

V.E.1. Durability Improvements through Degradation Mechanism Studies

Rod Borup¹ (Primary Contact),
Rangachary Mukundan¹, Dusan Spornjak¹,
Andrew Baker¹, Roger Lujan¹, David Langlois¹,
Rajesh Ahluwalia², Dennis. D. Papadias²,
Adam Weber³, Ahmet Kusoglu³, Shouwen Shi³,
Karren More⁴, Steve Grot⁵

¹Los Alamos National Laboratory (LANL)
MS D429, P.O. Box 1663
Los Alamos, NM 87545
Phone: (505) 667-2823
Email: Borup@lanl.gov

DOE Manager

Nancy Garland
Phone: (202) 586-5673
Email: Nancy.Garland@ee.doe.gov

Subcontractors & Collaborators

² Argonne National Laboratory, Argonne, IL
³ Lawrence Berkeley National Laboratory, Berkeley, CA
⁴ Oak Ridge National Laboratory, Oak Ridge, TN
⁵ Ion Power, New Castle, DE

Project Start Date: October 1, 2009

Project End Date: Project continuation and direction determined by DOE

- AST validation and new AST protocol development
- Metal bipolar plates
- Reversible degradation, transport (gas diffusion layer effects), shut-down/start-up (SD/SU)
- Durability evaluation of Pt alloys, effect of Pt and alloy migration on membrane durability/performance

Technical Barriers

This project addresses the following technical barriers from the Fuel Cells section of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan:

- (A) Durability
- (B) Cost

Technical Targets

- Transportation Durability: 5,000 hours (with cycling)
 - Estimated Start/Stop Cycles: 17,000
 - Estimated Frozen Cycles: 1,650
 - Estimated Load Cycles: 1,200,000
- Stationary Durability: 40,000 hours
- Transportation Survivability: -40°C
- Transportation Cost: \$30/kWe

FY 2015 Accomplishments

- Measurement of carbon corrosion during drive cycle operation
- Modeling of the measured carbon corrosion
- Measurement of membrane structural changes
- In situ and ex situ measurements of cerium migration
- Development of new ASTs
 - Combined chemical/mechanical AST with a better representation of membrane failure in the field
 - Catalyst AST and acceleration factor comparison with drive cycle tests
- Measurement of metal bipolar plate corrosion and effect on membrane durability

Overall Objectives

- Identify and quantify degradation mechanisms
 - Components, component interfaces, component interactions, and degradation mechanisms
 - Discern impact of electrode structure on durability and performance
- Develop advanced in situ and ex situ characterization techniques, accelerated stress test (AST) protocols
- Develop models related to degradation mechanisms
- Explore non-system related mitigation methods

Fiscal Year (FY) 2015 Objectives

- Catalyst layer morphology effect on durability (carbon corrosion during drive cycle)
- Membrane structural changes and cerium migration/stabilization
 - Changes in properties including: crystallinity, water up-take, transport, mechanical strength, swelling, crack propagation



INTRODUCTION

The durability of polymer electrolyte membrane (PEM) fuel cells is a major barrier to the commercialization of these systems for stationary and transportation power applications. By investigating cell component degradation modes and defining the fundamental degradation mechanisms of components and component interactions, new materials can be designed to improve durability. To achieve a deeper understanding of PEM fuel cell durability and component degradation mechanisms, we utilize a multi-institutional and multi-disciplinary team with significant experience investigating these phenomena.

APPROACH

Our approach to understanding durability and degradation mechanisms within fuel cells is structured in three areas: fuel cell testing (life testing, ASTs, and ex situ aging), characterization of component properties as a function of aging time, and modeling (component aging and integrated degradation modeling). The modeling studies tie together what is learned during component characterization and allow better interpretation of the fuel cell studies. This approach enables an increased understanding of fuel cell

degradation and development of materials to overcome durability limitations in fuel cell systems. This work is also being coordinated with other funded projects examining durability through the DOE Durability Working Group.

RESULTS

Carbon Corrosion at Normal Operating Fuel Cell Potentials

Corrosion of the carbon electrocatalyst support is one of the major contributors to degradation which leads to changes in the catalyst layer structure and reduces its activity. During FY 2015, we measured and quantified carbon corrosion during the drive cycle operation recommended by the Fuel Cell Tech Team (FCTT). We also evaluated the effect of varying the upper and lower potential limits used during drive cycle operation on the carbon corrosion rate.

A series of carbon corrosion spikes during potential cycling of a high-surface area carbon support (E-type carbon) are shown in Figure 1a where the upper potential was varied from 0.95 V to 0.60 V while the lower potential was kept constant at 0.40 V. Figure 1b illustrates the cathode carbon corrosion rate during the same FCTT drive cycle where

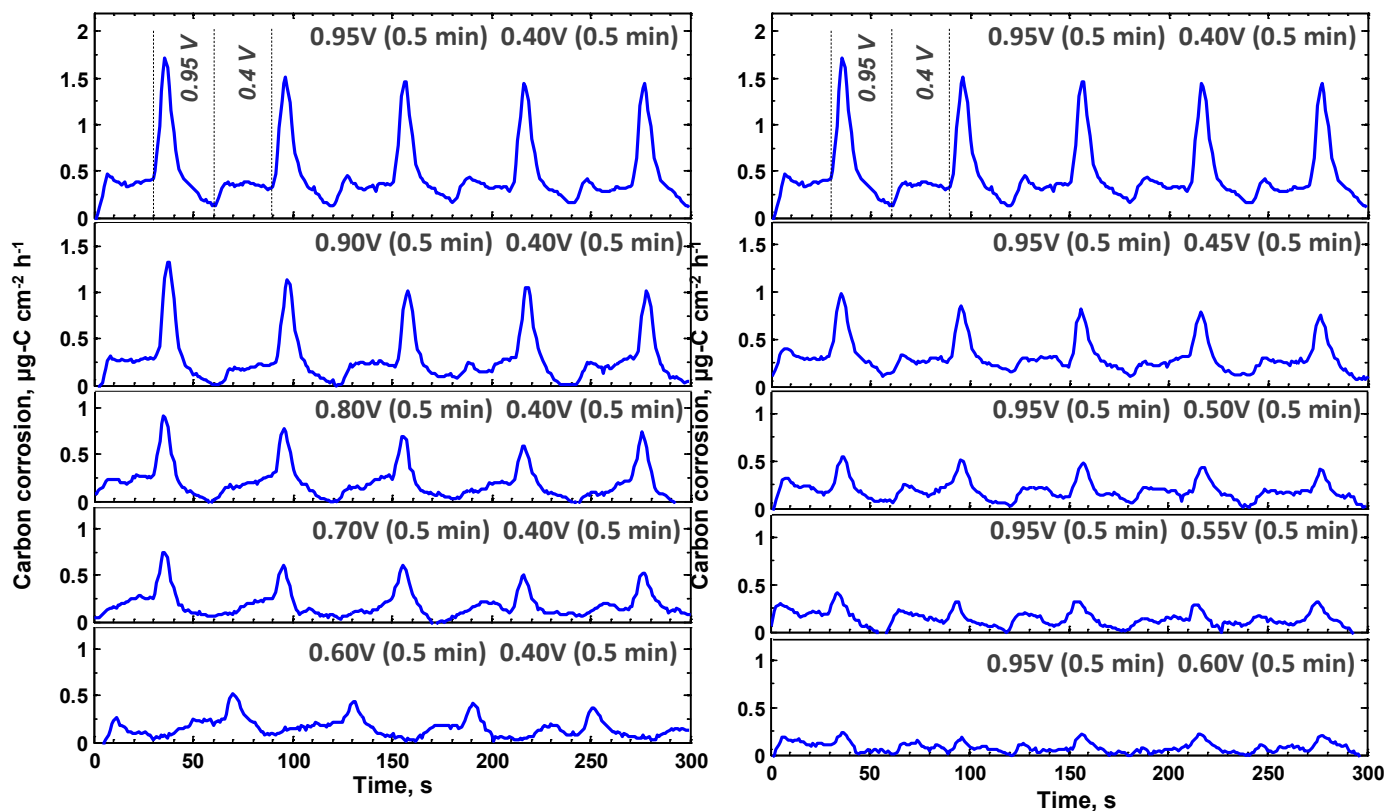


FIGURE 1. Carbon dioxide emissions from the fuel cell cathode for E-type support carbon during potential cycling operation for (a) varying upper potential limits (0.95 V, 0.90 V, 0.80 V, 0.70 V, 0.60 V) and constant lower potential of 0.4 V and (b) varying lower potential limits (0.40 V, 0.45 V, 0.50 V, 0.55 V, 0.60 V) and constant upper potential of 0.95 V. Operation at 80°C and 100% relative humidity (RH). Figure 1 has 0.5 min hold times at both upper and lower potential.

the lower potential was varied from 0.40 V to 0.60 V while keeping the upper potential constant at 0.95 V.

Sharp spikes in the carbon corrosion rate are observed during a step increase in cell potential during the simulated drive cycle operation, with the magnitude of the spikes decreasing as the upper cell potential is reduced from 0.95 V to 0.60 V. The peak in CO₂ evolution occurs when the cell potential increases from high power operation to low power operation near open circuit voltage (OCV). This correlates with the observation of CO₂ evolution during cyclic voltammograms (CVs), which occurs during the positive sweep at ~0.55 V to 0.60 V. The evolution of this CO₂ peak suggests that oxygen is adsorbed onto the carbon and/or CO is formed on the Pt surface.

The carbon corrosion rate at the upper cell potential (0.95 V) decreases with time at potential, indicating formation of passivating carbon surface oxides; this is indicated by the lower CO₂ evolution during longer durations at the higher potentials. The corrosion rate at lower cell potentials does not decrease with time, consistent with the absence of the oxide passivation layer at the lower potentials. Furthermore, a non-zero corrosion rate is also observed at low cell potentials (0.4 V).

For fixed upper cell potential (0.95 V), the magnitude of spikes decreases as the lower cell potential is raised from 0.4 V to 0.6 V (see Figure 1b). This reducing trend in carbon corrosion as the gap between upper and lower potential is reduced is similar to what was observed in Figure 1a, where the upper potential was reduced. This suggests that the corrosion rate strongly depends on the amplitude of the square potential cycle, not as much on the potential limits in this potential range. Keeping the carbon either in the oxidized or reduced states results both result in lower corrosion rates.

Catalyst support carbon corrosion occurs under normal fuel cell operating conditions and is exacerbated by the voltage cycling inherent in automotive drive cycle operation. Carbon corrosion was measured during the drive cycle measurements for all three types of carbon, with the relative carbon corrosion rates of high surface area > VULCAN® > graphitized. All types of carbon show a similar trend in reduction of carbon corrosion as the potential gap between upper and lower potential is reduced.

The amount of carbon corrosion is greater during drive cycle voltage cycling than at constant potential operation. The potential gap between upper and lower potentials appears to be more important than the absolute operating potentials in the normal operating potential regime (0.40 V to 0.95 V), as changes in the measured carbon corrosion are similar when the upper potential was lower compared to raising the lower potential. Catalyst layer thinning was observed during the simulated drive cycle operation, which had an associated decrease in catalyst layer porosity. This catalyst layer thinning is not due solely to carbon corrosion, although

carbon corrosion likely plays a role; much of this thinning must be from compaction of the material in the catalyst layer. The decrease in catalyst layer porosity leads to additional performance losses due to mass transport losses.

Cerium Migration in Polymer Electrolyte Membranes

Cerium ions enhance the durability of PEM fuel cell components by rapidly and reversibly scavenging degrading radical species which are generated during operation. However, during cell assembly, conditioning, and discharge, these ions migrate between the membrane and the catalyst layers of the membrane electrode assembly (MEA) [1,2]. Cerium migration in PEMs was induced through ex situ experiments and monitored during cell assembly and operation at OCV.

When a 1.5 V potential was applied across a 12.5 mm wide window cell, cerium migrated steadily towards the lower potential over 140.5 hours (Figure 2a). Plotting the change of mean peak location with time yields a migration rate of 0.09 mm/h. Based on these results, potential differences generated during fuel cell operation can cause cerium ions to migrate towards the lower potential. After 140.5 hours at 1.5 V, the potential difference was removed and the membrane was dried in air at room temperature for 24 hours. Figure 2b reveals that the cerium ions did not equilibrate across the cell after 24 hours of drying at 0 V at room temperature and ambient RH. This result suggests that at ambient temperature and RH, cerium ion diffusion is slow relative to its potential-driven mobility.

During long-term, 100% RH operation at OCV, water content gradients generate in-plane cerium concentration variations. At OCV, cerium washout at 30% RH was greater than at 100% RH, presumably due to increased membrane degradation. Side chain attacks may cause the detachment of sulfonic acid groups, which contain exchanged cerium cations. However, cerium washout reduced total MEA cerium inventory by only a projected 1.5% after 5,000 hours of low RH OCV operation. Results demonstrate rapid cerium movement within in the MEA due to electronic potentials and protonic currents, as well as diffusion due to heat and hydration.

Membrane Accelerated Stress Testing

The development of ASTs is a critical step in the evaluation and improvement of PEM fuel cell durability. The availability of validated component ASTs can lead to the accelerated development of materials with improved durability. However, the ASTs must faithfully reproduce the degradation mechanisms of interest while attempting to accelerate the degradation rates, without introducing new degradation modes. Furthermore, the acceleration factors need to be determined accurately with respect to the intended application, to provide manufacturers with a valuable tool

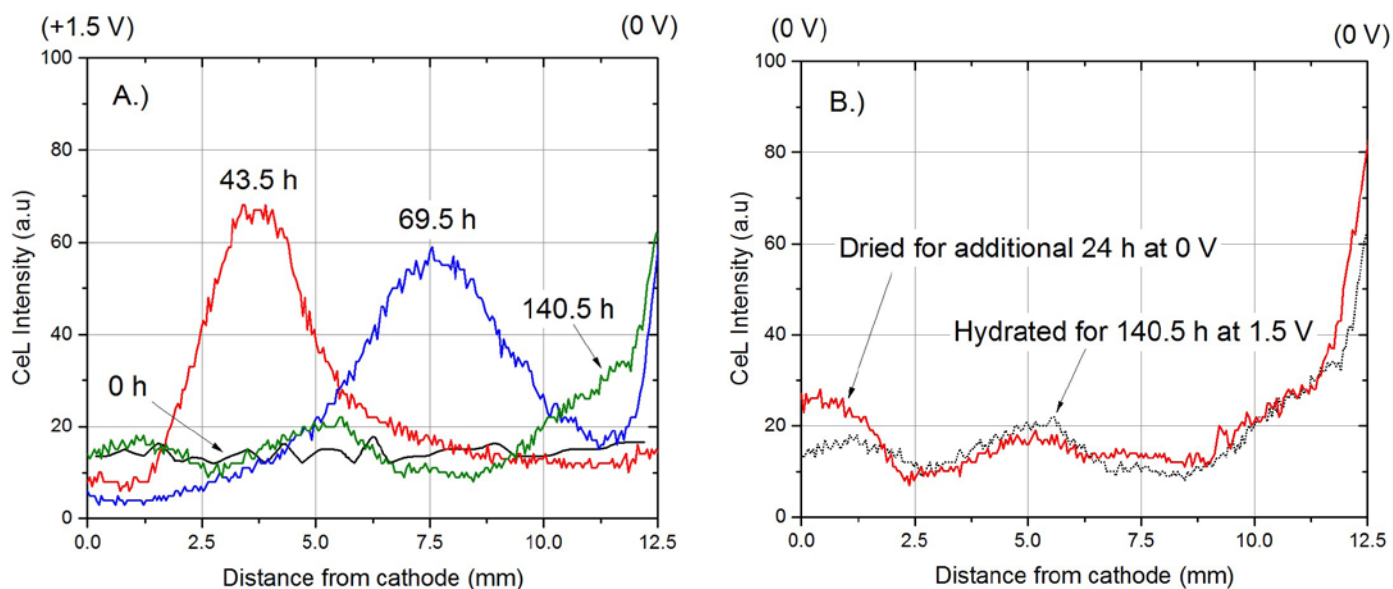


FIGURE 2. Cerium concentration measured over time via X-ray fluorescence in ex situ tests in hydrated Nafion XL[®] membranes at (a) 1.5 V and (b) 0 V

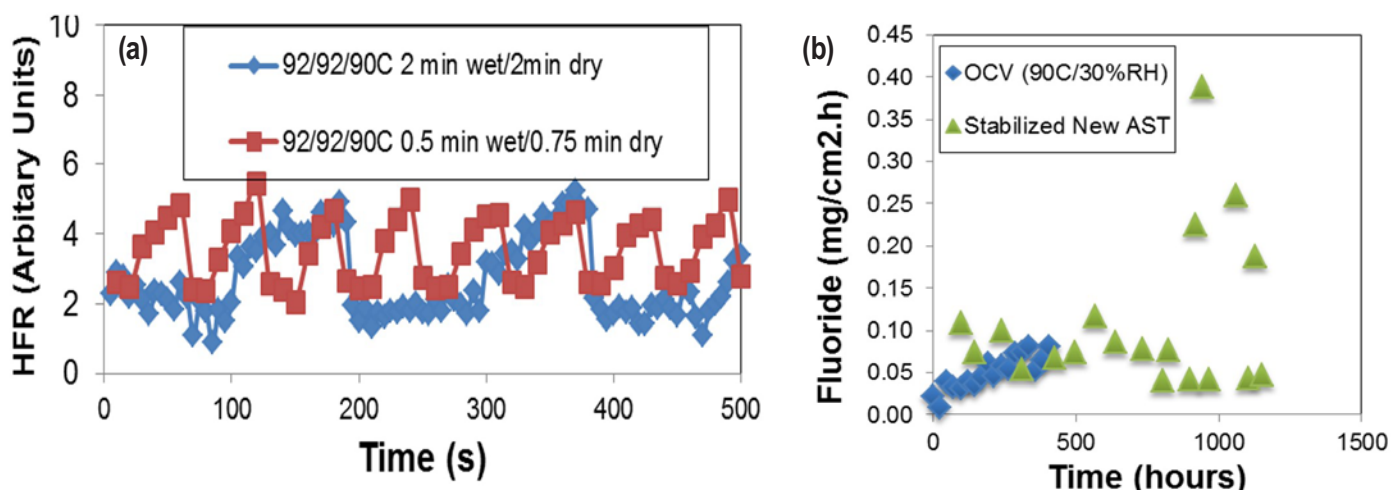


FIGURE 3. (a) High-frequency-resistance measurements during RH cycling comparing 2 minute wet/dry holds and 0.5 min wet/0.75 min dry hold times and (b) a comparison of fluoride emission rate during RH cycling at OCV (new AST) versus OCV hold at constant 30% RH

that enables informed decision making regarding cost, performance, and durability.

The FCTT has two recommended ASTs for membranes, with one focusing on chemical degradation (OCV hold at 90°C and 30% RH) and another focusing on mechanical degradation (RH cycling in air). Our previous results have indicated that both ASTs are not representative of membrane failure in the field [3]. During FY 2015, we developed a new AST that combines mechanical and chemical degradation and is more representative of field failure. This AST consists of cycling the RH between saturated and dry conditions in a H₂/air atmosphere at 90°C. The duration of the wet and dry

cycles are 30 s and 45 s, respectively and results in similar high frequency resistance values as the FCTT recommended 2 minute wet/dry cycles (Figure 3a). The fluoride release rate observed during this AST using DuPont XL[®] membranes, illustrated in Figure 3b, is comparable to the chemical AST and significantly higher than in the mechanical AST. A baseline MEA using an unstabilized membrane sample fails at ≈1,500–2,000 cycles, while a stabilized DuPont XL[®] MEA fails at ≈25,000–30,000 cycles (625 hours). Moreover the failure is primarily due to mechanical failure with some global thinning (chemical degradation) observed during the long term. This AST better reproduces drive cycle failure

and is a significant acceleration over the drive cycle and other membrane ASTs.

Catalyst Accelerated Stress Testing

The FCTT has recommended potential cycling from 0.6 V to 1.0 V at 50 mV/s as the electrocatalyst AST. This potential cycling is performed at 80°C in 100% RH with H₂ (anode) and N₂ (cathode) and results in Pt particle growth and loss in electrochemical surface area (ECSA). This loss in ECSA and associated performance loss correlates well to the degradation observed in the field and during simulated drive cycle operation. Figure 4 compares the degradation observed during this AST (old AST) to that observed during the wet portion of the FCTT Drive Cycle (current cycling from 0.02 A/cm² to 1.2 A/cm² at 80°C and saturated conditions). It is seen that this AST has a 5X acceleration factor for three different MEAs that were evaluated. In order to further accelerate this AST, we modified the triangle wave to a square wave and lowered the upper potential to 0.95 V. This new AST consisted of a square wave with upper and lower potentials of 0.95 V and 0.6 V with 3 seconds duration, and was based on literature reports [4], although a lower upper potential was used to lessen carbon corrosion. This new AST (Figure 4) demonstrated a 100X acceleration factor compared to the drive cycle which is a 20 time improvement over the old AST. Moreover, this new AST retained the Pt growth mechanism while minimizing carbon corrosion.

CONCLUSIONS

We have measured and quantified carbon corrosion during drive cycle operation and studied the effect of varying the upper and lower potential limits used in the drive cycle. Carbon corrosion is observed during the normal operating cycle potentials, although it's significantly less than corrosion induced by start/stop cycles.

Cerium migration in PEMs was induced through ex situ experiments and monitored during cell assembly and operation at OCV. Results demonstrate rapid cerium movement in the MEA due to electronic potentials and protonic currents, as well as diffusion due to heat and hydration.

New ASTs were developed for membranes and catalyst testing. These ASTs have higher acceleration factors and better represent failures observed during drive cycle testing than the current FCTT recommended ASTs.

FUTURE DIRECTIONS

- Ionomer/electrode stability and structural changes
 - Characterize and model performance losses (kinetic and mass transfer) in low-loaded MEAs
 - Effect of ionomer durability in ultra low-loaded catalyst layers
 - Chemical degradation of ionomer (increased radicals)
- Durability of catalysts
 - Alloy catalyst materials
 - Stability of PtCo/C or de-alloyed PtNi/C catalysts in CV, rotating disc electrode and aqueous dissolution apparatus
 - Effect of transitional metals (from alloys)
 - Support stability
 - Quantitative relationship between carbon corrosion and resulting changes in cathode catalyst layer structure (Pt/pore distributions, Pt utilization, ECSA)

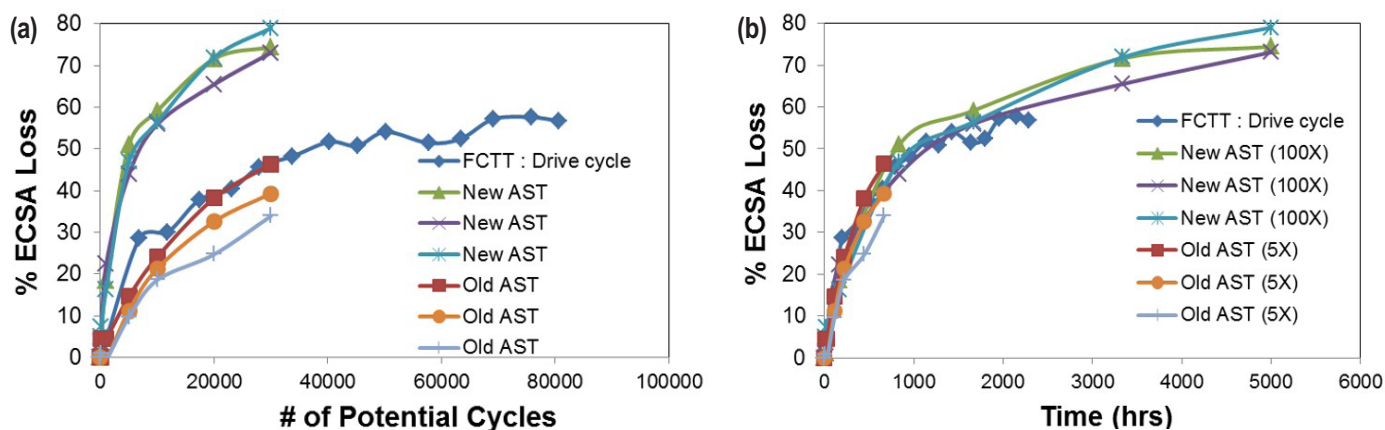


FIGURE 4. Comparison of the degradation rate (ECSA loss) observed during a FCTT drive cycle and two different catalyst ASTs as a function of (a) number of potential cycles and (b) time. Old AST: 0.6 V to 1.0 V @ 50mV/s @ 80°C and 100% RH. New AST: Square wave 0.6 V (3 s), 0.95 V (3 s) @ 80°C and 100% RH.

- Correlate catalyst and support durability
- Accelerated stress testing and protocol development
 - Finalize combined chemical/mechanical AST protocol
 - Compare fast RH cycling with water concentration cycling by induced current profiles
 - Metal bipolar plate in situ AST durability protocol
 - Freeze tolerance MEA protocol
- Benchmark durability status
 - State-of-art catalyst/MEAs in DOE/FCTT durability protocol
 - Un-mitigated SD/SU
 - Metal bipolar plates
 - Durability comparison of ultra-low loadings
- Bipolar plate durability
 - Corrosion of advanced bipolar plate materials
 - Transport of dissolved cations through catalyst ionomer and membrane
 - In situ protocol for AST testing of metal bipolar plates
- Membrane structural/chemical degradation and radical scavenger effect
 - Structural/chemical changes to membranes
 - Including the effect of RH and temperature, and flow rate
 - Crystallinity changes and crystallinity effect on proton and water transport and gas crossover
 - Mechanistic modeling of chemical degradation including effect of ceria
 - Fatigue crack-growth model for interfacial cracks and multiple defects
 - Effect of loading conditions, cell and membrane design
 - Validation with experimental AST data and scanning electron microscopy of failed membranes
 - Combined crossover/pinhole model with chemical-degradation effects
 - Cerium/radical scavenger migration
- Model ceria/cation migration in the membrane with reaction terms
- Measurement of cerium dissolution and migration
- Concentration profile (in situ mapping) during fuel cell operation in both x-y and z directions
 - Define driving forces behind cerium migration (RH, current, potential)
- Mitigation
 - Cerium stabilization and localization
 - Cerium encapsulation and time-release
 - Model mitigation strategies

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SPECIAL RECOGNITIONS & AWARDS/ PATENTS ISSUED

1. **Rod Borup (LANL)** 2015 Research Award by Energy Technology Division of the Electrochemical Society (ECS).
2. **Adam Weber (LBNL)** 2014 Charles W. Tobias Award, 2014 Kavli Fellow of the National Academy Sciences Award.

FY 2015 PUBLICATIONS/PRESENTATIONS

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12. A.M. Baker, D. Torraco, E.J Judge, D. Spornjak, R. Mukundan, R.L. Borup, S.G Advani, and A.K. Prasad, “Cerium Migration during PEM Fuel Cell Assembly and Operation,” submitted to *ECS Transactions*.

Presentations

1. A. Kusoglu, A.Z. Weber, “Synergistic Chemical/Mechanical Degradation of Polymer-Electrolyte Membranes,” MRS Meeting, San Francisco, CA. April 2015.

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14. R.L. Borup, “PEM Fuel Cells: Design and Durability at the Microstructural Level,” *Materials Capability Review: 2014 Materials for the Future*, Los Alamos, 2014.

15. R.L. Borup, “PEM Fuel Cells for Transportation: Design and Durability at the Microstructural Level,” (Invited), *226th ECS Meeting*, Cancun, October 2014.

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