In-line Quality Control of PEM Materials

SBIR Phase II and IIb

DOE Annual Merit Review, Crystal City, VA

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Project ID #: ta005

Contract No.: DE-SC0013774

PM: Nancy Garland

Overview

Timeline and Budget

SBIR Phase I/II/IIb

- June 2015 – August 2020
  - Phase II end: August 2018
- $2.15 MM
  - Total Project: $2.15 MM
  - Total recipient share: $0
  - Total DOE funds spent: $1.3MM

Technical Targets

Demonstrate a turnkey system that simultaneously measures:

Phase II

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

Phase IIb

- Defects in a moving web of PEM materials
- Material 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

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 Manufacturing R&D Activities**
  - 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
  - Improve manufacturing process by providing real time feedback on quality metrics
  - Scan the PEM materials with 100% coverage, marking and logging defective regions

In-line QC of PEM Materials

- Mainstream is developing a suite of instruments to provide full turnkey inspection for MEA production including catalyst coated membrane and other opaque materials
- In the Phase II, we developed and commercialized the Mantis Eye™ machine vision system for automated, continuous monitoring of transparent films on coating lines and web converting equipment
- In the Phase IIb we will integrate technology for opaque material inspection into the Mantis Eye™ platform to provide a complete quality control solution
- Patent-pending optical design provides simultaneous thickness mapping and defect detection
- Guiding principle of our product design is to provide the finest defect and thickness resolution with reasonably priced camera hardware

PhaseIIb Technical Approach

- Create defective membrane, GDLs, CCMs, and MEAs to identify defect size that leads to cell failure
- Transition technology from small-scale, offline use to real-time, full web analysis based on the Mantis Eye platform
- Determine defect and thickness limit of detection (LOD) with hardware for all PEM materials
- Develop hardware and automated software
- Test prototype system on partner’s weblines
- Validate performance criteria in-house
- Demonstrate prototype system on industry weblines
- Expand technique to alternative membranes

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Webline Measurements

- **Modular system can be installed in a variety of webline locations**
- **In-house Mantis Eye Inspection Station**
  - 500 ft/min
  - Automated defect marking for membrane and transparent materials
  - Cross-polarized optical arrangement for enhanced defect detection
  - Small test samples can be spliced into existing rolls for analysis
  - Continue commercialization efforts with thin and transparent films

Materials Examined

- **Examined three types of materials**
  - Membrane (Phase I/II)
    - Thicknesses from 17 – 250 microns
    - Nafion sheets and freshly cast
    - Supported and unsupported
    - A variety of alternative membranes for electrolysis and anion exchange
  - GDLs (PhaseIIb)
    - Base material
    - Pt/C catalyst ink in Nafion ionomer
  - CCMs (PhaseIIb)
    - Base membrane
    - Pt/C catalyst ink in Nafion ionomer
- **NREL will be providing additional material and commercial materials will also be tested**

Milestones

Phase II Final 2018 Milestones	Results
Commercialize the Mantis Eye system for transparent film	-Mantis Eye system is currently being used to evaluate multiple samples from industry for capability demonstrations
Phase IIb Final 2020 Milestones	**Current Results**
Identify defect size and type that leads to MEA failure	NREL prepared a variety of pinholes in membrane and CCMs and assembled for initial performance and life testing in MEAs
Detect defects down to 10 µm at 100 ft/min for all fuel cell materials (e.g., membrane, GDL, catalyst, electrodes)	-Pinholes down to 25 µm in both supported and unsupported membranes -Modified Mantis Eye found defects down to 100 µm in GDLs and CCMs, the smallest size tested -Smaller defect samples are being made and new hardware setup
Determine membrane thickness to 0.5 µm resolution	-Examined different GDEs and CCMs and determined catalyst loading from 0 – 0.25 mg Pt/cm^2, thickness testing ongoing
Create a turnkey prototype and demonstrate it on three weblines	-New reflectance hardware was setup and installed in a static orientation and software is in development, demos target late 2019 and 2020
Achieve a 5σ false-positive and false-negative rate	-Mantis Eye software and winder can be used to compare runs and determine identification rate and will be tested late 2019

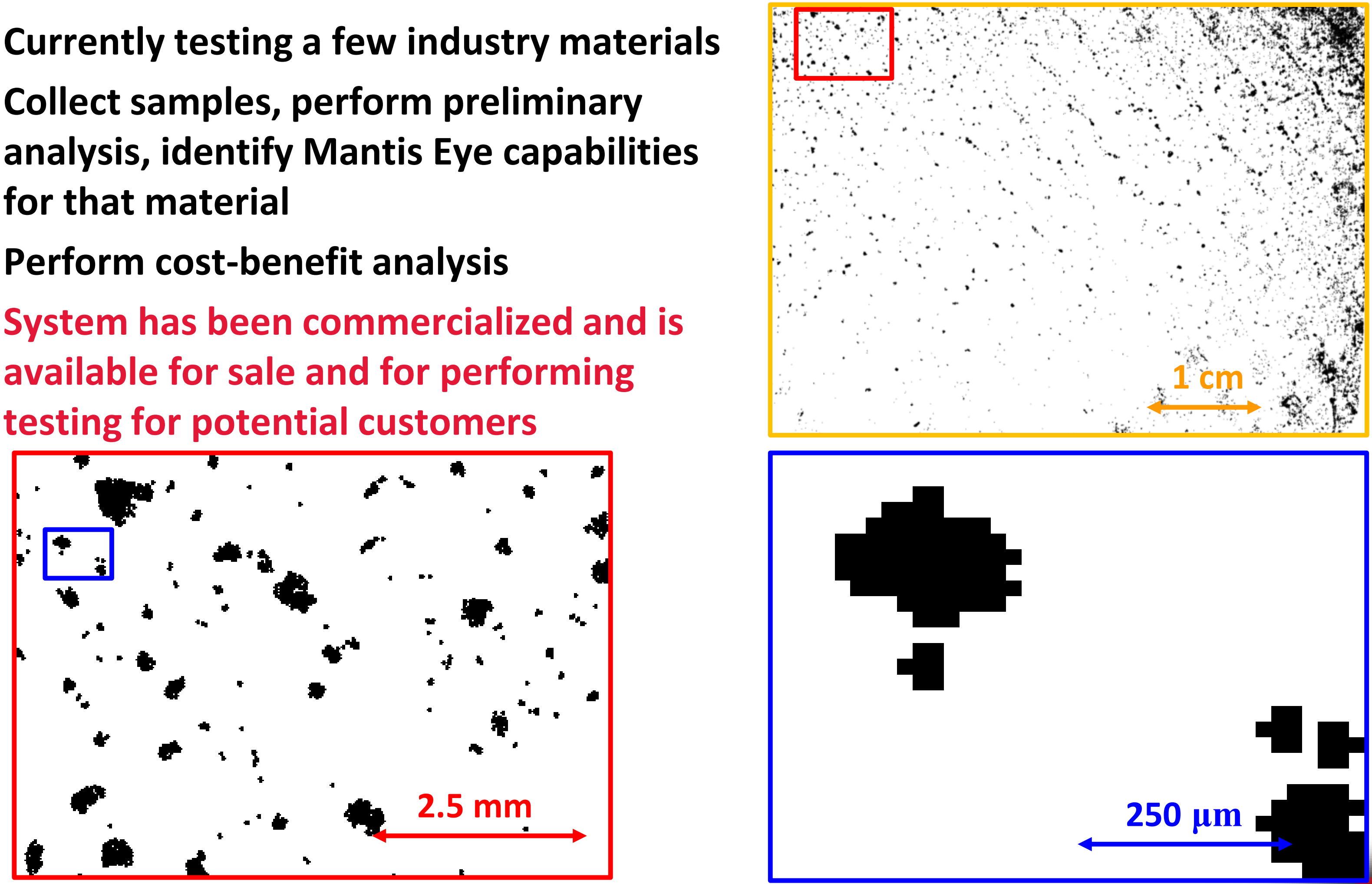
Mantis Eye Commercialization Efforts

Mantis Eye Inspection Station

- Setup website for dissemination of results and contact requests
- www.mainstream-engr.com/products/mantis-eye-optical-scanner/
- Integrated winder triggered from Mantis Eye inspection system
- Built-in polarized backlight and splice table
- Automatic tension control
- Forward and reverse winding
- Max web speed – 575 ft/min
- Max web width – 13.75 in.
- Demonstrated unit on multiple web lines
- Sold one unit to UConn for catalyst QC

Industry Sample Testing

- Currently testing a few industry materials
- Collect samples, perform preliminary analysis, identify Mantis Eye capabilities for that material
- Perform cost-benefit analysis
- System has been commercialized and is available for sale and for performing testing for potential customers



Example of testing a new commercial material and analysis capabilities at different scales – identified mesh plate impact

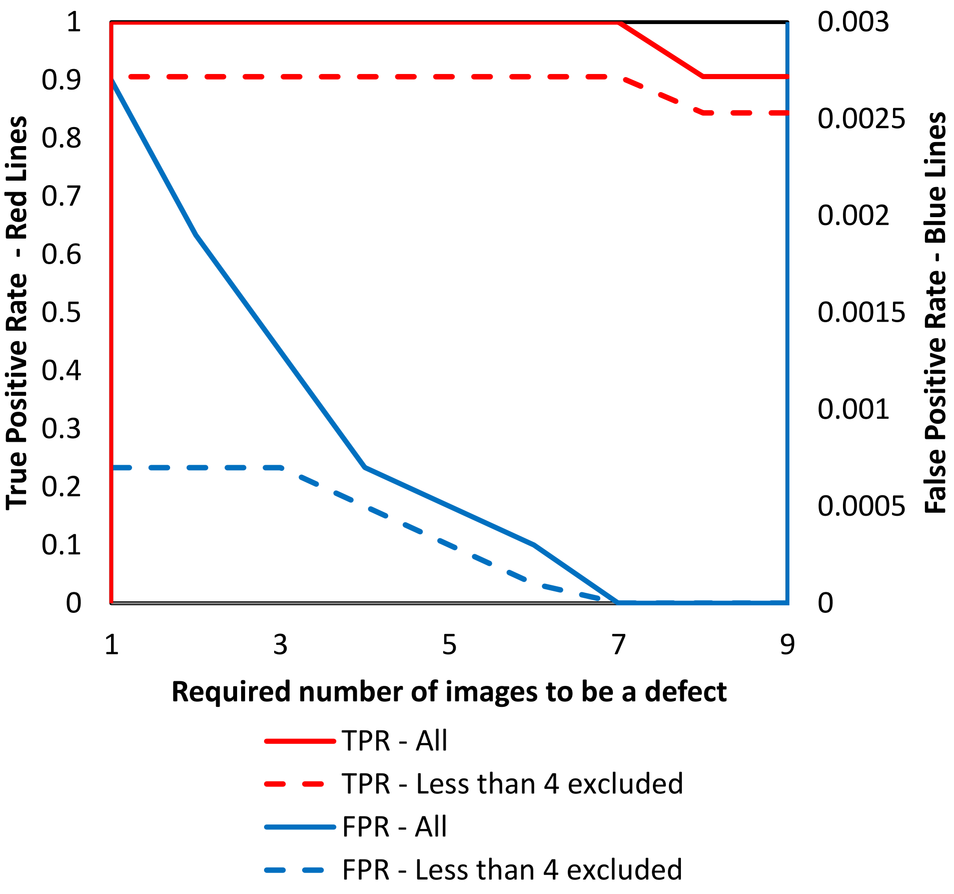
Reliability Statistics

- **False-positive and false-negative rate can be tuned based on cost to miss a defect, mark good material, web speed, and data management**
- **Statistics can be used to offer the optimal savings through QC installation**

Total population	Condition positive	Condition negative
Predicted positive	True positive	False positive
Predicted negative		

$$FPR = \frac{False\ positive}{Condition\ negative}$$

$$TPR = \frac{True\ positive}{Condition\ positive}$$



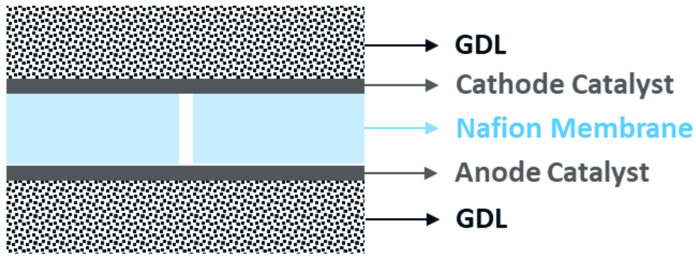
Defects in Nafion-211 at 30 ft/min based on number of images a defect is seen in

Impact of Defect Size and Type on MEAs

- **NREL prepared a variety of pinhole samples and testing is ongoing to determine necessary QC detection limits and identify cost trade-offs**

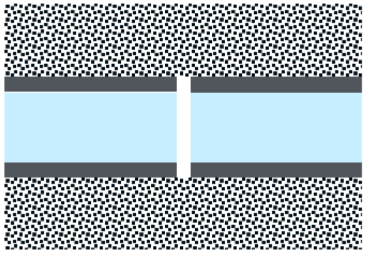
Pinhole configuration is related to the hole production step and the MEA fabrication procedure.

(a)



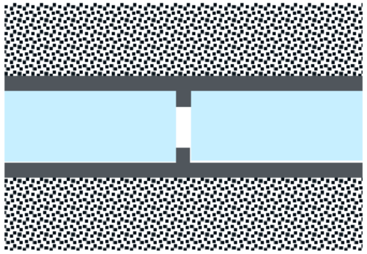
(1). Pinhole was produced during membrane casting
MEA fabrication: GDE without lamination

(b)



(2). Pinhole was produced through the CCM
MEA fabrication: CCM without lamination

(c)



(3). Pinhole was produced during membrane casting
MEA fabrication: CCM without lamination

(4). Pinhole was produced during membrane casting
MEA fabrication: CCM with lamination

(5). Pinhole was produced during membrane casting
MEA fabrication: GDE with lamination

(6). Pinhole was produced through the CCM
MEA fabrication: CCM with lamination

Case to be studied first: (b)-(2), (c)-(3)

Static Reflectance System

- **New reflectance hardware installed and software is under development**

Remotely located fanless industrial controller

Static reflectance camera setup

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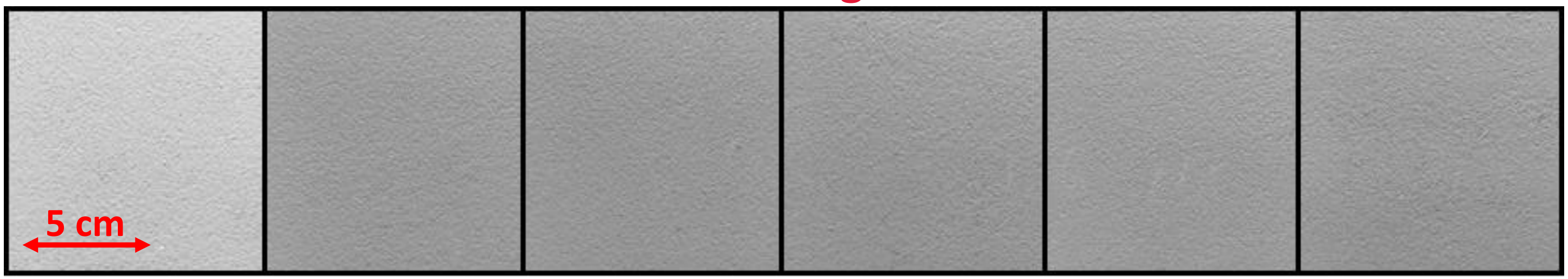
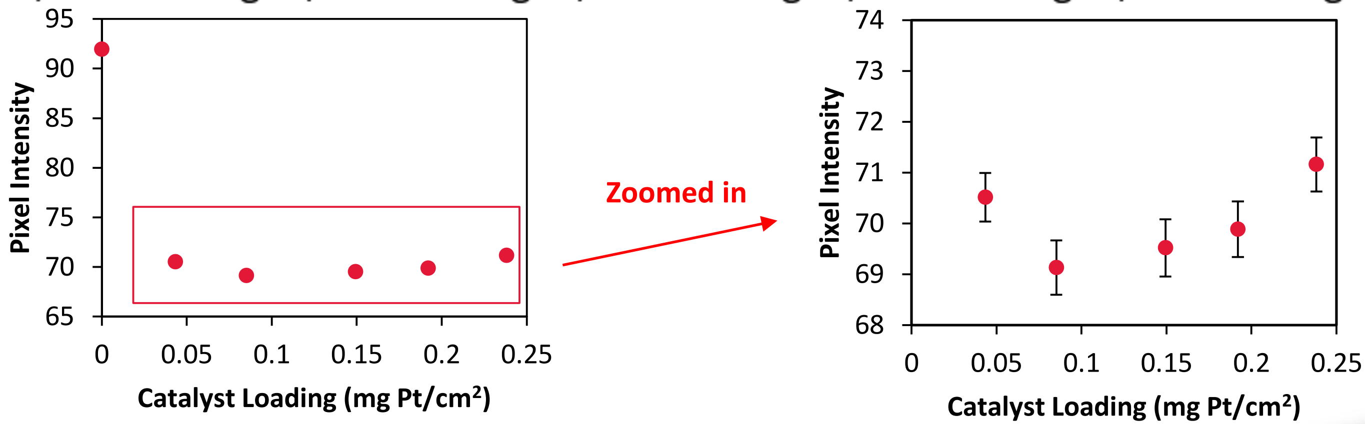
Gas Diffusion Electrode Materials

- **Different concentrations of Pt/C catalyst applied to GDLs**
- **System can detect changing concentration, pixel-to-pixel variation determines the smallest area a change can be seen on**



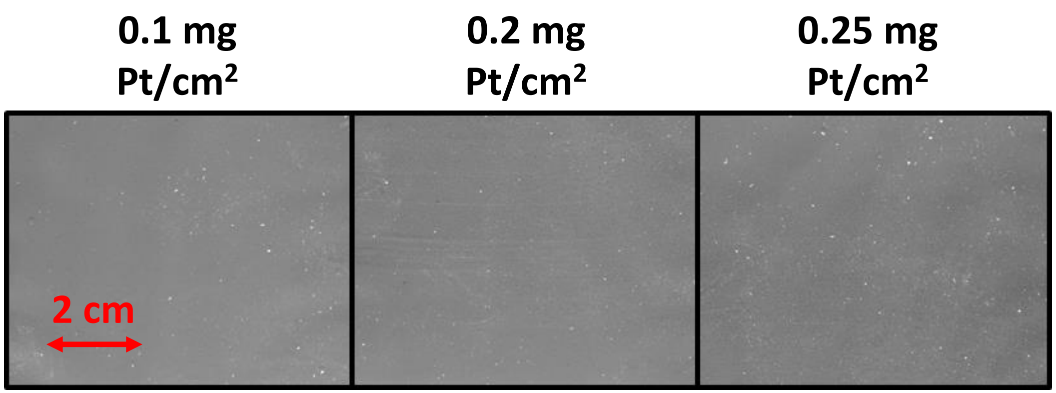
0 mg Pt/cm^2	0.04 mg Pt/cm^2	0.09 mg Pt/cm^2	0.15 mg Pt/cm^2	0.19 mg Pt/cm^2	0.24 mg Pt/cm^2



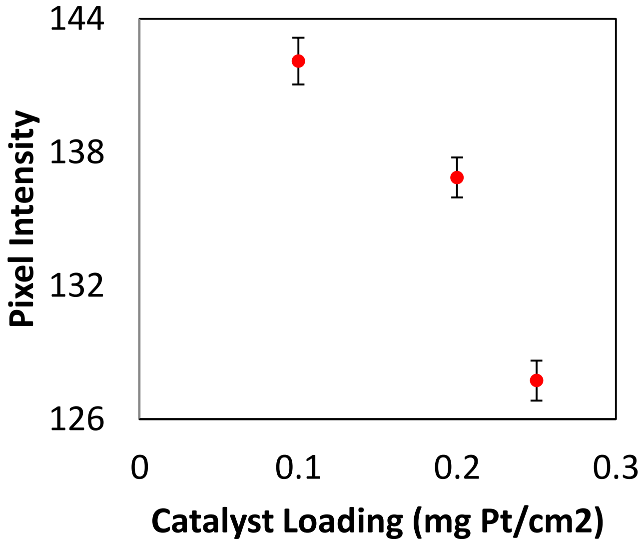
Images of different GDL catalyst loadings and their corresponding pixel intensities

Catalyst Coated Membranes

- **Different concentrations of Pt/C catalyst applied to Nafion membrane**
- **NREL will provide future reference samples as well as commercially available materials**
- **System can detect changing concentration, but each material will require a calibration**



Images of CCMs with different catalyst loading



Pixel intensity of different CCMs

Response to Reviewer Comments

Reviewer Comment	Response
• "Add an analysis of the spatial resolution limitations of the technique, as well as an analysis of the cost of the system as a function of the resolution"	• Developed cost functions that correlate how changes in defect size that can be detected, positive and negative identification rates, line speed, and material cost impact total cost of the system and cost benefit on the webline
• "Collaborations with membrane manufacturers would strengthen the project" • "Lack of input from customers" • "No commercial demonstrations have been executed yet" • "Earlier involvement by potential end users"	• Mainstream performed primary market research and received feedback on pain points • Setup a product website for disseminating results and reached out to multiple end users for feedback • Work led to collaboration with UConn • Continue to seek manufacturing partners
• Investigate alternative markets (e.g., PEM electrolyzers)	• Have evaluated multiple alternative materials for companies in need of improved QC and commercialization is ongoing
• "Main weakness is current detection limit of 25 microns"	• Limit of detection continues to improve as camera hardware is upgraded, but our approach is low cost and 100% web coverage

Collaborations

Institution	Type	Extent	Role and Importance
National Renewable Energy Lab	National Laboratory	Major	Fabricate defect samples and conduct testing to determine the smallest defect to cause cell failure, develop small-scale advanced techniques for identification of MEA material properties of interest, and facilitate webline demonstrations

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Remaining Challenges and Barriers

- **Remaining Objectives**
  - Transition reflectance technology and hit key loading, thickness, and defect milestones
  - Develop software for continuous, autonomous operation
  - Design turnkey prototype system
  - Demonstrate the unit on three industrial weblines
- **Key Barriers**
  - Increased image size and detail requires faster processing and improved data management
  - Relevant fuel cell materials with defects must be procured to develop and demonstrate the project to be relevant to manufacturers
  - The limit of detection that impacts MEA performance must be identified and proven
  - Calibration and installation must be simple to allow flexibility onto multiple lines
  - Cost of improved quality control must outweigh system cost

Proposed Future Work

- **Proposed Work**
  - Transition reflectance technology and demonstrate defect resolution to 10 µm in PEM materials (e.g., CCMs, GDLs)
  - Scale system to real-time measurements over 12-inch web
  - Demonstrate reliability of packaged system for defect detection on three industrial weblines
- **Methods to Mitigate Risk**
  - Convert proven technology for offline analysis to real-time
  - Conduct impact of defect size on MEA performance testing
  - Demonstrate prototype system for industry customers
  - Focus on reducing cost at current defect targets
  - Disseminate results and find manufacturing partners
- **Key Milestones**
  - 10 µm defects in CCMs and GDL
  - Operating in real-time at 100 ft/min
  - Identifying catalyst loading and thickness to 0.5 µm
  - 5σ false-positive and negative rate
  - Customizable and packaged turnkey prototype able to be deployed
  - Prototype demonstration on industry webline

Technology Transfer Activities

- Mainstream is using SBIR Phase IIb funding to develop the system into a turnkey product and commercialize it
- While the PEM fuel cell market is the primary focus, the Mantis Eye technology is well-suited to many types of toll coating and other polymer films and we are conducting product demonstrations with multiple companies that are interested in the system
- Mainstream received a subcontract from UCONN to leverage this technology developed in the Phase II to build and deliver a quality control system as part of a project for improved catalyst design
- 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

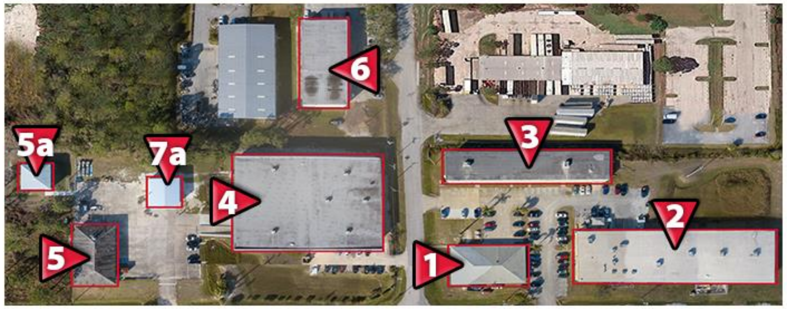
Summary

- Mainstream is commercializing the Mantis Eye inspection system through industry demonstrations and a dedicated website
- Reliability statistics were identified and can be used with a cost function to determine the benefit of improved quality control for each material and webline
- NREL has prepared multiple pinhole defect samples in membrane and CCMs to evaluate impact on MEA performance
- Demonstrated ability to determine catalyst loading on GDLs and membranes as well as see defects with a modified Mantis Eye setup
- New reflectance hardware has been brought online and software is in development

BACKUP SLIDES

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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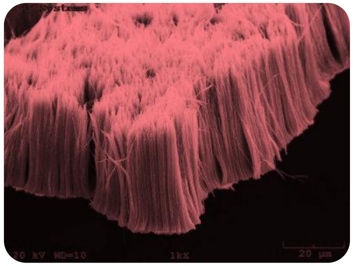
Mainstream's Focus Areas

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
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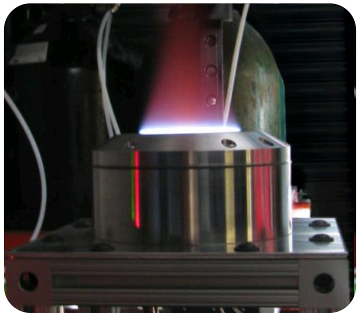
- Hybrid Power Systems
- Solar/Wind Electronics
- Pulse Power Supplies
- Battery Chargers



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