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

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### Partners:

- Lawrence Berkeley National Laboratory (LBNL), Berkeley, CA
- DJW Technology, Dublin, OH
- Colorado School of Mines, Golden, CO
- University of Hawaii, Hawaii Natural Energy Institute, Honolulu, HI
- DuPont Fuel Cells, Wilmington, DE
- BASF Fuel Cells, Somerset, NJ
- Arkema Inc., King of Prussia, PA
- 3M, St. Paul, MN

Project Start Date: July 16, 2007

Project End Date: December 31, 2009

### Objectives

NREL and its collaborators are developing capabilities and knowledge related to in-line quality control that will assist manufacturers of membrane electrode assembly (MEA) components in transitioning from low to high-volume manufacturing methods for polymer electrolyte membrane fuel cells.

- Evaluate and develop in-line diagnostics for MEA component quality control, and validate diagnostics in line.
- Investigate the effects of MEA component manufacturing defects on MEA performance and durability.
- Further develop and validate models to predict the effects of local variations in MEA component properties.
- These objectives have strong support from our industry partners. Note that the directions of this project will be adjusted as new technologies emerge and as the needs of the industry change. Accordingly, although the project end date listed is specifically for tasks related to component thickness, we have already broadened our activities

to investigate other critical material properties, in alignment with guidance from our industry partners.

### Technical Barriers

This project addresses the following technical barriers from the Manufacturing section (3.5) of the Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan:

- (A) Lack of High-Volume Membrane Electrode Assembly (MEA) Processes
- (F) Low Levels of Quality Control and Inflexible Processes

### Contribution to Achievement of DOE Manufacturing Milestones

This project will contribute to achieving the following DOE milestones from the Manufacturing section (3.5) of the Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan:

- **Milestone 1:** Develop prototype sensors for quality control of MEA manufacturing. (4Q, 2011)
- **Milestone 2:** Develop continuous in-line measurement for MEA fabrication. (4Q, 2012)
- **Milestone 3:** Demonstrate sensors in pilot-scale applications for manufacturing MEAs. (4Q, 2013)
- **Milestone 4:** Establish models to predict the effect of manufacturing variations on MEA performance. (4Q, 2013)

### Accomplishments

NREL has accomplished the following:

- Established facilities, capabilities, protocols, and methodologies for MEA component manufacturing research and development (R&D).
- Established an industry advisory panel to provide guidance and test materials for the project.
- Held a formal review of project directions with the industry advisory panel in January 2008.
- Completed an evaluation of commercially available diagnostic systems for in-line thickness measurement for their relevance to MEA component materials.
- Completed an evaluation of an NREL-developed optical diagnostic system for use with MEA components.

- After visits and communications with numerous well-known research groups, we initiated a collaboration with the University of Hawaii to develop segmented-cell test hardware to enable spatially resolved MEA performance and durability testing of the effects of manufacturing defects.



## Introduction

In Fiscal Years 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. The project will rely on and use the unique and well-established capabilities in NREL's National Center for Photovoltaics (PV) for developing and transferring diagnostic and process technology to the PV manufacturing industry.

Defects in MEA components will differ in type and extent depending on the fabrication process used. The effects of these defects will also differ, depending on their 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 manufacturing defects is necessary to be able to specify and/or develop diagnostic systems with accuracy and data acquisition/processing rates appropriate to the size and speed scales of high-volume continuous manufacturing methods. Furthermore, predictive capabilities for manufacturers are critical to assist in developing transfer functions, i.e., numerical relationships between measurements and physical properties, and to assess the effects of material and process changes.

## Approach

NREL and its partners are addressing the Manufacturing section milestones by establishing capabilities to measure and predict the effects of manufacturing defects in MEA components and by using that knowledge base to evaluate, develop, and validate in-line diagnostics for high-volume fabrication of MEA components. Diagnostic development is focused (at least initially) on the material properties found to be most critical by component manufacturers and on measurement capabilities not available commercially. The NREL-developed optical diagnostic will be a

platform for investigating in-line measurement of many of these critical material properties. These directions will be modified as test hardware and methods are developed to enable us to measure the impact that specific defects in each MEA component have on the performance and durability of MEAs. The developed diagnostics will be validated in a continuous fashion, using a research web-line to provide moving media, simulating an actual manufacturing line. Test data will be fed to LBNL, where researchers will validate existing models and make further refinements based on continued input from our industry partners.

## Results

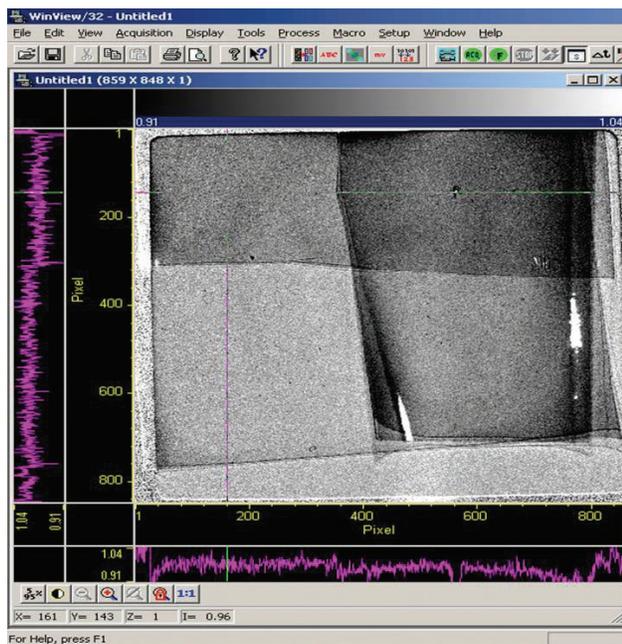
We completed a preliminary evaluation of the applicability of commercially available in-line measurement systems to MEA component materials. Suppliers evaluated small samples of a variety of MEA components, e.g., membranes, catalyst-coated membranes, gas diffusion layer media, and gas diffusion electrodes—having a range of material properties, when possible. The evaluation focused on component thickness as the basic characteristic of materials made as thin films or sheets—like MEA components—and on which the uniformity of many other material properties depend. For each component, at least one system was identified that could measure thickness. Not unexpectedly, though, we identified very few systems with the potential to measure other material properties in line. Table 1 summarizes the findings of this evaluation.

**TABLE 1.** Summary of Preliminary Investigation of Thickness Measurement Devices

Technique	Vendor	Status	Applicability
Optical/IR Reflection	Lumetrics, Infocus	Complete	Membrane
Laser Triangulation	Infocus	Complete	GDE, GDL, possibly CCM
Low-force Contact	Infocus	Complete	All, but in-line implementation may be difficult
Beta- /Gamma- / X-ray	Thermo Fisher, NDC, Mahlo	Need larger samples	Possibly all
X-ray Fluorescence	Fischer, Spectro	Complete	Catalyst, possibly membrane
Capacitance	Infocus	Complete	Questionable, but could measure conductance

IR - infrared  
 GDE - gas diffusion electrode  
 GDL - gas diffusion layer  
 CCM - catalyst coated membrane

We completed an initial investigation of the applicability of an NREL-developed optical reflectometer for use as an in-line diagnostic for MEA components. This system was developed for—and is being used in—continuous manufacturing of solar cells. Optical characterization of a sample set similar to that mentioned above was performed to identify what material properties of each MEA component were potentially measurable by the device. We identified numerous material properties across all of the MEA components. Follow-on development work to date has focused on membrane properties. We have demonstrated simultaneous measurements indicative of membrane thickness and composition. If proven, this function would enable membrane manufacturers to measure two material properties that relate directly to process variables, and to use them for in-line process feedback. We have also completed a feasibility assessment of the use of this device for two-dimensional imaging of membrane thickness. This function is well developed for measuring thin films on silicon wafers, and initial work looks promising for transferring this capability to membranes. Figure 1 shows an image of multiple layers of 1-mil membrane using the device. We are acquiring a new detector and optical hardware



**FIGURE 1.** Two-Dimensional Image of Membrane Thickness (Overlapping layers of a 1-mil membrane are shown. The lower left corner has a single layer, the adjacent corners have two layers, and the upper right corner has three layers. The data graphs along the left side and bottom of the image indicate the measured signal along the pink and green lines in the image, e.g., the graph along the bottom shows the signal along the green horizontal line that traverses the two-layer and three-layer (top) portion of the sample area. Total sample area is about 16 in<sup>2</sup>. The data shown are qualitative.)

that will enable a quantitative assessment of the measurement and development of a transfer function for thickness.

## Conclusions and Future Directions

Our conclusions include the following:

- Continued support and guidance from our industry partners is critical.
- Existing in-line diagnostic systems typically provide for point measurements and, in some cases, are relatively slow in data acquisition and signal processing rates. As much as possible, the diagnostic development aspect of this project should focus on addressing these potential limitations.
- Very few existing in-line diagnostic systems measure material properties—beyond thickness—of critical interest for MEA component manufacturing. This should be another focus of this project.
- The NREL-developed optical reflectometer has the potential to measure many different MEA component material properties and to provide area, rather than point, measurements of some properties. Industry has shown strong interest in this capability.

Future directions include the following:

- Install and commission the research web-line for in-line validation of diagnostic systems.
- Investigate the use of the optical reflectometer for measuring various material properties, including catalyst loading, porosity, and surface structure.
- Modify the configuration of the optical reflectometer for measuring wide webs of thin-film or sheet materials, i.e., coated and uncoated membranes and media for gas diffusion layers.
- Develop fuel cell test hardware and methods for critical defect analysis.
- Study and validate other diagnostics of interest to our partners and collaborators, including existing diagnostics, e.g., X-ray fluorescence for measuring Pt content.
- Use material characterization and fuel cell test data to further refine the LBNL model.

## FY 2008 Publications/Presentations

1. Fuel Cell Manufacturing R&D: Preliminary Assessment of Measurement Techniques, report to DOE, September 2007.
2. DOE Hydrogen Program Annual Merit Review, oral presentation, June 2008.