VI.6 In-line Quality Control of PEM Materials

Paul Yelvington (Primary Contact), Andrew Wagner
Mainstream Engineering
200 Yellow Place
Rockledge, FL 32955
Phone: (321) 631-3550
Email: pyelvington@mainstream-engr.com

DOE Managers:
Nancy L. Garland
Phone: (202) 586-5673
Email: Nancy.Garland@ee.doe.gov
Jesse Adams
Phone: (720) 356-1421
Email: Jesse.Adams@ee.doe.gov

Contract Number: DE-SC0013774

Subcontractors:
- National Renewable Energy Laboratory, Golden, CO
- Professor Tequila Harris, Georgia Institute of Technology Atlanta, GA

Project Start Date: Phase I: June 8, 2015, Phase II: August 1, 2016
Project End Date: Phase I: March 7, 2016, Phase II: July 31, 2018

Overall Objectives

- Identify membrane defect size that leads to cell failure.
- Create a production-intent prototype automated vision system to perform quality control and demonstrate it on a full-speed membrane web line.
- Detect defects down to 4 µm at 100 ft/min.
- Determine membrane thickness to 0.5 µm resolution.
- Achieve a 5σ false-positive and false-negative rate.

Fiscal Year (FY) 2017 Objectives

- Create defective membrane and identify defect size that leads to cell failure.
- Create calibration samples to identify the smallest detectable defect.
- Bring new hardware online and fully automate software for membrane thickness mapping and defect identification.
- Scan the membrane with 100% coverage, marking and logging defective regions.

Technical Barriers

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

(E) Lack of Improved Methods of Final Inspection of Membrane Electrode Assemblies (MEAs)

(H) Low Levels of Quality Control

Contribution to Achievement of DOE Manufacturing R&D Milestones

This project will contribute to achievement of the following DOE milestones from the Manufacturing R&D section of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan.

- Milestone 1.3: Develop continuous MEA manufacturing processes that increase throughput and efficiency and decrease complexity and waste. (4Q, 2017)
- Milestone 1.6: Develop fabrication and assembly processes for PEMFC MEA components leading to an automotive fuel cell stack that costs $20/kW. (4Q, 2020)
- Milestone 5.2: Demonstrate improved sensitivity, resolution, and/or detection rate for MEA inspection methods. (4Q, 2016)
- Milestone 5.4: Design and commercialize an in-line quality control device for PEMFC MEA materials based on National Renewable Energy Laboratory's (NREL) optical reflectance technology. (4Q, 2017)
- Milestone 5.6: Demonstrate methods to inspect full MEAs and cells for defects prior to assembly into stacks in a production environment. (4Q, 2018)
- Milestone 5.8: Implement demonstrated in-line quality-control techniques on pilot or production lines at PEMFC MEA material manufacturers. (4Q, 2020)

FY 2017 Accomplishments

- Defective membranes have been made for testing and calibration that include bubbles and 5 µm, 10 µm, and 25 µm pinholes.
- NREL and Georgia Tech have tested defective samples and preliminary results indicate that defects less than

10 µm have no immediate effect on performance, while larger than 300 µm cause decreased performance.

- Software development is complete, hardware has been selected, and packaging has begun.
- A static membrane test station was made for repeatable measurements of membrane sheets.
- Mainstream's small-scale (6-inch web width) winder/unwinder was improved to run at 100 ft/min.

✧ ✧ ✧ ✧ ✧

INTRODUCTION

Fuel cells stand on the cusp of commercialization for large-scale applications such as zero-pollution automotive systems. They are held back by high manufacturing costs and expensive catalysts. The membrane alone accounts for as much as 45% of the total material cost of a commercial fuel cell system [1]. Moreover, manufacturing defects in the membrane not only lead to wasted expensive materials but they also cause cell failures that can cascade into complete stack failure. This requires additional labor to rework the stack as well as results in the loss of expensive catalyst and gas diffusion electrode materials. Current inspection methods look for defects after batch production of the membrane, leading to delayed correction of issues with the membrane and membrane electrode fabrication process. Reaching the quality targets for fuel cell system manufacturing requires a new, high-efficiency, real-time quality control system. Mainstream Engineering is developing a real-time optical quality control system that provides significant benefits with increased resolution, improved accuracy, and increased detection speeds for the examination of fuel cell and other membranes.

APPROACH

Mainstream's overall approach to create a prototype automated vision system for quality control was to rigorously prove out the patent-pending optical technique with a wide-range of commercially available membranes, select upgraded hardware for Nafion®, prove out the defect detection abilities of the hardware, automate the software, and build the prototype. A wide range of typical defects were induced and examined in the Nafion® membranes and characterized with Mainstream's machine vision system. Pinholes down to 5 µm were the focus during the first year and the limits of the upgraded hardware were determined. Then the software used for defect detection was fully automated to image the membrane, detect defects, log the defects, track the defect location, and print an identifying marker beside the defect. The software and hardware were validated on Mainstream's 6-inch web-line at speeds up to 100 ft/min. During this time, NREL and Georgia Tech created defective membrane samples and tested them to determine the smallest defect that impacts cell performance. These results are guiding our determination of an appropriate limit of detection for detect size.

RESULTS

Created Controlled Membrane Defects

Mainstream obtained a variety of Nafion® samples with controlled defects of varying sizes, including bubbles and pinholes, for equipment optimization and validation. Georgia Tech produced membrane sheet materials with a range of thicknesses and a variety of as-cast defects. These membranes were created under several off-design coating conditions that represent real, non-reinforced, polymer electrolyte membrane (PEM) membrane materials. In general, the as-cast defects produced were on the order of several hundred microns in size and were mainly bubbles and color defects. In addition, Georgia Tech created Nafion® samples with pinhole defects of 10, 50, and 100 microns using electron-beam lithography. Mainstream also created calibration samples using laser drilling to make a set of membrane sheets consisting of Nafion-115®, Nafion-211®, and Nafion-HP® that were laser drilled to create a 12 in × 12 in array of 5 µm, 10 µm, and 25 µm holes. A schematic of the sheet and microscope image of a nominally 5 mm laser machined hole are shown in Figure 1.

Analyzed Membrane Defects for Impact on Cell Performance

The pinhole and bubble defect samples were characterized, and in situ performance testing was conducted. The defective membranes were characterized with optical microscopy, assembled in cells, tested in NREL's segmented cell system for spatially resolved initial performance, and then tested for spatial hydrogen crossover with NREL's infrared cell hardware. Two examples of this series of testing are given in Figure 2 for a pinhole and a surface cut, in which the local performance impact (center images) correlates with the known location of the defect in the cell and with increased hydrogen crossover (right images). Samples with very small pinholes were also tested, but the cells did not show any local or total cell initial performance impact or increased hydrogen crossover. These preliminary results indicate that defects smaller than 10 µm have no immediate impact on cell performance, while defects larger than 300 µm immediately degrade cell performance.

Developed and Tested Upgraded Hardware and Fully Automated Software

Mainstream conducted a trade study of performance and cost (e.g., line speed, limits of detection) of over 30 cameras, 50 lenses, 10 polarizers, 10 machine vision lights, three software platforms, and four data transfer protocols alone and

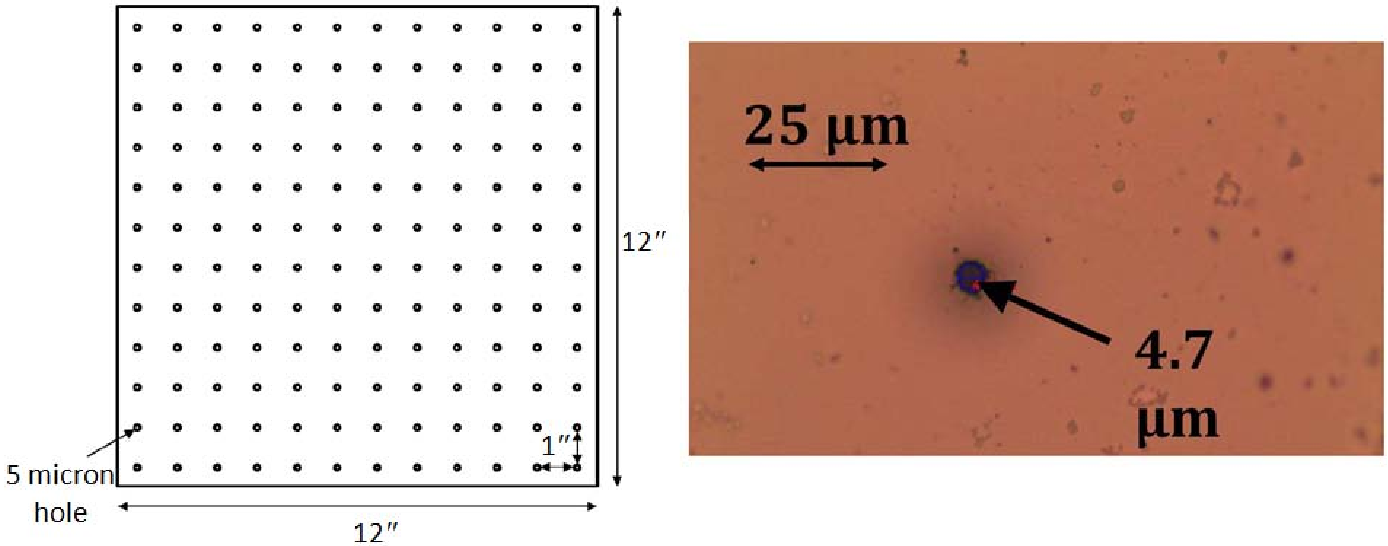

FIGURE 1. Schematic of a membrane calibration sheet and a microscope image of a laser machined hole

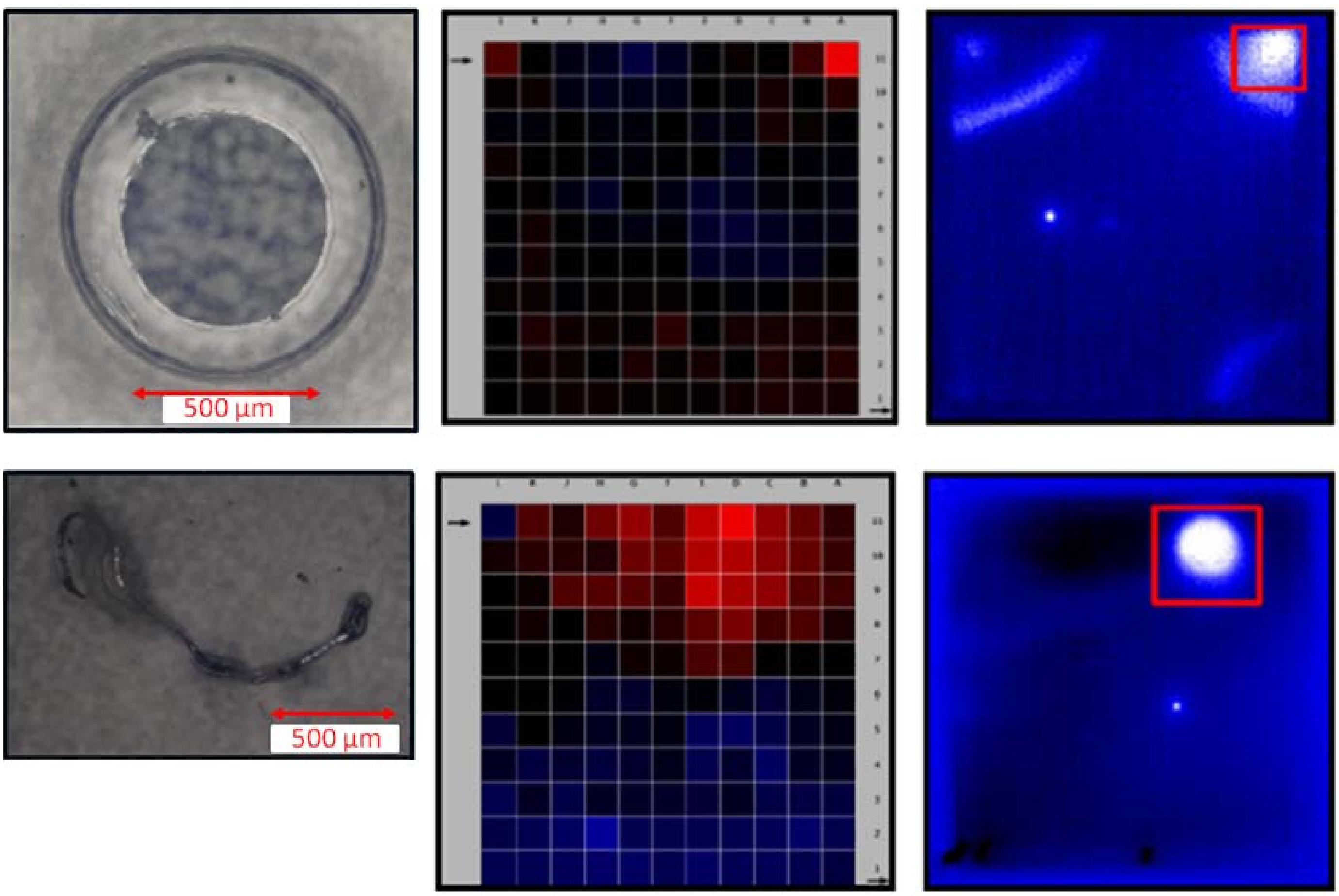

FIGURE 2. Optical microscopy, spatial performance, and spatial hydrogen crossover imaging of two as-cast membrane defects

working as a system. The best equipment within initial cost targets was procured and assembled into an upgraded Phase II system shown in Figure 3. An alpha release of a LabVIEW program for detecting defects in fuel cell membranes was then completed.

The LabVIEW program contains three basic loops, each of which perform different functions and run with different priority levels. The basic architecture is shown in Figure 4. Images are acquired by the camera and pre-processed in the Acquisition Loop. The images are then placed in a buffer. The Field-Programmable Gate Array (FPGA) Loop performs additional processing on the images as well as interfaces with the encoder and printer. The image is sent back to the controller central processing unit, and the machine vision algorithms are performed in the Processing Loop. This loop also performs all image saving and data recording functions, as well as graphical user interface updates. The Acquisition Loop provides an interface between the camera and the software program. Images are acquired at speeds up to 16 frames/s and are pre-processed before being sent to the buffer. The user can select many different camera settings such as scaling, frame rate, image size, exposure time, and gain. The Acquisition Loop also performs adjustments to the brightness, contrast, and gamma of the image, as well as selecting the region of interest. After the image is sent to the buffer, the FPGA Loop reads the oldest image in the buffer. Typically, the buffer never has more than two images at steady-state operation since the Acquisition Loop is acquiring and storing a new image while the previous image is being processed. On the FPGA, the image is deconstructed into a pixel stream, and up to eight pixels are processed at a time via parallel loops. Most of the machine vision algorithm is implemented on the FPGA. The FPGA Loop also interfaces with the encoder and printer. The Image Processing Loop receives the image from the FPGA and performs all the image analysis and recording functions. Once processing of an image is complete, the Image Processing Loop records all relevant data. The combined software and hardware was tested on Mainstream's 6-in web line and operated successfully up to the maximum line speed of 100 ft/min.

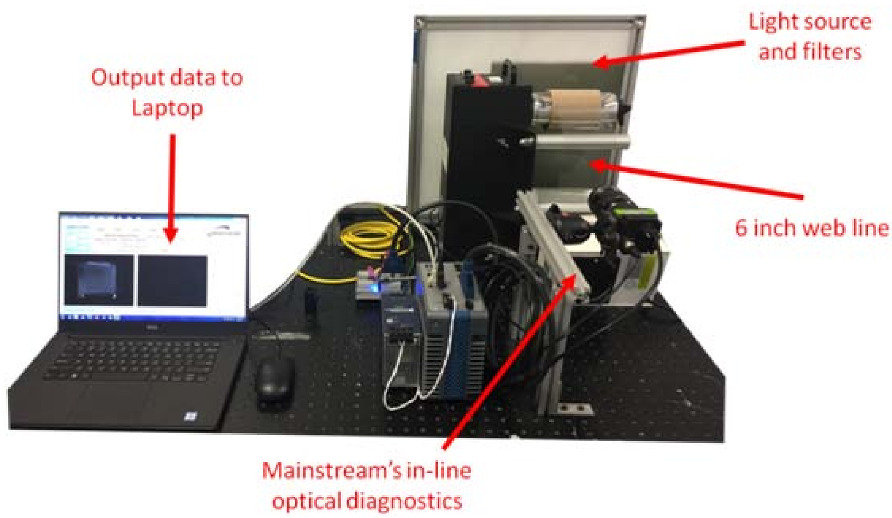

FIGURE 3. Small-scale winder/unwinder inspection station with photosynthetic electron transport film

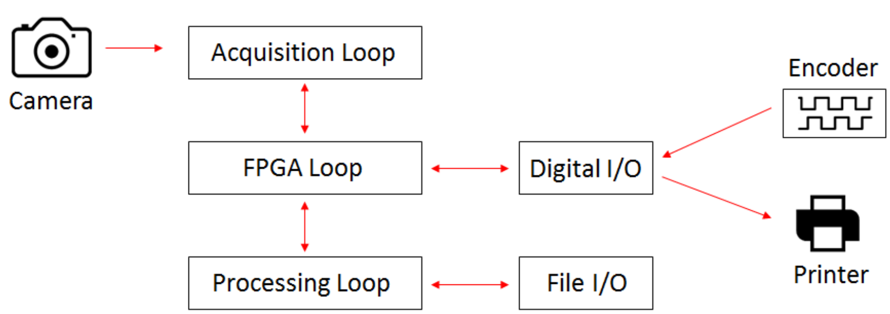

FIGURE 4. Image processing software architecture

CONCLUSIONS AND UPCOMING ACTIVITIES

The overall goal of the Phase II program is to research, develop, and commercialize an in-line quality control system for roll-to-roll membrane manufacturing. Mainstream Engineering developed a low-cost, real-time optical detector for quality control using continuous analysis of membranes for polymer electrolyte membrane fuel cell MEAs. The inspection system samples, logs, and marks every location on the roll of material such that defects in MEA materials can be removed prior to assembly into complete cells. An upgraded prototype system was built, and an initial version of the fully automated software was developed. The system was tested on Mainstream's 6-in web line at speeds up to 100 ft/min. The device will identify and mark defects as well as monitor membrane thickness in real time to improve line efficiency and to reduce waste. For FY 2018, the main goals will be to produce new defective membranes for use in determining defect size that leads to cell failure, to package a prototype system for a 24-in web, to validate the system, and to demonstrate it on multiple web lines including on-site manufacturing demonstrations.

SPECIAL RECOGNITIONS & AWARDS/ PATENTS ISSUED

1. Invention Disclosure Attorney Docket No. 62988US titled, "Apparatus and Method for Cross-polarized, Optical Detection of Polymer Film Thickness and Defects Using Polarimetric Thickness Mapping."

FY 2017 PUBLICATIONS/PRESENTATIONS

1. Wagner, A., Lasko, T., Yelvington, P.E., "In-line Quality Control of PEM Materials," presented, DOE 2017 Annual Merit Review, Washington, D.C., June 2017.

REFERENCES

1. Kleen, G.J., "Membrane Development in the U. S. DOE Fuel Cell Technologies Program," Fuel Cell Seminar and Exhibition, Orlando, FL, 2011.