In-line Quality Control of PEM Materials

SBIR Phase II

DOE Annual Merit Review, Washington D.C.

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Author Andrew Wagner
E-mail awagner@mainstream-engr.com
Author Tom Lasko
E-mail tlasko@mainstream-engr.com
Author Paul Yelvington (P.I.)
E-mail pyelvington@mainstream-engr.com

Mainstream Engineering Corporation
200 Yellow Place
Rockledge, FL 32955
www.mainstream-engr.com

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

Overview

Timeline and Budget

SBIR Phase II

- June 2015 – August 2018
- $1.15 MM
  - Total Project: $1.15 MM
  - Total recipient share: $0
  - Total DOE funds spent: $450K

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
- Georgia Institute of Technology: Dr. Tequila Harris

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 membrane with 100% coverage, marking and logging defective regions

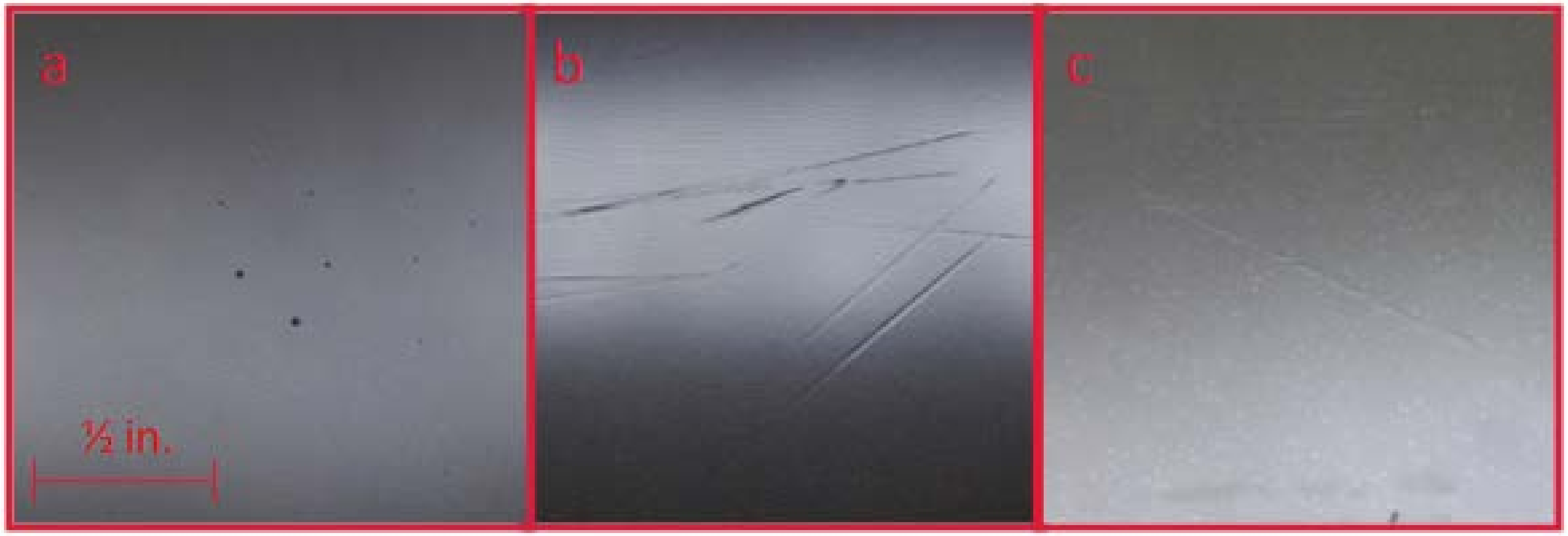
In-line QC of PEM Materials

- Create defective membrane and identify defect size that leads to cell failure
- Determine defect and thickness limit of detection (LOD) with new hardware
- Develop and package automated, real-time software
- Determine trade-offs in equipment sensitivity and cost
- Design and fabricate full-scale prototype system
- Demonstrate prototype system on full speed webline
- Explore viability for alternative membrane and film applications

Membrane Defect Types

- **Examined three primary types of defects**

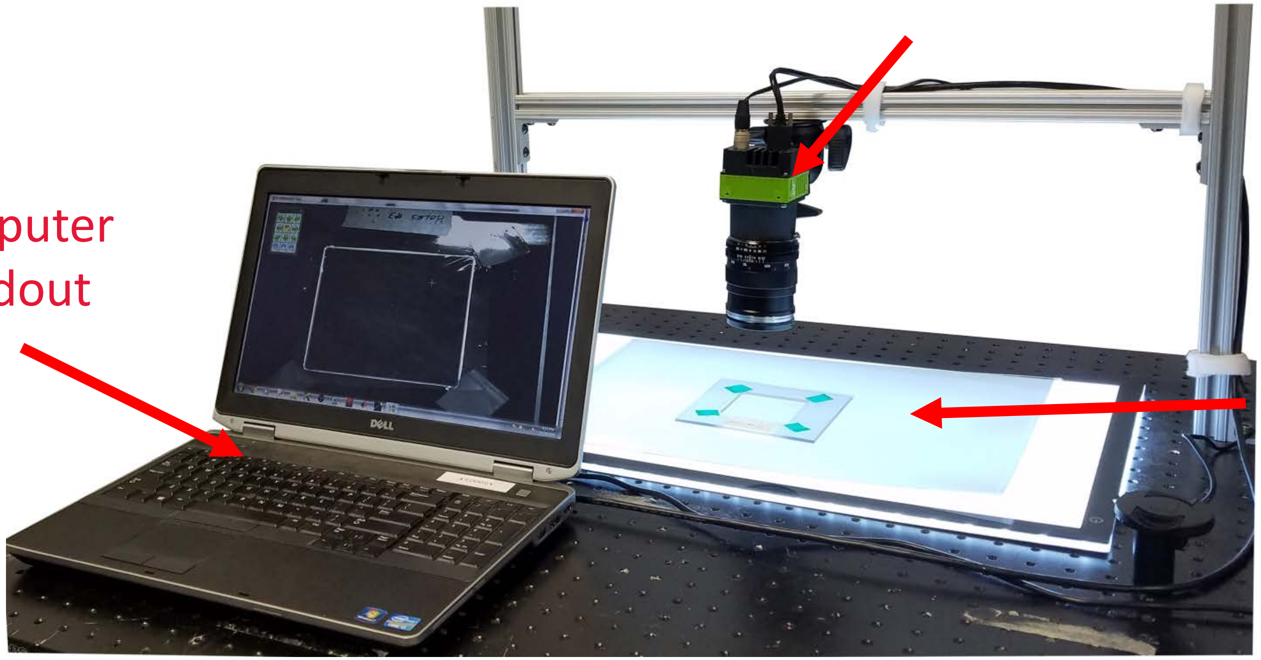
Pinholes **Scratches** **Folds**



Images taken with edge-lit compact camera

Static Measurements

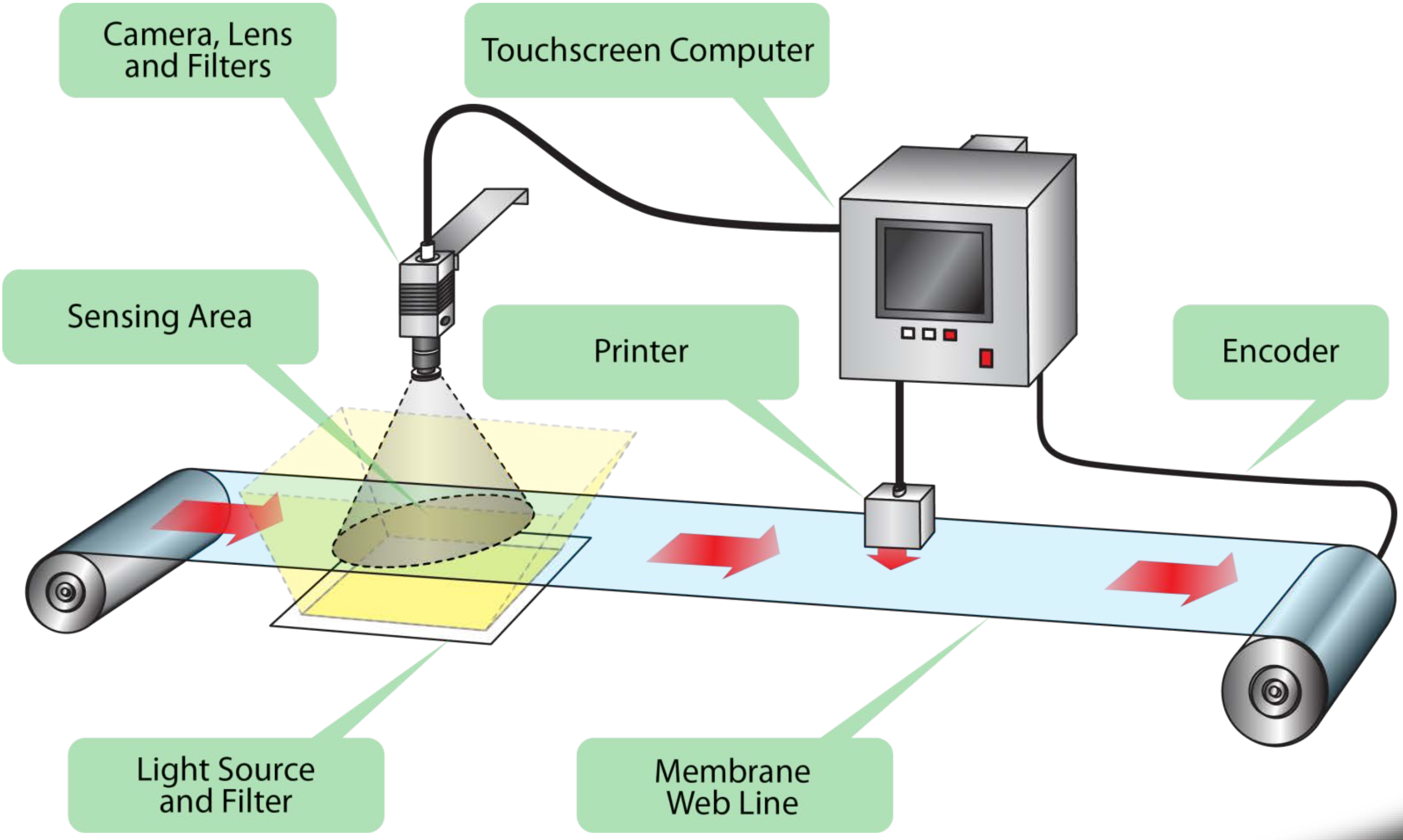
- **Determination of thickness and defect detection limits for the current optical hardware**



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

Modular Setup

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



Solutions Through Advanced Technology®
MAINSTREAM ENGINEERING

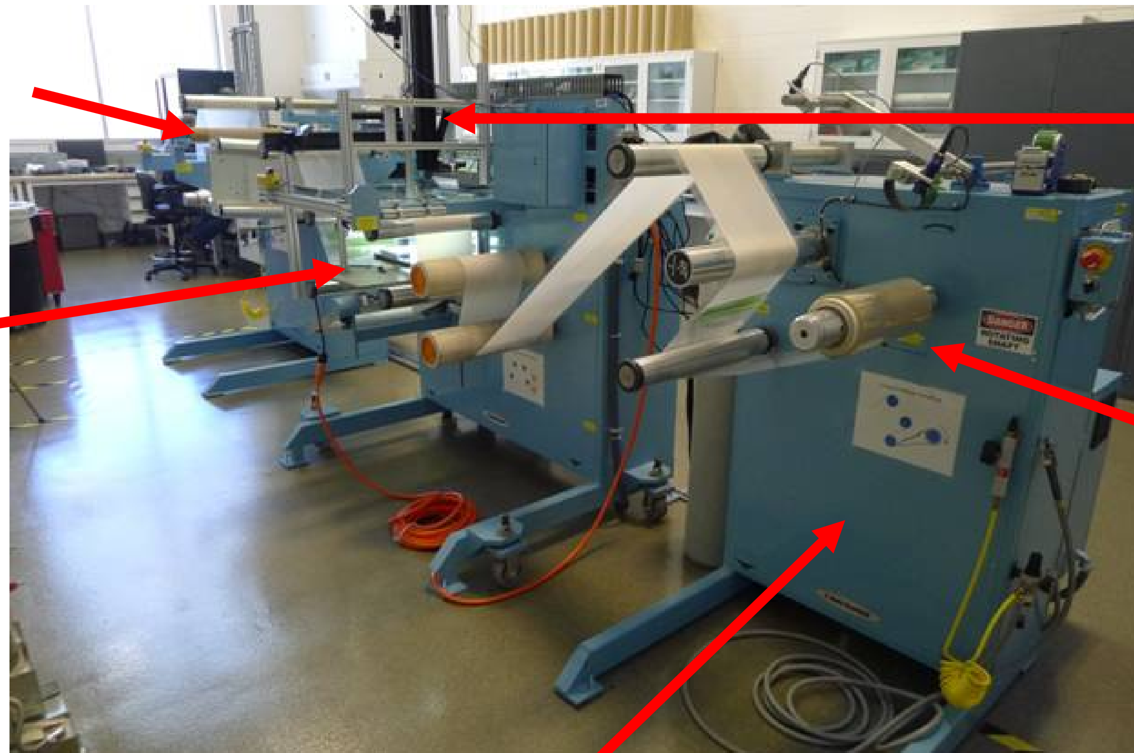
Moving Web Line Measurements

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

Rewind Station with web steering

Mainstream's in-line optical diagnostics

Light source and filters

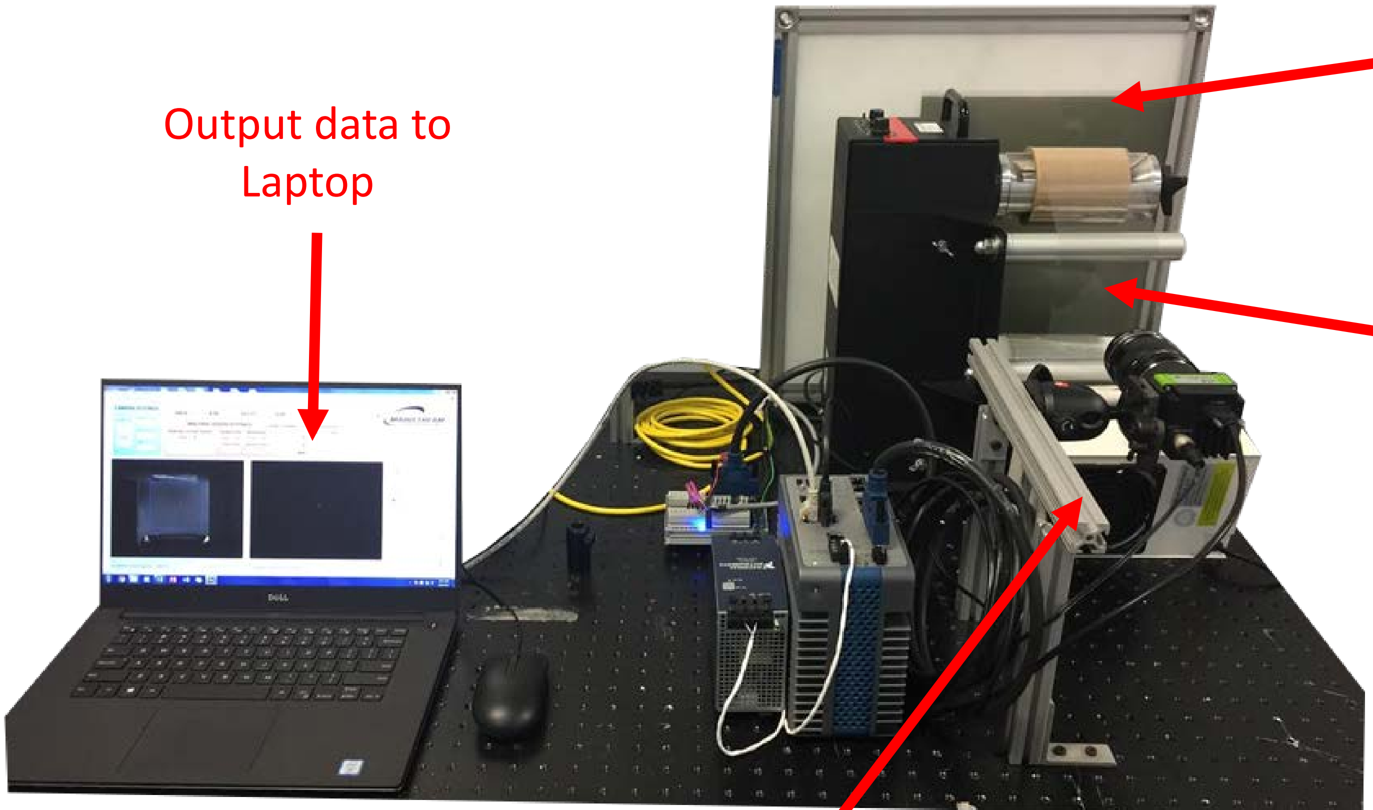


Membrane web with tension control

Unwind Station

Moving Web Line Measurements

Mainstream's 6 inch web line that runs up to 110 ft/min



Milestones

Phase II Milestones (Final Milestones)	Phase II Results (Year 1 of 2)
Detect defects down to 4 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 50 ft/min for Nafion®-115 in real-time
Identify membrane defect size that leads to cell failure	NREL and Georgia Tech have prepared defective samples and found defects less than 10 microns have no immediate effect, while larger than 300 microns cause 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 development is complete, hardware has been selected, packaging has begun
Achieve a 5σ false-positive and false-negative rate	▪ Calibration rolls have been made with specific defects that will be run continuously to determine quality metrics

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

Calibration Samples

- **9 calibration sheets were made**
  - Nafion®-HP, 211, and 115
  - 5, 10, and 25 micron holes

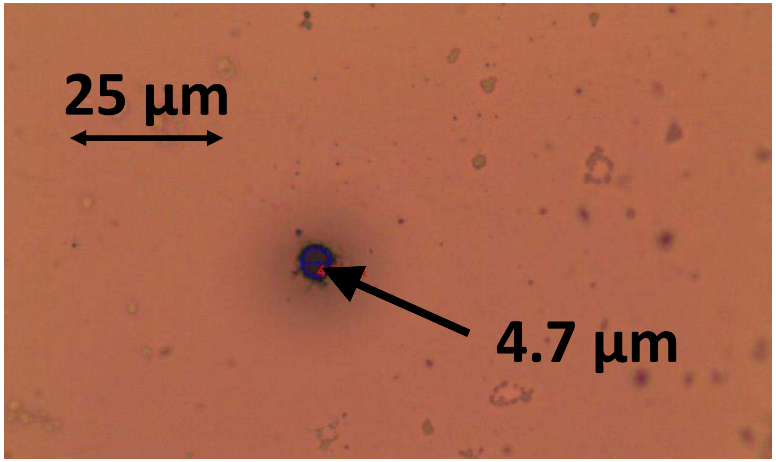
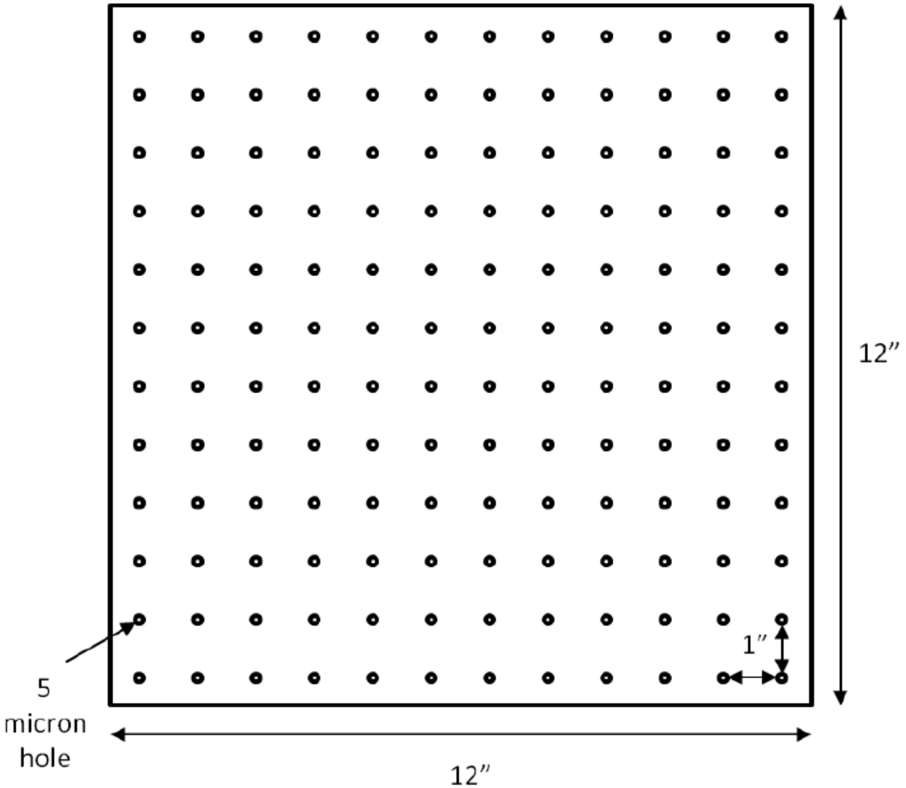


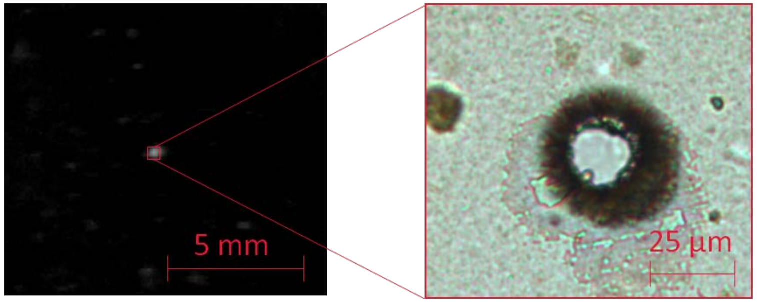
Image of Nafion®-211 with a 5 mµ hole



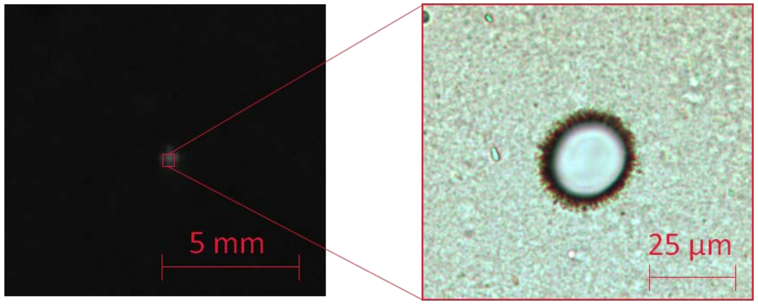
Schematic of calibration sample grid

Defect Limit-of-Detection

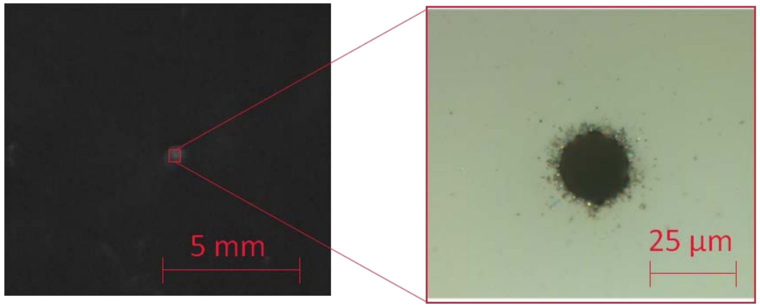
- **25 micron defects found for both supported and unsupported Nafion®, where the left image is cropped from the Mainstream's detector and the right is from a high-powered optical microscope**



25 µm Pinhole defect in Nafion®-211



25 µm Pinhole defect in Nafion®-HP



25 µm Pinhole defect in Nafion®-115

Membrane Thickness Mapping

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

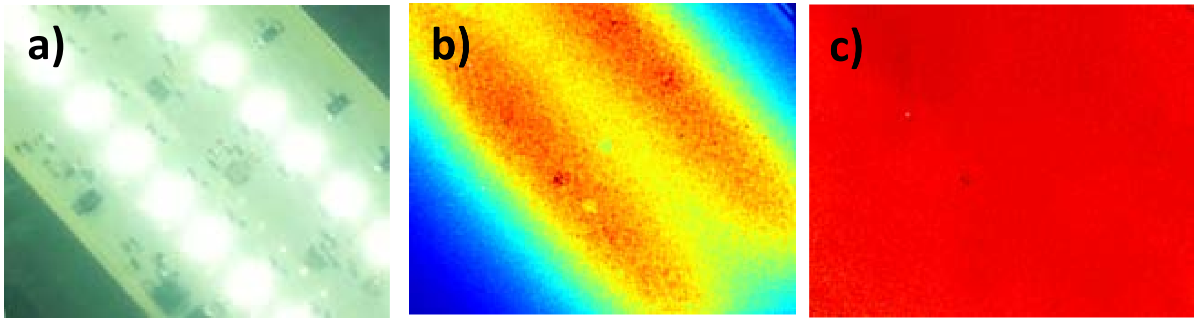
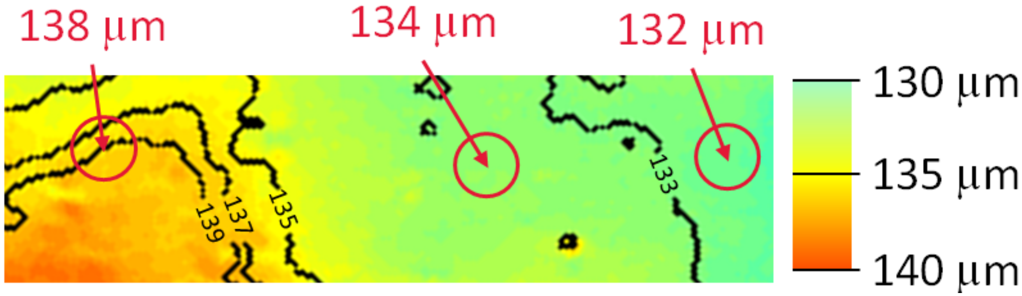


Image of Nafion®-115: (a) regular backlit photograph, (b) colorized image from Phase I area-scan camera, (c) image with background compensation



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

Image Analysis

- **Custom software and optical enhancement provides improved defect resolution**

The software process

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

Resultant image

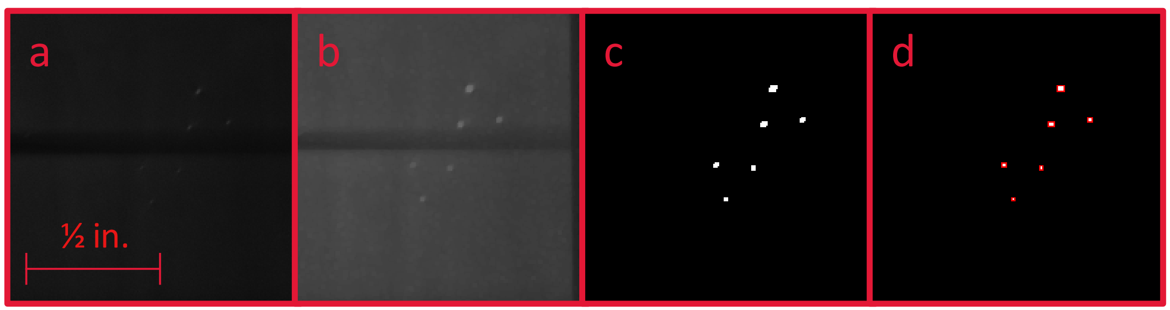


Image Processing

- LabVIEW program consists of 3 main loops that operate in parallel
- Acquisition and Processing Loops operate on Real-Time Module
- FPGA Loop runs on embedded Xilinx Kintex-7 FPGA

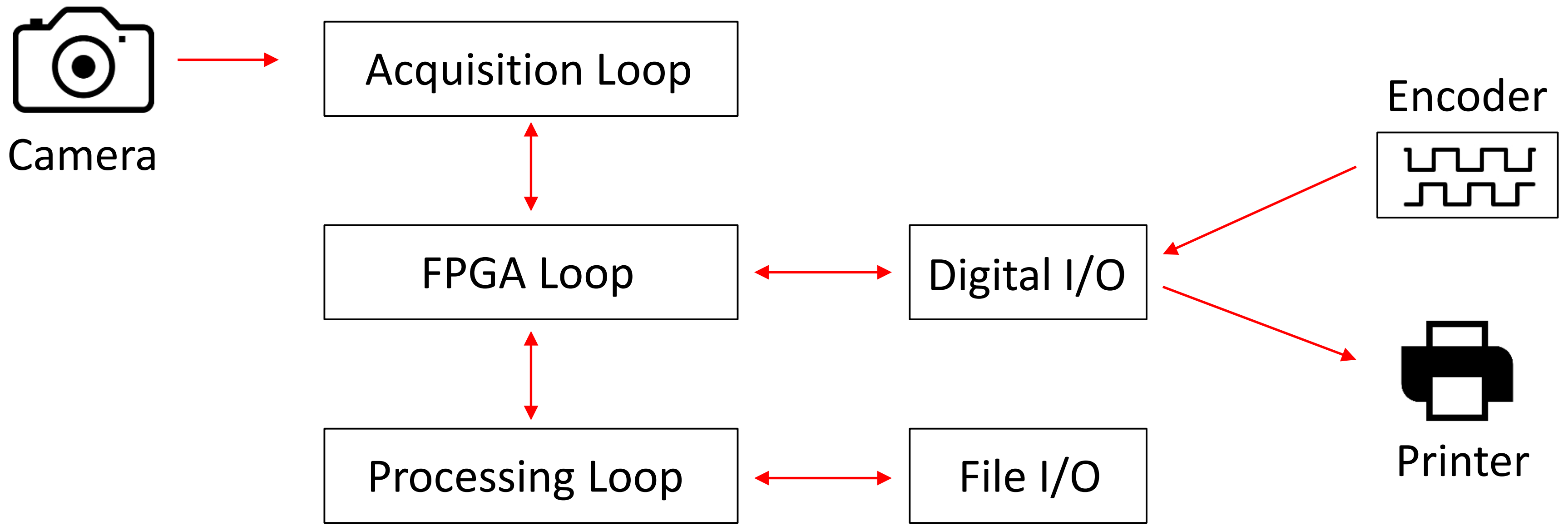


Image Processing

- **Acquisition Loop**
  - Get image from camera
  - Perform pre-processing of image
    - Brightness, Contrast, Gamma adjustment
    - Mask image based on Region of Interest
  - Send image to FPGA
- **FPGA Loop**
  - Apply Gaussian filter
  - Grayscale Morphology (Open, Close, Erode, or Dilate)
  - Binary Conversion based on threshold pixel brightness value
  - Digital I/O (Input from Encoder, Output to Printer)
- **Image Processing**
  - Get image from FPGA
  - Particle Analysis (size, shape, area, location)
  - Particle Filtering
  - Write image files and defect data

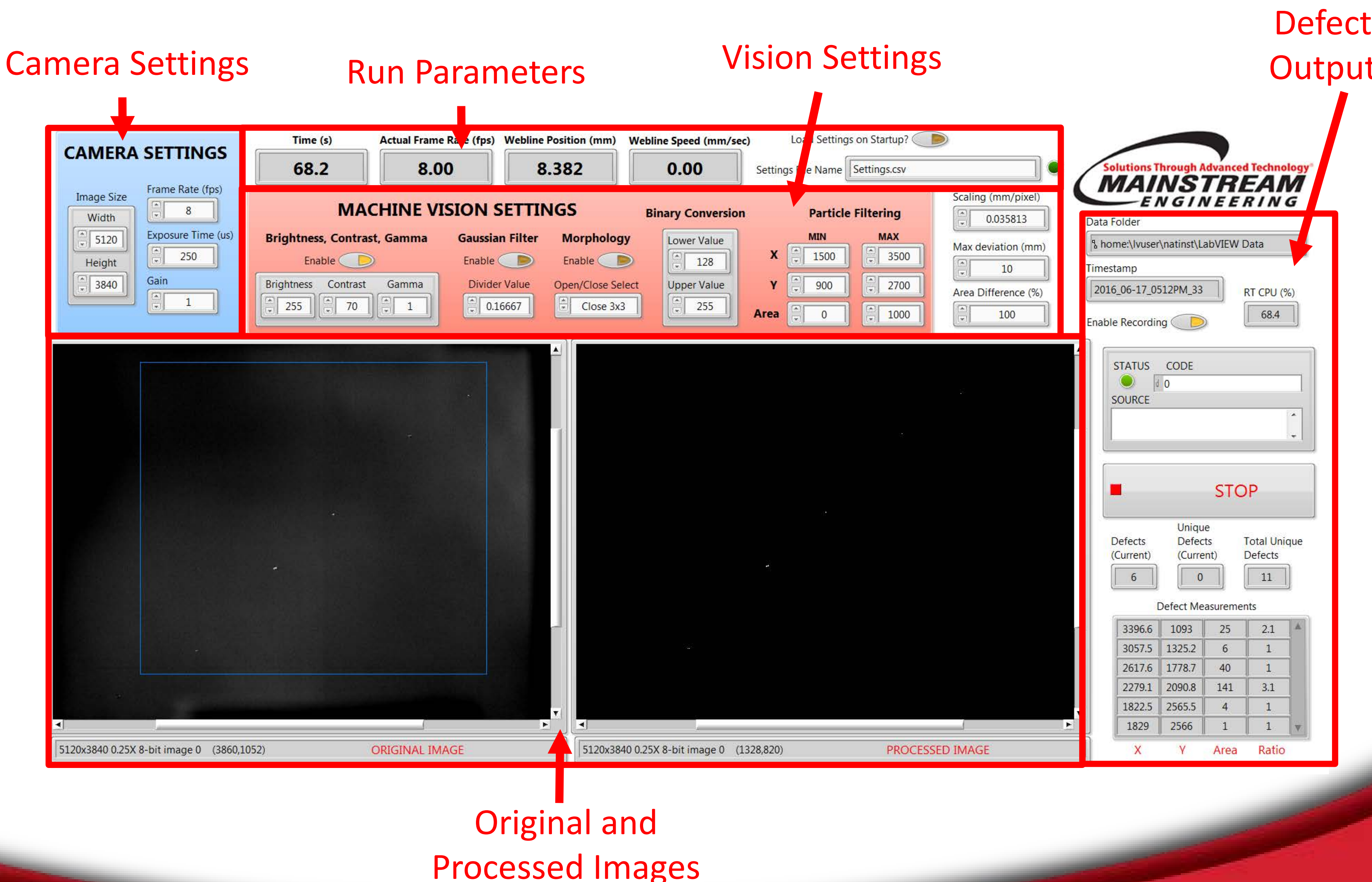
Image Processing Rate

- **Acquisition Loop**
  - Camera is limited to 16 fps
  - Pre-processing can be done while camera is acquiring next image
- **FPGA Loop**
  - Limited to 40 million pixels per second (onboard clock is 40 MHz)
  - Image processing algorithms limited to 8 pixels per clock cycle
  - At max resolution (5120 x 3840), the processing speed in 16 fps
- **Processing Loop**
  - If no images are saved (just defect locations), this step is limited to 12 fps
  - If all images are saved this step is limited to 8.4 fps

CPU Times for Image Processing

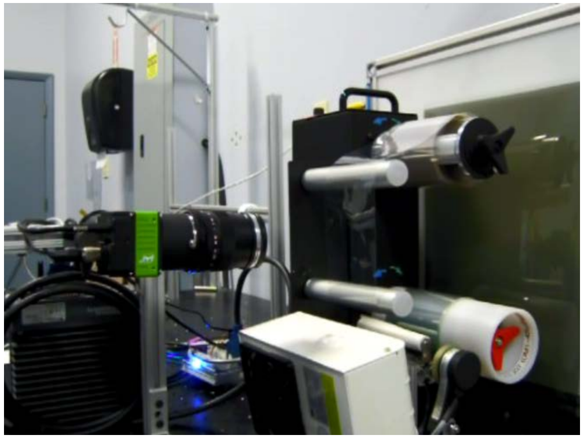
Processing Step	Time (msec)
Write Image File to Disk	73
Particle Filtering	16
Analyze Defects	29
Write Data Files	0.5
Other Functions	0.5
Total	119

Prototype Image Analysis UI

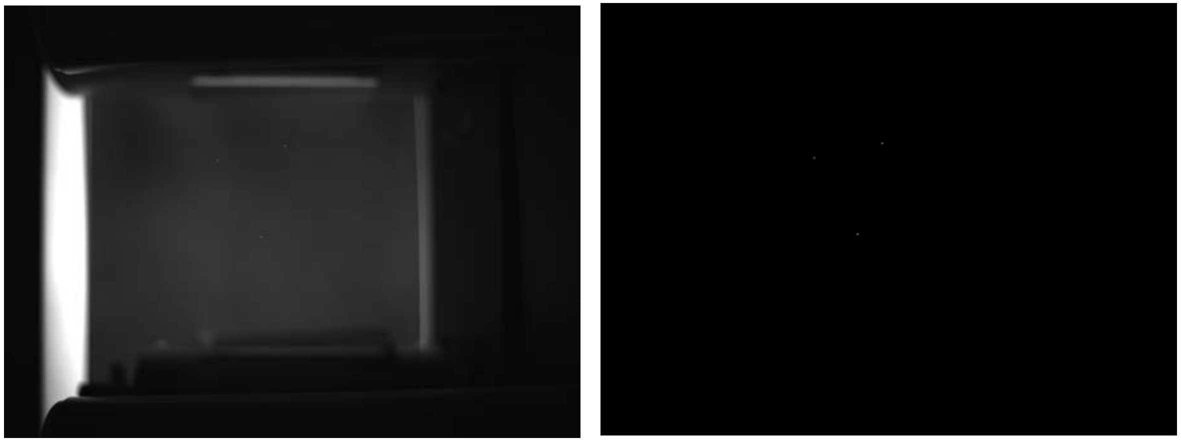


Defect Detection up to 100 ft/min

- Defects accurately detected in a range of supported and unsupported PEM membranes including Nafion-HP, Nafion-211, Nafion-115
- 63 of 63 100-μm pinhole defects identified in real-time at 50 ft/min



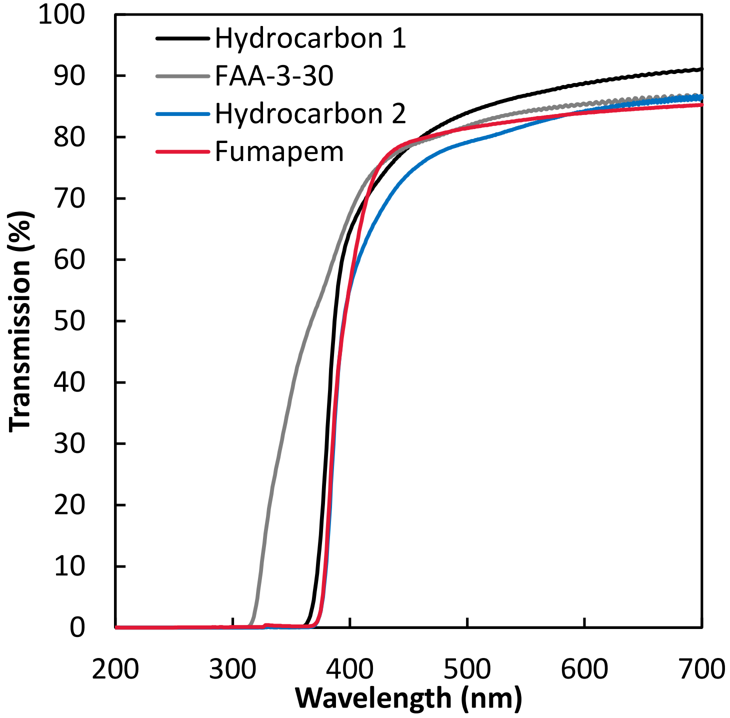
Video of webline rolling

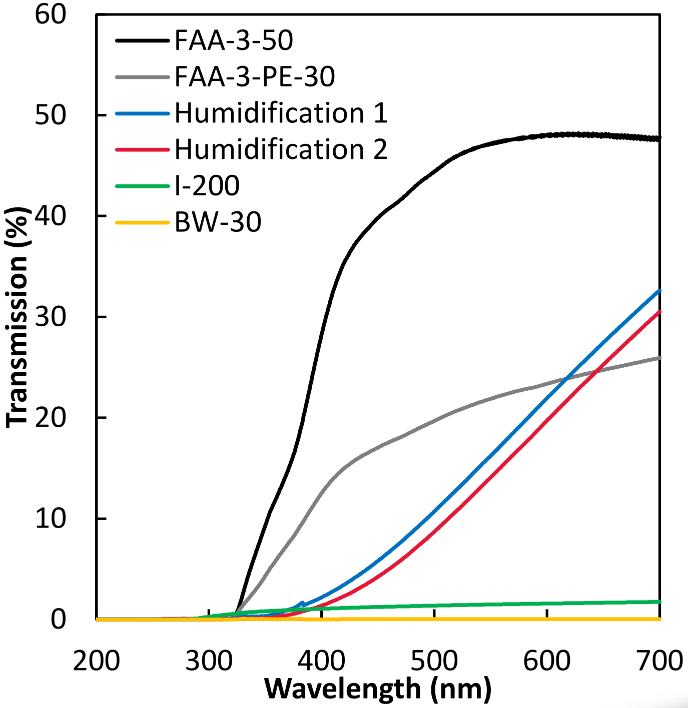


Slow motion video showing defect identification at 2 ft/min, raw image (left), and processed image with defects circled (right)

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)**





Response to Reviewer Comments

- **This project was not reviewed last year.**

Collaborations

Institution	Type	Extent	Role and Importance
National Renewable Energy Lab	Federal Laboratory	Major	Providing testing and technical assistance with determining the smallest defect to cause cell failure
Georgia Institute of Technology	University	Major	Providing pristine and defective membrane samples for QC testing and failure testing

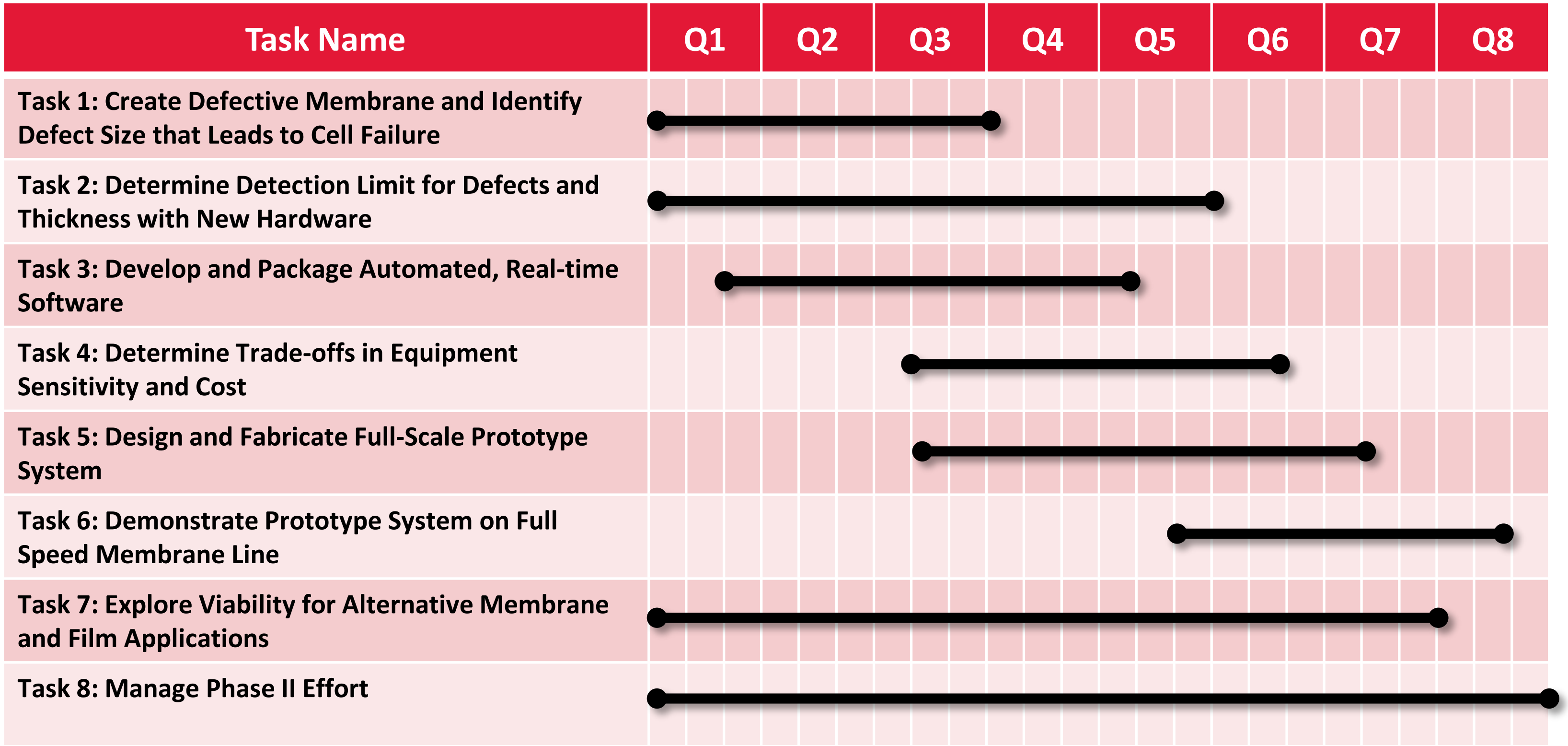
Remaining Challenges and Barriers

- **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 using SBIR Phase II funding to develop the system to a TRL 7 and commercialize the product
- 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
- Mainstream submitted a patent "Apparatus and Method for Cross-polarized, Optical Detection of Polymer Film Thickness and Defects." U.S. Patent Application Serial No. 15/170,360.

Proposed Future Work



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
- **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
- **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)

Summary

- 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 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®-115 at 50 ft/min on 6 inch webline
- Software development is complete, hardware has been selected, packaging has begun

SUPPORTING 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-Administrative Offices
4-Production
2-Research and Development
5-Product Development
3-Research and Development

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.

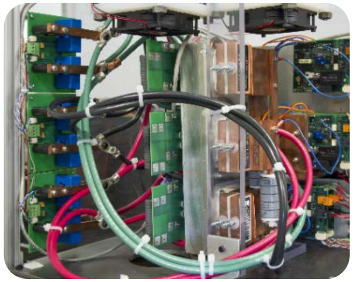
SBIR Successes and Awards

- **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

Mainstream's Focus Areas

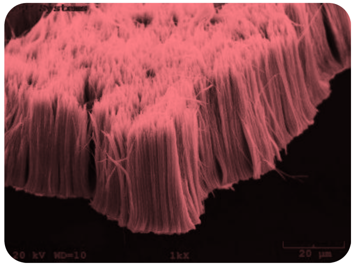
THERMAL CONTROL
- High Heat Flux Cooling
- Thermal Energy Storage
- Directed Energy Weapons
- Rugged Military Systems

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

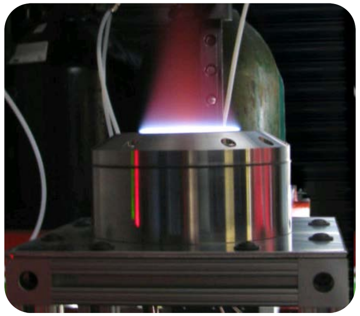


POWER ELECTRONICS
- High Speed Motor Drives
- Hybrid Power Systems
- Solar/Wind Electronics
- Pulse Power Supplies
- Battery Chargers

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



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



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