In-line Quality Control of PEM Materials

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Project ID #: MN016
Contract No.: DE-SC0013774
PM: Nancy Garland
Overview

Timeline and Budget

SBIR Phase I

- June 2015 – March 2016
- $150,000
  - Total Project: $150,000
  - Total recipient share: $0
  - Total DOE funds spent: $141,118

Technical Targets

Build a prototype system to simultaneously measure:

- Defects in a moving membrane web
- Membrane thickness over the full web width

Barriers Addressed

- E. Lack of Improved Methods of Final Inspection of MEAs
- H. Low Levels of Quality Control

Partners/Collaborators

- National Renewable Energy Laboratory: Mike Ulsh, Peter Rupnowski
Relevance

- **DOE Objectives**: Improved quality control to improve reliability and reduce automotive fuel cell stack costs to $20/kW by 2020 at 500,000 units/year

- **DOE Targets**
  - Develop in-line diagnostics for component quality control and validate performance in-line
  - Increasing the uniformity and repeatability of fabrication
  - Reduce labor costs and improve reproducibility by increasing automation
  - Identify cost drivers of manufacturing processes

- **Mainstream Engineering Targets**
  - Demonstrate real time automated in-line defect and thickness mapping on NREL web line
  - Improve manufacturing process by providing real time feedback on quality metrics
  - Scan the membrane with 100% coverage, marking and logging defective regions
In-line QC of PEM Materials

- Demonstrate membrane defect detection using in-line machine vision optical techniques
- Develop membrane thickness mapping capable of real time measurement across the full web
- Determine membrane rejection criteria
- Develop software to automate analysis, defect logging and real time identification of critical defects
- Fabricate and test a prototype incorporating an optical sensor system
- Apply methods to an array of membrane materials at web speeds up to 100 ft/min
Membrane Defect Types

- Examined three primary types of defects

**Pinholes**

**Scratches**

**Folds**

Images taken with edge-lit compact camera
Static Measurements

- Determination of thickness and defect detection limits for the current optical hardware

- Mainstream’s cross-polarized near-UV-Vis optical arrangement improves the defect resolution
Moving Web Line Measurements

Mainstream’s system tested on NREL’s web line up to 100 ft/min

Rewind Station with web steering

Light source and filters

Unwind Station

Mainstream’s in-line optical diagnostics

Membrane web with tension control
## Milestones

<table>
<thead>
<tr>
<th>Phase I Milestones</th>
<th>Phase I Result</th>
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<tbody>
<tr>
<td>Identify the smallest discernible defect size and characteristics for PFSA membranes</td>
<td>Unsupported membranes ▪ 25 µm diameter pinhole ▪ 10 µm width scratch ▪ 100 µm width fold or crease</td>
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<tr>
<td>Determine membrane thickness to ±1 µm for a 25 µm thick membrane</td>
<td>Nafion®-115: ±1 µm for 132 µm film by polarimetry ▪ Nafion®-211: ±0.5 µm for 25 µm film by absorption</td>
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<tr>
<td>Demonstrate defect and thickness analysis in real-time up to 60 ft/min</td>
<td>Demonstrated at up to 30 ft/min for Nafion®-211 with real-time processing; 100 ft/min with image post-processing</td>
</tr>
<tr>
<td>Develop membrane defect criteria and identify defects on a moving web</td>
<td>Found 100% of 100 µm pinholes in Nafion®-211 at 30 ft/min in real-time; 100 ft/min with post-processing</td>
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<td>Integrate an encoder and printer to mark defects locations in real time</td>
<td>Marked 35-of-35 defects in real-time. Printed every 1 foot for 50 feet at variable web speed from 1 to 60 ft/min.</td>
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</table>

Optical arrangement provides a significant improvement in the defect resolution for a given camera pixel count
Defect Limit-of-Detection

- Smallest detectable defects where the left image is from the Mainstream’s detector and the right is from a high-powered optical microscope.

- Pinhole defect in Nafion®-211 at 25 μm
- Fold defect in Nafion®-211 at 100 μm width by 500 μm length
- Scratch defect in Nafion®-211 at 10 μm width by 100 μm
Membrane Thickness Mapping

- High resolution thickness mapping by polarimetry across the membrane web

Image of Nafion®-115: (a) regular backlit photograph, (b) colorized image from Phase I area-scan camera, (c) image with background compensation

Thickness Map of a deformed Nafion®-115 sample, where the red circles are micrometer measurements

138 μm 134 μm 132 μm
Image Analysis

- Custom software and optical enhancement provides improved defect resolution

### The software process

<table>
<thead>
<tr>
<th></th>
<th>Description</th>
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<tbody>
<tr>
<td>a</td>
<td>Image acquisition and transfer from camera to computer</td>
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<tr>
<td>b</td>
<td>Image enhancement effects</td>
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<tr>
<td>c</td>
<td>Image conversion to binary image</td>
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<tr>
<td>d</td>
<td>Defect detection and logging</td>
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</tbody>
</table>

### Resultant image

- a: Image acquisition and transfer from camera to computer
- b: Image enhancement effects
- c: Image conversion to binary image
- d: Defect detection and logging

![Resultant Image](image_url)
Prototype Image Analysis UI

**Processed Image**

- Monochrome image represented as a color gradient with defects marked in the black region

**Real-time Output**

- **Number of Defects**
  - 0

- **Roll Speed (ft/min)**
  - 10.1346

**Accomplishments**

- **Total Defects Found**
  - 0

- **Line roll speed for determining necessary camera parameters and marking defects**

- **Total of defects found since start of the roll**

- **Inspection status** – green indicates no defects present

**Mainstream Engineering Corporation**
Defect Detection up to 100 ft/min

- Defects accurately detected in a range of supported and unsupported PEM membranes
- 40/40 100 µm and 40/40 500 µm pinhole defects identified
  - In real-time up to 30 ft/min, with post-processing up to 100 ft/min

Stop-frame time series of images showing roll-to-roll defect detection in Nafion®-211 at 30 ft/min
Defect Location Printing

- PET defects detected at 10 ft/min marked by the printer
- Encoder used to measure roll speed and determine printer timing

Five 500 µm pinhole PET defects, highlighted with black circles, automatically detected and marked by Mainstream’s setup at 10 ft/min
Other Membrane Applications

- Alternative membranes for reverse osmosis, anion exchange, hydrocarbon PEM, and electrolysis
- All transmit over 10% in the UV/Vis except for I-200 (AEM) and BW-30 (reverse osmosis)
## Collaborations

<table>
<thead>
<tr>
<th>Institution</th>
<th>Type</th>
<th>Extent</th>
<th>Role and Importance</th>
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<tbody>
<tr>
<td>National Renewable Energy Lab</td>
<td>Federal Laboratory</td>
<td>Major</td>
<td>Provided technical assistant with patented technique, full-scale web line for testing up to 100 ft/min</td>
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Remaining Challenges and Barriers

Remaining Objectives
- Knowledge of smallest required limit of detection
- Testing of smallest defect with upgraded hardware
- Full automation of software and hardware
- Data on real web-lines
- Trade-offs between cost and accuracy
- Alternative membrane application testing

Key Barriers
- Access to industry web-lines
- Testing on most relevant membranes
- Full understanding of system requirements
Technology Transfer Activities

- Mainstream is pursuing SBIR Phase II funding to develop the system to a TRL 7 and commercialize the product.
- Mainstream has an option to license two patents from NREL.
- Plan to demonstrate the prototype system on two industrial web lines in addition to NREL.
- While the PEM fuel cell market is the primary focus, the technology is applicable to other markets such as reverse osmosis, electrolysis, and protective films.
## Proposed Future Work

<table>
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<tr>
<th>Task Name</th>
<th>Q1</th>
<th>Q2</th>
<th>Q3</th>
<th>Q4</th>
<th>Q5</th>
<th>Q6</th>
<th>Q7</th>
<th>Q8</th>
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<tbody>
<tr>
<td>Task 1: Create Defective Membrane and Identify Defect Size that Leads to Cell Failure</td>
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<td>Task 2: Determine Detection Limit for Defects and Thickness with New Hardware</td>
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<td>Task 3: Develop and Package Automated, Real-time Software</td>
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<td>Task 4: Determine Trade-offs in Equipment Sensitivity and Cost</td>
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<td>Task 5: Design and Fabricate Full-Scale Prototype System</td>
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<td>Task 6: Demonstrate Prototype System on Full Speed Membrane Line</td>
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<td>Task 7: Explore Viability for Alternative Membrane and Film Applications</td>
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<td>Task 8: Manage Phase II Effort</td>
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Proposed Future Work

Proposed Work
- Improve resolution to 4 µm incorporating high-resolution camera and high-speed processor
- Scale system to real-time measurements of thickness over 24-inch web
- Demonstrate reliability of packaged system for defect detection up to 100 ft/min

Key Milestones
- 4 µm defects at 100 ft/min
- 0.5 µm thickness resolution
- 5σ false-positive and negative rate
- Fully packaged prototype (TRL 7)

Methods to Mitigate Risk
- Leverage NREL experience
- Leverage expertise from other projects
- Involve potential customers early in the development process
- Design a low-cost variant for applications with looser tolerances
- Explore alternative applications to broaden market and drive down cost

Go/No Go Decisions
- Full-width thickness mapping across a 24-inch web at 30 ft/min
- Defect detection across 24-inch web at 30 ft/min
Summary

- Pinholes as small as 25 µm were successfully identified in static samples with the low cost camera system.
- Demonstrated thickness mapping to a resolution of ± 1 µm for Nafion®-115 and Nafion®-211.
- Demonstrated the performance of the enhanced optical techniques with 18 membranes and films including a variety of supported and unsupported membranes.
- Real-time identification of 100% of 100 µm induced defects in Nafion®-211 at 30 ft/min on NREL’s web line.
- Defect type and position successfully logged electronically and location printed on the web.
SUPPORTING SLIDES
Mainstream Engineering Corporation

- Small business incorporated in 1986
- 100+ employees
- Mechanical, chemical, electrical, materials and aerospace engineers
- 100,000 ft² facility in Rockledge, FL
- Laboratories: electric power, electronics, materials, nanotube, physical and analytical chemistry, thermal, fuels, internal combustion engine
- Manufacturing: 3- and 5- axis CNC and manual mills, CNC and manual lathes, grinders, sheet metal, plastic injection molding, welding and painting

**Capabilities**
- Basic Research, Applied Research & Product Development
- Transition from Research to Production (Systems Solution)
- Manufacture Advanced Products

**Mission Statement**
To research and develop emerging technologies. To engineer these technologies into superior quality, military and private sector products that provide a technological advantage.
SBIR Successes and Awards

- 95% DOD Commercialization Index
- SBIR spinoffs – QwikProduct Line
- SBIR spinoffs – Military Product Line
- Honors
  - 2014 DOE’s SBIR/STTR Small Business of the Year
  - 2013 Florida Excellence Award by the Small Business Institute for Excellence in Commerce
  - Winner Florida Companies to Watch
  - Blue Chip Enterprise Initiative Awards
  - Job Creation Awards
  - Two SBA’s Tibbetts Awards for Commercialization
  - State of Florida Governor’s New Product Award
  - SBA’s Small Business Prime Contractor of the Year for the Southeastern U.S.
  - SBA’s Administrator’s Award for Excellence
Mainstream’s Focus Areas

THERMAL CONTROL
- High Heat Flux Cooling
- Thermal Energy Storage
- Directed Energy Weapons
- Rugged Military Systems

ENERGY CONVERSION
- Combustion
- Diesel/JP-8 Engines
- Biomass Conversion
- Alternative Fuels
- Fuel Cells

TURBOMACHINERY
- Compressors
- Turbines
- Bearings/Seals
- Airborne Power Systems

MATERIALS SCIENCE
- Thermoelectrics
- Batteries/Ultrapacitors
- Hydrogen Storage
- E-Beam Processing
- Nanostructured Materials

POWER ELECTRONICS
- High Speed Motor Drives
- Hybrid Power Systems
- Solar/Wind Electronics
- Pulse Power Supplies
- Battery Chargers

CHEMICAL TECHNOLOGIES
- Heat Transfer Fluids
- Catalysis
- Chemical Replacements
- Water Purification
- Chemical Sensors