

# MEA Manufacturing R&D

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DOE Hydrogen and Fuel Cells Program  
2020 Annual Merit Review and Peer Evaluation Meeting

Project ID: ta001

# Overview

## Timeline

Start: July 2007

End: Project continuation and direction determined annually by DOE

% complete: N/A

## Barriers

Barriers	Target
E: Lack of Improved Methods of Final Inspection of MEAs	\$20/kW (2020) at 500,000 stacks/yr
H: Low Levels of Quality Control	

## Budget and Funded Partners

Fiscal Year	Total Funding*	LBNL	CSM
2019 (received)	\$0	\$0	\$0
2020 (planned)	\$800,000	\$295,000	\$80,000

\* Total funding is the sum of NREL and funded university partners (but not LBNL funding), and includes the work shown in S. Mauger (ta008)

# Relevance

- HFTO Multi-year R&D Plan – Manufacturing R&D milestones
- International Workshop on Quality Control series
  - NRC-Canada, Fraunhofer ISE, NREL co-organizing
  - Meetings in May 2019 (Vancouver) and September 2019 (Freiburg)
  - ~175 total attendees at the three international meetings to date
  - *NREL planning U.S. meeting*
- Path to hydrogen competitiveness – A cost perspective (Hydrogen Council, January 2020)
  - “Our findings suggest that **scale-up will be the biggest driver of cost reduction, notably in the... manufacturing of system components.** This will deliver significant cost reductions...”
  - “The **cost of low-carbon and/or renewable hydrogen production will fall drastically** by up to 60 per cent over the coming decade. This can be attributed to... **scaling up of electrolyzer manufacturing...**”
- FCTO FOA 2229: H2@Scale New Markets, Topic 1, Electrolyzer Manufacturing R&D
  - “Other innovative manufacturing approaches such as... **advanced real-time metrology techniques for QC/QA also have potential to enable overall system cost reductions.**”
  - “Developing best practices for... **quality control, in situ diagnostics/inspection (at required production rates, in-line), reducing reject rates, addressing non-uniformities...**”



**Attendees at Freiburg  
QC Workshop**

# Approach

- Understand quality control needs from industry partners and forums
  - *Engage LTE/H<sub>2</sub>@Scale community*
- Develop diagnostics
  - *Study underlying physics of excitation and material response*
  - *Use multi-physics modeling to guide development*
  - *Use a unique suite of in-situ testing capabilities to understand defect thresholds*
- Validate diagnostics in-line
- Transfer technology

## \*Annual Milestone Criteria:

- Line speed of 4-30 ft/min
- Minimum web length of 20 m

*Milestone completion impacted by COVID19 lab closure*

Date	Milestone/Deliverable (status as of 3/4/19)	Complete
3/19	Perform initial inspection studies with LTE materials	100%
6/19	Perform multi-spectral thickness imaging of PEMFC and LTE membranes	100%
9/19	Perform in situ and ex situ studies to understand the impact of pinholes	100%
12/19	Complete evaluation of LTE in situ defect study goals and needs	100%
3/20	Evaluate the impact of pinholes experimentally and computationally	95%
6/20	Understand the impact of line speed on y-resolution of thickness imaging*	10%
9/20	Perform work toward membrane thickness imaging for thick membranes	50%

# Collaborations

## Industry Collaborators

*Objective: ensure we continue to get detailed input on manufacturing QC needs, prioritization of diagnostic development, feedback on technique capabilities, and pursue tech transfer*

- **GM (CRADA):** development of in-line inspection techniques
- **Mainstream Engineering (CRADA):** development of in-line inspection techniques, in situ membrane defect studies, mapping XRF studies
- **Gore:** membrane inspection methods and full-roll scanning
- **Nel/Proton:** QC development for LTE MEA materials and structures and understanding MEA defect effects

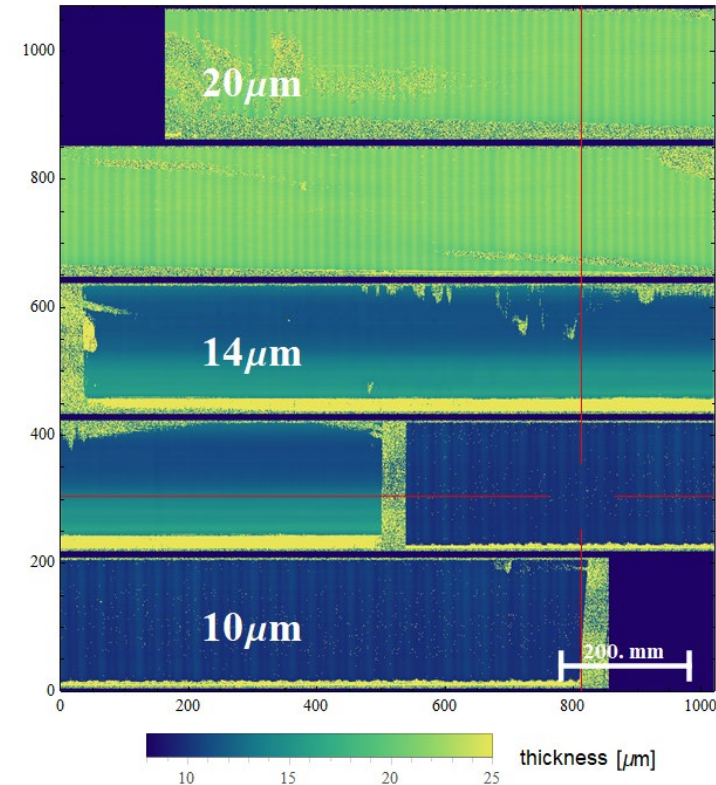
*Leveraging separately funded activities with **3M, Nel/Proton, Giner, GM, Plug Power, and Lynntech** to further understand industry directions and challenges*

## Labs and Academia

- **Lawrence Berkeley National Lab:** model development and integration, x-ray computed tomography
- **Colorado School of Mines:** diagnostics R&D, MEA ex situ characterization
- **National Research Council-Canada (NRC):** membrane inspection, CCM fabrication and testing
- **CEA-Liten:** QC development and validation for electrode manufacturing
- **NRC and Fraunhofer-ISE:** international workshops and collaboration on QC and defect studies

### Membrane thickness mapping: Web-line run

- Objectives: run longer web lengths, utilize new GPU-based image processing, validate impact of web speed on down-web resolution
- Ran 11 different polymer films, including fuel cell and electrolysis membranes from Chemours, 3M and Gore, totaling over 100 m (and 11 TB of data!)
- Results:
  - Successful imaging of single-layer and several multi-layer membranes less than 30  $\mu\text{m}$  thick
  - For thicker films, in most cases the wavelength range and/or wavelength resolution of the current hyperspectral imager were not sufficient
  - Given the set FOV of the detector (6 inches) and the detector pixel density (2000 pixels), the x resolution was 100  $\mu\text{m}$
  - Given the web-speed (1-4 ft/min) and camera frame rate (100 Hz), maximum y-resolution was 50  $\mu\text{m}$
  - The estimated error in the thickness measurement was approximately 0.2  $\mu\text{m}$
  - New NREL-developed GPU-based scheme enabled near-real time image processing



**Real-time thickness imaging results from three membranes showing: small-scale thickness chatter in the 10 and 20  $\mu\text{m}$  webs, and a persistent cross-web thickness variation in the 14  $\mu\text{m}$  web**

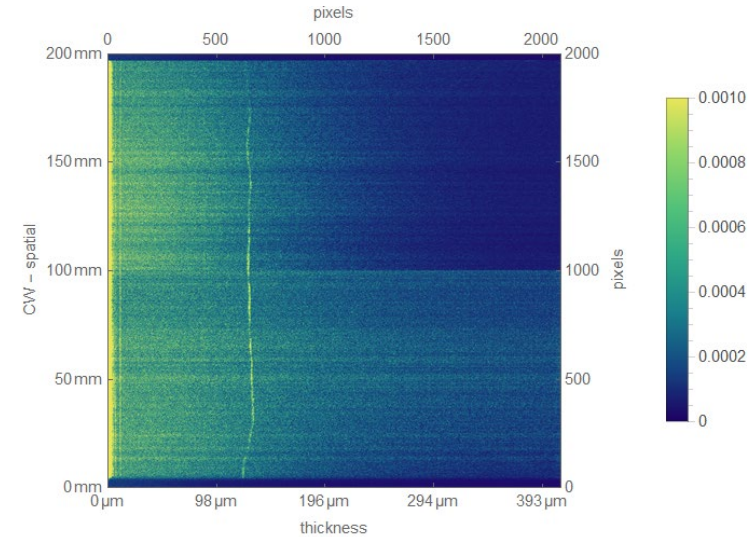


# Accomplishments

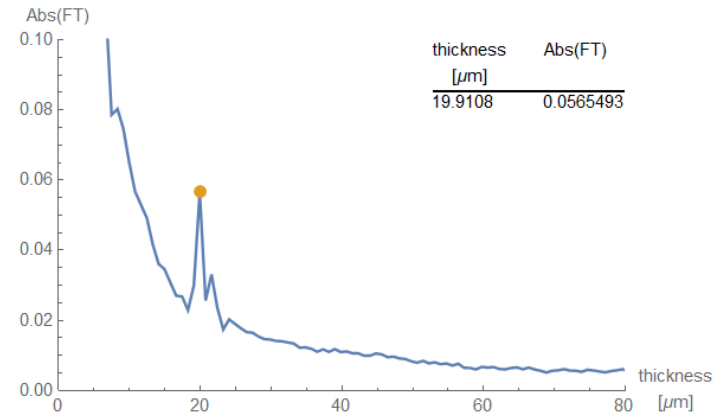
## Studied membrane thickness imaging for LTE and reinforced membranes

### Membrane thickness mapping: thick (LTE) and reinforced/multilayer membranes

- N115 (125  $\mu\text{m}$ ) imaging data using standard data processing algorithm did not successfully measure thickness
- Upon further manual analysis, weak fringes were obtained in the 800-1000 nm (near-IR) spectral range
  - Hyperspectral detector with better  $\lambda$  resolution in this range would improve S/N
- Nafion XL and Gore reinforced membranes successfully measured
- Rough surface on Nafion HP makes measurement difficult in UV/Vis range
  - However, we can successfully measure with NIR detector



**N115: bright vertical line represents the thickness as a function of cross-web position**



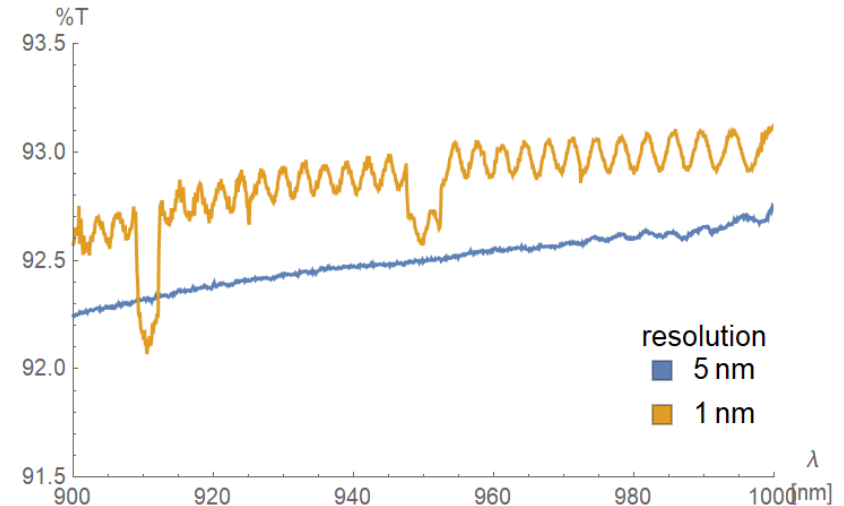
**Nafion HP: Fourier transform peak fitting from NIR spectra, showing thickness peak**

# Accomplishments

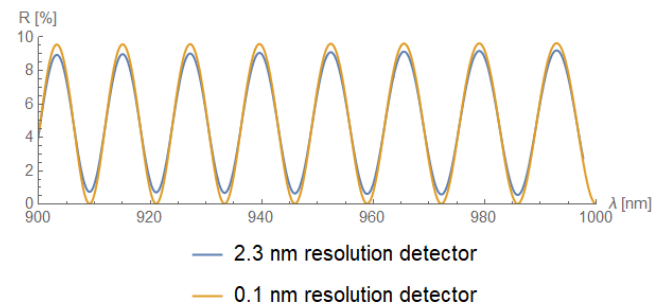
Detectors with higher wavelength resolution enable faster, cheaper measurement

## Membrane thickness mapping: Impact of detector wavelength resolution

- For membranes thicker than  $\sim 30\text{-}50\ \mu\text{m}$ , fast UV/Vis detectors with low wavelength resolution often cannot detect interference fringes
- We studied the impact of detection with higher wavelength resolution using a benchtop spectrophotometer to understand if different detector design could enable improved measurement
  - Result: higher wavelength resolution would enable the use of cheaper, faster, and higher spatial resolution UV/Vis detectors (compared to detectors in the IR region)
- We modeled the response of the optical detector as a function of wavelength resolution and membrane thickness
  - Enables the prediction of needed specifications for in-line detectors



Comparison of UV/Vis spectra between 1 nm and 5 nm wavelength resolution detectors for a 100  $\mu\text{m}$  membrane



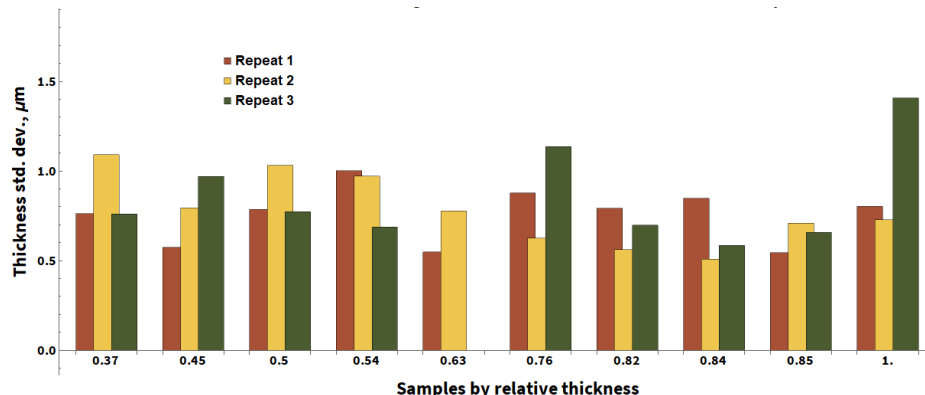
Optical modeling results showing the impact of wavelength resolution for 25  $\mu\text{m}$  (top) and 100  $\mu\text{m}$  (bottom) membranes



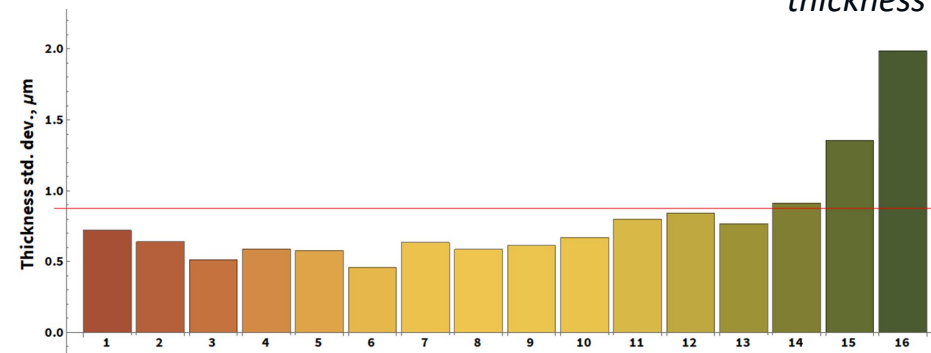
### Update on thermal scanning for measurement of membrane thickness in half-cells (GM CRADA)

- Analyzed the error of the measurement with same-orientation repeats
  - Mean standard deviation (SD) of the measurement, for all thicknesses, is  $\sim 0.8 \mu\text{m}$
- Assessed the potential for cross-web mapping by parsing full-sample data into 16 cross-web lanes
  - SD within each lane is comparable to overall sample SD
  - Indicates potential for mapping
- Working on further improvements to method signal:noise

*Assessment of error includes systematic error as well as actual deviations in the membrane thickness*



SD and repeatability of thickness measurement for all half-cell samples

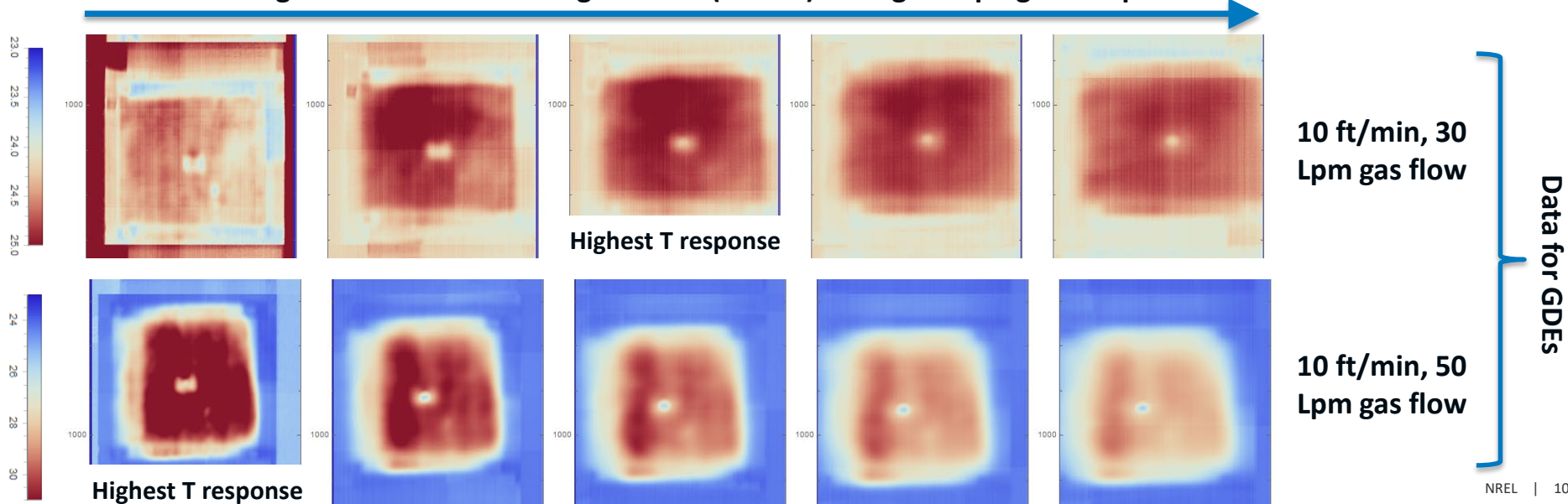


SD in each of 16 cross-web lanes of data for one half-cell sample

## In-line validation of QC methods (RIF and Optical)

- Leveraged CEA collaboration to access different electrode manufacturing method (screen printing) for QC validation
  - Gas-diffusion electrodes (GDE)
  - Catalyst-coated decals (CCD)
- Demonstrated multi-modal imaging (simultaneous IR and optical)
- Reactive Impinging Flow (RIF) learnings
  - Temperature rise (signal) is a function of line speed, H2 flow rate and distance of datum from impingement point -> *Need to optimize configuration for sensitivity*

Increasing distance of data taking location (datum) from gas impingement point



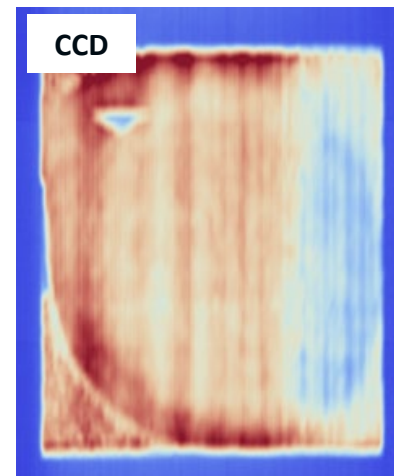
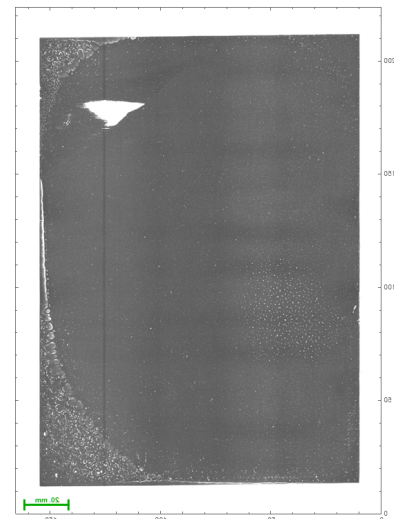
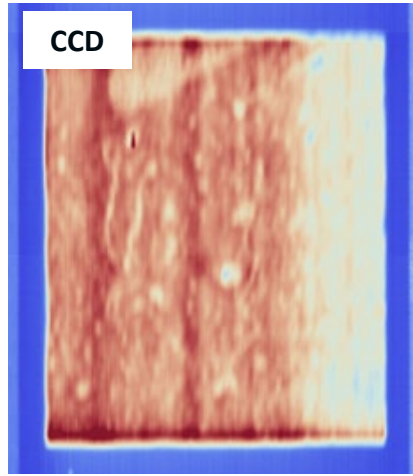
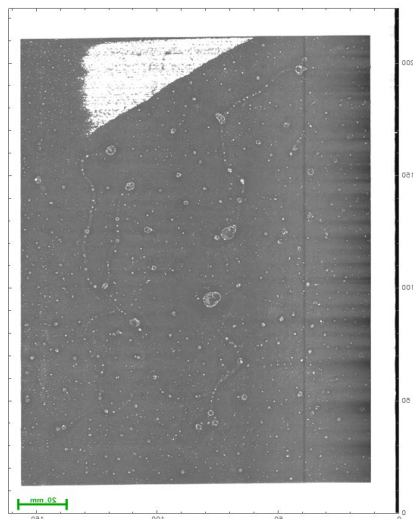
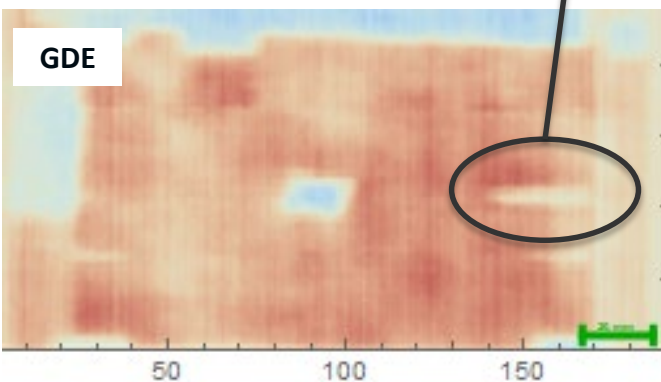
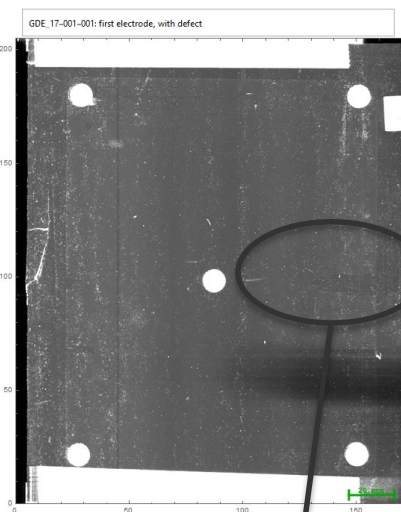
# Accomplishments

Leveraged CEA collaboration for further RIF demonstration and learning

## In-line validation of QC methods (RIF and Optical)

- Explored correlation between optical and IR methods for the detection of process-created and intentionally created irregularities

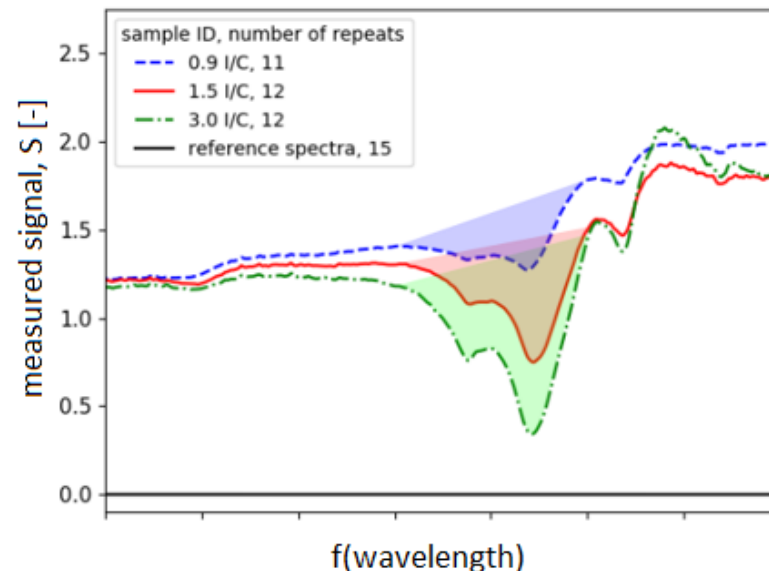
Faintly visible change in grayscale in optical image, more visible as cooler (thus lower loading) location in IR image



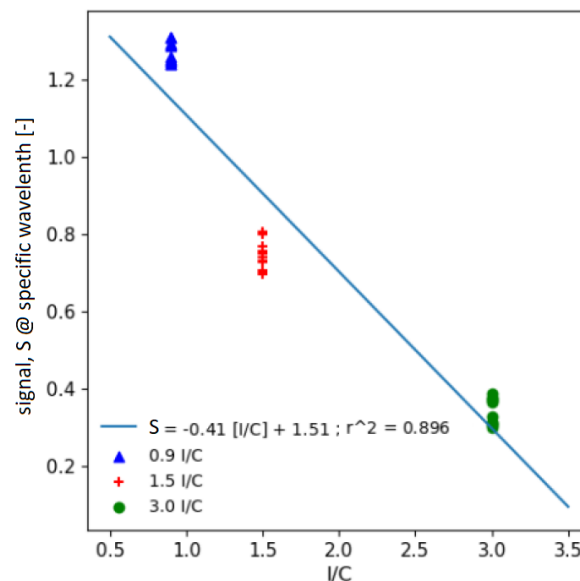
Examples of correspondence between optical (top) and IR (bottom) methods for process-created irregularities: circular features likely related to drying (left) and non-uniformity related to squeegee wiping (right)

### Nafion spectroscopy studies

- Motivation: in-line measurement of ionomer content in electrodes and inks has been identified as a quality inspection need
  - No current methods are known
- Explored multiple spectroscopy methods (UV-Vis, IR, ATR-FTIR, spectrofluorometry, capacitance spectroscopy)
- As an example, we identified a spectral region with signal proportional to ink ionomer loading
  - Similar result for a powdered catalyst layer
  - Exploring possible in-line configurations



Measured spectra for inks with three ionomer:carbon ratios



Measured signal minima for inks with three ionomer:carbon ratios



# Accomplishments

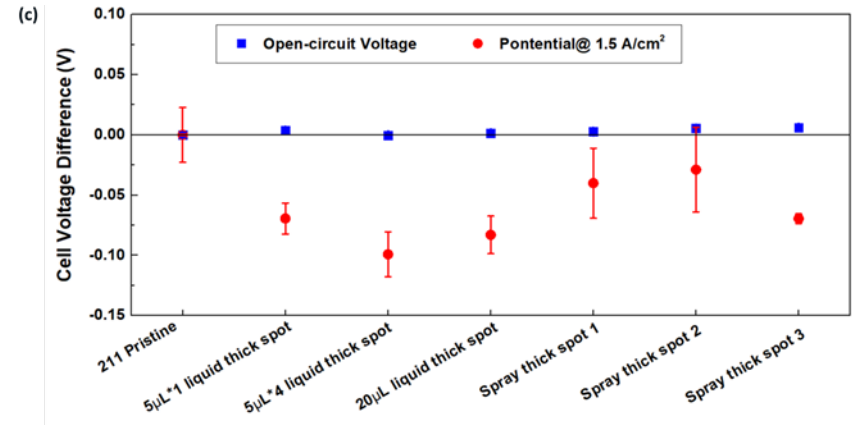
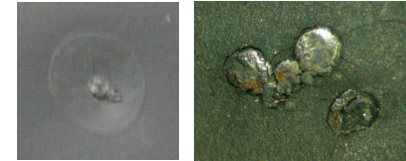
## Electrode thick spots can impact MEA morphology and initial performance

### Effects of electrode thick spots

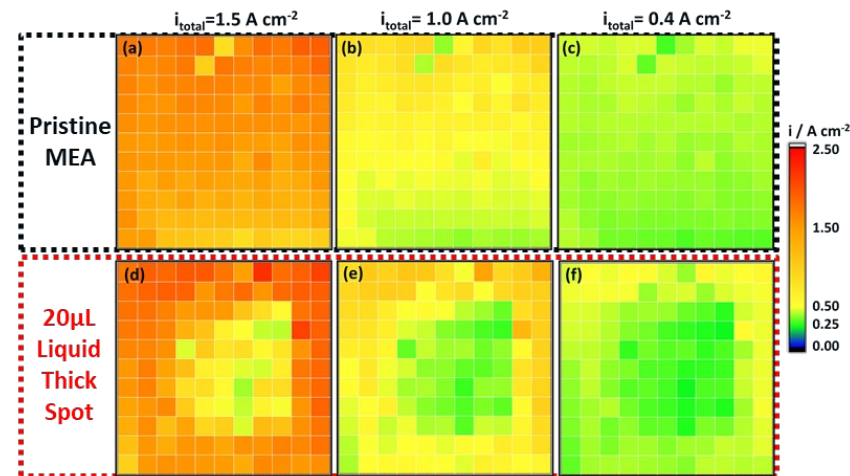
- Ongoing study
  - Addressing known process defects
  - Used drop-casting and spraying with mask to create thick spots
- SEM (after hot press but before testing) shows potential extensive morphology change due to thick spot
- Initial performance testing (on NRE211)
  - No impact on OCV
  - Significant impact (up to 100 mV) at 1.5 A/cm<sup>2</sup>
  - Spatial in situ testing shows large area of impact around the thick spot
  - *No initial performance impacts for thick spots on Nafion XL*

ECS Transactions 2019, 92 (8), 351-359  
J Power Sources 2020, 466, 228344

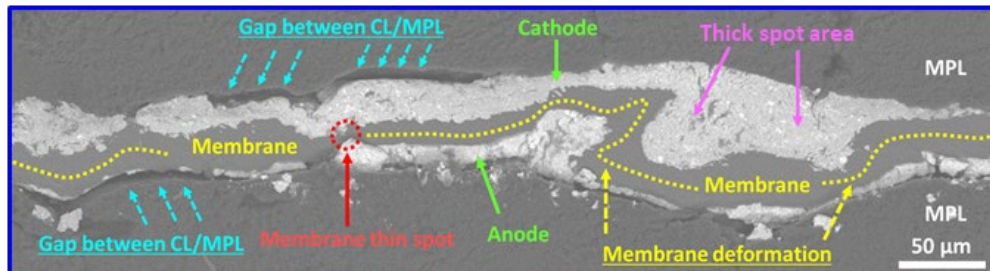
Reminder:  
These actually happen!



Impact of thick spots on initial performance



Segmented cell data at three current densities showing wide area of performance impact



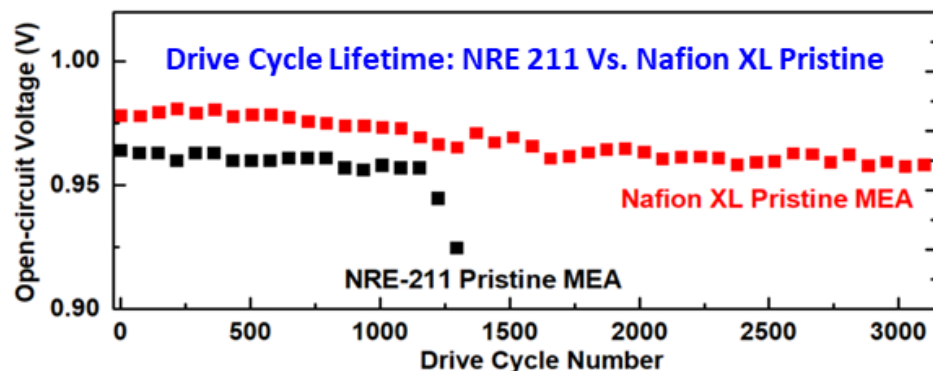
SEM of hot pressed MEA of drop-cast '4x 5 µL' thick spot showing significant morphology impact (CSM)

# Accomplishments

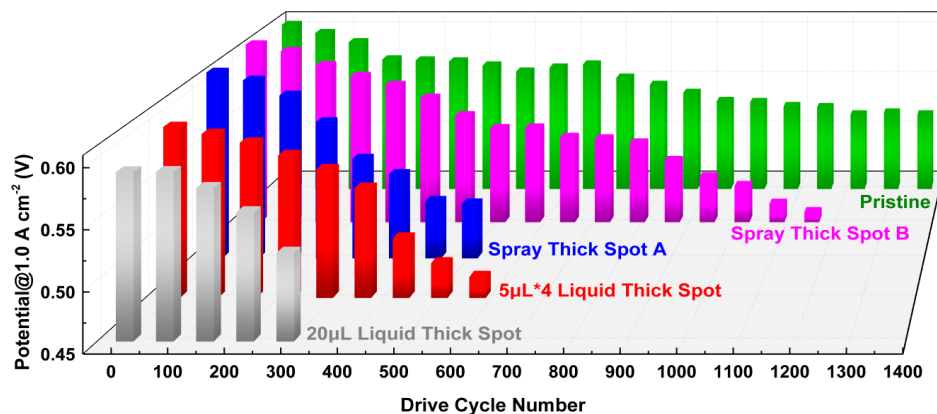
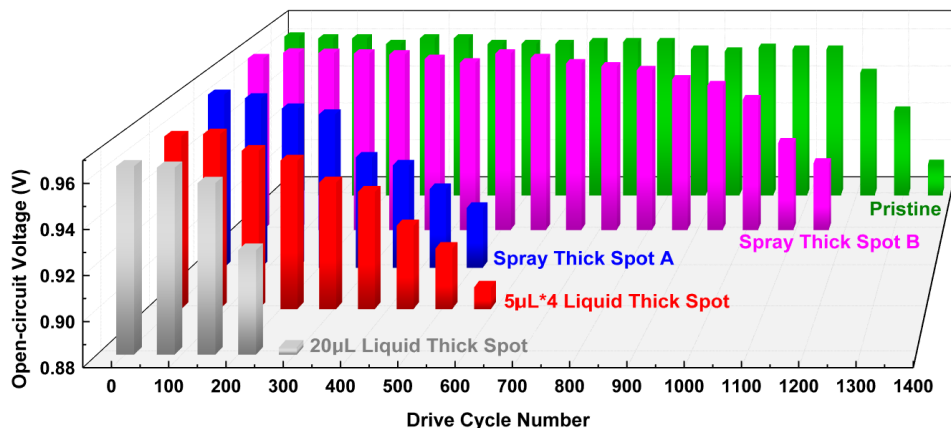
## Electrode thick spots impact MEA performance over time

### Effects of electrode thick spots

- Drive cycle testing
  - ‘New European Drive Cycle’
  - Observe clear reduction of performance over time via both OCV and voltage measurements at 1 A/cm<sup>2</sup>
  - Testing of thick spots using Nafion XL is ongoing



Comparison of cycling results between pristine MEAs: using NRE211 (black), using Nafion XL (red)



Drive cycle testing results vs. pristine for all thick spot cases (NRE 211 membrane): OCV (left), voltage at 1 A/cm<sup>2</sup> (right)

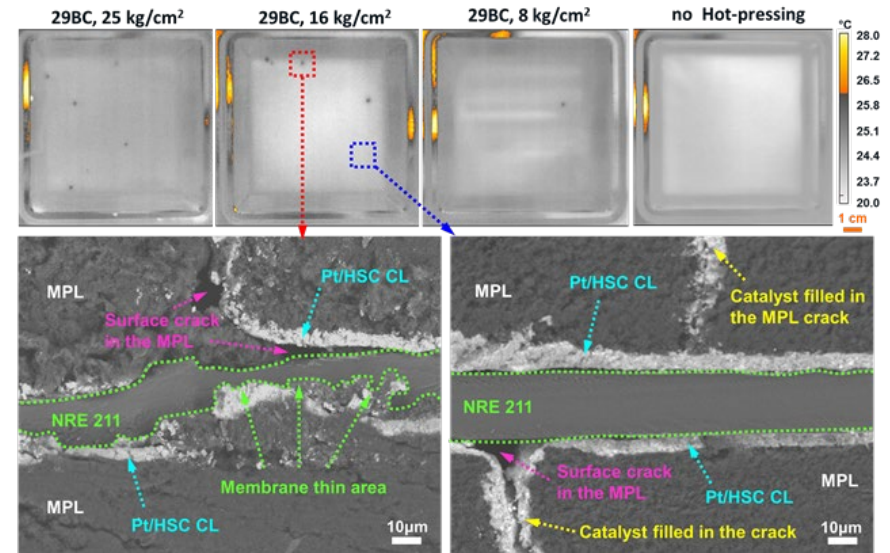


# Accomplishments

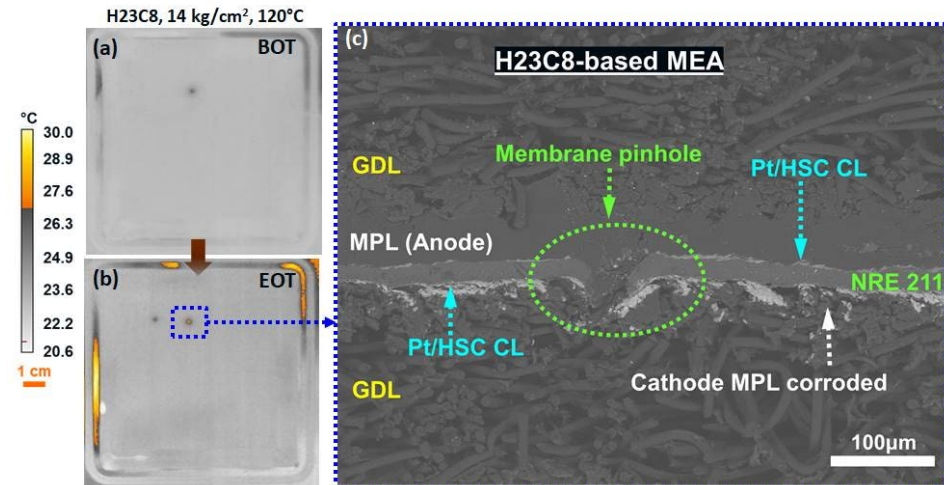
## Identified intrinsic irregularities in GDE MEAs that can lead to failure points

### Effects of MEA manufacturing methods

- Identified ‘intrinsic’ irregularities while establishing baseline conditions for pinhole study (using GDE-based MEAs)
- Points of locally increased hydrogen crossover using BOT infrared imaging (H2XO/IR)
  - ‘Warm spots’
  - Frequency is greatly impacted by hot-pressing pressure and temperature, and MPL roughness
  - SEM shows significant morphology disruption at warm spot due to: MPL cracks, GDL fibers, CL agglomerates
- These ‘warm spots’ result in failure points in AST testing



H2XO/IR mapping and SEM (CSM) showing BOT irregularities, impact of hot-pressing conditions, and local morphology



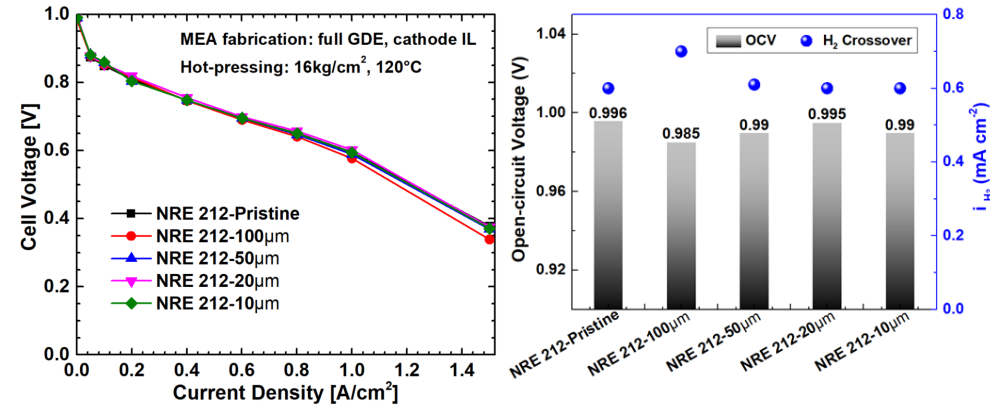
BOT and EOT H2XO/IR mapping and SEM (CSM) showing that BOT warm spots can lead to failure points

# Accomplishments

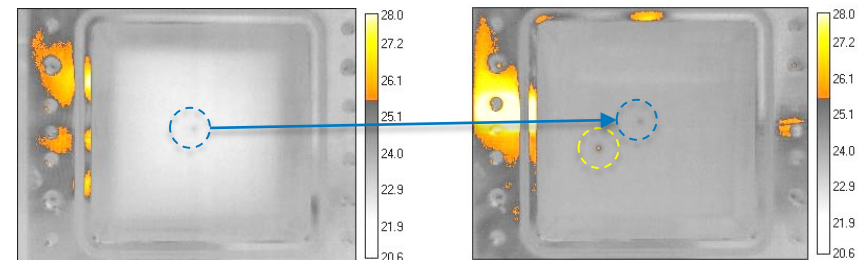
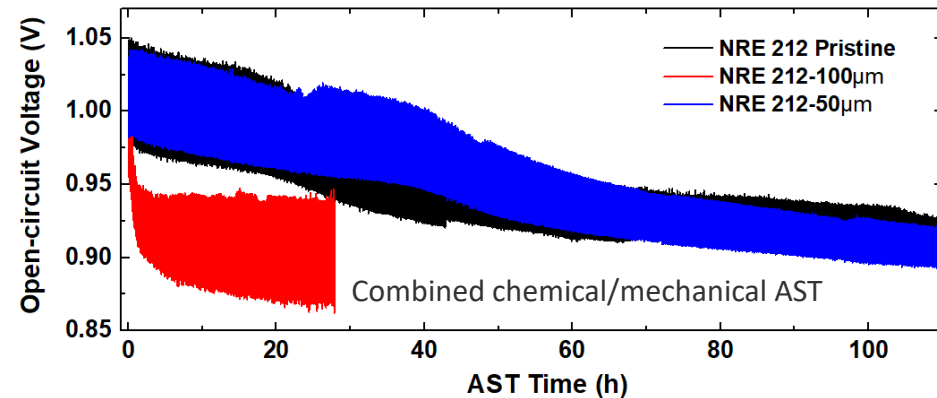
Found minimal initial performance impact except for largest pinhole

## Effects of membrane pinholes

- Objectives
  - Understand impact on performance and lifetime of
    - Pinhole size (using laser drilling): 10, 20, 50, and 100  $\mu\text{m}$
    - Membrane type and thickness
  - Explore differences in impact based on MEA fabrication process (GDE vs. CCM)
- NRE212 with pinholes
  - Minimal impact on initial performance for all but largest pinhole
  - Lifetime is severely shortened for 100  $\mu\text{m}$  pinhole
- Will study NRE211 and Nafion XL



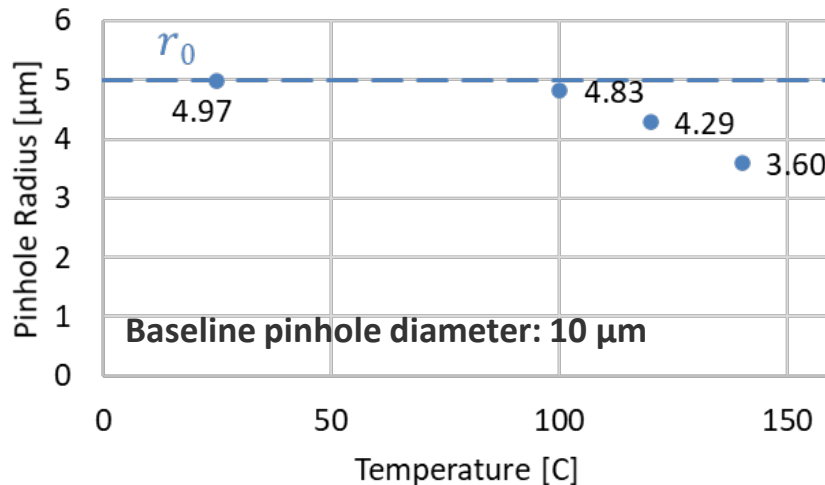
## Initial performance impact as a function of pinhole diameter



AST comparison (above) and EOT H<sub>2</sub>XO/IR showing development of separate failure point (below)

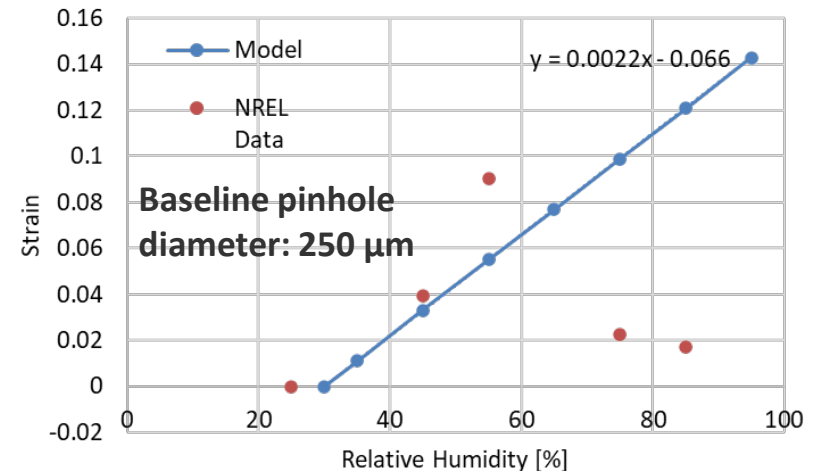
## LBNL mechanical modeling activities to understand and predict behavior of membrane pinholes

### 1. Impact of hot-pressing on pinhole size



- At constant pressure, pinhole radius is predicted to have a non-linear decrease with temperature
- In a similar analysis at constant temperature, pinhole radius is predicted to decrease linearly with pressure

### 2. Impact of temperature and humidity without membrane constraint



- Prediction matches NREL microscopy data (see backup slide) at low humidity
- Data diverge at high humidity due to measurement problems
- NREL is working on improved measurement configuration

# Accomplishments

Obtained industry inputs on critical LTE component defects, and assessed in situ testing tool needs

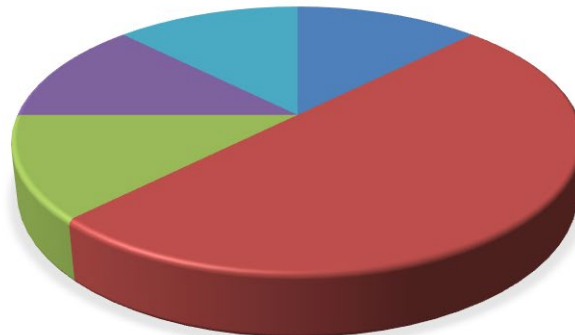
## Understanding key challenges related to in situ defect studies for low-temperature electrolysis (LTE) MEAs

- Want to expand our fuel cell in situ defect capabilities and studies to include LTE, but need to understand:
  - Different materials and structures
  - Different operating conditions
- Received input from four US companies developing LTE MEAs and/or components related to:
  - Manufacturing and assembly (not durability)
  - Key aspects to study
- Identified research tools to use, add or develop

### Inputs

- *Most frequent/critical defects?*
- *Main reasons for cell failure?*
- *Critical component properties?*
- *Typical cell operating conditions?*

**Example result:**  
Irregularities that occur in LTE MEA manufacturing (pie segments indicate frequency of responses)



- Electrode Misalignment
- Voids/nonuniform catalyst layer
- Point defects (GDL)
- Particulates
- Nonuniform PTL

# Technology Transfer Activities

- SBIR Phase II-b collaboration with Mainstream Engineering (see ta005)
  - Advance QC prototype device to commercializable configuration
- FCTO FOA #1874 project with 3M (see ta026)
  - LTE CCM and membrane processing and QC
- H2@Scale CRADA project with HyET (see h2006)
  - EHC MEA manufacturing
- *FCTO FOA #1874 project with Nel/Proton*
  - *LTE CCM processing and QC*
- *Nel/Proton Technical Services Agreement (TSA)*
- *Lynntech TSA*
- Advanced Materials Manufacturing Roll-to-roll (R2R) Consortium (see ta007)
  - Multi-lab consortium focused on material development, synthesis, process development, and QC/controls development for energy materials
  - *New FCTO-funded multi-lab activity with participation from GM, Plug Power, Nel/Proton and Giner*
  - *New multi-lab CRADA with Nel/Proton*
- GM collaboration (discussed above)
- U.S. Patent 10,480,935 B2, “Thickness mapping using multispectral imaging,” P. Rupnowski, M.J. Ulsh; November 19, 2019.

*Italics: new  
projects in  
FY2020*

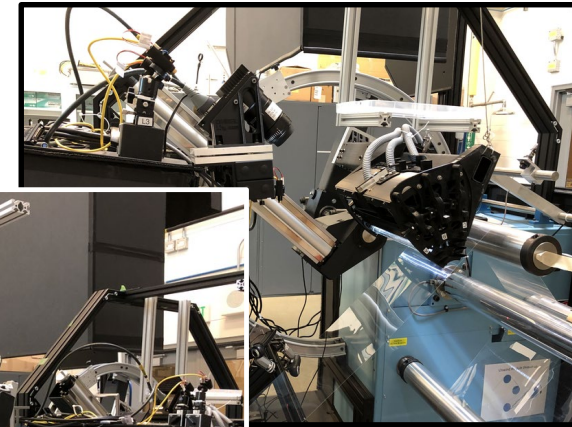
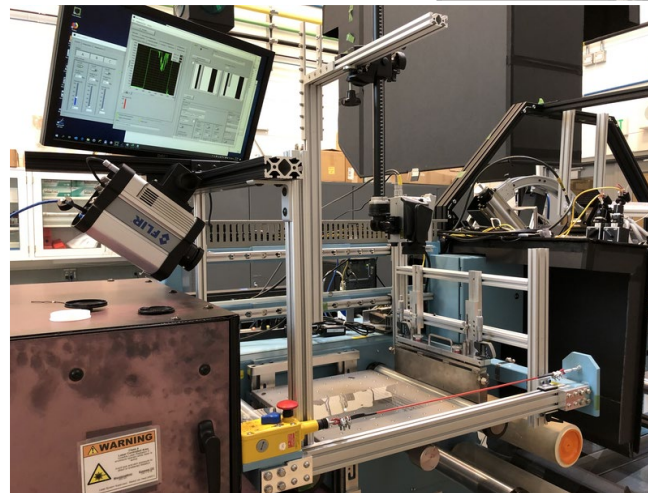


# Technology Transfer Activities

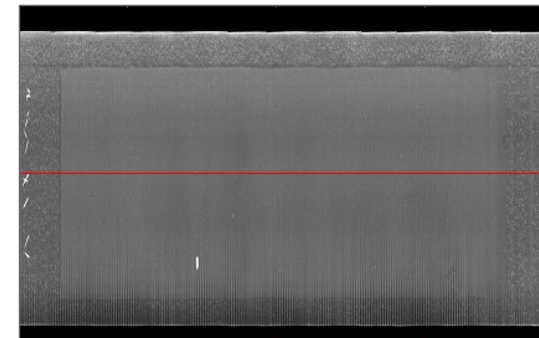
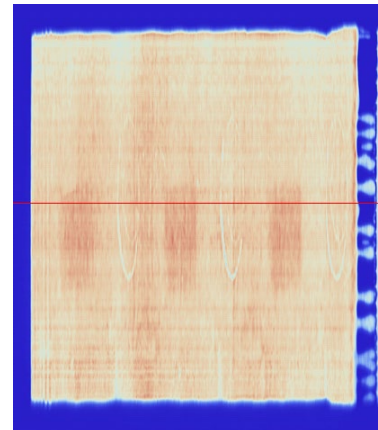
## Lynntech TSA project

- Validate in-line QC techniques for R2R GDE manufacturing
  - Used NREL-coated GDE webs
- First (at NREL) simultaneous multi-modal web-line inspection of roll materials
  - Optical reflectance
  - Reactive Impinging Flow (RIF) using MIR and FIR cameras
- Validates multi-modal data acquisition for roll materials

Optical reflectance (right) and RIF (below) setup on web-line



Examples of in-line RIF (left) and optical reflectance (right) imaging



*Partner required statement: "This material is based upon work supported by the USAMRAA under Contract No. W81XWH-16-C-0035. Any opinions, findings and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the USAMRAA."*



# Proposed Future Work

“Any proposed future work is subject to change based on funding levels.”

## Barriers and Needs

- We actively engage with partners to understand their needs, based on their specific processes, materials and MEA constructions
- We attended the recent QC Workshops in Canada and Germany, where our understanding of barriers and needs was confirmed and expanded
- We will pursue the barriers and needs documented in the MYRD&D Plan

## Future Focus

- Continue to work with and gather current information on challenges from as many industry partners as possible in the fuel cell and electrolysis space
  - Seek opportunities to implement diagnostics in industry
  - Initiate new technical activities to address new/unaddressed needs
- Further demonstrate in-line membrane thickness mapping for LTE membranes
- Explore in-line configurations for spectroscopic techniques
- Explore techniques for multilayer structures, leveraging AMO activities
- Study the effects of relevant defects on cell performance and lifetime
  - Expand focus to priority applications, e.g. LTE and Heavy-duty FC
- Continue to develop and apply predictive models to assist in the understanding of the performance and lifetime impacts of defects

# Summary

- Relevance
  - These R&D topics continue to garner domestic and international interest
- Approach and Collaborations
  - Continued detailed information exchange with industry partners on QC priorities
  - *Several milestones were impacted by COVID19 lab closure*
- Accomplishments
  - Further in-line demonstration of membrane thickness imaging
    - Long samples, impact of web speed, tested many membranes
    - Further development of technique for reinforced and thick (LTE) membranes
    - Identified improvements through increased detector wavelength resolution
  - Continued development of thermal scanning for half-cell membrane thickness measurement
  - Demonstrated in-line multi-modal (optical and IR) imaging of coated electrodes on multiple projects
    - Gained further process understandings for the RIF technique
  - Continued in situ studies of the effects of electrode thick spots and membrane pinholes
  - Explored the formation of failure points in MEAs due to fabrication conditions
  - Continued predictive model development for membrane response to RH, pressure and temperature
  - Obtained industry information on LTE defects and needed testing tools
- Progress
  - Explored spectroscopic methods for in-line measurements
- Tech Transfer
  - Leveraging HFTO investments for many industry-focused projects

# Responses to Previous Year Reviewers' Comments

Comments: *"Once there is a pinhole, it quickly propagates, so if the minimum acceptable size is unknown, it is hard to know whether the detection is good enough."*

Response: We totally agree with the reviewer. That is why we have the in situ defect study aspect of the project, in addition to the development of the QC methods themselves. The objective of the in situ defect studies is exactly to determine what those thresholds for detection are.

Comments: *"The post-mortem work is good, but it seems many investigators have already published these techniques."*

Response: We assume that the reviewer is referring to the work using infrared methods to identify failure points and extents in already-tested (in an operated stack) LTE CCMs. We agree with the reviewer that using IR methods to evaluate failed cells from stacks is known in the literature. However, in all of these cases known to us, these were fuel cell stacks, not LTE stacks. Part of our efforts over the last two years have been to validate the inspection methods developed for fuel cell materials now on LTE materials. In some cases the methods will work as-is. But when this is not the case, we need to understand why, and pursue modifications to the existing methods, or develop new methods, to meet the inspection need.

Comments: *"The team has an excellent team of collaborators, but it is unclear what the team is doing with General Motors."*

Response: The fuel cell group at NREL has had a multi-topic CRADA with GM for many years. Manufacturing R&D is one of the topics, and GM's main interest in this space has been the development of in-line QC methods for MEA structures. GM has provided us feedback on the relevance and direction of our work, regarding specific materials, defect types, and methods that we have and are exploring. Our AMR presentations for the last three years show the progression of the development of a specific QC technique to measure the thickness of a membrane in a half-cell configuration. This work has been identified as being in collaboration with GM. We also work with and receive guidance from GM through other related projects.

Comments: *"It is not certain that Gore or other PEM manufacturers currently have a problem with thickness mapping. Clearly, the thin-film industry has methods to measure this already."*

Response: We are certainly in agreement with the reviewer that it should be one of our main goals (and it is) to continuously ensure that our activities are addressing industry needs. We do this through our direct interaction with industry as well as through venues such as the International QC Workshops. We without question have received inputs from multiple partners, as well as through these broader venues, that thickness mapping is of interest. We also assert, with good knowledge of the roll-to-roll industry, that there is no existing capability to map the thickness of membranes in real-time, in-line. Single-point measurements exist, yes. But not 100% areal mapping.

# Acknowledgement

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Josias Ochoa  
Jason Pfeilsticker



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Derek Jacobsen  
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Samantha Medina



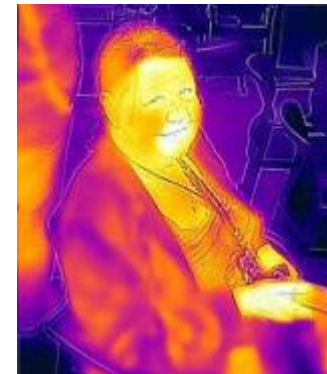
## LBNL

Adam Weber  
Ahmet Kusoglu  
Claire Arthurs



## DOE

Nancy Garland



# Thank You

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[www.nrel.gov](http://www.nrel.gov)

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# Technical Back-Up Slides

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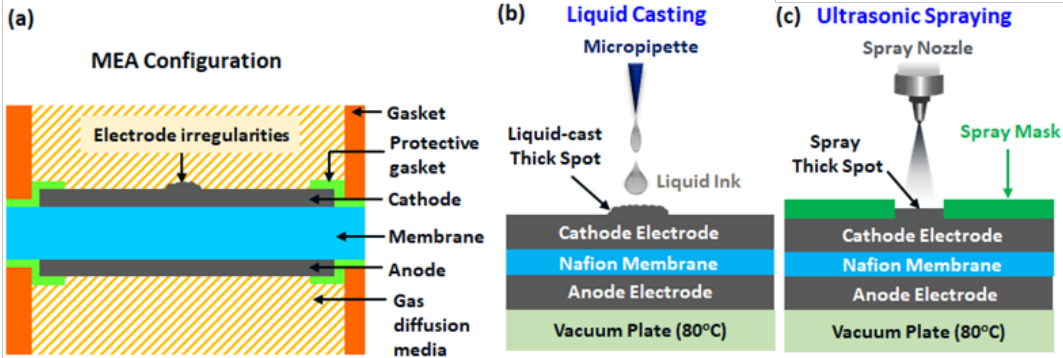
(Include this “divider” slide if you are including back-up technical slides [maximum of five]. These back-up technical slides will be available for your presentation and will be included in Web PDF files released to the public.)



# Accomplishments

## Additional information on in situ defect studies

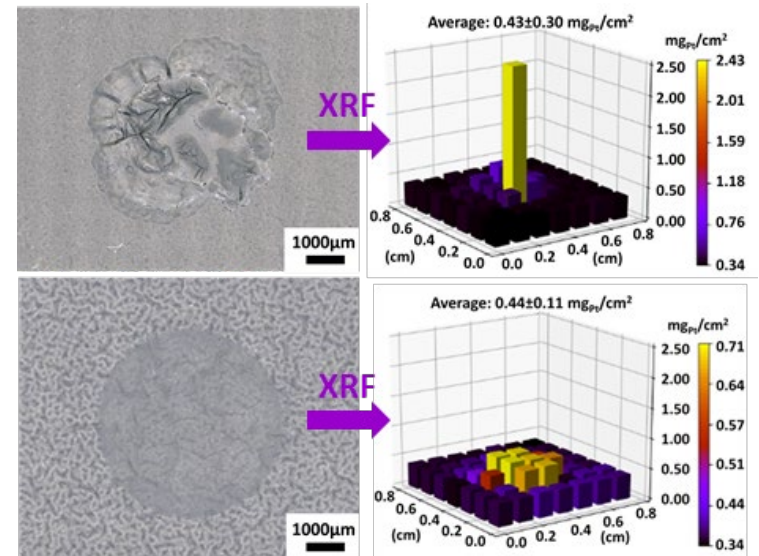
### Thick spot studies



✓ Developed MEA edge protection for PEMFC defect study.

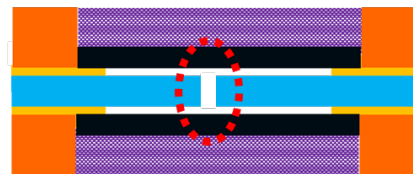
✓ Publication: *ECS Transactions* 2019, 92 (8), 351-359.

Anode and cathode loading: 0.2/0.2 mg<sub>Pt</sub>/cm<sup>2</sup>; I:C ratio: 0.9; Hot pressing : edge-protection, 25kg/cm<sup>2</sup>, 125°C

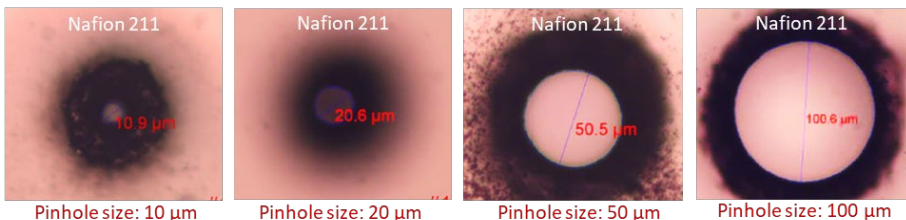


### Pinhole studies

#### Laser Pinhole MEAs



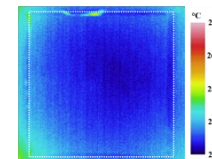
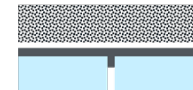
#### Nafion Membrane (NRE 211, 212, XL) with Laser Induced Pinholes



#### Preliminary study: impact of MEA configuration on pinholes

##### Half CCM-based MEA:

29BC + Cathode coated 212 with 100µm pinhole (Hot pressed)

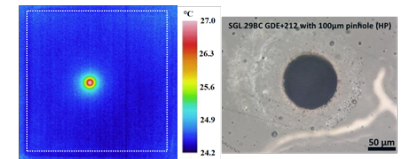
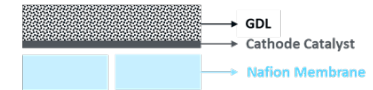


100 SCCM H<sub>2</sub>; exposure time 200s

No thermal responses observed at this condition, suggesting the pinhole was closed during spray coating.

##### Half GDE-based MEA:

29BC GDE+212 with 100µm pinhole (Hot pressed)



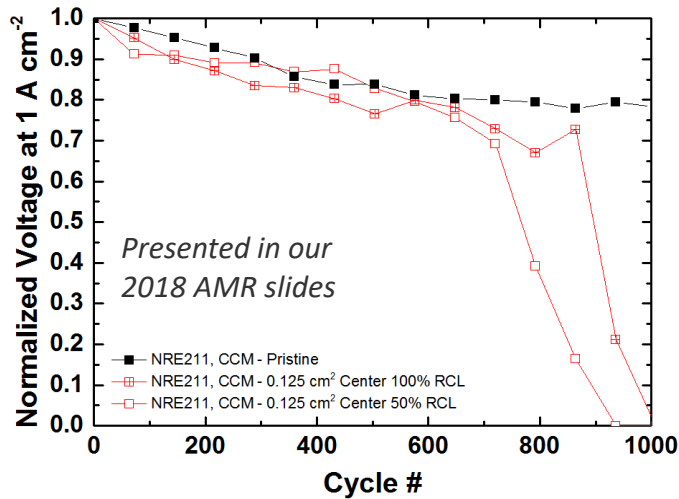
100 SCCM H<sub>2</sub>; exposure time 200s

Pinhole was observed in both pinhole detector (PDA) and optical microscope.

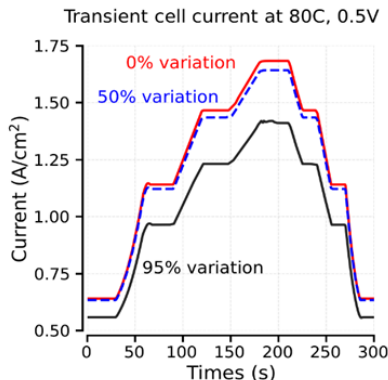
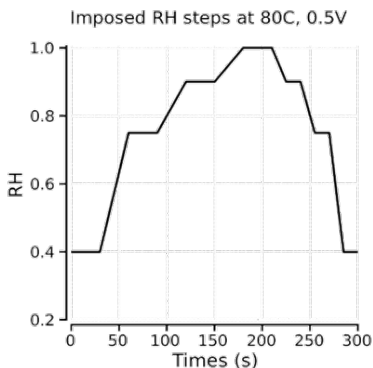
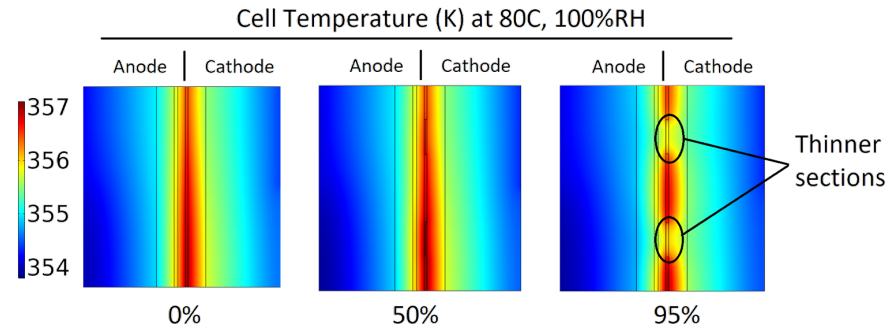
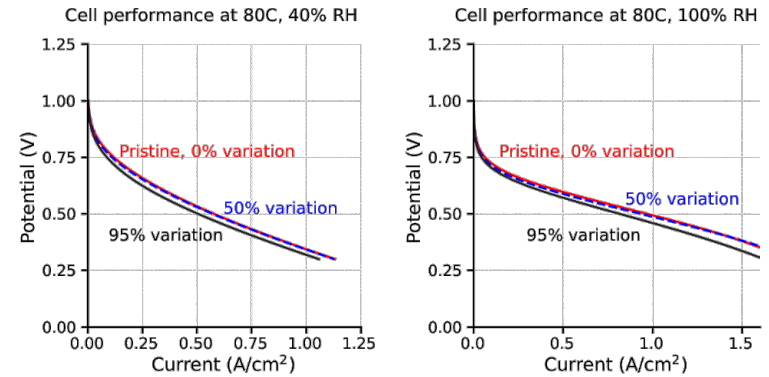
# Accomplishments

Modeling predicts low performance impact but potential failure development from electrode thin spots

## LBNL modeling of the performance impact of electrode thin spot defects



Steady-state model: predicts small impact of thin spot irregularities on total cell performance (top right). This is similar to prior NREL initial performance testing results (not shown). However, the thermal impact of the thin spots (bottom right) could lead to faster failure, as observed in NREL AST testing (top left).



Transient model: predicts decrease in cell performance (right) during cell humidity cycle (left), with increasing loss as the electrode thin spot becomes more extreme (e.g. “95% variation” indicates a 95% reduction in the electrode thickness at that location)

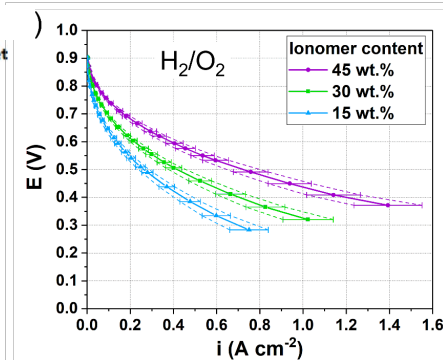
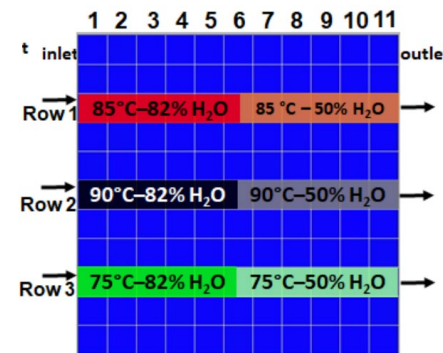
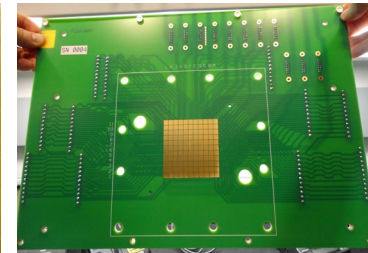
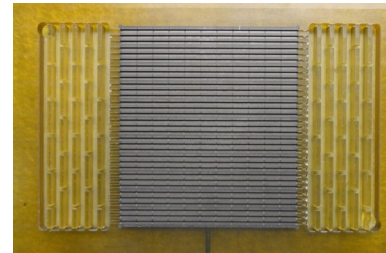
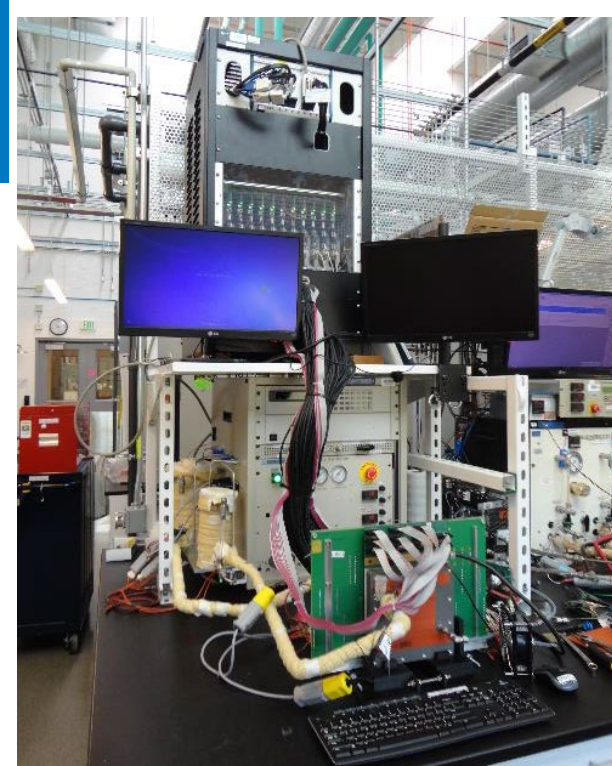
# Accomplishments: Segmented Cell GEN 2 Upgrade Completed

## Key elements and capabilities of NREL segmented cell system

- 121 segments in 50 cm<sup>2</sup> area (0.413 cm<sup>2</sup>/seg)
- Current density up to 2.4 A/cm<sup>2</sup>
- Custom software with visual and numerical data presentation & analysis features
- Cell operates as a true single cell
- Spatial 4-wire sensing of current and voltage distribution
- Interchangeable flow-fields
- Improved cell sealing

## Examples of advanced experimental applications

- Increased throughput for long term experiments
  - Long term operation at single cell station
  - Periodic spatial interrogation at segmented cell station
- High throughput combinatorial research
  - Strip cell approach enables differential cell operation with combinatorial samples



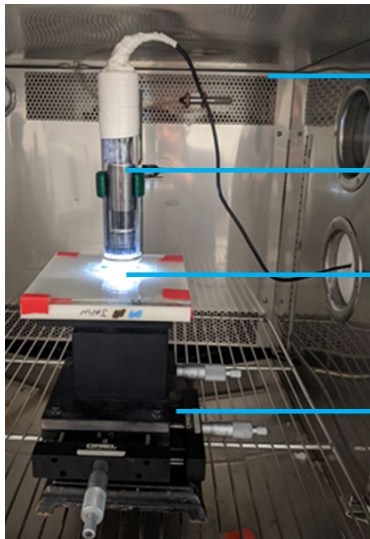


# Progress

Established capability to observe effect of RH and temperature on pinholes

Understand membrane pinhole swelling behavior under different temperature and relative humidity conditions

## Experiment Setup



Environmental Chamber

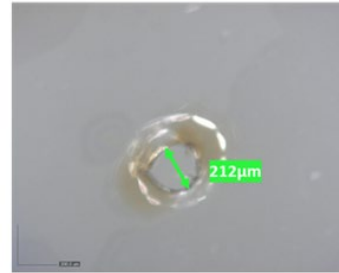
USB Microscope

Membrane sample

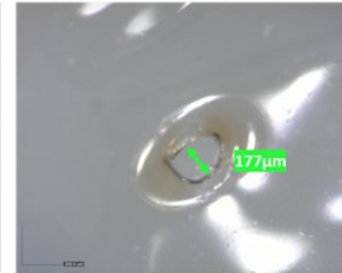
Sample stage

**Sample:** Nafion 211 membrane with a 250  $\mu\text{m}$  pinhole

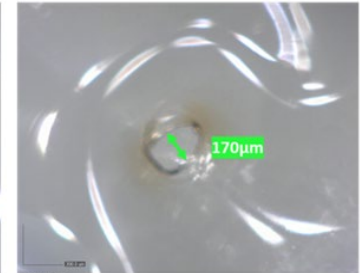
a) Room Temp/RH



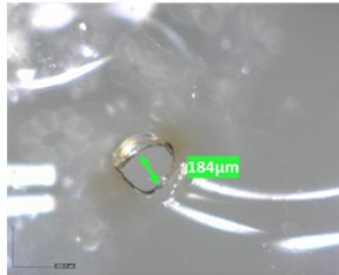
b) 80°C, 25% RH



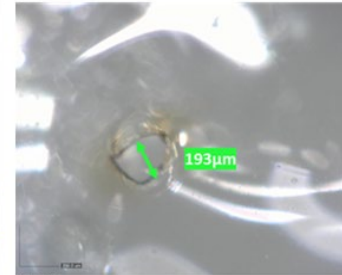
c) 80°C, 35% RH



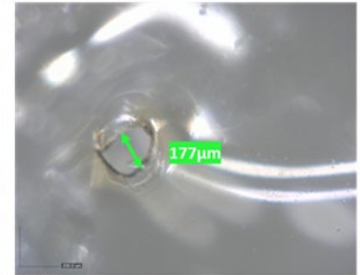
d) 80°C, 45% RH



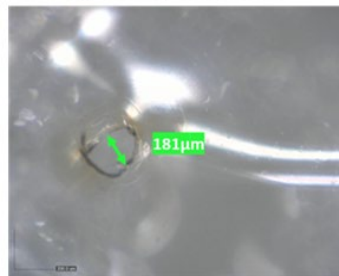
e) 80°C, 55% RH



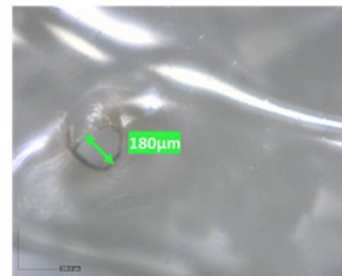
f) 80°C, 65% RH



g) 80°C, 75% RH



h) 80°C, 85% RH



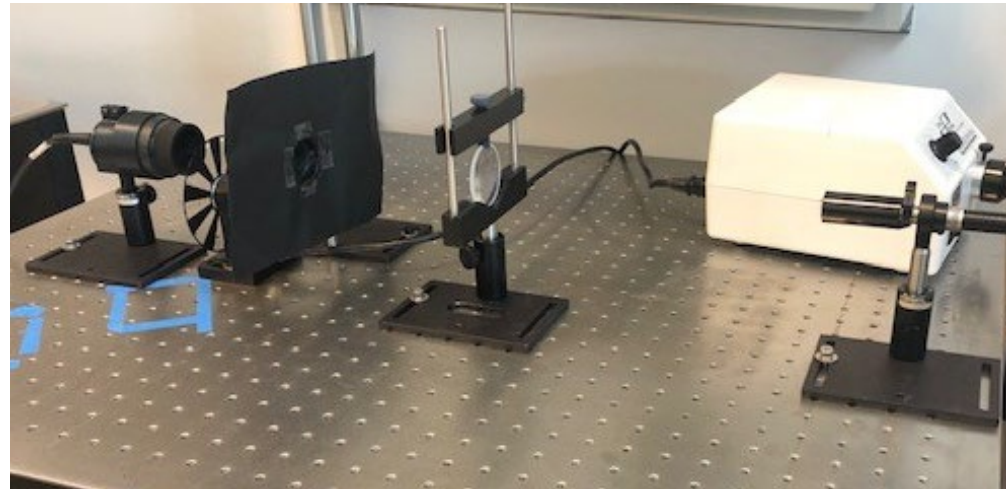
i) 80°C, 95% RH



- Established experimental setup
- Completed preliminary membrane imaging results
- Working on improving the setup
  - Holding/constraining membrane
  - Improved positioning relative to microscope
- Will study additional membranes
- Effort coordinated with LBNL modeling

### New optical transmission testbed

- Catalyst-containing layers are optically opaque
- However, transmission should be proportional to loading, if enough transmitted light is enabled
- New test stand with optical and electronic components to study optimization of signal/noise (S/N) for low transmission measurements
- Of interest for:
  - Catalyst-coated membranes
  - Membranes with H<sub>2</sub> crossover mitigation
- Exploring various light sources to improve S/N



**New low-transmission test-bed for development of fast measurement techniques for catalyst loading in various membrane-containing structures**

