
In-Line Quality Control of Polymer Electrolyte Membrane Materials

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Subcontractors:

- National Renewable Energy Laboratory, Golden, CO
- Georgia Tech, Atlanta, GA

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Project End Date: Phase I and II: July 31, 2018,
Phase IIb: August 26, 2020

Overall Objectives (Phase I/II)

- 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 microns at 100 ft/min.
- Determine membrane thickness to 0.5-micron resolution.
- Achieve a 5σ false-positive rate (FPR) and false-negative rate (FNR).

Fiscal Year (FY) 2018 Objectives

- Determine impact of line speed on detection rate and autonomous software operation.
- Demonstrate prototype on a full-speed membrane web line and a coating line.

- Conduct web-line runs to determine FPRs and FNRs.
- Identify final image analysis parameters for defect and thickness resolution.

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 (FCTO MYRDD) Plan¹:

- Lack of Improved Methods of Final Inspection of Membrane Electrode Assemblies (MEAs)
- 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 FCTO MYRDD 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 polymer electrolyte membrane fuel cell (PEMFC) membrane electrode assembly (MEA) components leading to an automotive fuel cell stack that costs \$20/kW at high volume (500,000 units/year). (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 the National Renewable

¹ <https://www.energy.gov/eere/fuelcells/downloads/fuel-cell-technologies-office-multi-year-research-development-and-22>

Energy Laboratory's (NREL's) 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 2018 Accomplishments

- The smallest detectable defect was identified as 10 microns in Nafion-211 and Nafion-HP.
- Mainstream brought online an inspection winder that could run up to 550 ft/min.
- The inspection system was tested and demonstrated operating in real time on two coating web lines up to 10 ft/min and on two inspection web lines up to 550 ft/min.
- FPRs and FNRs were achieved at 4σ under all conditions for defects 250 microns and larger, and at 5σ at 30 ft/min with a 127-micron-thick Nafion-115 membrane

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 at 1,000 units/year and 12% at 500,000/year [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 and 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, which we call the Mantis Eye, that provides increased resolution, improved accuracy, and increased detection speeds over existing optical scanners for the examination of fuel cell and other membranes.

APPROACH

Mainstream's overall technical approach to create the Mantis Eye prototype was to rigorously prove out the patent-pending optical technique with a wide range of commercially available membranes, upgrade to imaging hardware tailored 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 the machine vision system. Pinholes down to 5 microns were the focus during the first year and the limits of detection were determined for the upgraded hardware. During this time, NREL and Georgia Tech created defective membrane samples and tested them to determine the smallest defect that impacts cell performance. Then the custom vision software 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 machine vision software was programmed on an industrial microcontroller. The system was then deployed and tested on multiple web lines and coating lines to demonstrate its capabilities and identify areas needing refinement. Many defects including pinholes, scratches, folds, ribbing, and streaking were identified on the rollstocks. The Mantis Eye prototype was tested with freshly cast membrane and on a custom inspection winder at speeds up to 550 ft/min.

RESULTS

Developed and Tested High-speed Inspection Winder

The Mantis Eye optical scanner was originally developed with static samples and a small 3-inch web line in Phase I. Operation on the small web line showed that tension control and web steering are critical in web converting equipment, especially for the quality-control section. To achieve the best web imaging, the web needs to be free of wrinkles and sagging, have equal tension across the web width, and run straight to maintain proper camera alignment and eliminate roll telescoping. Mainstream had a custom winder/unwinder built to our specifications that is capable of bidirectional winding and unwinding a 14-inch membrane roll thousands of feet long (depending on thickness) at speeds up to 550 linear ft/min. In Figure 1, the inspection winder is shown with the Mantis Eye mounted on it. The winder uses active tension control to maintain proper tension and avoid any of the aforementioned web-line issues. The machine includes a removable splice table and a pneumatically actuated, backlit inspection platform. It is capable of maintaining tension and winding in the forward and reverse directions. The main plate has provisions for mounting the Mantis Eye camera, encoder, printer, and machine vision light. In addition, the web material passes extremely close to the light source. This provides the best possible vision conditions and allows for imaging at a realistic line speed. Using this inspection machine, detailed statistics were generated to allow calculation of the FPR and FNR. For dust control, the machine was installed in a soft-walled ISO Class 7 clean room.



Figure 1. Mainstream's Mantis Eye prototype detector installed on Mainstream's inspection winder

The Mantis Eye system was tested on multiple materials on the inspection winder with a focus on polyethylene terephthalate (PET) film due to its availability in long rolls and ease of handling compared to Nafion. The field of view, line speed, and vision algorithms were tested to optimize system performance and defect resolution as well as determine reliability statistics. The system successfully operated in real time and marked 100- μm defects at up to 550 ft/min. As the speed increased beyond 300 ft/min, the camera's exposure time began to limit defect detection as motion blur caused the defects to cover multiple pixels per exposure. This effect is seen in Figure 2; when the line speed exceeded 300 ft/min, the defect appears dimmer and larger. Thus the camera hardware became the limiting element at high line speeds. If speeds above 300 ft/min are required, cameras capable of shorter exposure times or multiple cameras can be used to prevent motion blur. The defect logging and printer were able to operate up to 550 ft/min with no issues. Reliability test rolls of PET and Nafion were made with known defects of different sizes and were run on the inspection winder.



Figure 2. Pinhole defect images in PET taken with the Mantis Eye detector at different speeds

Reliability Statistics

The FPR and FNR were determined by inducing a wide range of defects across a roll of material and scanning it with the Mantis Eye. A 1,000-ft roll had defects induced in a 100-ft test section. Zero to three pinhole defects that were 250, 500, or 640 microns in diameter were randomly induced every 4 inches. A key was made for the location and size of the defects. The roll was then run for the entire length and the location and number of defects logged. The run was repeated multiple times with the camera set up to image a full 14-inch-wide web. A data set was generated and analyzed to find the optimized image processing and logging settings. For these runs, a no-defect negative condition was defined as one inch of web with no defects assuming that is the minimum practical length that could be removed if a defect was found. The FPR was then defined as the number of incorrect positive defect marks (“false alarms”) for every inch of web divided by the total number of inches of web without a defect. The FNR was defined as the number of incorrect negative marks (“misses”) for every inch of web divided by the total number of inches of web with a defect. Using this information, the FPR and the FNR were identified. For PET at 100 ft/min, the FPR and FNR hit 4σ levels (0.62%) for 250–640-micron defects. For Nafion at 30 ft/min, the FPR and FNR hit 5σ levels (0.023%) for 100-micron defects. The FPR and FNR depend on algorithm parameters, such as the brightness/size thresholds or requirements for multiple image captures that can be optimized based on a cost function that appropriately weights false positives (false alarms) or false negatives (misses) for the specific manufacturing process.

Coating Line Deployment and Testing

The prototype Mantis Eye system was installed on Georgia Tech’s lab-scale coater and NREL’s pilot coating line. On Georgia Tech’s system, Nafion was coated at up to 10 ft/min with observed defects including ribbing, streaking, pinholes, blobbing, and dust. The optical scanner was able to accurately observe defects as they were generated, but at the time of the Georgia Tech deployment these defects had not been observed previously by the team and were not part of the real-time processing capabilities. The software was upgraded to identify these larger defects and not just small pinholes. In addition, multiple edge-detection features were added to determine spots and streaking. The deployment onto NREL’s line allowed for the Nafion web to be dried before or after the inspection location and is shown in Figure 3. This allowed for images of freshly cast Nafion both wet and dry. In addition, the NREL deployment had the camera underneath the web, imaging through the PET, which was the first time it was deployed in this manner. Freshly cast, defective membrane is shown before and after image processing in Figure 4. The streaking and ribbing were successfully outlined and identified.

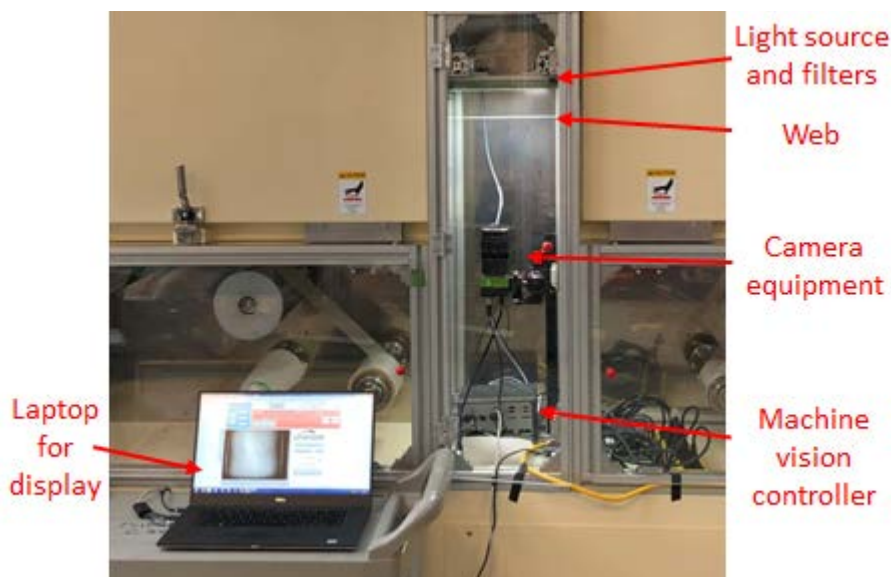


Figure 3. Mainstream's Mantis Eye prototype on NREL's pilot coating line

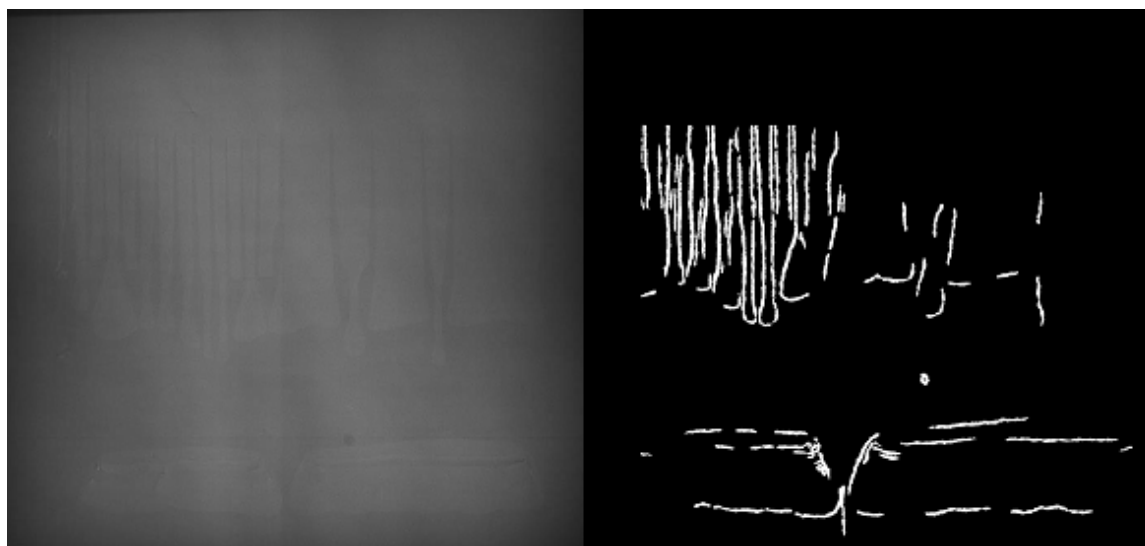


Figure 4. Raw image (left) and processed image (right) from Mantis Eye scanner on freshly cast Nafion membrane with streaking defects

CONCLUSIONS AND UPCOMING ACTIVITIES

The overall goal of the Phase II program was to research, develop, and commercialize an in-line quality control system for roll-to-roll membrane manufacturing. Mainstream Engineering developed the Mantis Eye inspection system, a low-cost, real-time optical detector for quality control using continuous analysis of membranes for PEMFC MEAs. The inspection system samples, logs, and marks the location on the roll of material such that defects in MEA materials can be addressed prior to assembly into complete cells. A prototype system was developed and tested on Mainstream's inspection winder, NREL's research web line, Georgia Tech's research coating line, and NREL's pilot-scale coating line. These deployments allowed for optimization of the software to detect both small and large defects. In addition, NREL determined that pinhole defects smaller than 10 microns caused no immediate cell performance issues, while defects larger than 50 microns did. For the Phase IIb project, the overall goal is to develop and commercialize a suite of instruments that provides a full turnkey inspection package for MEA production, including analysis of catalyst-coated

membrane and other opaque materials. For FY 2019, the goal will be to transition reflectance technology from NREL and use it on a web line at full production speed for MEA material quality control.

SPECIAL RECOGNITIONS AND AWARDS/PATENTS ISSUED

1. P.E. Yelvington and A. Wagner, “Method and Apparatus for Cross-Polarized, Optical Detection of Polymer Film Thickness and Defects Using Polarimetric Thickness Mapping,” U.S. Patent Application Serial No. 15/894,172 (2018).

FY 2018 PUBLICATIONS/PRESENTATIONS

1. A. Wagner, T. Lasko, and P.E. Yelvington, “In-Line Quality Control of PEM Materials,” presented at the DOE Hydrogen and Fuel Cells Program 2018 Annual Merit Review and Peer Evaluation Meeting, Washington, D.C., June 2018.

REFERENCES

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