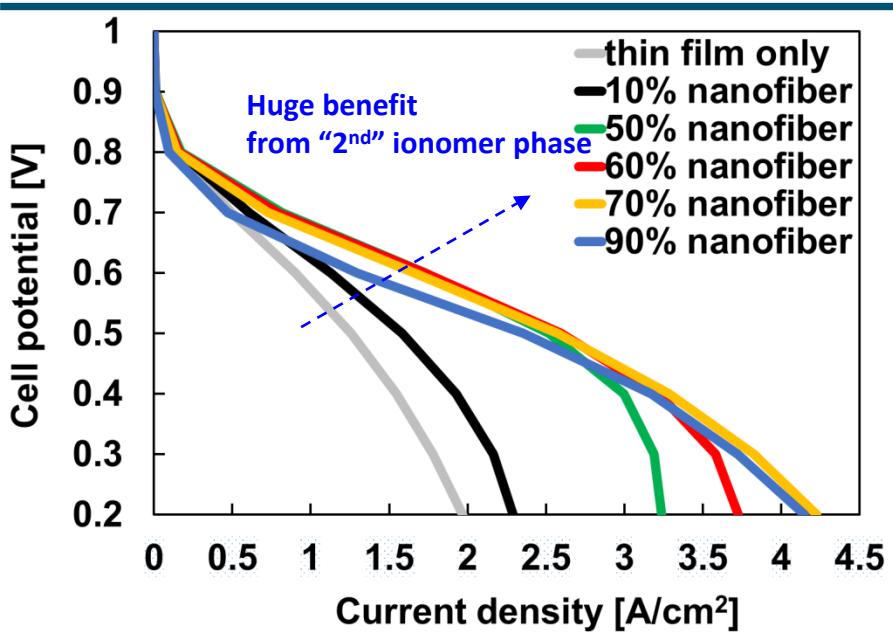
$$i = i_0 \left(\frac{p_{o2}}{p_{o2,\mathrm{ref}}} \right)^{\gamma} (1-\theta) \exp\left(\frac{-\alpha F \eta}{RT} \right) \exp\left(-\frac{\omega\theta}{RT} \right)$$

	Pt Loading [mg_{Pt}/cm^2]	γ	$i_{o,s}$ [A/cm^2_{Pt}]	ω	$i_m^{0.9V}$ [mA/mg_{Pt}]
oxide kinetics - Pt/Vu (lit*)	0.06	0.7	3.0E-05	3000	125
oxide kinetics - Pt/Vu (NREL[a])	0.046	0.62	2.3E-05	3733	109
oxide kinetics - Pt/HSC (NREL[a])	0.045	0.53	6.4E-05	4003	295

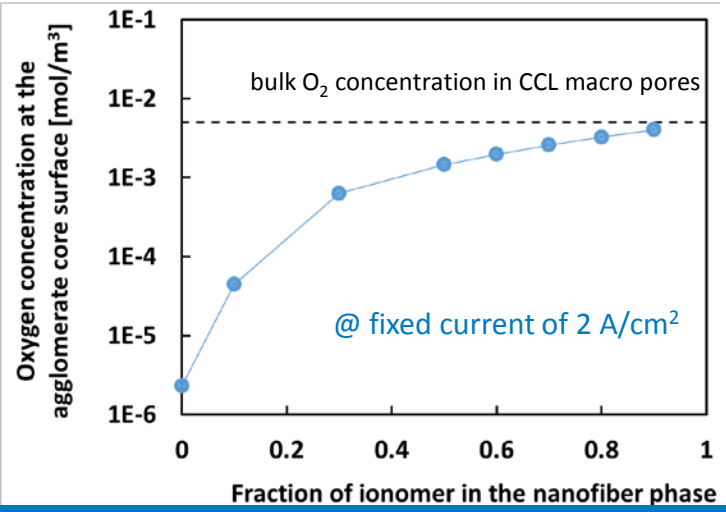
*Subramanian, N. P., et al. *JECS* 159.5 (2012): B531-B540.

Will attempt to apply to Pt-alloys going forward

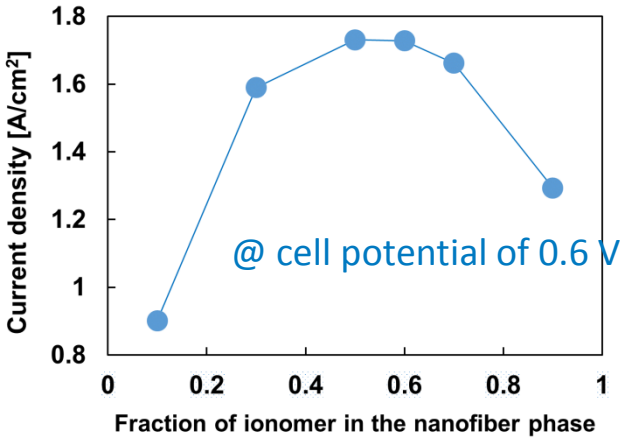
Thin film/Nanofiber Catalyst Layer Model



- 70% nanofiber (30% film) attains a maximum limiting current (with an optimal film thickness of 1.5 nm = 5 nm * 30%). At a higher fraction of nanofiber, the limiting step switches from O_2 diffusion to H^+ conduction in the thin film.
- At 0.6 V, a maximum current of 1.7 A/cm² is obtained from the case of 50% nanofiber (50% film).



- Oxygen diffusion through the film is slow for low nanofiber fractions due to small O_2 concentration at the agglomerate core surface.
- O_2 concentration at the catalyst/film interface approaches its bulk value for high nanofiber fractions.

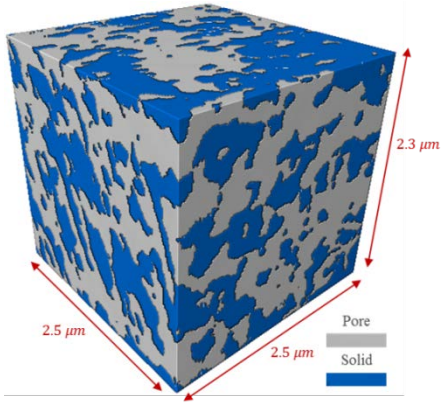


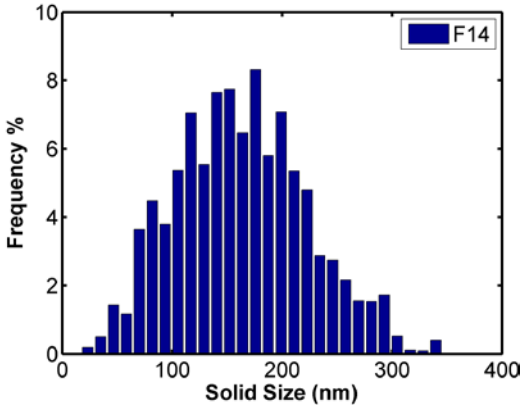
Nanofiber fraction	Rate-liming transport
0% (thin film only)	O_2 diffusion through film
10%	O_2 diffusion through film
50%	O_2 diffusion through film
60%	O_2 diffusion through film
70%	H^+ conduction in film
90%	H^+ conduction in film

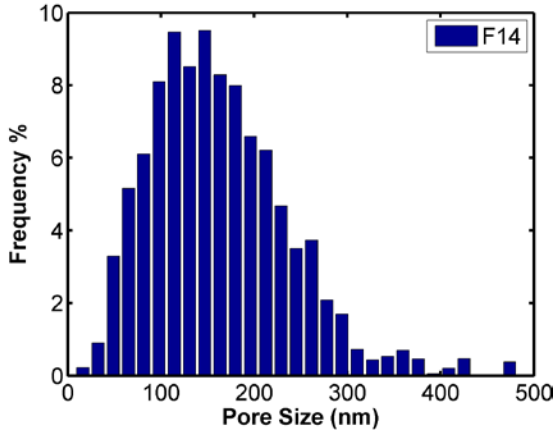
Optimization of the distribution of the two ionomer phases critical

Microstructure Model

1. X-ray tomography for solid and macro pore size distribution and connectivity

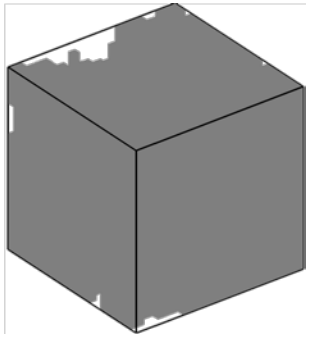




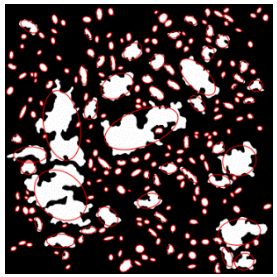


2. Numerical reconstruction algorithm of the electrode structure using XCT and TEM/porosimetry data

XCT Solid

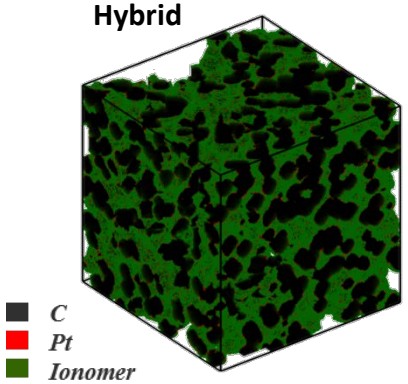


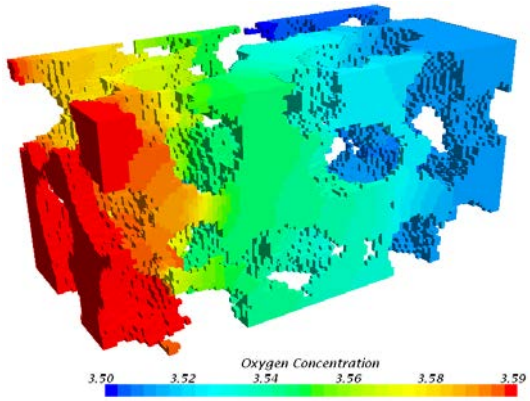
TEM



3. Multi-physics model of H^+, e^-, and O_2, transport in carbon, ionomer and pore phases in the electrode microstructure

Hybrid





4. Next steps: Complete reconstructions of all XCT data. Correlate electrode structure with performance. Attempt to distinguish ionomer phase from carbon. Model liquid water movement.

Microstructure model development with input from experiments is ongoing

Collaborations

Institutions	Role
FC-PAD Consortium	ANL, LBNL, ORNL, LANL, NREL
Umicore	Supply SOA catalysts and MEAs for evaluation
IRD	Supply SOA catalysts and MEAs for evaluation
GM	Supply SOA MEAs for evaluation
TKK	Supply catalysts for evaluation
NEChemCat	Supply SOA catalysts and/or MEAs for evaluation

Summary

- **Relevance:** Electrode layers optimization with mitigation of transport issues at rated power are vital to meet 2020 DOE targets.

- **Approach:** Our approach involves identifying SOA catalysts, optimizing them in catalyst layers, developing diagnostics to help resolve the high current density/low loading problem and mitigating the problem through the use of novel electrode design, novel components, novel diagnostics techniques all complemented with modeling.

- **Accomplishments and Progress:** All 3 SOA catalyst layers evaluated have met the DOE MA target of 440 mA/mgPt. Progress has been made on understanding transport through the layer using diagnostic tools and modeling.

Proposed Future Work

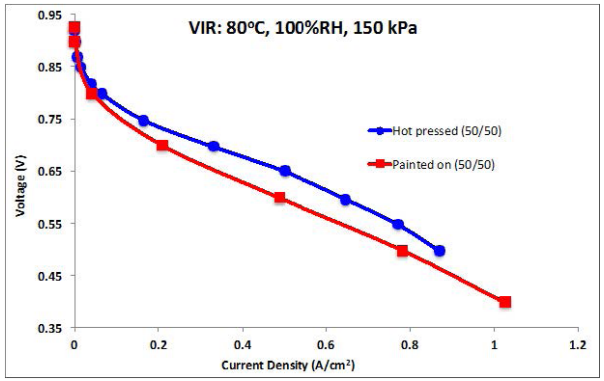
- **Plans for the remainder of FY16**
  - MEA screening of remaining SOA catalyst materials already identified
    - Optimize catalyst layer to achieve peak BOL performance for promising candidates
    - Implement alternative designs for cathode catalyst layer
- **Plans for FY 17**
    - Confirm whether kinetics actually comes into play at high current densities
    - Identify and implement alternative ionomers in catalyst layers to examine effects on performance
    - Model performance diagnostics data at high current densities
    - Identify alternative designs for cathode catalyst layer that enhance performance and durability
    - Conduct durability studies/ASTs on catalyst CCM that meet DOE target of performance.

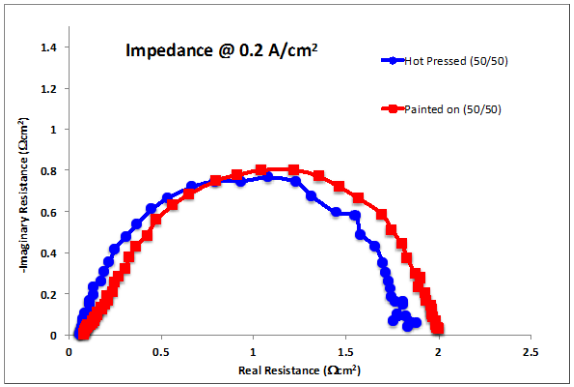
END

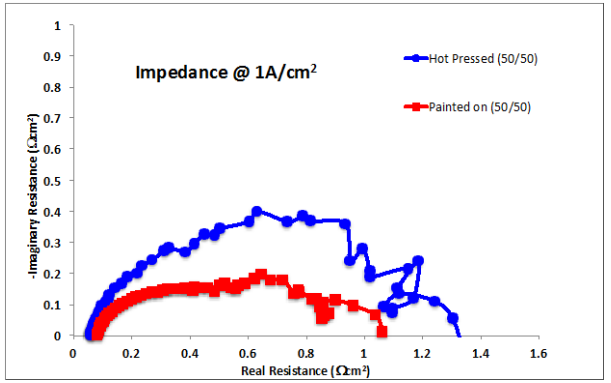
Supplemental Slides

Electrode Design

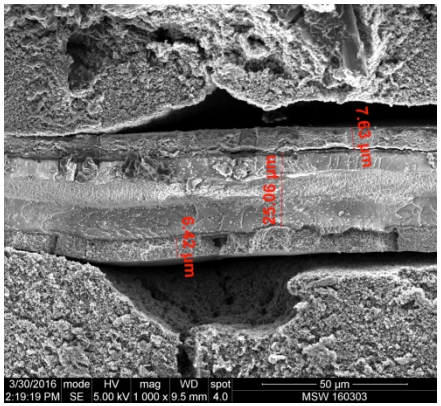
Hot pressed versus painted on electrode (50% Nafion as fibers)

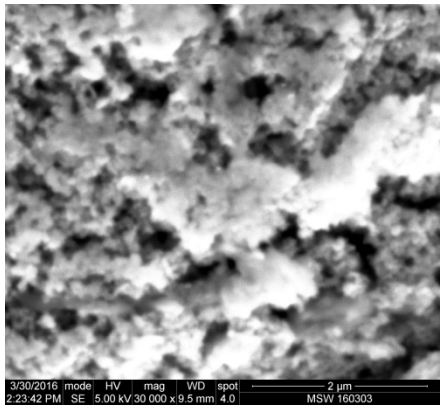






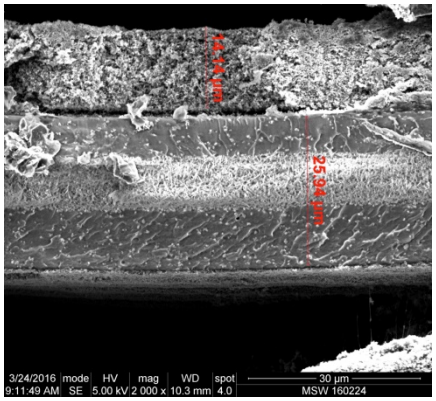
Hot pressed Electrode

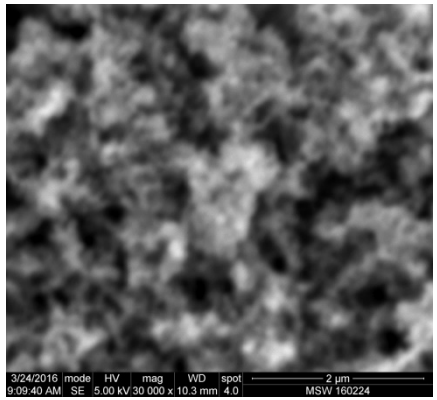




More uniform and thinner electrode.
Better kinetics.

Painted on Electrode

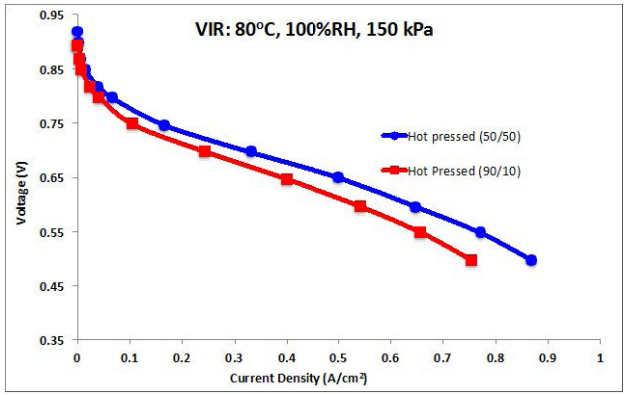


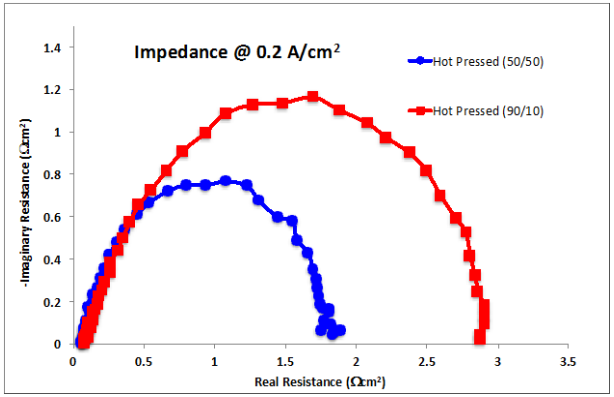


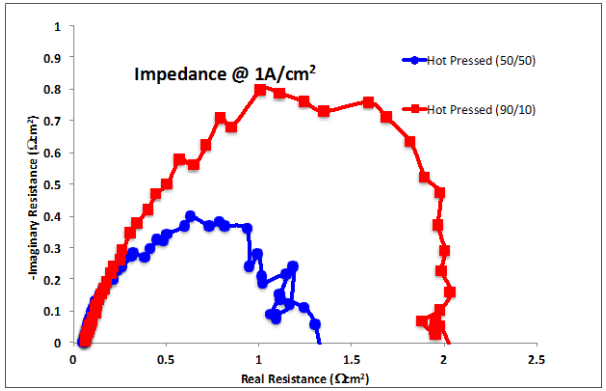
≈ 80% greater porosity (measured by MIP) in the sub 100nm range. Better Mass transport.

Electrode Design

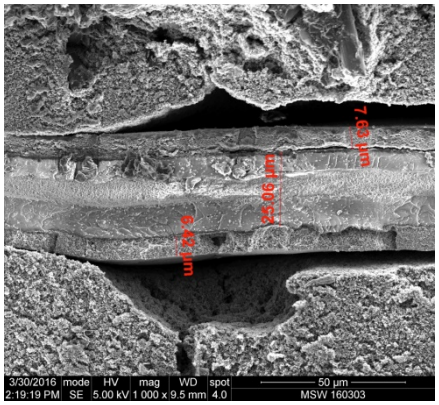
Hot pressed electrode (50% Nafion as fibers vs 90% Nafion as fibers)

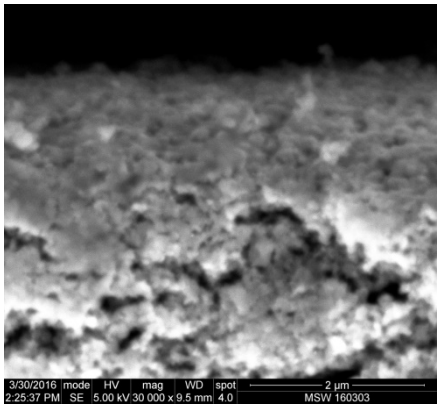




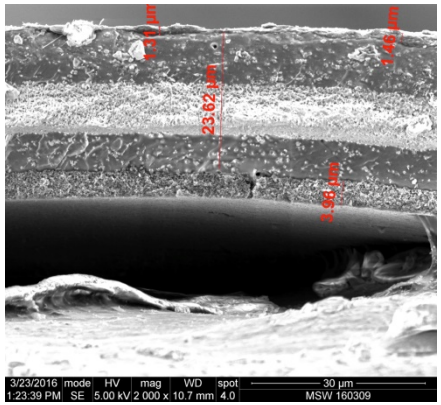


90/10 electrode : painted on





hot pressed



- Worse kinetics in 90/10 sample: access to catalyst limited by 10% amorphous Nafion)
- Worse mass transport in 90/10 sample : Denser electrode structure due to collapse of fibers

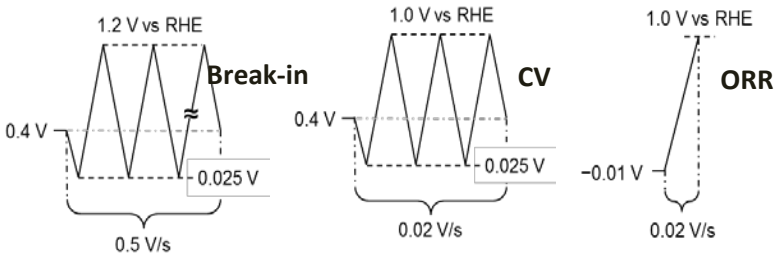
Dense areas where fibers have collapsed (more so when hot pressed)

Supplemental Slides

1. Protocol used for TF-RDE measurements
2. TF-RDE screening results on SOA catalysts
3. Protocol for MEA/sub-scale fuel cell measurements
4. Inter-lab and intra-lab reproducibility of data
5. Other diagnostics not discussed in present.
6. Facilities/test stands used for diagnostics
7. MEA Fabrication set-up

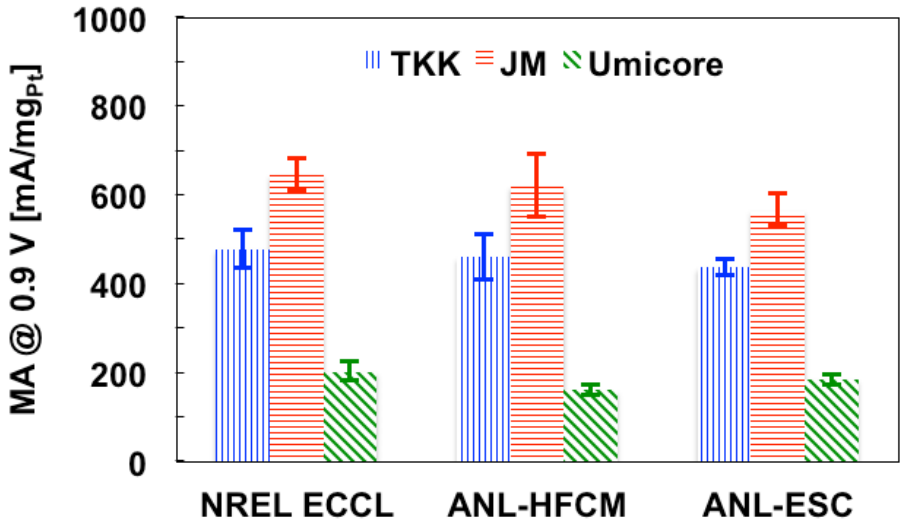
TF-RDE Protocols & Benchmarking

TF-RDE Standard Protocols



Gas	N_2 or O_2
Temperature	r.t.
Rotation Rate [rpm]	1600
Potential Range [V vs. RHE]	−0.01 to 1.0 (anodic)
Scan Rate [V/s]	0.02
R_{sol} measurement method	i-interrupter or EIS (HFR)
iR compensation	during measurement
Background Subtraction	LSV (O_2)−LSV (N_2)

Inter-lab Comparison; Pt/C;
N-RAD Technique



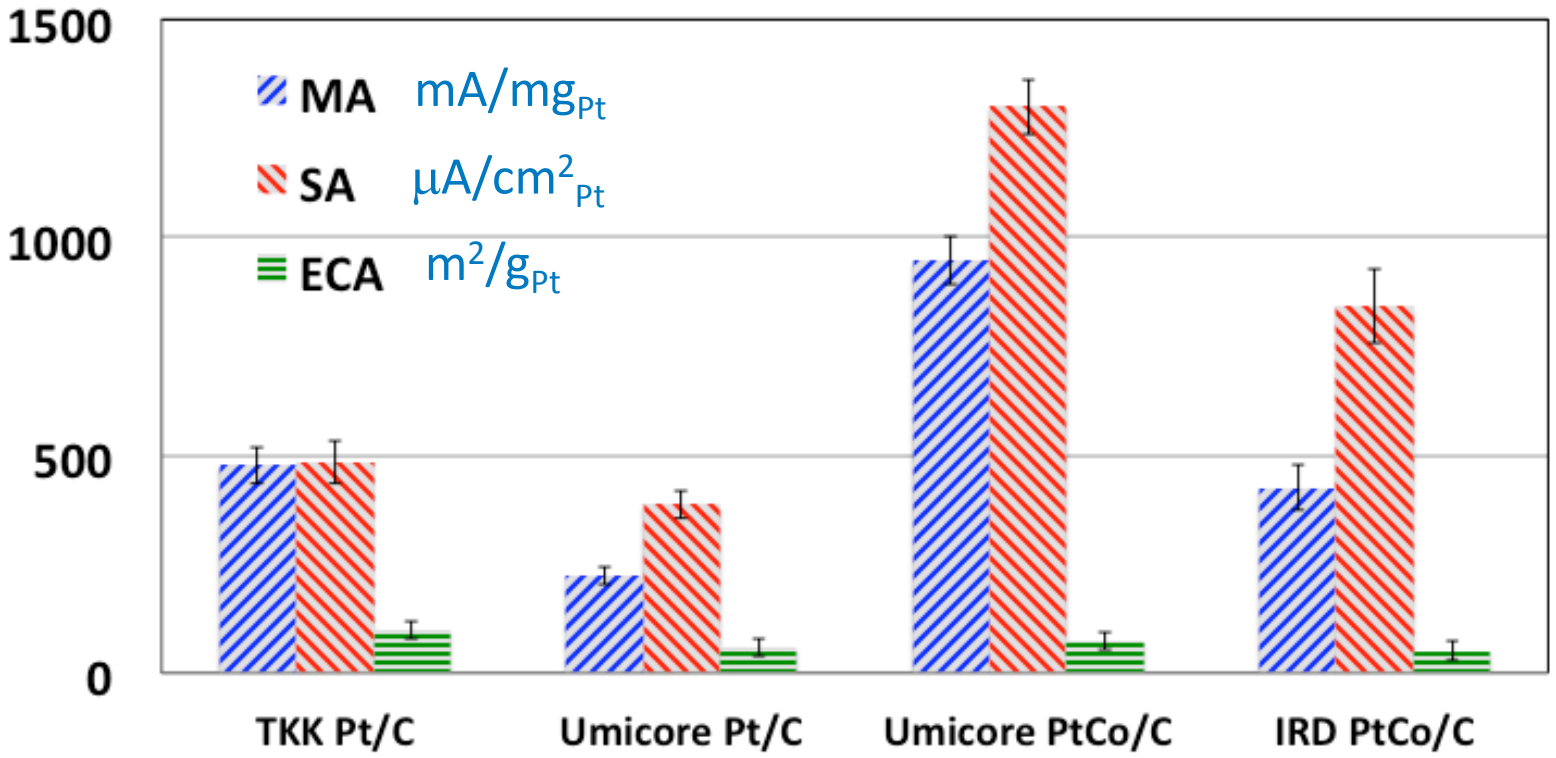
Shinozaki, Kazuma, Jason W. Zack, Ryan M. Richards, Bryan S. Pivovar, and Shyam S. Kocha. "Oxygen Reduction Reaction Measurements on Platinum Electrocatalysts Utilizing Rotating Disk Electrode Technique I. Impact of Impurities, Measurement Protocols and Applied Corrections." *Journal of The Electrochemical Society* 162, no. 10 (2015): F1144-F1158.

Shinozaki, Kazuma, Jason W. Zack, Svitlana Pylypenko, Bryan S. Pivovar, and Shyam S. Kocha. "Oxygen Reduction Reaction Measurements on Platinum Electrocatalysts Utilizing Rotating Disk Electrode Technique II. Influence of Ink Formulation, Catalyst Layer Uniformity and Thickness." *Journal of The Electrochemical Society* 162, no. 12 (2015): F1384-F1396.

Shinozaki, Kazuma, Jason W. Zack, Svitlana Pylypenko, Ryan M. Richards, Bryan S. Pivovar, and Shyam S. Kocha. "Benchmarking the oxygen reduction reaction activity of Pt-based catalysts using standardized rotating disk electrode methods." *International Journal of Hydrogen Energy* 40, no. 46 (2015): 16820-16830.

Shyam S. Kocha, Kazuma Shinozaki, Jason W. Zack, Deborah Myers, Nancy Kariuki, Tammi Nowicki, Vojislav Stamenkovic, Yijin Kang, Dongguo Li, and Dimitrios Papageorgopoulous "Best Practices and Testing Protocols for Benchmarking ORR Activities of Fuel Cell Electrocatalysts using Rotating Disk Electrode": to be published.

RDE Benchmarking of SOA Catalysts



Umicore PtCo/HSC catalyst exhibited ~x2 higher MA in RDE evaluations in perchloric acid compared to baseline TKK Pt/C.

Overall FC-PAD FC Test Protocol Summary

Initial Diagnostics

Ensure that fuel cell does not have a severe short or pinhole and catalyst is accessible

1. Electrical Short Measurement
2. Hydrogen X-over Measurement
3. Cyclic Voltammogram

Break-in/Conditioning

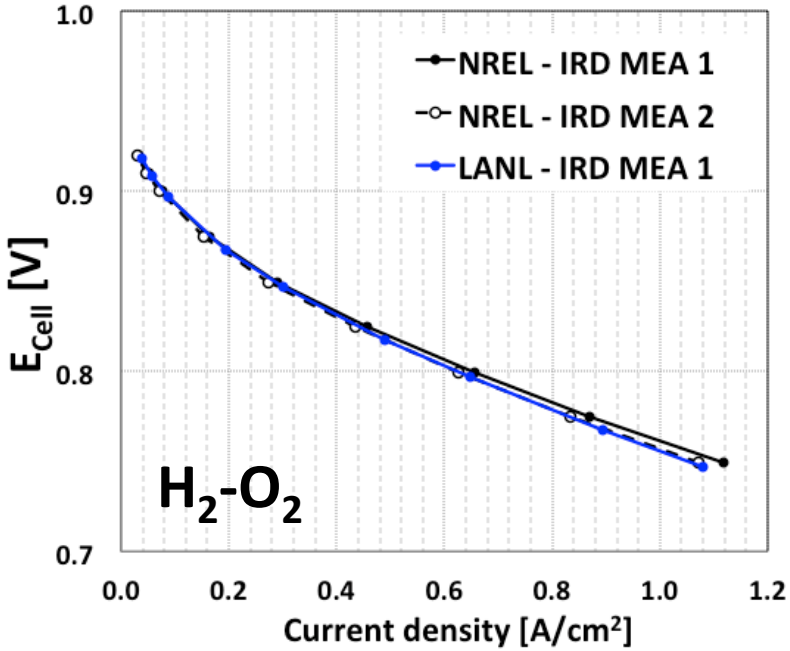
ORR Activity and H_2–Air Performance

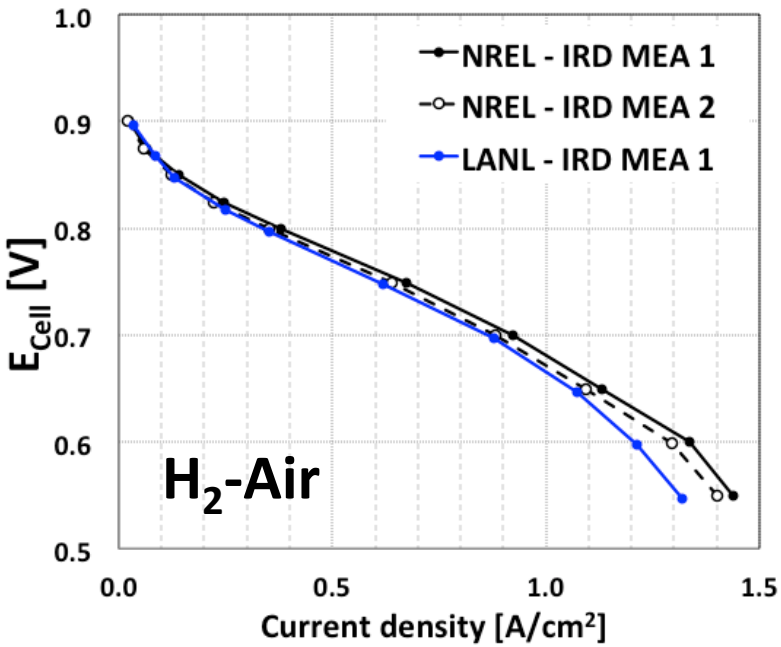
1. O_2 Curve; 100% RH, 150 kPa [PO_2= 100 kPa]
2. One-point ORR Activity
3. Wet Air Curve (high I to low i) 100% RH, 150 kPa
4. Dry Air Curve (high I to low i) 42% RH, 150 kPa
5. H_2 X-over (single point) at 80°C, 100%RH, 150 kPa
6. ECA (HUPD or CO stripping), 30-35°C

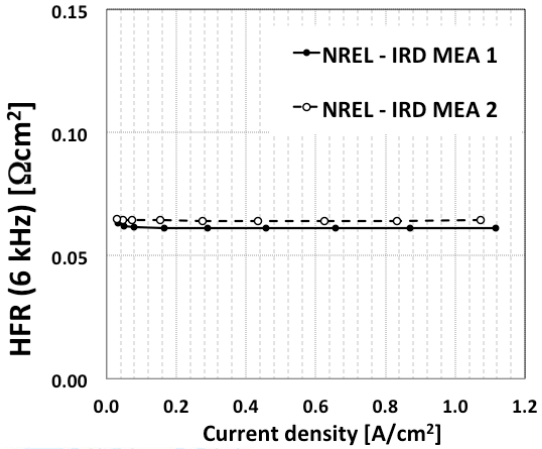
Optional Selected Diagnostics

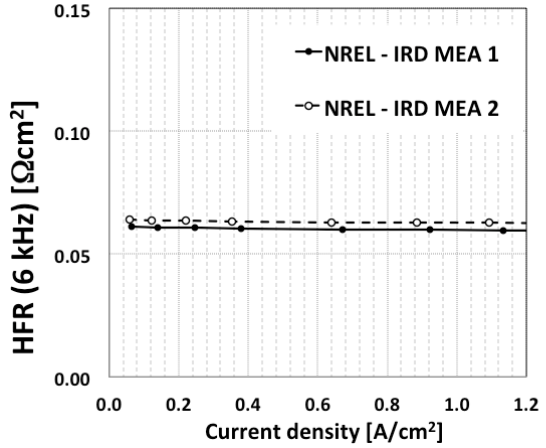
1. Catalyst Durability Cycling: 0.60–1.0V; based on DOE protocol
2. Support Durability Cycling: 1–1.5 V based on DOE protocol
3. Other: limiting currents, EIS, lab-specific diagnostics, etc.,

Inter-Lab FC Performance Reproducibility





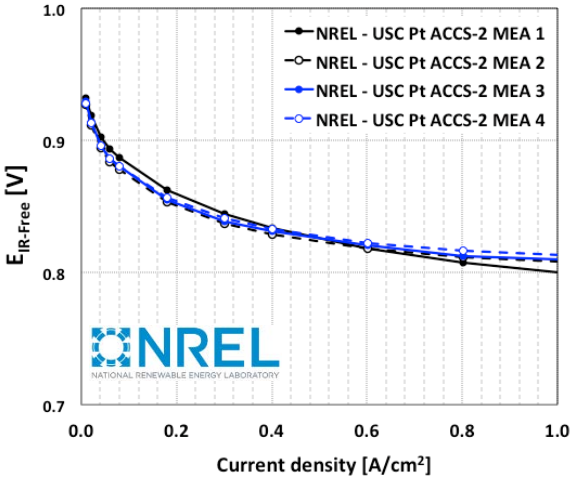




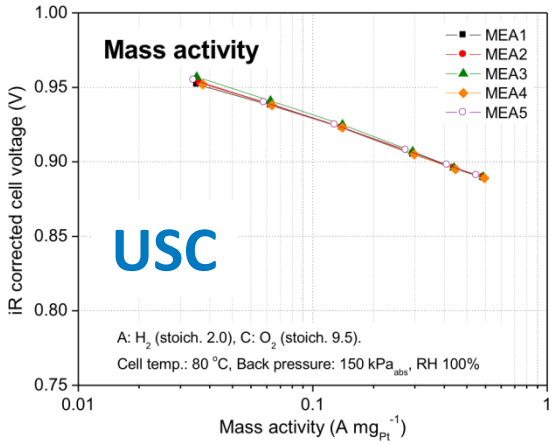
Inter-Lab FC Performance Reproducibility

USC

NREL NATIONAL RENEWABLE ENERGY LABORATORY



H_2-O_2



Pt*/ACCS-2 catalyst

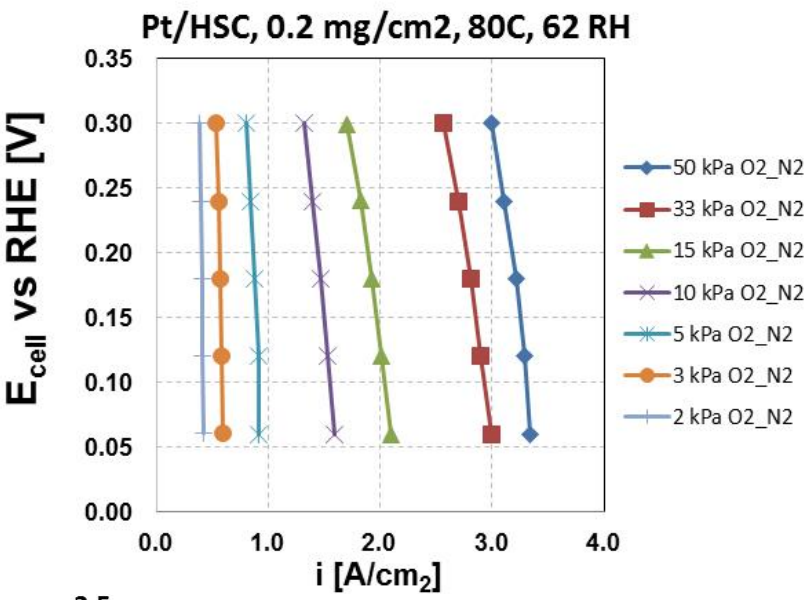
Pt* stands for suppressed platinum lattice catalyst synthesized with Co doped platinum

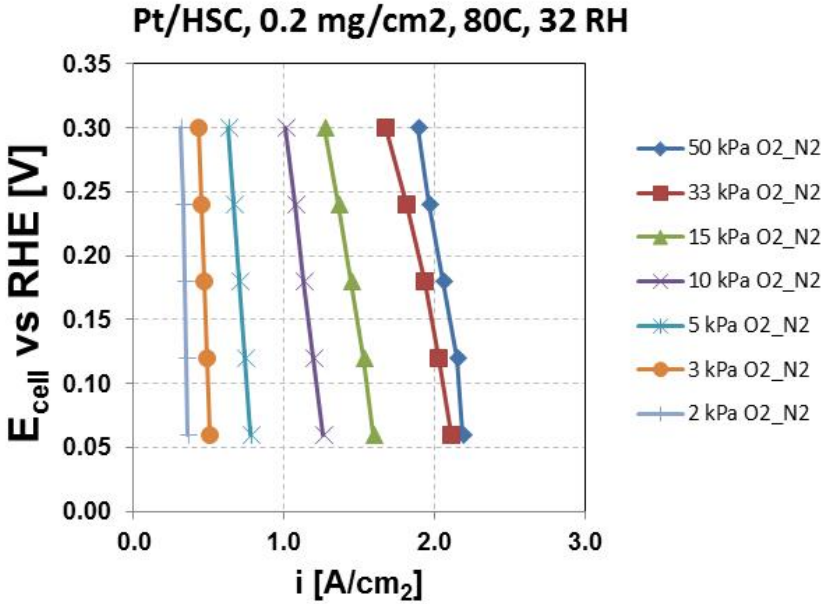
#	MA NREL	MA USC	SA NREL	ECA NREL
MEA1	503	334	714	70.4
MEA2	364	341	707	51.4
MEA3	387	348	655	59
MEA4	389	331	552	70.4

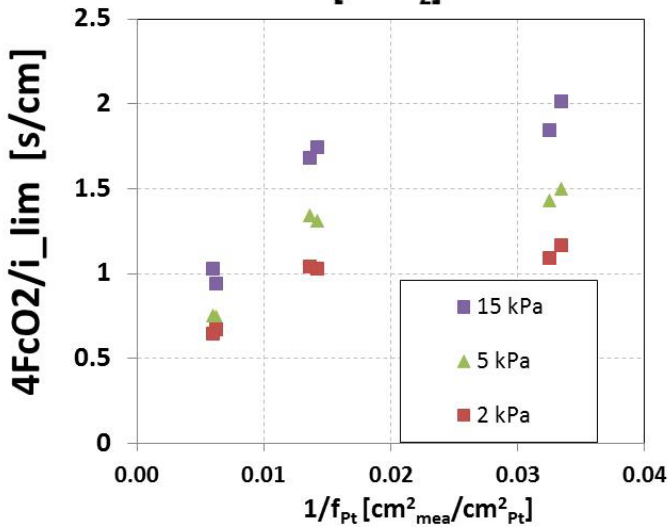
MA= mA/mg_{Pt}; SA= $\mu A/cm^2Pt$; ECA= m^2/g_{Pt}

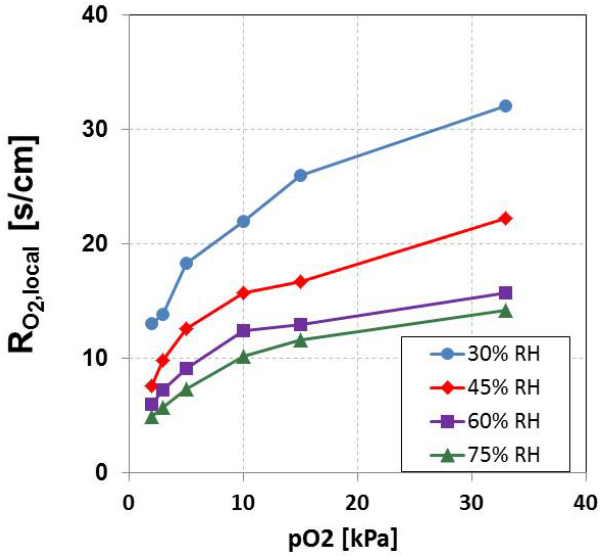
FUEL CELL FCPAD PERFORMANCE AND DURABILITY

O_2 limiting current measurements

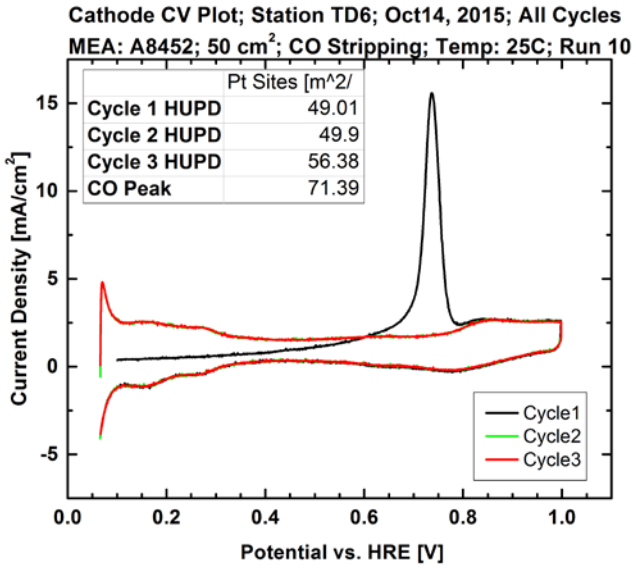


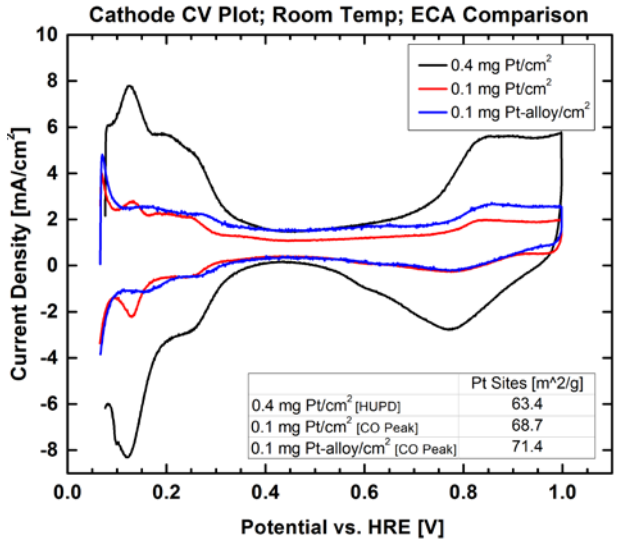






Accomplishments – Baselining low loading MEAs





Cathode Catalyst	ECA [m²/g] [HUPD]	ECA [m²/g] [CO Peak]
0.4 mg Pt/cm$^{2\$}$	63.3±2.2	N/A
0.1 mg Pt/cm$^{2\#}$	61.5±0.7	68.7
0.1 mg Pt-alloy/cm^{2*}	47.3±2.5	69.7±2.3

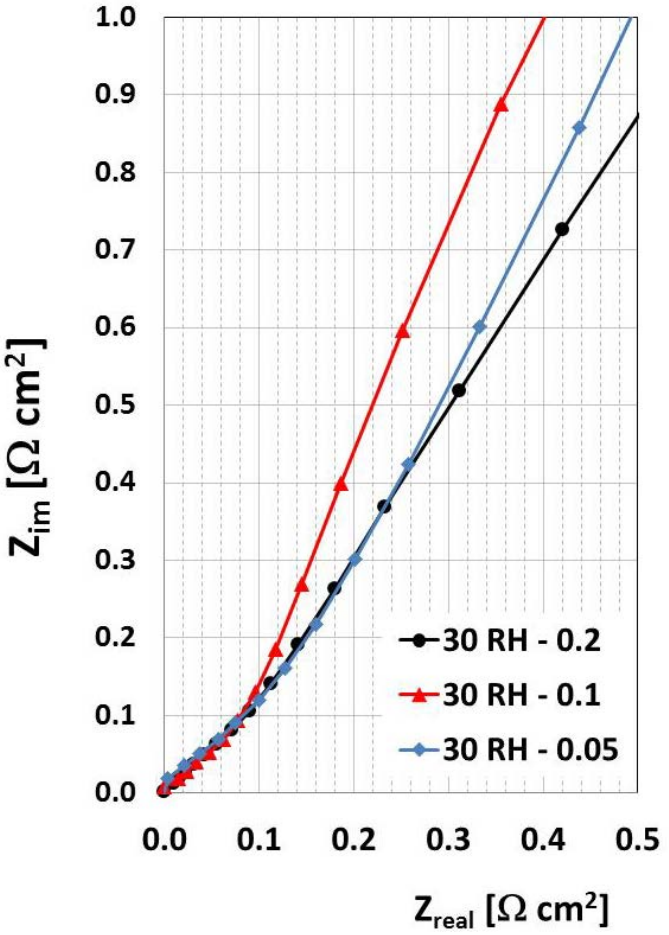
CV obtained after BOT

ECAs measured at 25C
$ average of 7 MEAs
# average of 2 MEAs
* average of 3 MEAs

Standard Diagnostics

Supplemental

- HFR
- EIS
- O2 limiting currents
- H2 limiting currents



Automated Diagnostics

Automated gas mixing for oxygen limiting current and the development/investigation of CO limiting current as a diagnostic

Automated Gamry potentiostats

-ideal for durability studies

-voltage cycling and automated CV collection

-very helpful for Pt oxide measurements

(automated hold and sweep, temp, RH etc in one program)

-useful for CO limiting current measurements

HFR-Free Potential Control

(1 stand, only requires software upgrade to be utilized on 3 more stands)

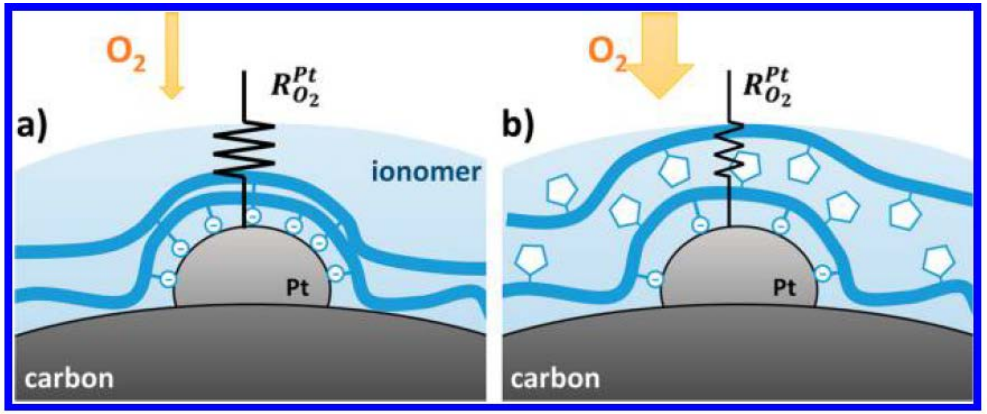
-Used to match potentials where kinetic data and oxide coverage data is taken

Technical Back-Up Slides

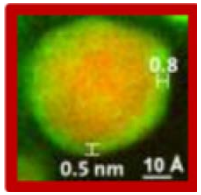
Mitigation Strategies

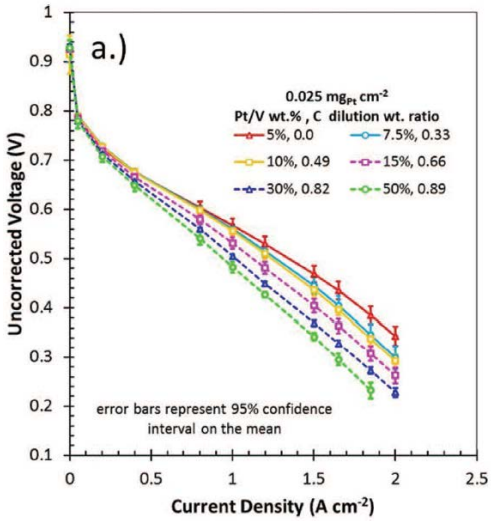
- **Alter ionomer structure**

  A. Kongkanand and M. F. Mathias, *JPC Letters*, **7**, 1127 (2016)



- **Increase Pt Electrocatalyst Surface Area**
  - Pt monolayer electrocatalysts





- **More Disperse Electrocatalysts**
  - Lower Pt wt%
- **Electrospun Ionomer Electrodes**
  - Reduce ionomer amount via

  Creation of H^+ superhighways