In-line Quality Control of PEM Materials

SBIR Phase II

DOE Annual Merit Review, Washington D.C.

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

Overview

Timeline and Budget

SBIR Phase I/II

- June 2015 – August 2018
- $1.15 MM
  - Total Project: $1.15 MM
  - Total recipient share: $0
  - Total DOE funds spent: $900K

Technical Targets

Demonstrate a prototype system that simultaneously measures:

- 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: Michael Ulsh, Peter Rupnowski, Scott Mauger
- Georgia Institute of Technology: Dr. Tequila Harris

Relevance

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

- **<u>DOE Manufacturing R&D Activities</u>**
  - 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

- **<u>Mainstream Engineering Targets</u>**
  - Demonstrate real time automated in-line defect and thickness mapping
  - 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

- Mainstream is developing the Mantis Eye^{TM} machine vision system for automated, continuous monitoring of films
- Can be used on coating lines and other converting equipment
- Patent-pending optical design provides simultaneous film thickness mapping and defect detection
- Samples the entire web width in real-time (100% inspection)
- Guiding principle of our product design is to provide the finest defect and thickness resolution with reasonably priced camera hardware (i.e., better bang for your buck than brute force method of using very expensive, high-res cameras)
- This approach is analogous to the eyes of the remarkable mantis shrimp, which can perceive the polarization state of light and multi-spectral images.

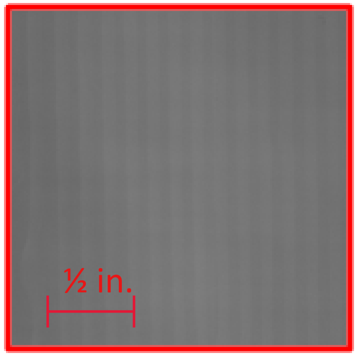
Technical Approach

- Create defective membrane and identify defect size that leads to cell failure
- Determine defect and thickness limit of detection (LOD) with hardware in freshly cast and production membranes
- Conduct surveys with manufacturers of membrane and MEAs to identify key equipment performance and cost targets
- Beta test hardware and software
- Test prototype system on partner's weblines
- Validate performance criteria in-house
- Demonstrate prototype system on industry weblines
- Expand technique to alternative membranes

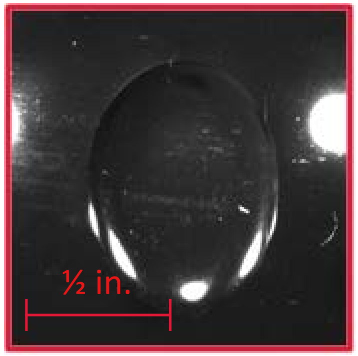
Membrane Defect Types

- **Examined four types of defects in freshly cast membrane and three types in production membrane**

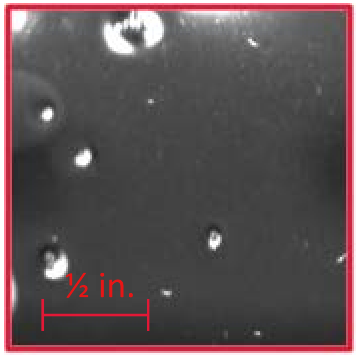
Streaking



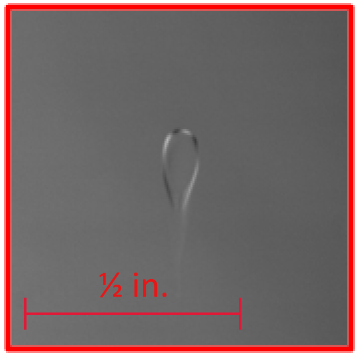
Spotting



Pinholes

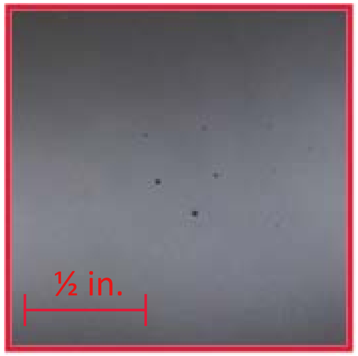


Blob

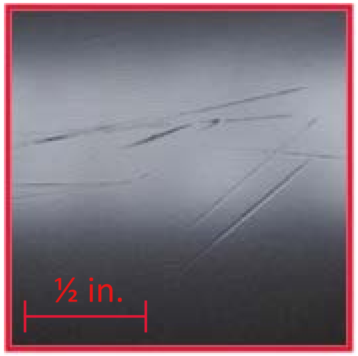


Defects in freshly cast membrane

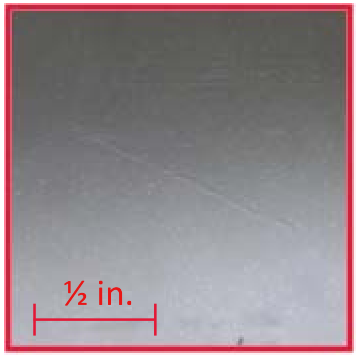
Pinholes



Scratches



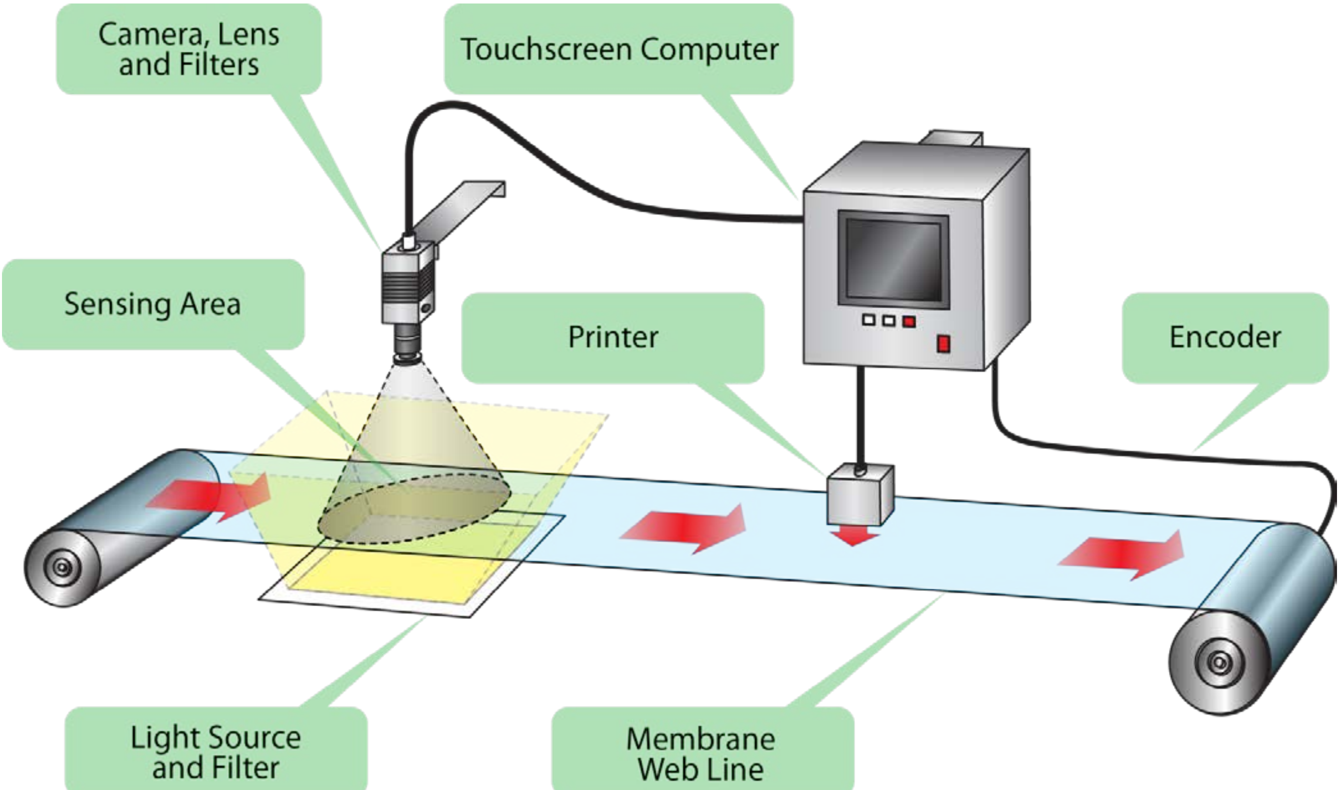
Folds



Defects in membrane after production

Modular Setup

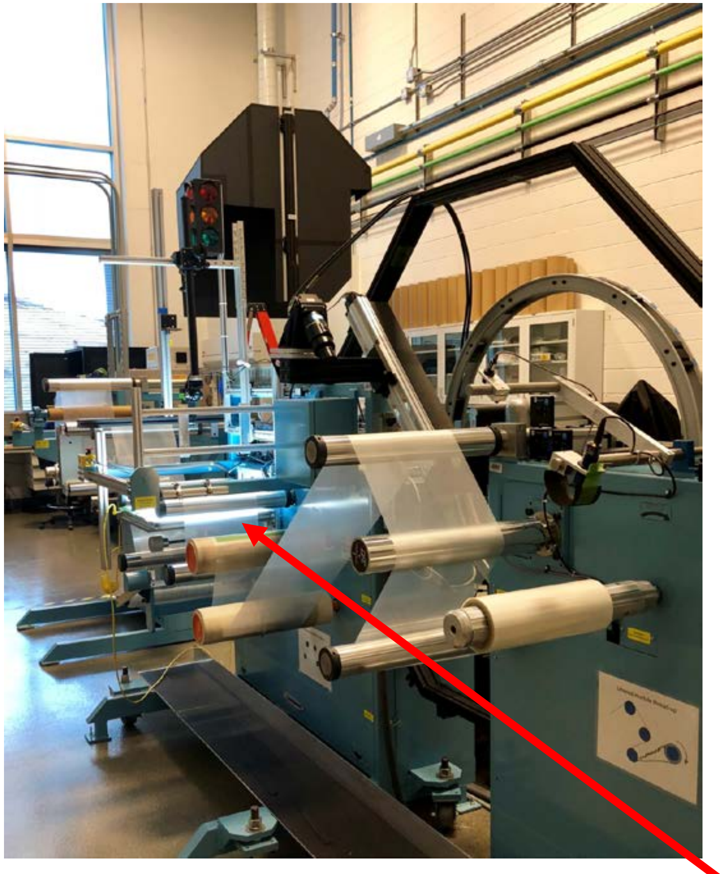
- **Simple system that can be setup in a variety of webline locations**



- **Mainstream's cross-polarized near-UV-Vis optical arrangement improves the defect resolution**

Webline Measurements

NREL web line (100 ft/min)

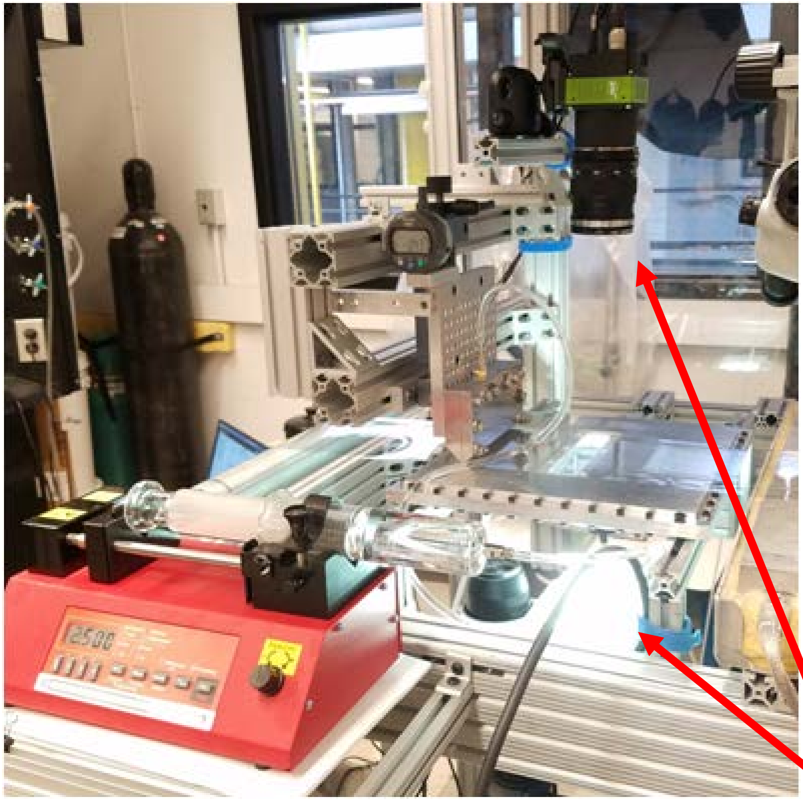


In-house inspection station (500 ft/min)

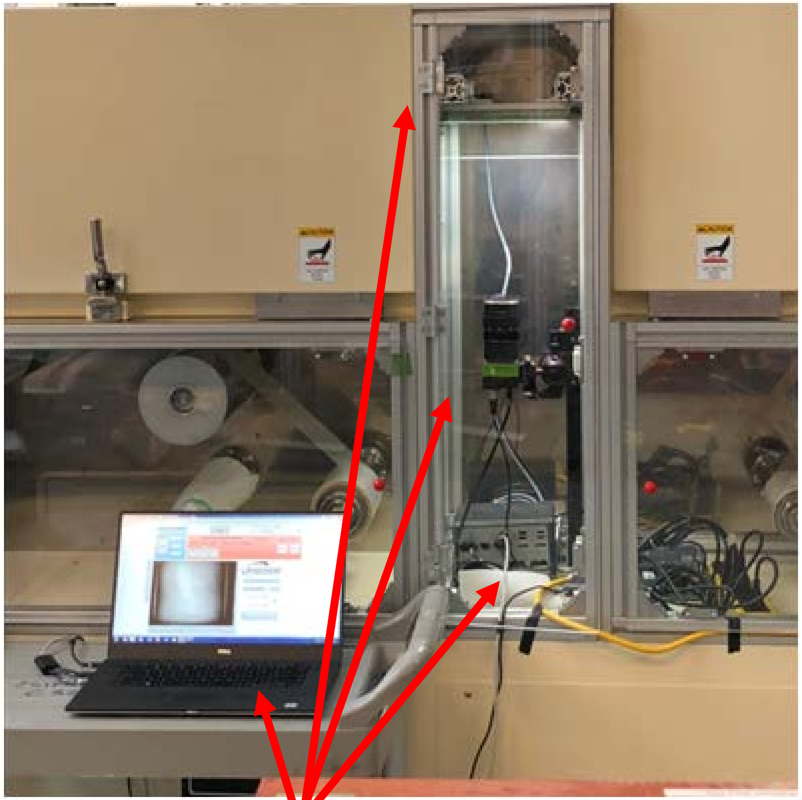
Mantis Eye in-line optical diagnostics

Coating Line Measurements

Georgia Tech's coating line (mounted above)



NREL's coating line (mounted below)



Mantis Eye in-line optical diagnostics

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Milestones

Final Milestones	Results
Detect defects down to 10 µm at 100 ft/min	-For both supported and unsupported membranes - 25 µm diameter pinhole - 10 µm width scratch - 100 µm width fold or crease -Demonstrated at up to 300 ft/min for PET and 100 ft/min for Nafion®-115 in real-time -Identified defects in freshly cast membrane
Identify membrane defect size that leads to cell failure	NREL determined defects up to 10 microns have no immediate effect, while larger than 40 microns decreased performance
Determine membrane thickness to 0.5 µm resolution	-Nafion®-115: ±1 µm for 132 µm film by polarimetry -Nafion®-211: ±0.5 µm for 25 µm film by absorption
Create a packaged prototype and demonstrate it on a web line	-Software is being beta-tested in-house and by commercial partners and custom installations have been developed
Achieve a 5σ false-positive and false-negative rate	-Software was developed to compare identical runs and determine identification rate – roll testing is ongoing

Optical arrangement provides a significant improvement in the defect resolution for a given camera pixel count

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Membrane and MEA manufacturer

- **Industry survey of manufacturers provided the following feedback**
  - Defects do occur and improved quality control is necessary
  - Customers frequently want tighter performance or tolerance metrics that are difficult for manufacturer to comply with or measure continuously
  - Full width thickness inspection is critical feature
  - Cost is the most important metric assuming a specific defect resolution is met
  - Data management is important

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Freshly cast defects identified

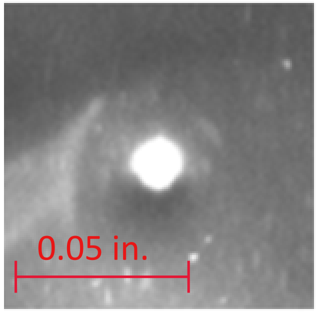
- **Defects in freshly cast Nafion were identified at Georgia Tech**

Edge of cast membrane

Freshly cast membrane

Pinhole defect

Image taken with compact camera

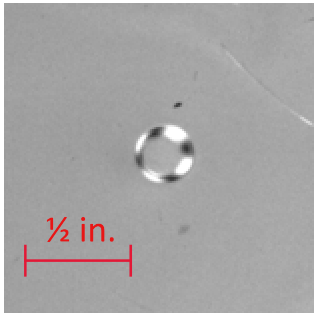


300 micron Pinhole

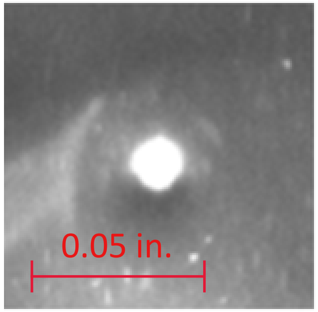
Image from machine vision camera

Membrane defects identified

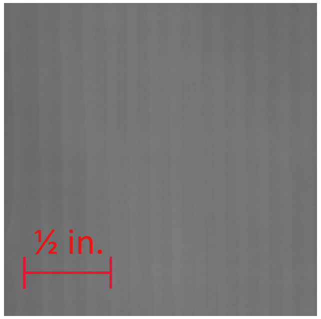
- **Defects can be identified in machine vision based on their unique properties in the software**



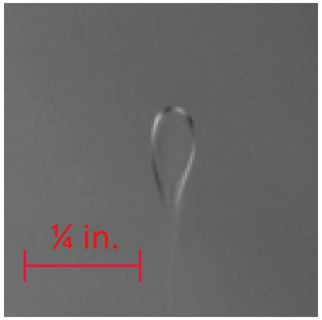
Bubble – alternative light



Pinhole – white hole



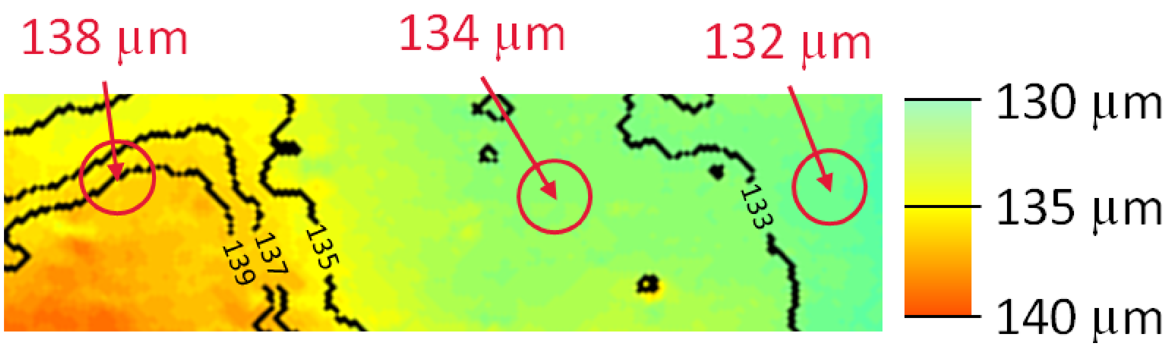
Streak – see dark and light border



Blob – similar to bubble, usually less defined

Membrane Thickness Mapping

- **High resolution thickness mapping by polarimetry across the membrane web**

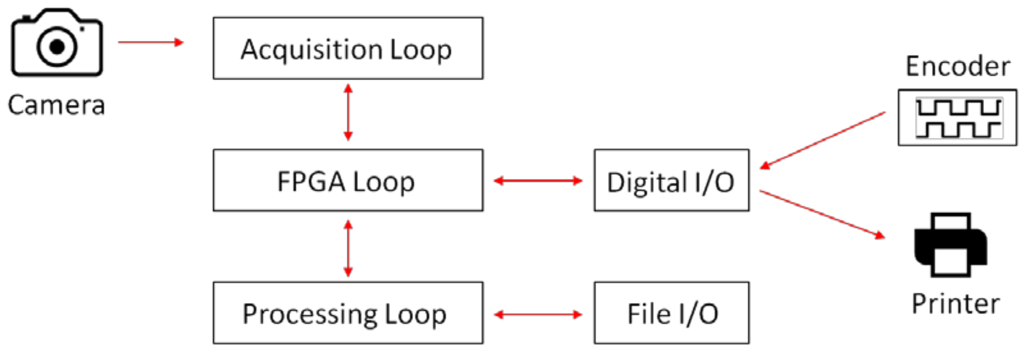


Thickness Map of a deformed Nafion®-115 sample, where the red circles are micrometer measurements

- **Thickness determination of freshly cast Nafion membrane by absorption with an accuracy of 1 micron on 10 micron, 25 micron, and 50 micron coatings**

Image Processing

- LabVIEW program consists of 3 main loops that operate in parallel
- Acquisition and Processing Loops operate on Real-Time Module



- Acquisition loop takes image from the camera, performs pre-processing and sends it to the FPGA
- FPGA performs image analysis and converts to binary
- Processing loop identifies key information about image and writes file
- Total loop time enables 8 fps, allowing over 500 ft/min at a 24 inch FOV
- 2nd camera can be added to the acquisition loop, but due to FPGA loop limitations causes small reduction in speed

Prototype Deployments

- **Two weblines**
  - NREL’s pilot webline at speeds up to 100 ft/min
  - Mainstream’s winder/unwinder at speeds up to 300 ft/min
- **Two coating lines**
  - Georgia Tech’s coating line for Nafion on 6 inch width PET at up to 6 ft/min
  - NREL’s pilot coater for Nafion for Nafion on 6 inch width PET at up to 3 ft/min
- **Successfully recorded images at all speeds and found defects including pin holes, streaking, spotting, folds, and scratches**

Custom Designed Inspection Station

- **Integrated polarized backlight and splice table**
- **Winder triggered from Mantis Eye™ inspection system**
- **Automatic tension control**
- **Forward and reverse winding**
- **Max web speed – 575 ft/min**
- **Max web width – 13.75 in.**
- **3 in. pneumatic mandrels**
- **Housed in ISO Class 7 softwall clean room**

Freshly cast defect detection

- **Defects accurately detected in a range of freshly cast Nafion membrane including streaking, spotting, and pinholes**
- **Nafion from 10 – 25 micron thick was cast onto 1 mil PET at speeds up to 1 m/min**

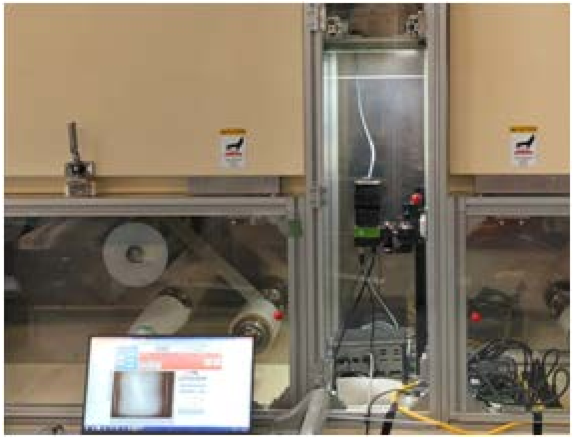


Image of the setup at NREL

Slow motion video showing defect identification at 1 m/min

Defect Detection up to 500 ft/min

- **Defects detected in PET web up to 500 ft/min**
- **Reliability statistics will be calculated for 50 micron, 100 micron, and 250 micron holes to determine false-positive and false-negative rates**

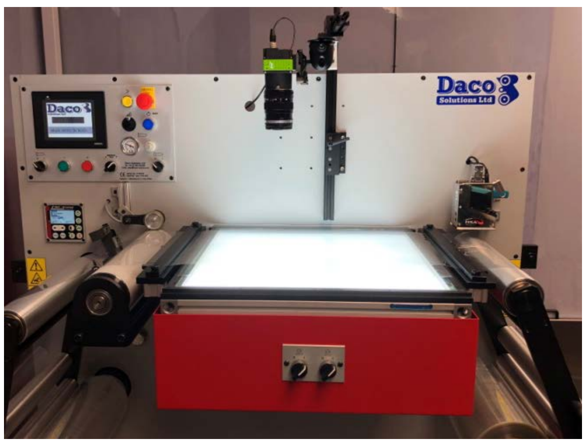
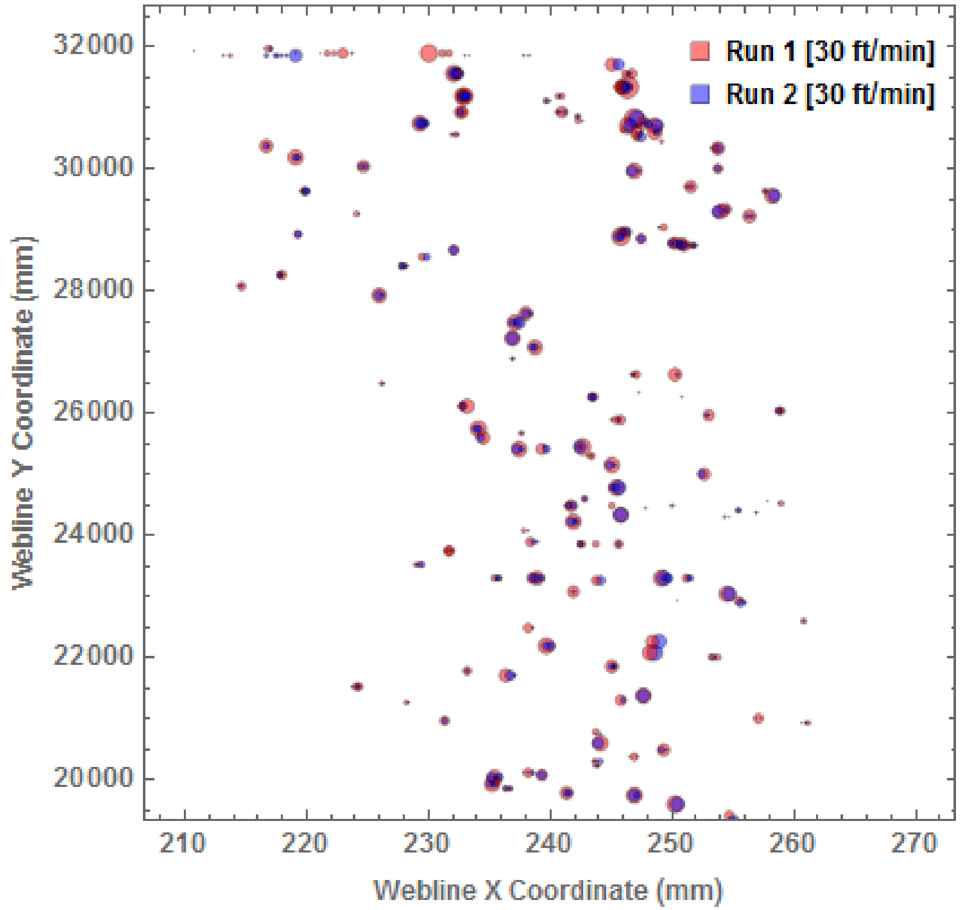


Image of webline setup

Slow motion video showing defect identification at up to 500 ft/min

Defect Detection Statistics

- Custom software for web location registration
- Bubble chart used to visually compare multiple runs
- Bubbles represent defects and are scaled to be seen visually
- Defects missed during a run can then be examined and algorithms improved
- Calibrations rolls can be run to determine false-positive and false-negative rate



Defects in Nafion-115 run at 30 ft/min

Response to Reviewer Comments

Reviewer Comment	Response
• "There was no discussion about alternative approaches"	• Alternative techniques include beta, X-ray, or gamma irradiation, interference fringe reflectance analysis, or off-line micrometers (these techniques do not sample 100% of the web)
• "Collaborations with MEA manufacturers would be appreciated" • "Closer collaboration with MEA manufacturers would be appreciated in order to validate technology on real web lines" • "Collaboration with a membrane manufacturer would strengthen the project"	• Mainstream has two demonstrations planned with membrane and film manufacturers • Mainstream and Dawnbreaker performed primary market research and have received feedback on pain points and what they desire for quality control
• Membrane thickness and the impact on performance should be further investigated	• Membranes as thin as 10 microns cast on one mil PET were tested; interference from reinforcement was examined more in-depth

Collaborations

Institution	Type	Extent	Role and Importance
National Renewable Energy Lab	Federal Laboratory	Major	Provide testing on webline and pilot scale coating line as well as conduct testing to determine the smallest defect to cause cell failure
Georgia Institute of Technology	University	Major	Provide pristine and defective coated membrane samples, provided pilot coating line for vision QC system testing

Remaining Challenges and Barriers

- **Remaining Objectives**
  - Beta test system in-house and at a production facility
  - Demonstrate the unit on two industrial weblines
  - Based on the demonstrations, refine system as necessary to better commercialize the product
- **Key Barriers**
  - Few players in the R2R film manufacturing space meaning customers are in the stronger position in negotiations
  - Requirements vary from company to company and project to project making the necessary targets for a commercial product subject to variability
  - Some potential applications are for opaque materials

Proposed Future Work

- **Proposed Work**
  - Improve defect resolution to 10 μm incorporating smaller FOV optics and high-speed processor
  - Scale system to real-time measurements of thickness over 24-inch web
  - Demonstrate reliability of packaged system for defect detection on two industrial weblines
- **Methods to Mitigate Risk**
  - Conducted membrane and MEA manufacturer survey to identify key areas of interest
  - Demonstrate prototype system for industry customers
  - Focus on reducing cost at current defect targets
  - Explore alternative applications to broaden market and drive down cost
- **Key Milestones**
  - 10 μm defects at 100 ft/min
  - 0.5 μm thickness resolution
  - 5σ false-positive and negative rate
  - Customizable and packaged prototype able to be deployed
  - Prototype demonstration on industry webline

*Any proposed future work is subject to change based on funding levels

Technology Transfer Activities

- Mainstream is using SBIR Phase II funding to develop the system into a product and commercialize it
- Demonstrated and collected data at our partner’s facilities
- While the PEM fuel cell market is the primary focus, the technology is well-suited to many types of toll coating and other polymer films
- Mainstream has submitted two patents on this project
  - “Apparatus and Method for Cross-polarized, Optical Detection of Polymer Film Thickness and Defects.” U.S. Patent Application Serial No. 15/170,360.
  - “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

Summary

- NREL determined defects up to 10 microns have no immediate effect, while larger than 40 microns decreased performance
- Pinholes as small as 25 µm were successfully identified in both supported and unsupported membranes
- Demonstrated thickness mapping to a resolution of ± 1 µm for Nafion®-115, Nafion®-211, freshly cast Nafion from 10 – 25 microns, and PET
- Deployed prototype system onto two weblines and two coating lines and successfully collected defect data including pin holes, streaking, spotting, folds, and scratches
- Collected quality metrics at speeds up to 300 ft/min on PET
- System is in the process of being beta tested

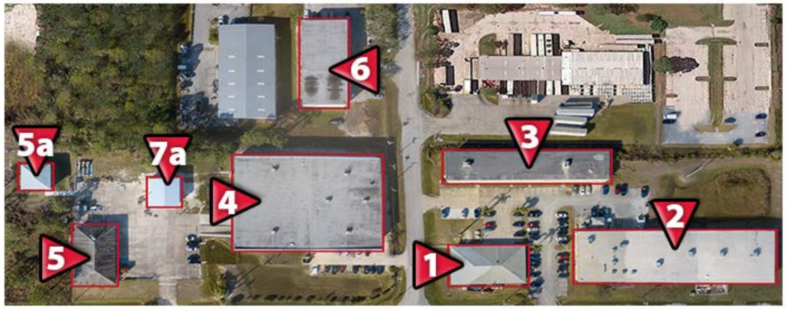
BACKUP SLIDES

Roll Comparison Software

- **Algorithm to automatically compare runs**
  - Consider two runs: Run 1 (i=0) with m defects and Run 2 (i=1) with n defects
  - Index each defect in both runs based on location (x and y pixel values) and run number: {x,y,i}
  - For Run 2, add an offset value y_0 to each y pixel value
  - Combine all defects into single matrix of size (m+n) by 3 and sort by y pixel value
  - Compute successive differences of column 3 (index column) and take absolute value of each entry, resulting in vector of 0's and 1's of length (m+n-1)
  - Take the sum of all elements in resulting vector, giving a single scalar quantity T
  - Find offset value y_0 that maximizes the quantity T
  - Repeat similarly for x pixel offset x_0

Mainstream Engineering Corporation

- Small business incorporated in 1986
- 100+ employees
- Mechanical, chemical, electrical, materials and aerospace engineers
- 100,000 ft^2 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



1 - ENGINEERING OFFICES
2 - RESEARCH & DEVELOPMENT
3 - RESEARCH & DEVELOPMENT
4 - PRODUCTION FACILITY
5 - PRODUCT DEVELOPMENT
5a - MAINSTREAM EBEAM
6 - ADMINISTRATIVE OFFICES
7a - ROTOMOLD PRODUCTION - 1

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**
  - **Two SBA’s Tibbetts Awards for Commercialization**
  - 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
  - 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

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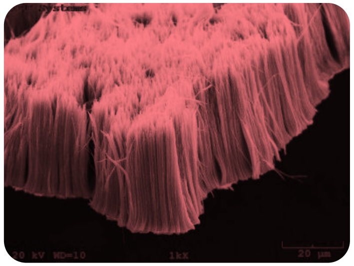
Mainstream's Focus Areas

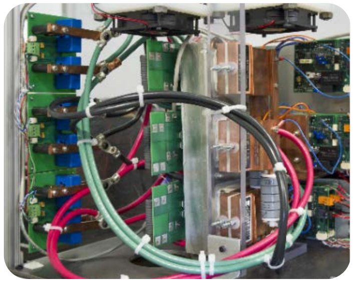
THERMAL CONTROL
- High Heat Flux Cooling
- Thermal Energy Storage
- Directed Energy Weapons
- Rugged Military Systems

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

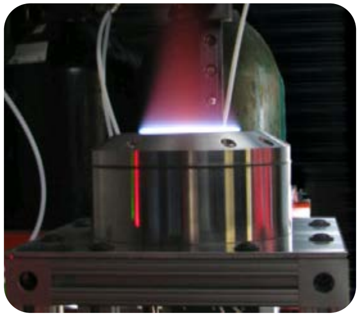
TURBOMACHINERY
- Compressors
- Turbines
- Bearings/Seals
- Airborne Power Systems



MATERIALS SCIENCE
- Thermoelectrics
- Batteries/Ultracapacitors
- 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