V.A.7 Technical Assistance to Developers

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Project Start Date: October 2003
Project End Date: Project continuation and direction determined annually by DOE

Objectives

- Support technically, as directed by DOE, fuel cell component and system developers
- Assess fuel cell materials and components and give feedback to developers
- Assist the DOE Durability Working Group with the development of various new material durability testing protocols
- Provide support to the U.S. Council for Automotive Research (USCAR) and the USCAR/DOE Fuel Cell Technology Team
- Fiscal Year (FY) 2012 Specific Technical Objectives:
  - Evaluate novel micro-porous layer (MPL) materials
  - Develop of startup/shutdown protocol
  - Test the impact of hydrophobic treatment on graphite bipolar plates
  - Perform complete diagnostics on metal bipolar plates for corrosion
  - Participate and lead efforts in the DOE Working Groups

Technical Barriers

This project addresses the following technical barriers from the Fuel Cells section (3.4.4.2) of the Fuel Technologies Program Multi-Year Research, Development and Demonstration Plan:

(A) Durability
(B) Cost
(C) Performance

FY 2012 Accomplishments

- Evaluation of novel MPL materials showing improvements in mass transport and durability after hydrophilic treatment including the addition of C-nanotubes into the MPL.
- Successful protocol development and completion of startup/shutdown protocol tests.
- Testing of graphite bipolar plate hydrophobic treatment showing better performance than an untreated graphite plate at low current densities (i.e. <500 mA/cm^2).
- Application of several different diagnostics tools to study the titania-coated stainless steel bipolar plates corrosion resistance including fuel cell testing using a drive cycle protocol, alternating current (AC) impedance spectroscopy, and X-ray fluorescence (XRF) elemental imaging. Participation in the DOE Fuel Cell Technical Team, co-chairing DOE Working Group meetings on Durability and Transport Modeling Working; presenting data and leading discussions on protocol development.

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Introduction

This task supports the allowance of technical assistance to fuel cell component and system developers as directed by the DOE. This task includes testing of novel materials and participation in the further development and validation of single-cell test protocols. This task also covers technical assistance to DOE Working Groups, USCAR and the USCAR/DOE Driving Research and Innovation for Vehicle efficiency and Energy sustainability (U.S. DRIVE) Fuel Cell Technology Team. Assistance includes technical validation of new fuel cell materials and methods, single-cell fuel cell testing to support the development of targets and test protocols, and regular advisory participation in other working groups and reviews. This assistance is made available to polymer electrolyte membrane (PEM) fuel cell developers by request and DOE approval.

Approach

The LANL fuel cell team has extensive knowledge and in-house analytical capabilities. These capabilities along with the personnel uniquely allow us to conduct thorough diagnostics and confirm results of existing and novel materials. In FY 2012, several requests were approved by the DOE to be completed under this task. Requests granted were the testing of novel MPL materials, development and testing a new startup/shutdown protocol, investigation of the impact of the hydrophilic treatment of a graphite bipolar plate material, and validation of enhanced corrosion protection of titania-coated stainless steel bipolar plate materials. Detailed highlights of these projects will be further discussed in the following.

Results

In FY 2012, we completed testing, analysis provided feedback to both the collaborator and our DOE managers in four major component areas. Some selected findings were as follows:

- Novel MPL materials were evaluated in a 50-cm^2 fuel cell operating at 80ºC, 100% relative humidity (RH), 50% air utilization and 28.4 psig back pressure. We tested three different cathode gas diffusion layers (GDLs) with varying MPL amounts and different carbon-fiber substrates. In particular, the GDLs were a standard MPL with carbon/Teflon®/binder (25BC), a standard MPL with a hydrophilic treatment (25BL), and a standard MPL with carbon nanotubes (25BN). The findings indicated that GDLs 25BL and 25BN both improved in the mass transport region compared to 25BC, but only 25BN resolved durability issues that surfaced in the others.
- The startup/shutdown protocol was developed in collaboration with Ballard Power and initial tests were conducted. A graphical representation of the start up/shut down protocol is shown in Figure 1.
- Figure 2 shows pictures of a plain bipolar plate (top) and a hydrophobic-treated bipolar plate (bottom). Water

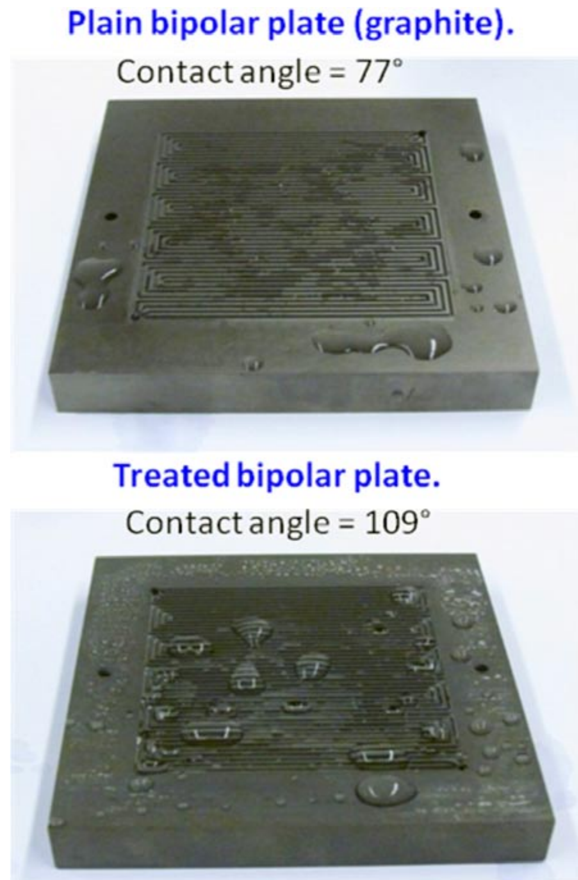

FIGURE 2. Illustration comparing the contact angles of graphite bipolar plates: plain vs. treated

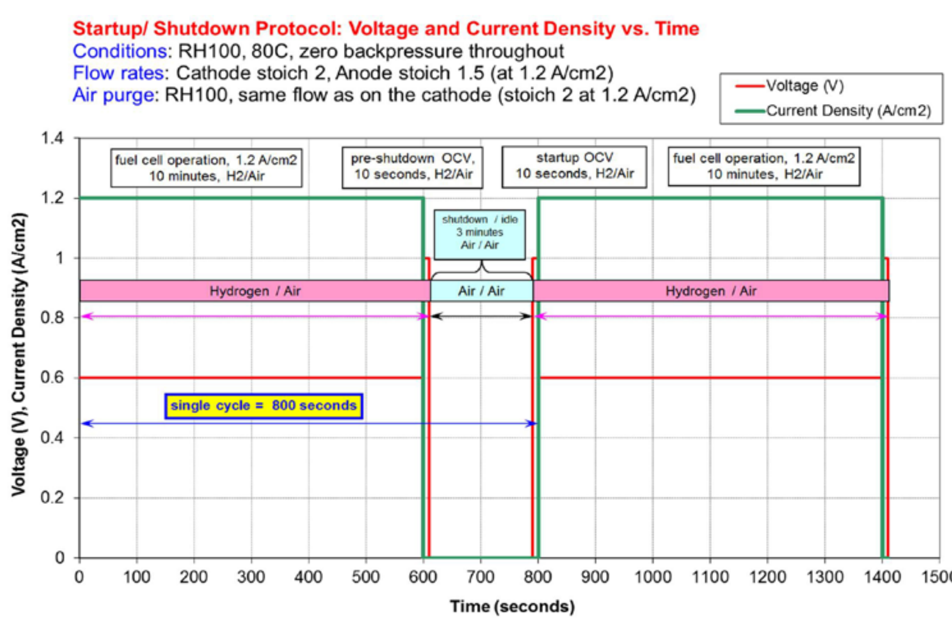

FIGURE 1. Test results using newly developed startup/shutdown protocol

droplet contact angle measurements clearly demonstrated that the treated plate is more hydrophobic. Here we focus on their impact on PEM fuel cell performance when the treated bipolar plates were used on the cathodes. We used identical test materials, varying cathode plate types only, to allow for a direct comparison between the plates. An identical test protocol was performed on both plates. The test protocol included a 2 hr break-in period, voltage-current tests (V-Is), and several full impedance spectra at various current densities using 100 and 25% RH (Figure 3). The V-Is showed at low currents densities (<500 mA/cm^2) the hydrophobic plate performs slightly better, while extensive flooding was observed at the higher currents. In order to further investigate this phenomenon, impedance spectra in the different regions of the V-I were probed. At low current densities (<20 mA/cm^2), the hydrophobic plate keeps the cathode catalyst layer and MEA more hydrated. This results in improvement in high-frequency resistance and decreased catalyst sheet resistance. Product water keeps the catalyst layer hydrated especially at drier inlet RH operation. At higher current densities (>1 A/cm^2), the use of a hydrophobic flow field became a detriment since it led to increased mass transport resistance due to less efficient water removal from the cathode catalyst layer and GDL.

- We conducted a systematic study using several different diagnostics to test coated metal bipolar plates for enhanced corrosion protection. This task was requested after the observation of small discolorations in the metal bipolar plates after they were manufactured and coated and fuel cell tested. Initial speculation was that they were due to galvanic corrosion; however, our X-ray elemental mapping results did not indicate materials losses from corrosion. In fact, no significant change in the elemental composition of the titanium oxide coating or the underlying stainless steel was observed.

  The long-term corrosion resistance of the treated plates still needs confirmation. Laboratory corrosion tests were developed to further characterize the corrosion resistance of the treated plates. However, the uncertainty of this material after being subjected to an aggressive drive cycle conditions in an actual fuel cell remained. There are currently no accelerated stress tests for corrosion testing bipolar plates; however an existing DOE drive-cycle was modified and used in this task. The drive cycle called for 30K cycles going from 1 A to 60 A with a 30 seconds settling time at each current for a total of 500 hours. The fuel cell operates with hydrogen and air fixed flows (669 and 1,773 sccm) at 80ºC and slightly oversaturated humidification conditions and ambient back pressure. We performed beginning-of-test and end-of-test diagnostics for comparisons, which included digital imaging, XRF elemental mapping of plates and MEA, initial and final voltage-current-resistance tests (VIRs), AC impedance and contact resistance measurements. The VIRs behaved similarly for the metal and graph plates. The digital imaging showed visible discoloration for the metal plates, more significant at the anode outlets. These changes were compared with the neutron imaging of a similar plate tested under similar conditions. The location of liquid water imaged by neutron scattering coincided with the regions of discoloration observed on the metal plates. Elemental mapping at the anode outlet show titanium loss from the outer layer. This is depicted in Figure 4. Analyses of bipolar plates (post test) indicates corrosion present on anode plate, typically where large amounts of liquid water were present and minimal corrosion present on cathode plate (but not zero). Analysis of MEAs shows small levels of metal contamination of GDL/MEA which correlates to approximately ~5% to ~14% of the sulfonic acid sites if all of the cations reside inside the membrane; the cationic concentration was also higher where liquid water was present. In addition the contact resistance increased of the cathode plate.

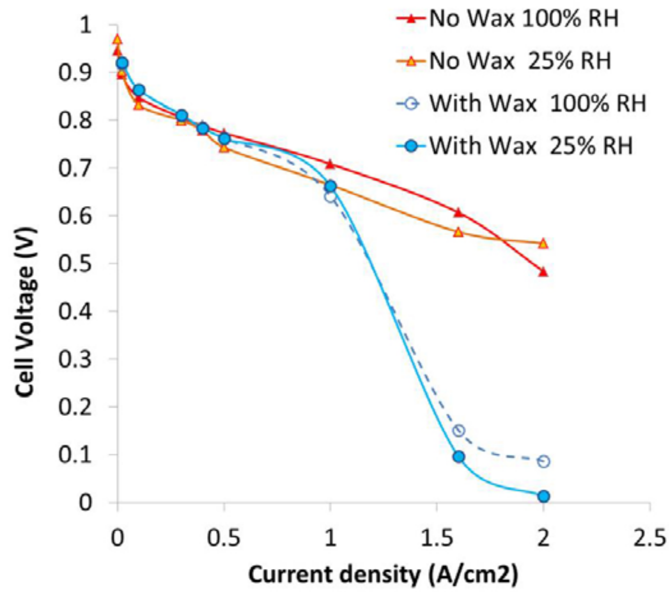

FIGURE 3. Polarization curves measurements from a plain vs. treated bipolar plate operating at 100 and 25% RH

Conclusions and Future Directions

In FY 2012 LANL:

- Completed testing of new novel MPL layers with hydrophilic fibers to analyze the changes in mass transport.
- Interacted with various organizations to discuss the proper protocols for start up/shut down in terms of durability testing (with results presented from the University of Nancy at the AMR).

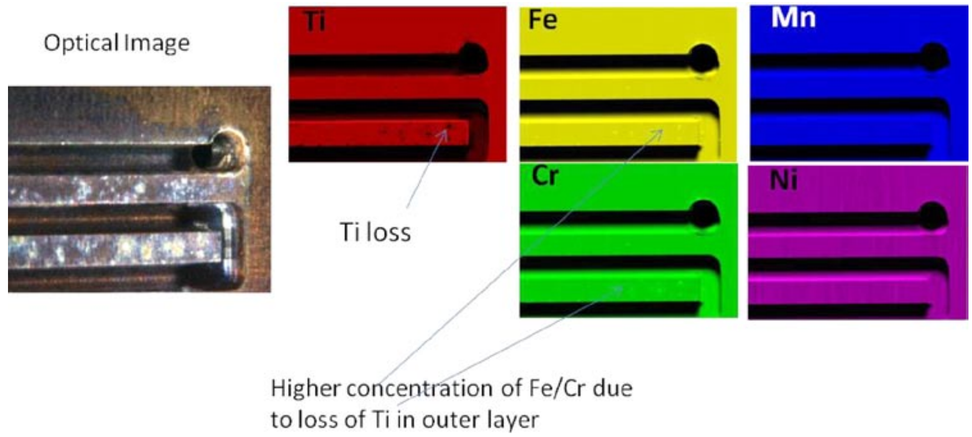

FIGURE 4. Elemental mapping images of metal bipolar plates taken after completing a DOE drive cycle protocol

- Measure the performance of novel hydrophobic bipolar plate flow field coatings.
- Performed characterization on tested metal bipolar plates, and performed in situ testing of metal bipolar and presented these results to DOE and the U.S. DRIVE Fuel Cell Tech Team.
- Provided support for program interaction with DOE; such as the support for the co-chair of the DOE Fuel Cell Technologies Durability Working Group and the co-chair of the DOE Transport Modeling Working Group, and permanent representative to the DOE Fuel Cell Technical Team.

For FY 2013, we will continue to support fuel cell developers as directed by DOE to provide capabilities that exist at LANL not readily available to many developers.