VI.1 Fuel Cell Membrane Electrode Assembly Manufacturing R&D

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Partners

- Lawrence Berkeley National Laboratory (LBNL), Berkeley, CA
- Colorado School of Mines, Golden, CO
- New Jersey Institute of Technology, Newark, NJ
- Automotive Fuel Cell Cooperation, Burnaby, BC
- General Motors, Pontiac, MI
- Ion Power, New Castle, DE
- W.L. Gore and Associates, Elkton, MD

Project Start Date: July 16, 2007 Project End Date: Project continuation and direction determined annually by DOE

Overall Objectives

- Evaluate and develop in-line diagnostics for cell and component quality control and validate diagnostics inline
- Investigate the effects of membrane electrode assembly (MEA) component manufacturing defects on MEA performance and durability to understand the required performance of diagnostic systems and contribute to the basis of knowledge available to functionally determine manufacturing tolerances for these materials
- Use established models to predict the effects of local variations in MEA component properties, and integrate modeling of the operational and design characteristics of diagnostic techniques into the design and configuration of in-line measurement systems
- These objectives have strong support from the industry. Specifically, the outcomes of the 2011 NREL/DOE Hydrogen and Fuel Cell Manufacturing R&D Workshop and the Office of Naval Research-funded Manufacturing Fuel Cell Manhattan Project confirmed the importance of continued development of in-line quality control techniques for cell manufacturing. Our specific development activities have been and will continue to

be fully informed by direct input from industry. As new technologies emerge and as the needs of the industry change, the directions of this project will be adjusted.

Fiscal Year 2014 Objectives

- Implement the infrared/direct-current (IR/DC) diagnostic on a production electrode coating line
- Make a Go/No-Go decision for further work to implement the capacitance technique for electrode ionomer-to-carbon ratio on moving sheet material
- Demonstrate the IR/reactive impinging flow (IR/RIF) technique on the research web-line

Technical Barriers

This project addresses the following technical barriers from the Manufacturing R&D section (3.5) of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan:

- (A) Lack of High-Volume Membrane Electrode Assembly Processes
- (E) Lack of Improved Methods of Final Inspection of MEAs
- (K) Low Levels of Quality Control.

Contribution to Achievement of DOE Manufacturing Milestones

This project contributes to the achievement of the following DOE milestone from the Manufacturing R&D section (3.5) of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan:

• Milestone 6.4: Demonstrate methods to inspect full MEAs and cells prior to assembly into stacks (4Q, 2014).

FY 2014 Accomplishments

NREL accomplished the following in FY 2014:

- Implemented our IR/DC technique on industry partner Ion Power's production electrode coating line and demonstrated successful detection of intentionally created defects and process variability at their process conditions
- Used the coated sheets made during the Ion Power implementation on our research web-line to identify IR/DC excitation conditions for successful detection of electrode defects at speeds up to 60 foot per minute (fpm)
- Demonstrated the sensitivity of our IR/RIF technique to variations in electrode platinum loading and defects

- Performed modeling of the impinging flow dynamics of the IR/RIF technique to help understand the effects of the main process variables and process configuration
- Designed, fabricated, and installed new hardware to enable the demonstration of the IR/RIF technique on our research web-line with gas diffusion electrode (GDE) sheet material
- Demonstrated detection of a variety of membraneelectrode sub-assembly defects, including debris and scratches, using our optical reflectance technique
- Performed detailed studies of the capacitance technique and recommended a No-Go decision to move forward with testing using moving materials
- Continued collaboration with our industry partners, including the last of DOE's competitively awarded Manufacturing R&D projects, in accordance with our project charter
- Led the planning and organization of, and hosted a workshop on in-line quality control for two-dimensional engineered surfaces, which focused on identifying needs and synergies across DOE Office of Energy Efficiency and Renewable Energy technologies (e.g., solar, fuel cells, batteries, building materials, and window films)

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INTRODUCTION

In FY 2005–2007, NREL provided technical support to DOE in developing a new key program activity: manufacturing R&D for hydrogen and fuel cell technologies. This work included a workshop on manufacturing R&D, which gathered inputs on technical challenges and barriers from the fuel cell industry, and subsequent development of a roadmap for manufacturing R&D. In late FY 2007, NREL initiated a project to assist the fuel cell industry in addressing these barriers, initially focusing on in-line quality control of MEA components.

Defects in MEA components differ in type and extent depending on the fabrication process used. The effects of these defects also differ, depending on size, location in the cell relative to the reactant flow-field, cell operating conditions, and which component contains the defect. Understanding the effects of these different kinds of defects is necessary in order to specify and/or develop diagnostic systems with the accuracy and data acquisition/processing rates required for the speed and size scales of high-volume continuous manufacturing methods. Furthermore, predictive capabilities for manufacturers are critical to assist in the development of tolerances and to enable assessment of the effects of material and process changes.

APPROACH

NREL and its partners are addressing the DOE manufacturing milestones listed above by evaluating, developing, and validating (in-line) diagnostics that will support the use of high-volume manufacturing processes for the production of MEAs and MEA component materials. Prioritization of this work is based on inputs from our industry partners on their critical manufacturing quality control needs. We are focusing on diagnostic capabilities not addressed by commercially available in-line systems; in particular we are evaluating methods to make areal rather than point measurements such that discrete defects can be identified. We are also developing test methodologies to study the effects of the size and/or extent of each important type of variability or defect. These results will assist our industry partners in validating manufacturing tolerances for these materials, ultimately reducing scrap rates and cost, and improving supply chain efficiency. Finally, predictive models are being used at LBNL to understand the operational and design characteristics of diagnostic techniques by simulating the behavior of MEA components in different excitation modes. These results are being fed back to our design effort in configuring the diagnostics for in-line implementation. MEA models are also being utilized to understand the in situ behavior of defected MEAs to guide and further elucidate experiments.

RESULTS

As noted in the approach section, our ultimate goal is to transfer in-line inspection techniques to industry. Taking another step in that direction, our major accomplishment over the past year was the implementation of our IR/DC technique on Ion Power's production electrode coating line. We set up our equipment directly on their line and operated it while they coated under standard production conditions. Figure 1 shows an example of the successful demonstration, in this case detection of a thin scratch in the electrode. While all of the defects were intentionally created or allowed to occur, many of the defects were fairly ubiquitous for the coating technique used, for example the "die line" in Figure 1. We then extended the value of this demonstration by taking the coated sheets back to NREL and running them on our research web-line to identify IR/DC excitation conditions (i.e. voltage set-points) for higher line speeds. All of the defects created in the electrode sheets were detected at speeds up to 60 fpm; an example of this is given in Figure 2, where the same defects are detected over a factor of six increase in speed.

Another important effort was advancing our reactive excitation technique for detection of GDE defects closer to a web-line demonstration. Formerly referred to as reactive flow-through, we renamed the technique to RIF (reactive impinging flow) to capture the open-environment conditions



FIGURE 1. IR/DC data from demonstration on Ion Power coating line, showing detection of scratch in electrode surface and die line.



FIGURE 2. IR/DC data from Ion Power coated sheets run on NREL web-line, showing detection of defects over a range of speed.

under which it now operates. Using commercially available GDEs as well as materials fabricated in-house with our automated ultrasonic spraying system, we explored the sensitivity of the technique to platinum loading and different sizes and extents of defects. Figure 3 shows a sample with different sizes of electrode bare spots on the GDE, and the infrared thermographic line data indicating detection of those defects. These studies were performed at speeds up to 30 fpm. In support of these studies, our partners at LBNL developed new steady-state and transient models of the technique. These models are providing insights into the physics of the impinging flow that will help us optimize operating conditions and system configuration. Finally, new hardware was designed, fabricated, and installed in preparation for demonstration of the IR/RIF technique on our research web-line by the end of the fiscal year.



FIGURE 3. Commercial GDE sample with created defects of different size, and detection of those defects using IR/RIF at 30 fpm (2% H_2 and 1% O_2 in N_2 , 1 mm knife-to-GDE gap, 20 slpm flow).

In other work, we performed detailed studies of an exploratory measurement using capacitance (via ex situ alternating current impedance) as an indicator of the electrode ionomer-to-carbon ratio. While some of the results were promising, we were not able to show sufficient sensitivity to small changes in ionomer-to-carbon ratio or acceptable repeatability of the data, which ultimately led to a No-Go decision for further work at this time. We also continued to apply our optical reflectance technique to different MEA and sub-assembly configurations of interest to our industry partners. As many of these partners scale up their manufacturing processes for commercialization (e.g., for automotive fuel cells), optimization of MEA components and configurations leads to new structures on which to validate these techniques. Known defects such as carbon powder debris on surfaces and inadvertent scratches on membranes during handling and assembly were detected.

FUTURE DIRECTIONS

- Apply our toolbox of techniques to industry-relevant MEA constructions and sub-assemblies, particularly focusing on near-term commercialization opportunities such as scale up of automotive fuel cells
- Use modeling and experimental studies to refine and improve the performance of the IR/RIF technique for GDE defect detection

- Continue to use predictive modeling and single and segmented cell test methods to study the effects of asmanufactured defects on MEA performance and lifetime using standard or accelerated stress tests
- Continue to work toward the implementation of more of our techniques on industry production lines

FY 2014 PUBLICATIONS AND PRESENTATIONS

1. Ulsh, M. "Fuel Cell MEA Manufacturing R&D." DOE Hydrogen Program Annual Merit Review, Washington, D.C., June 2014.

2. Das, P.K.; Weber, A.Z.; Bender, G.; Manak, A.; Bittinat, D.; Herring, A.M.; Ulsh, M. "Rapid detection of defects in fuel-cell electrodes using infrared reactive-flow-through technique." *Journal of Power Sources* (261), 2014; pp. 401-411. **3.** Bender, G.; Felt, W.; Ulsh, M. "Detecting and localizing failure points in proton exchange membrane fuel cells using IR thermography." *Journal of Power Sources* (253), 2013; pp. 224-229.

4. Reshetenko, T.V.; St-Pierre, J.; Rocheleau, R. "Effects of local GDL gas permeability variations on spatial PEMFC performance." *Journal of Power Sources* (241), 2013; pp. 597-607.

5. Bittinat, D.C.; Bender, G.; Ulsh, M. "Defect detection in fuel cell gas diffusion electrodes using infrared thermography." ECS Fall Meeting, San Francisco, CA, October, 2013. *ECS Transactions* (58:1), 2013; pp. 495-503.

6. Reshetenko, T.V.; St-Pierre, J.; Artyushkova, K.; Rocheleau, R.; Atanassov, P.; Bender, G.; Ulsh, M. "Multi-analytical study of the PTFE content local variation of the PEMFC gas diffusion layer." *Journal of the Electrochemical Society* (160:11), 2013; pp. F1305-F1315.