# FC-PAD: Fuel Cell Performance and Durability Consortium

Rod Borup[1] (Primary Contact), Adam Weber,[2] Deborah Myers,[3] K.C. Neyerlin,[4] Ahmet Kusoglu,[2] Rajesh Ahluwalia,[3] Rangachary Mukundan,[1] Karren More[5]
*Author list reflects FC-PAD directors and thrust coordinators only*
[1]Los Alamos National Laboratory
MS D429, P.O. Box 1663
Los Alamos, NM 87545
Phone: 505-667-2823
Email: Borup@lanl.gov

DOE Managers: Dimitrios Papageorgopoulos
Phone: 202-586-5463
Email: Dimitrios.Papageorgopoulos@ee.doe.gov

Gregory Kleen
Phone: 720-356-1672
Email: gregory.kleen@ee.doe.gov

Subcontractors:
- [2] Lawrence Berkeley National Laboratory, Berkeley, CA
- [3] Argonne National Laboratory, Lemont, IL
- [4] National Renewable Energy Laboratory, Golden, CO
- [5] Oak Ridge National Laboratory, Oak Ridge, TN

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## Overall Objectives

Advance performance and durability of polymer electrolyte membrane fuel cells (PEMFCs) at a *pre-competitive* level.

- Improve high-current-density performance at low Pt loadings:
  - Improved electrode structures
  - Reduced mass transport losses
  - Loading: ≤0.125 mg Pt/cm$^2$ total
  - Performance @ 0.8 V: 300 mA/cm$^2$
  - Performance @ rated power: 1,800 mW/cm$^2$.
- Develop knowledge base for more durable and high-performance PEMFC components:
  - Understand science of component integration (e.g., understand ionomer interactions with carbon/interfaces between electrodes and gas diffusion layer or membranes)
  - Improve component durability (e.g., limit catalyst ripening and carbon corrosion; membrane stabilization, self-healing, electrode-layer stabilization).
- Develop new diagnostics, characterization tools, and models.
- Provide support to DOE-funded FC-PAD projects:
  - Support projects from DOE DE-FOA-0001412 (3M Company, General Motors, United Technologies Research Center, Vanderbilt University)
  - Add support for new projects from DOE DE-FOA-0002044 (Nikola Motor Company, General Motors, Carnegie Mellon University).
- Develop knowledge base for both industrial and academic developers.
- Expand work supporting light-duty fuel cell vehicles to heavy-duty fuel cell applications.

## Fiscal Year (FY) 2019 Technical Objectives

- Understand electrode layer structures:
  - Understand effect of catalyst ink properties in terms of catalyst-ionomer-ink-solvent-composition and define variable contributions to limiting performance.
  - Develop methodology for determining relative ionomer coverage on carbon and Pt.
  - Demonstrate electrode reconstruction method from analyzing nano-X-ray computed tomography (nano-XCT) data related to catalyst/carbon particle agglomeration.
  - Conduct direct imaging of catalyst layer cross-sections measuring ionomer layer thickness and carbon agglomerates.
  - Measure cation effect on thin film ionomer structure.

  - Quantify bulk electrode and local transport resistance as a function of ink composition, processing, and fabrication method.
- Define/measure degradation mechanisms:
  - Measure durability of alloy catalysts and effect of leaching on ionomer properties.
  - Compare membrane electrode assembly (MEA) durability in terms of catalyst accelerated stress test (AST).
  - Coordinate microstructural analysis of at least three new catalyst alloy MEAs before and after catalyst-cycling and C-corrosion ASTs.
- Begin to understand implications and limitations of current fuel cell materials related to heavy-duty fuel cell applications.

## Technical Barriers

This project addresses the following technical barriers from Table 3 of the Fuel Cell Technical Team Roadmap:

- A. Decrease Cost
- B. Increase Power Density
- C. Simultaneously Improve Durability.

## Technical Targets

This project develops MEAs that meet the targets in Table 1.

## FY 2019 Accomplishments

- Measured durability performance as a function of ultra-low loading and with recovery protocols.
  - Experimental measurements of square-wave AST, impedance, and polarization performance demonstrated a more significant and rapid decay below 0.10 $mg_{Pt}/cm^2$.
  - Modeled durability results to define voltage-loss-breakdown identifying $R_f$ (ionomer film resistance) as the majority cause of the increased performance loss below 0.10 $mg_{Pt}/cm^2$.
- Measured transport limitations related to MEA conditioning effects.
  - Monitored carbon support effect during conditioning.
  - Compared Pt particles size distribution during conditioning and measured significantly higher and broader particle growth for Vulcan carbon compared with high-surface-area carbon (HSAC).
  - Evaluated degradation mechanisms during conditioning by small-angle X-ray scattering (SAXS), transmission electron microscopy, and extended X-ray absorption fine structure; observed the Vulcan carbon having higher surface mobility.
- Characterized aggregates and agglomerates.
  - Utilized nano-XCT, atomic force microscopy, scanning transmission electron microscopy, and X-ray scattering (ultra-small angle and small angle) to measure ionomer and carbon aggregates and agglomerates.
  - Reconstructed agglomerate includes porous C, Pt, and ionomer distributions from absorption contrast images.
  - Mesoscale transport modeling identified that cylindrical agglomerates show lower $O_2$ transport resistance than spherical agglomerates of the same equivalent diameter (500 nm).
- Examined catalyst inks and solvent effect of MEA structure and performance.
  - Measured ionomer coverage for different ink-solvent ratios and identified slightly water-rich catalyst ink concentrations as optimal to minimize diffusion resistance and maintain good proton conductivity at 0.5 V.
  - Measured particle size distributions for ink-solvent ratios with Vulcan and HSAC carbons showing post-conditioning average particle sizes of 3.2 and 4.0 nm, respectively.
- Evaluated cation migration and diffusion in MEAs.
  - Evaluated cation effect on Nafion structure and performance demonstrating reduced conductivity of 40% at a level of 20% Ce doping level (80°C).

- Evaluated nanodomain effect on rate of ionomer swelling and identified that the rate of swelling is inversely proportional to nanodomain orientation.

Table 1. Technical Targets: MEAs for Transportation Applications

| Characteristic | Units | 2025 Targets |
|---|---|---|
| MEA cost | $/KW | 10 |
| Platinum group metal (PGM) total content | g/kW rated | ≤0.10 |
| Performance @ 0.8 V | mA/cm$^2$ | 300 |
| Performance @ rated power | mA/cm$^2$ | 1,800 |
| Durability with cycling | hours | 8,000 |
| Loss in performance at 0.8 A/cm$^2$ | mV | ≤30 |
| Loss in performance at 1.5 A/cm$^2$ | mV | ≤30 |

## INTRODUCTION

Although fuel cells are being deployed in cars in limited commercialization, they still fall short of the DOE targets for this technology, which are required for widespread consumer acceptance. The FC-PAD consortium works to advance performance and durability of PEMFCs at a pre-competitive level to further enable their commercialization. This consortium coordinates national laboratory activities related to fuel cell performance and durability, provides technical expertise, and harmonizes activities with industrial developers. The consortium serves as a resource that amplifies the Office of Energy Efficiency and Renewable Energy's impact by leveraging the core capabilities of several labs in conducting low-technology-readiness-level research.

The major challenge addressed by this consortium is to develop the knowledge base and understanding to optimize electrode structures for more durable, high-performing PEMFC component technologies, while simultaneously reducing cost. Current research focuses on achieving high performance and durability in low-Pt-loaded PEMFCs.

## APPROACH

The FC-PAD consortium incorporates national laboratory investigators with proven experience (developed in prior projects) related to durability, transport, and performance, and combines them into one highly coordinated effort. The consortium formalizes already existing and effective collaborations among the national laboratories that have established leadership in PEMFC performance and durability research and development.

Three thrust areas are related to components: (1) Electrocatalysts and Supports, (2) Electrode Layers, and (3) Ionomers, Gas Diffusion Layers, Bipolar Plates, Interfaces; and three thrust areas are cross-cutting in nature: (4) Modeling and Validation, (5) Operando Evaluation: Benchmarking, ASTs, and Contaminants, and (6) Component Characterization and Diagnostics.

FC-PAD is an integrated five-national-laboratory consortium with a large number of contributing staff scientists, research technicians, post-docs, and students. For FY 2019, FC-PAD contributors, in addition to the listed FC-PAD thrust coordinators, included:

- **Argonne National Laboratory:** Nancy Kariuki, Dennis Papadias, C. Firat Cetinbas, J-K Peng, Xiaohua Wang, Jeremy Kropf, Jaehyung Park, and Evan Wegener
- **Lawrence Berkeley National Laboratory:** Lalit Pant, Meron Tesfaye, Anamika Chowdhury, Sarah Berlinger, John Petrovick, Andrew Crothers, Peter J. Dudenas, Victoria Ehlinger, Grace Lau, Clayton Radke, Gregory Su, Isvar Cordova, and Claire Arthurs
- **Los Alamos National Laboratory:** Andrew Baker, Siddharth Komini Babu, Kavitha Chintam, Derek Richard, David Langlois, Roger Lujan, Mike Workman, Sergio Herrera, Dongguo Li, and Xiaojing Wang

- **National Renewable Energy Laboratory:** Sadia Kabir, Tim Van Cleve, Guanxiong Wang, Andrew Star, Ellis Klein, Guido Bender, and Mason Mooney
- **Oak Ridge National Laboratory:** David Cullen and Shawn Reeves.

Four FC-PAD projects from DE-FOA-0001412 were announced by DOE during FY 2017. Those projects are led by 3M Company, General Motors, United Technologies Research Center, and Vanderbilt University. The core national lab team supports those four projects with an equal level of effort utilizing national lab capabilities available in FC-PAD.

Three new FC-PAD projects from DE-FOA-0002044 were announced during FY 2019. Those projects will begin during FY 2020, and will be led by General Motors, Nikola Motor Company, and Carnegie Mellon University. Initial discussions have commenced about support to these projects from the core national lab team.

## RESULTS

During FY 2019, FC-PAD conducted work in all six thrust areas. Much of the work is related to fundamental understanding of the electrode structure, different effects during fabrication, the relative distribution of materials, and how ionomer and ionomeric cations interact with the catalyst and catalyst support. This includes significant characterization of the electrode structure and modeling to determine the relative impact on performance and durability. Below is a selected subset of these activities during FY 2019.

### Ink-Solvent Interactions

To identify and correlate catalyst-electrode ink interactions with different solvents and how they affect catalyst-layer morphology and subsequent performance, FC-PAD examined solvent-ink interactions. Ex situ and operando experiments were used to explore impact of different solvents in MEA inks and fuel-cell performance. This examination included comparing catalyst inks of n-propanol rich (24% $H_2O$), water rich (83% $H_2O$), and a baseline solvent ratio (62% $H_2O$), results of which are shown in Figure 1 for Pt/Vu.

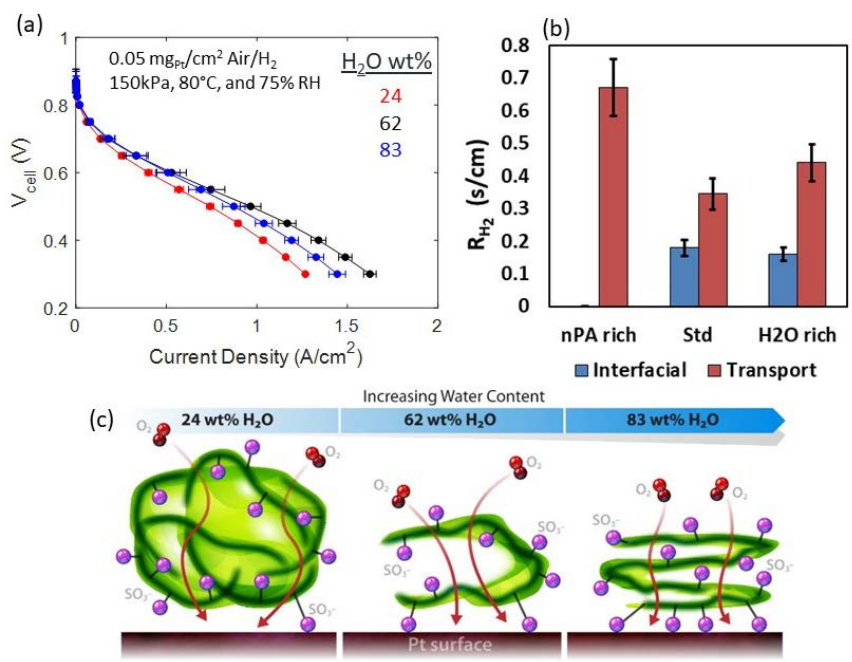


Figure 1. (a) Polarization behavior as a function of water/alcohol solvent ratio. (b) Breakdown of the limiting current resistance between interfacial and transport effects, with the transport through the ionomer being dominant. (c) Schematic in terms of how the ionomer interacts with the Pt on Vulcan carbon as a function of solvent ratio.

The water/alcohol ratio had a minimal effect on the carbon aggregate or agglomerate structure but a significant influence on performance. Water/alcohol ratios slightly above 50% yielded the best performance, which is likely due to the best ionomer conformation in the resultant catalyst-layer structure. Transport through the ionomer layer, which is the dominant resistance, falls at higher water content in the ink. Interfacial resistance due to $SO_3^-$ adsorption, a secondary resistance, increases at higher water content.

### Agglomerate Structure Impact on Oxygen Transport in PEMFC Cathodes

We used nano-XCT to simulate the microstructure of electrodes with porous carbon-supported catalysts. This allows simulation of the carbon morphology effect and catalyst-ionomer agglomerate size on reactant-product transport and electrode performance. Catalyst-ionomer agglomerates were extracted from nano-XCT images and simulated by spherical agglomerates (Figure 2a). This identifies the optimum agglomerate size and structure for the highest electrode performance under various cell operating conditions. When combined with experimental methodology for controlling agglomerate size and structure, this can optimize stack power density for a given material set (Figure 2b, 2c). This model shows that cylindrical agglomerates provide lower oxygen transport resistance at high relative humidity when primary pores of porous carbon support are flooded and that smaller agglomerates are preferred at high relative humidity conditions to decrease transport resistance to catalyst particles in pores.

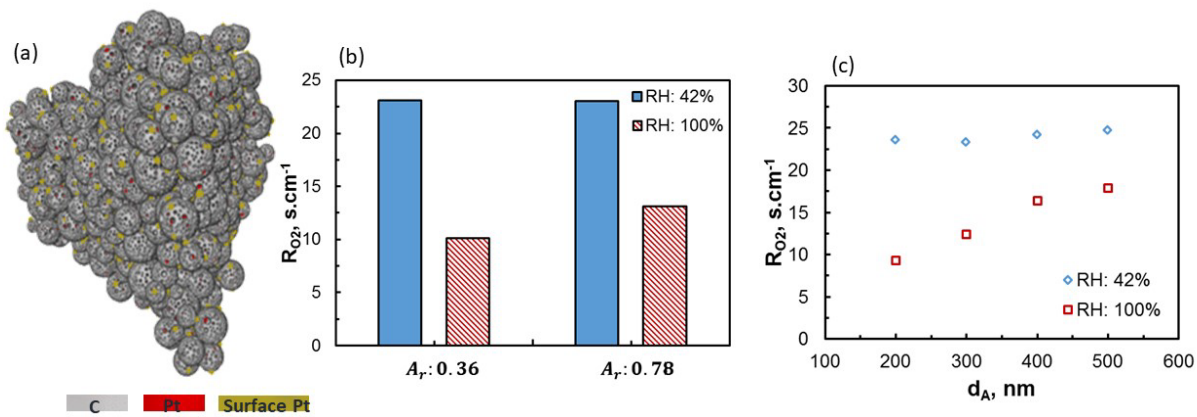


Figure 2. (a) Reconstruction of agglomerates with porous carbon with distribution of Pt; (b) resulting modeling predicting of local $O_2$ transport resistance for different agglomerate aspect ratios ($A_r$); and (c) model predictions of the local $O_2$ transport resistance for different agglomerate radius and relative humidity (RH)

### Cation Effects on MEA Performance and Ionomer Structure

Cations such as cerium (used in membranes as radical scavengers) and cobalt (which leach out of cathode PtCo alloy catalysts) are known to migrate through the ionomer and can affect fuel cell performance and change ionomer structure and water uptake. An in situ fuel cell design was made so that cation (cerium) concentration profiles and migration could be measured in situ using confocal X-ray fluorescence. Experimental observations of cerium migration and the resulting structural change of Nafion were made (Figure 3a, 3b). Cerium concentration profiles were fitted to a 1-D Nernst-Planck equation to calculate diffusivity. Concentrated solution theory was utilized to evaluate the high concentration of ions. Constitutive relations were used to relate transport coefficients to measured properties of the cations. This modeling and experimental analysis details the migration of Ce to the fuel cell cathode during operation and provides details on cation movement within operating fuel cells due to both potential-induced migration and diffusion. The experimental data was also used for accurate model predictions of water uptake and membrane conductivity, which are detailed with Ce concentration.

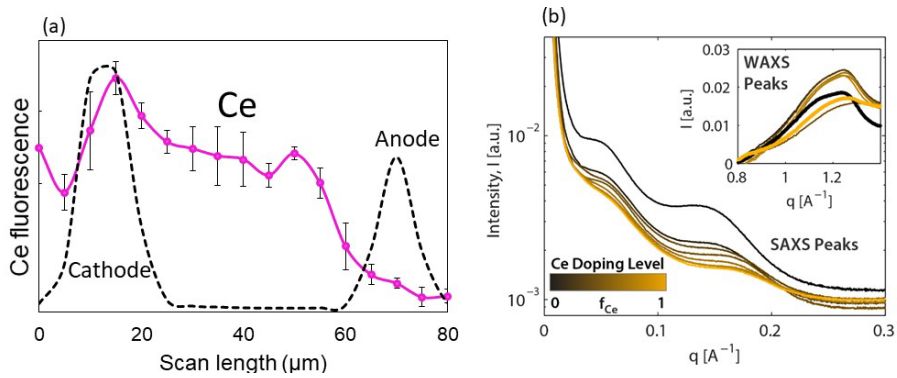


Figure 3. (a) In situ cross-sectional experimental observation of cerium concentration profile in an operating fuel cell, and (b) Nafion structural change measure by SAXS and wide-angle X-ray scattering (WAXS).

## Heavy-Duty Applications

FC-PAD is in the initial stages of placing new emphasis on heavy-duty applications. The fuel cell MEA targets for heavy-duty applications are less well developed compared with light-duty applications; however, it is clear that heavy-duty applications require much longer-term durability. Late in FY 2019, we began to apply FC-PAD capabilities toward heavy-duty fuel cell applications with a specific focus on higher efficiency and better durability at higher Pt loadings, with initial durability catalyst ASTs being extended to 75,000 cycles (Figure 4). Experimental measurements established linkage of Co leaching to lowered efficiency at current densities <1 A/cm$^2$. While PtCo particles increased in size less than the Pt particles (5.2 to 6.8 nm for PtCo versus 4.3 to 7.5 nm for Pt), the performance was reduced both at low current densities (kinetic region) and at high current densities (transport regime). This identifies a pathway to retaining benefits of alloying over a much longer operational lifetime as required by heavy-duty applications. PtCo-alloy catalysts show higher efficiency at low current; however, Pt catalysts show better performance at high current (higher roughness). Postmortem characterization shows that Co completely leaches out of small particles while the larger core-shell particles are more stable (Pt shell, PtCo core) (see Figure 4b). Examining the Pt concentration profile in the MEA after durability AST, a depletion of platinum is observed, which is approximately 1.5 micron thick next to the membrane (see Figure 4c, 4d).

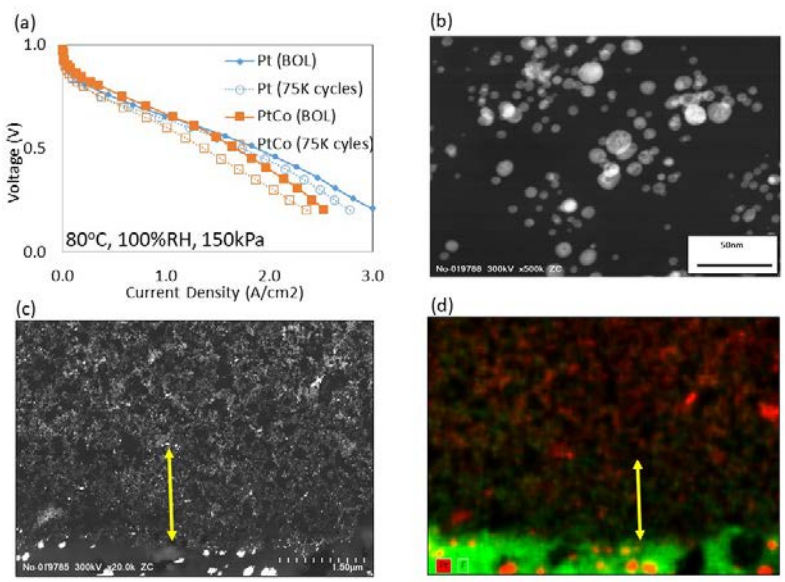


Figure 4. (a) Beginning of life (BOL) performance and after 75,000 cycles for Pt and PtCo-alloy catalysts; (b) transmission electron microscopy post-characterization of PtCo particles; (c) MEA cross-section of back-scattered electrons; and (d) elemental profile of MEA cross-section.

## CONCLUSIONS AND UPCOMING ACTIVITIES

The FC-PAD consortium has conducted significant analysis of MEA electrode structures and analyzed the structures to understand the performance and durability losses. New emphasis has started related to heavy-duty applications. The results and understandings from FC-PAD have been disseminated through peer-reviewed publications and numerous presentations. In addition, the FC-PAD consortium provided support to the four light-duty DOE-funded projects.

The FC-PAD consortium's planned future work concentrates on developing the foundational science to understand MEA component performance and durability with new emphasis on heavy-duty applications.

- Initial FC-PAD workscope related to heavy-duty applications
  - Understand the heavy-duty fuel cell operating space and prioritize research directions.
  - Refine applicable models, characterization, and diagnostics to heavy-duty applications.
  - Develop refined ASTs for extended lifetime prediction for heavy-duty operating conditions.
- Material and characterization studies
  - Conduct catalyst alloy studies including dissolution and high-potential kinetics.
  - Directly measure ionomer film properties and morphology on operating electrodes.
- Catalyst layer studies
  - Explore different catalyst layer structures with low and moderate Pt loadings.
  - Perform microstructural reconstruction and modeling for catalyst layers including multiphase flow.
  - Understand the size and impact of Pt/C aggregates and agglomerates.
- Durability
  - Evaluate system component contaminants (e.g., Fe++) and mechanism for transport to MEA.
  - Characterize electrode microstructural changes as a result of aging.
  - Analyze resistance related to impact of alloy catalyst leaching on ionomer transport.

## FY 2019 PUBLICATIONS/PRESENTATIONS

**Publications Relevant to FC-PAD from Consortium Members**

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