NREL
Transforming ENERGY

MEA Manufacturing R&D

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National Renewable Energy Laboratory
DOE Annual Operating Plan WBS 10.1.0.501
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DOE Hydrogen Program
2023 Annual Merit Review and Peer Evaluation Meeting

AMR Project ID: TA001

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Project Goal

Project vision (as modified in FY 2021): to **provide focused and proactive work on quality control understanding and development that supports industry scale-up of PEM fuel cell and electrolyzer MEAs** and stacks, in concert with activities within the M2FCT and H2NEW consortia

Project goals:

- Continue to develop quality inspection and defect threshold methods and understandings, focusing on heavy-duty fuel cell and low-temperature electrolysis applications
- Improve and update our core capability platforms to continue to enable this unique knowledge development

Outcomes: transferring knowledge and methods to industry to accelerate commercialization

Overview

Timeline and Budget

- Project start date: July 2007
- Project continuation and direction determined annually by DOE

Barriers

Barriers	Target*
E: Lack of Improved Methods of Final Inspection of MEAs	$20/kW (2025) at 100,000 stacks/yr
H: Low Levels of Quality Control	

*https://www.hydrogen.energy.gov/pdfs/20005-automotive-fuel-cell-targets-status.pdf

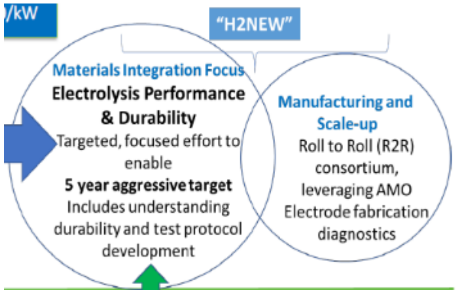
Budget and Funded Partners

Fiscal Year	NREL	LBNL
2022 (received)	$800,000	$200,000
2023 (planned)	$119,000	$0

Relevance and Potential Impact

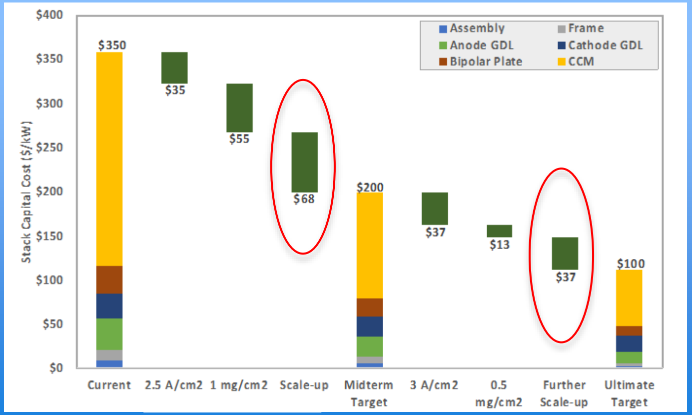
2020 DOE Hydrogen Program Plan: "…R&D efforts can help **achieve economies of scale** in manufacturing… key opportunities include: Technologies for **in-line diagnostics and quality control/quality assurance**; Sensors and other technologies to **reduce manufacturing defects** in high-throughput production…"

Excerpt from 2020 HFTO Lab Call

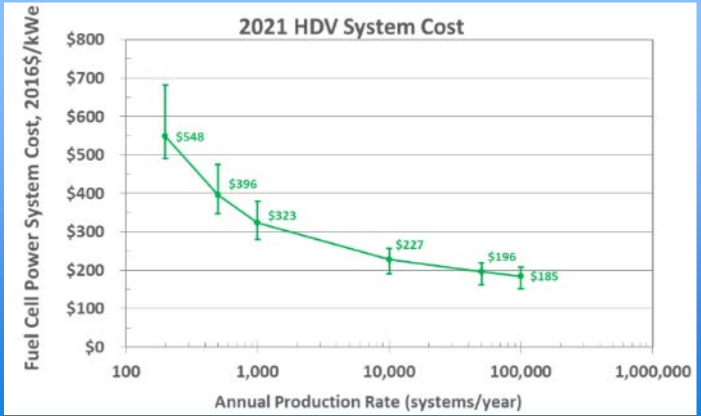


2022 DOE Fuel Cell Technologies Overview: "Substantial improvements needed in power density, durability, stack and BOP components, and **manufacturing**."

Pivovar, H2NEW, 2022 Hydrogen AMR



Papageorgopoulos, FC technology overview, 2022 AMR



Addressing DOE Goals: **Supporting manufacturing scale-up challenges, like quality control**, for fuel cell and electrolysis MEA and stack technologies **assists U.S. manufacturer competitiveness** and **job creation**, and contributes to the commercialization of technologies that will **lower pollutant emissions** and **enable clean energy power and fuel infrastructures** in the U.S.

Relevance

NREL Transforming ENERGY

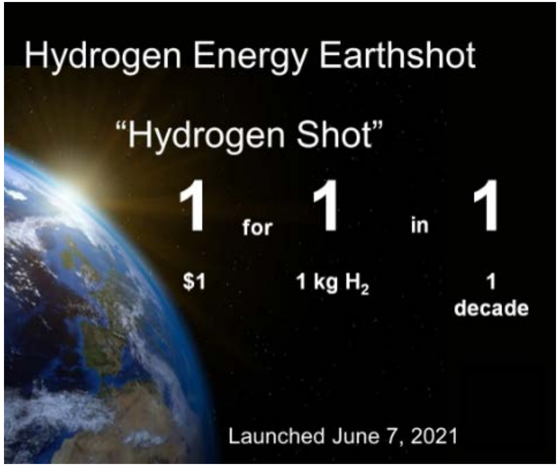
Hydrogen Energy Earthshot: Key Enablers for Lower Cost Electrolytic H2

- Low-cost electricity
- High electrical efficiency
- Low-cost capital expense
- **Increased durability-lifetime**
- **Low-cost manufacturing processes**
- **Manufacturing at MW-scale**

Stetson, Hydrogen Program Annual Merit Review plenary, 2021

Addressed by MEA Manufacturing R&D

ENERGY earthshots U.S. DEPARTMENT OF ENERGY | Hydrogen



Bipartisan Infrastructure Law (BIL)

- Total of $9.5B authorized and appropriated for Clean Hydrogen in Nov. 2021 over 5-year period (FY22-FY26)
- Section 815: $0.5B Clean Hydrogen Manufacturing and Recycling
    - "**RD&D projects to advance new clean H2** production, processing, delivery, storage and use equipment **manufacturing technologies and techniques**"
- Section 816: $1.0B Clean Hydrogen Electrolysis Program
    - Goal: To **reduce the cost of hydrogen produced using electrolyzers** to less than $2 per kilogram of hydrogen by 2026

Approach

- Leverage industry relationships and international forums to understand needs and challenges
- Establish synergies and lay framework for supporting HFTO consortia – develop and improve R&D capabilities
- Focus on technology development, demonstration and transfer
  - Feed technology and methods into industry via SPP, FOA, and other DOE-funded/cost-shared projects

***Annual Milestone Criteria:**
- Two cameras
- 3 membrane rolls
- each at least 10m long
- Speed >=5 ft/min

Date	Milestone/Deliverable (status as of 4/10/23)	Complete
6/22	Complete fabrication of LTE spatial diagnostic boards	100%
9/22	Development of the optical transmission method for highly absorbing materials	100%
12/22	Propose concepts and estimate cost to improve the day-to-day repeatability for electrode thickness monitoring based on chromatic confocal probes	100%
3/23	Perform UV-VIS-NIR spectroscopy study to enable mapping of the free radical scavenger concentration	100%
3/23	Complete development of data acquisition interfaces and control software for spatial LTE diagnostics	100%
3/23	*Discuss with HFTO and create an initial plan to explore engagement with vehicle OEMs*	100%
6/23	In-line mapping of loading/thickness/surface topography of opaque materials	20%
6/23	*Get broader LTE community feedback on the LTE MEA failure modes assessment*	0%
9/23	**Interference fringe based thickness mapping for transparent membranes***	**5%**
9/23	Spatial LTE diagnostic study. Demonstrate functionality of segmented cell using reference cells and cells with electrode inhomogeneities	0%

Collaboration and Coordination

Industry Collaborators

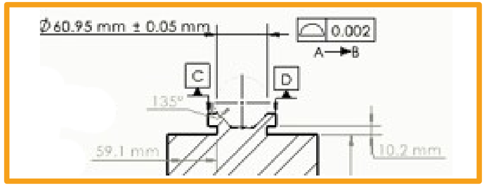
Objective: ensure we continue to get detailed input on manufacturing QC needs, prioritization of diagnostic development, feedback on technique capabilities, and pursue tech transfer

- We leverage separately funded activities with **Chemours, 3M, Nel Hydrogen, DeNora, Giner** and **Fortescue Future Industries** to understand industry directions and challenges (partners provide materials and QC requirements)
- We are teaming with numerous commercial entities to pursue a diverse set of funding opportunities for future work
- **Advent Technologies** loaned NREL's XRF system for in-line monitoring of Pt loading

Labs and Academia

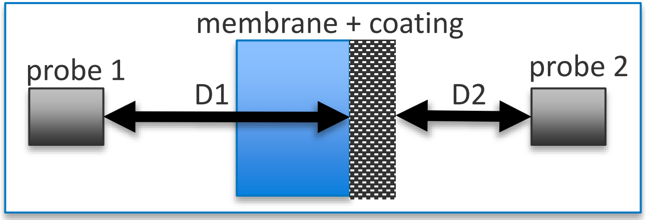
- **Lawrence Berkeley National Lab:** LBNL provides model development and integration, e.g., to understand the impact of defects and irregularities in MEA materials
- **National Research Council-Canada (NRC) and Fraunhofer-ISE:** Co-organizers and sponsors of international meetings and coordination on QC and defect studies
- **UCLA:** Ad hoc collaboration for initial exploration of TeraHertz imaging methods
- **UMass Amherst:** Collaboration on in-line metrology and AI/ML; hosting interns

Accomplishments & progress



Tight tolerancing for surfaces supporting probes

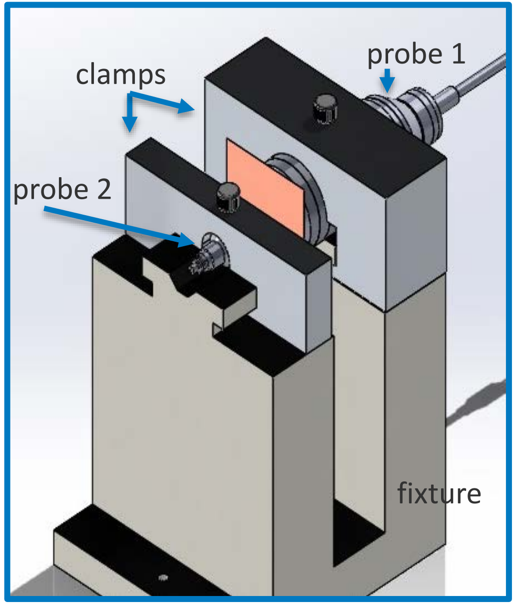
Finalized design of a mechanically stabilized fixture for chromatic confocal probes (FY23Q1 QPM)



Double probe configuration.
Coating thickness = const – D1 – D2

Objective: Enable single-point, high-rate electrode coating thickness monitoring based on chromatic confocal probes (thickness range 1-15µm)

- Relevant for in-line monitoring of thickness and porosity
- The initial experimental setup exhibited day-to-day instability that resulted from:
  - thermal expansion coupled with typical temperature variations
  - poor mechanical rigidity
- A new fixture design was developed to mitigate the identified issues.



New experimental fixture design

- **Key design improvements** to mitigate observed drawbacks of the initial setup.
  - material selection: nickel-ion alloy, called Invar, with the thermal expansion coefficients approximately 10 times smaller than for an ordinary steel
  - the stiffness of new fixture parts is increased by eliminating plastic elements and by significantly increasing cross-section of the structure
- **Sensors' alignment strategy** is needed to provide micron level positioning accuracy
  - precision shimming will first be utilized
  - more advance concepts were also developed and will be implemented if needed
- Mechanical design of parts **has been completed**. We are collecting quotes from machines shop and fabrication is expected to commence soon.

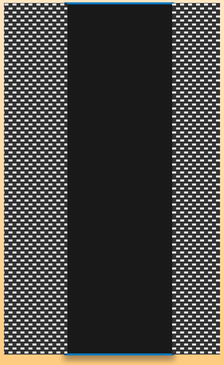
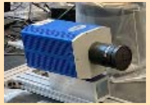
Accomplishments & progress

Figure of merit was the signal to noise ratio:

$$SNR(img_{light\ on}, img_{light\ off}) = \frac{MEAN(img_{light\ on} - img_{light\ off})}{SD(img_{light\ off})}$$

Further development of the optical transmission method for highly absorbing CCMs (FY22Q4 QPM)

Objective: Determine requirements needed to reliably acquire optical transmittance signal. If we find it feasible, the method can potentially be used for in-line monitoring quality (uniformity, defects) of 3-layer CCMs for LTE applications

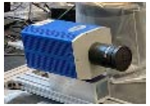
3 light sources	3-layer CCM	3 cameras			
				tested settings	fixed settings
LED 			cooled Si 2D array (Coolsnap)	integration times: 10, 100 and 1000 ms	
class II red laser 			standard line camera (P3)	line rates: 2.5, 13 and 23.5 klps	1x binning
tungsten-halogen bulb 			Time Delay and Integration camera (HS)	binning: 1x, 2x, 4x and 8x	TDI=96, line rate =3.5 klps

- Electrolyzer CCM specimen consisting of two electrodes deposited on an opaque membrane with Pt-mitigation
  - anode: 0.3 mgIr/cm^2, cathode: 0.08 mgPt/cm^2, PEM: 90µm thick, mitigated

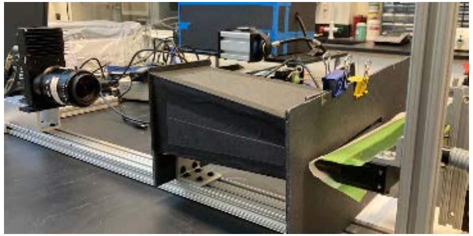
Accomplishments & progress

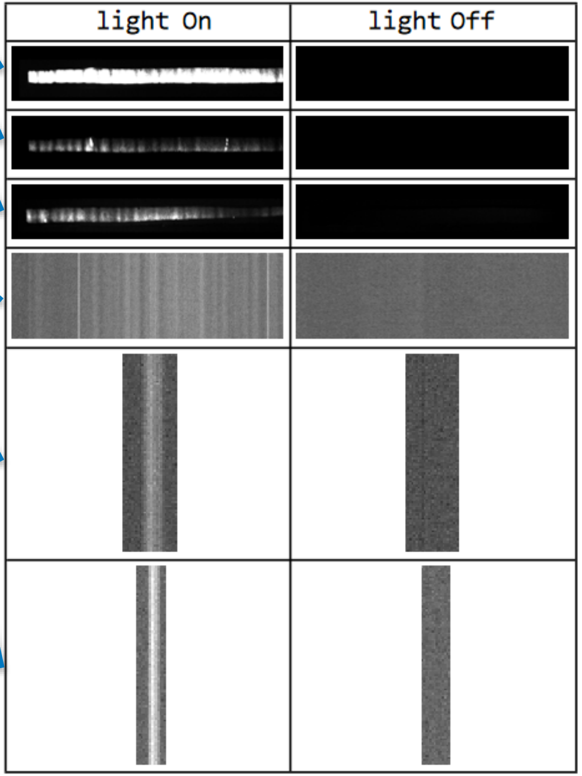
Key learnings from the experiment:

- The 2D array camera (CoolSnap) acquired best quality images
  - for integration times of 0.1 and 1 sec
- The tungsten-halogen lamp is effectively 6 times stronger comparing to LED source
- HS line camera with TDI yielded SNR of 3.9
  - this is state of the art for in-line monitoring in poor light conditions
- The laser illumination reached similar signal quality level as tungsten-halogen one
  - this was achieved only in one location where the laser beam was focused
- **Overall, the optical transmission inspection of the double sided, very absorbing CCM requires high sensitivity specialized sensors and rather long exposure times.**

camera	source	settings	SNR
CoolSnap	Halogen	1000ms	205.4
		100ms	22.49
		10ms	1.89
	LED	1000ms	3.02
		100ms	1.34
		10ms	0.02
Line Camera HS	Halogen	1x Binning	0.39
		2x Binning	0.71
		4x Binning	1.44
		8x Binning	2.6
	Laser	1x Binning	0.58
		2x Binning	1.21
		4x Binning	2.17
		8x Binning	3.9
	LED	1x Binning	0.07
		2x Binning	0.05
		4x Binning	0.27
		8x Binning	0.43
Line Camera P3	Halogen	13000lps	0.
		23500lps	
		2500lps	
	Laser	13000lps	
		23500lps	
		2500lps	
	LED	13000lps	
		23500lps	
		2500lps	

The experimental setup with line camera

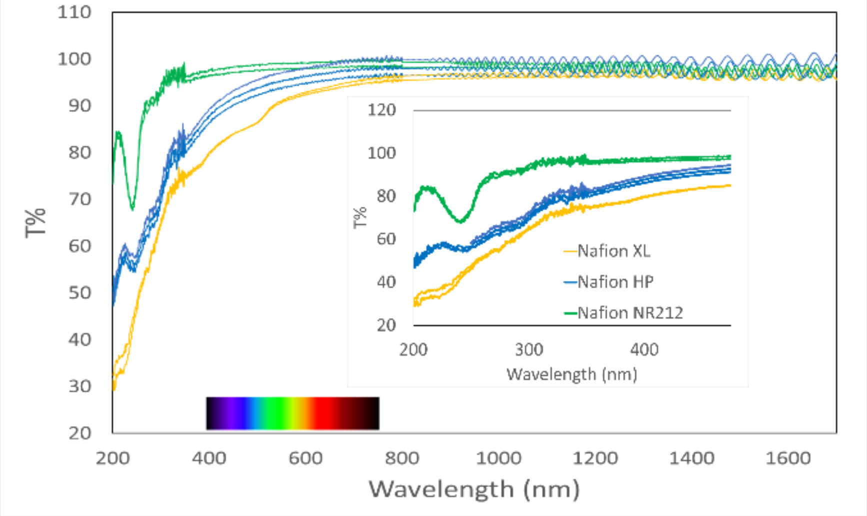




Accomplishments & progress

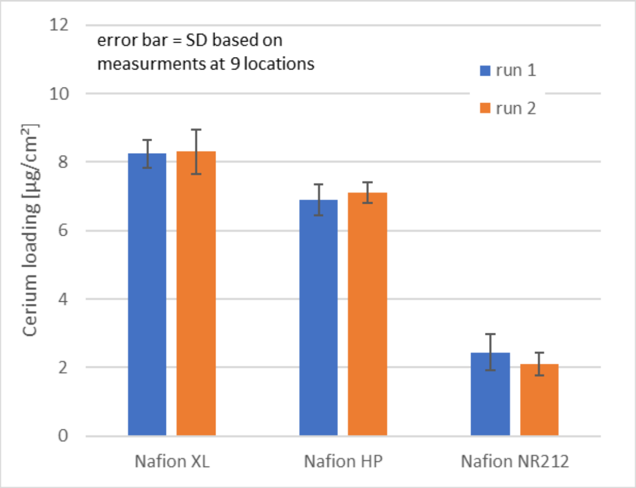
Performed UV-VIS-NIR spectroscopy study for monitoring of free radical scavengers (FY23Q2 QPM)

Membrane spectra indicating measurable differences between specimens with varying Cerium concentrations



XRF results showing concentration of Cerium

(the results may be off by 2µg/cm²)



- Successful step towards monitoring of concentration of Cerium, which is often added as a free radical scavenger
- The objective is to develop an in-line mapping solution
- In 200 to 500nm wavelength range the spectra were found to strongly depend on the membrane type. Clearly, the higher Ce concentration, as measured using XRF, the more absorbing material is in the UV-VIS wavelength range.
  - the difference in optical properties may also come from other physical dissimilarity, e.g., presence of reinforcement

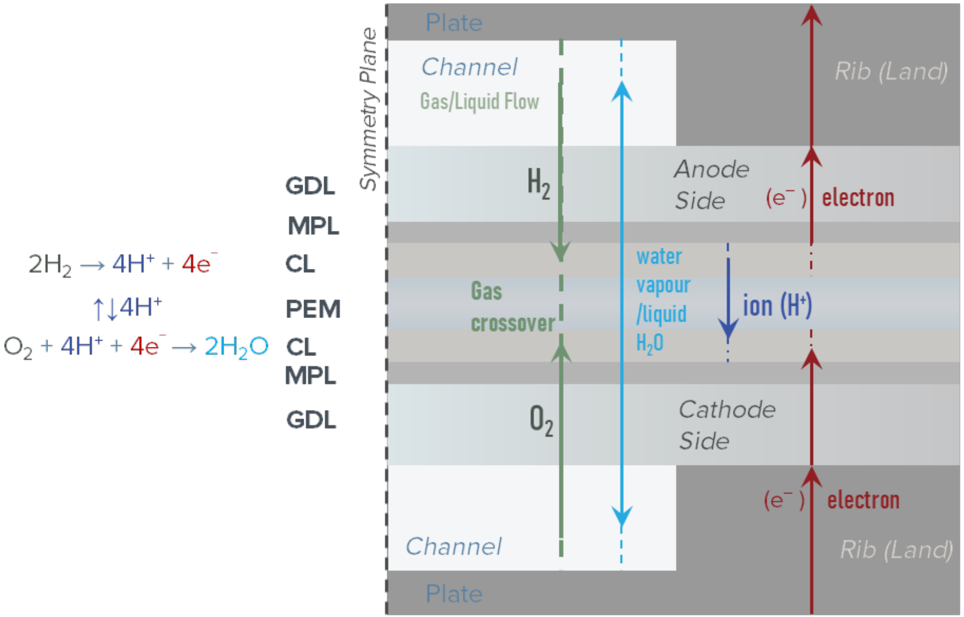
Accomplishments & progress

Key outcomes from the first US meeting in the series of International Quality Control Workshops that took place on May 5-6, 2021

- Electrolysis
  - Identify, evaluate and prioritize MEA failure mechanisms in order to identify key development needs for quality control
  - Actions to date
    - Initial assessment of MEA failure modes by NREL complete
    - Failure Modes Working Group core team formed, assessment of initial failure modes document and survey for larger industry feedback complete
    - Outreach to broader community to validate failure modes and implications for QC development ongoing
    - **This activity is planned to resume in Q4 of the fiscal year 2023**
- Fuel Cells
  - Explore engagement with vehicle OEMs to gather defect and failure data obtained from R&D as well as actual system testing, e.g., in an activity similar to the past Technology Validation effort, where OEM system operations data was aggregated and analyzed, but non-attributed
  - Actions to date (FY23Q2 QPM)
    - **Reviewed with HFTO the key aspects of the former Technology Validation project**
    - **As a next step the plan is to get feedback from potential FC OEMs to evaluate feasibility of this concept**

Accomplishments & progress

Using LBNL model of a PEMFC, the effect of membrane thickness variations was determined

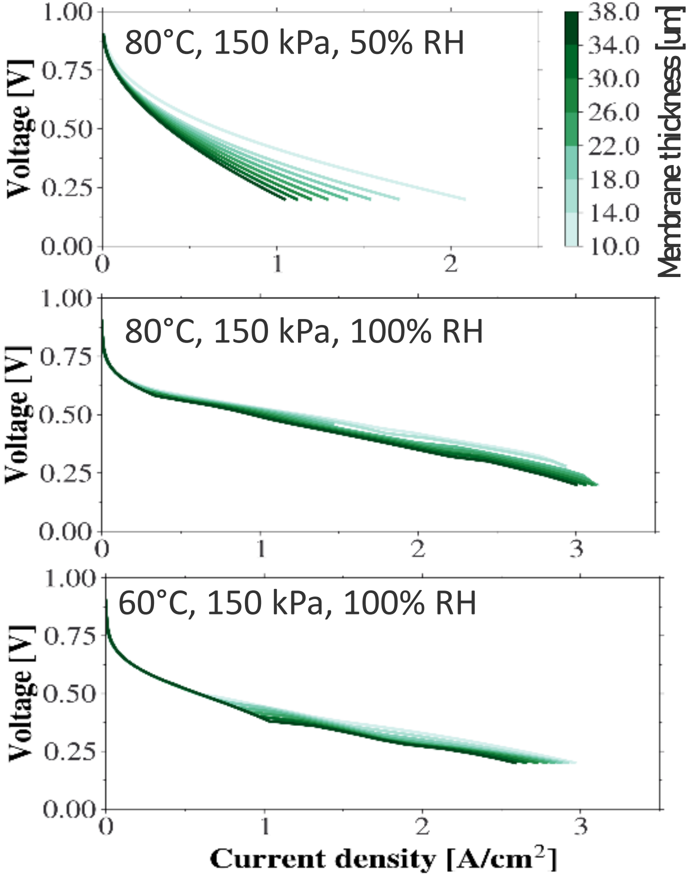


Representative unit of PEMFC

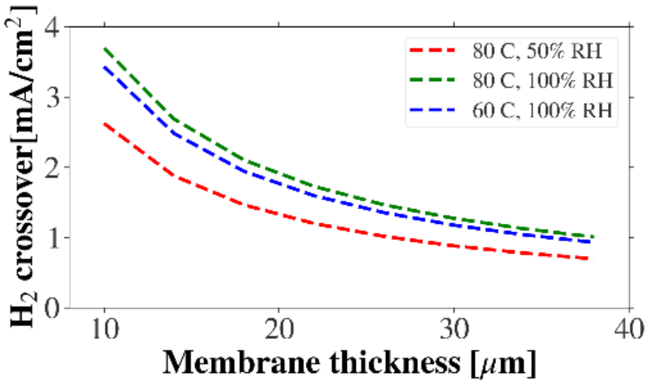
Electrochemical modeling of PEMFC response

- The model helps to understand the significance of membranes variations that need to be monitored and controlled
- LBNL standard model consisted of 7 layers and 2 bipolar plates with both channel and rib(land) sections
  - encompasses performance in a 2D with non-isothermal conditions and multiphase flow
  - includes explicit crossover of hydrogen and oxygen
  - thickness-dependent membrane transport properties
- It allowed for prediction of polarization curves and H_2 cross-over as a function of membrane thickness which varied from 10 to 38µm

Accomplishments & progress



- We quantified performance and polarization behavior based on the thickness where there is a tradeoff in crossover and associated water fluxes with lower resistance for thinner membranes
- Variations on the order of a micron, when thickness is around 10 microns , could have large impacts on performance, especially at high current density
- This is exacerbated when at low RH and to a lesser extent the temperature. It is driven more by the changes in the water management rather than the hydrogen crossover, where the latter is impacted at higher RH and shows at lower current densities.

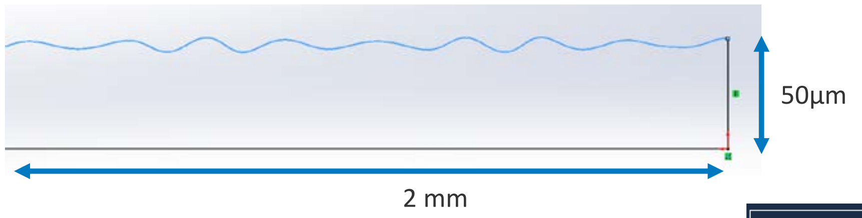


Plots showing effect of membrane thickness on the polarization curve (left) and on the H_2 crossover (right)

Accomplishments & progress

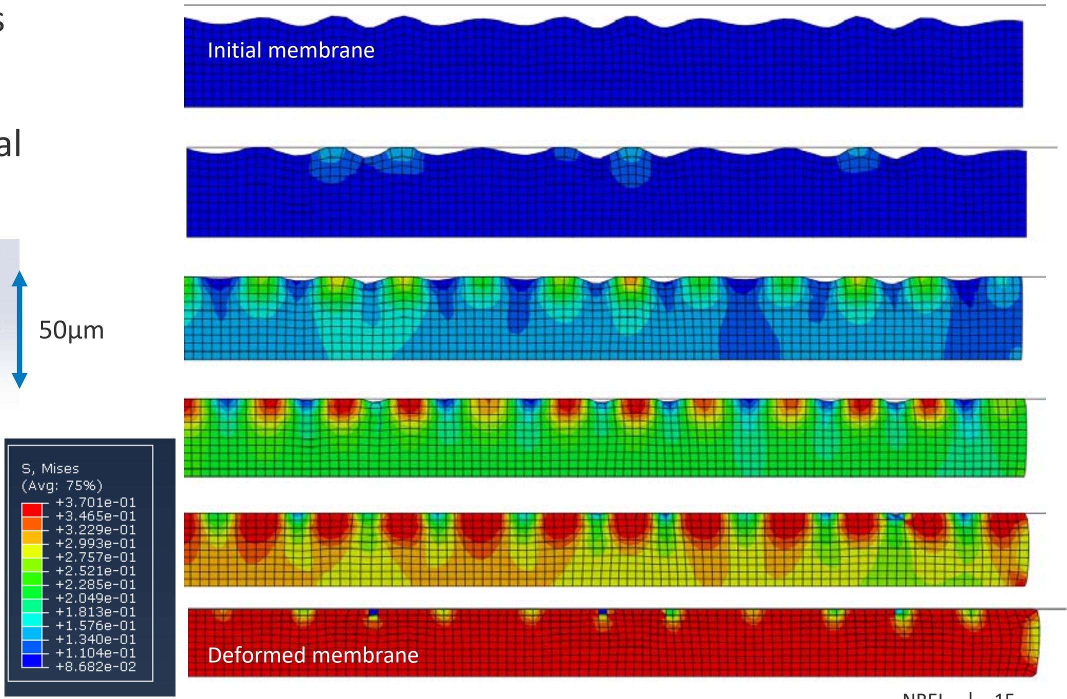
Computational mechanics modeling of membrane with thickness variations was performed

- **Compression of a membrane with user-defined nonuniform surface is modeled**
  - Stress-strain response of Nafion at 25C is used as material model for deformation under a rigid body compression
  - 2D Profile is generated from experimental data of Nafion surface roughness (NREL)



  - A rigid body compressed the membrane by 15 micron, and resulting deformation is simulated to monitor strain-stress fields as well as the change in surface nonuniformity

- **Preliminary results show deformation of PEM under a smooth rigid surface resulting in increased stress**



Accomplishments - Segmented Cell Development

Milestone:
Complete development of data acquisition interfaces and control software and integrate them with the hardware. Test, debug and troubleshoot the tool to make it ready for experimental study.

NREL design

Active Area: 25 cm^2

Segment Size: 1 cm^2, 5x5 layout

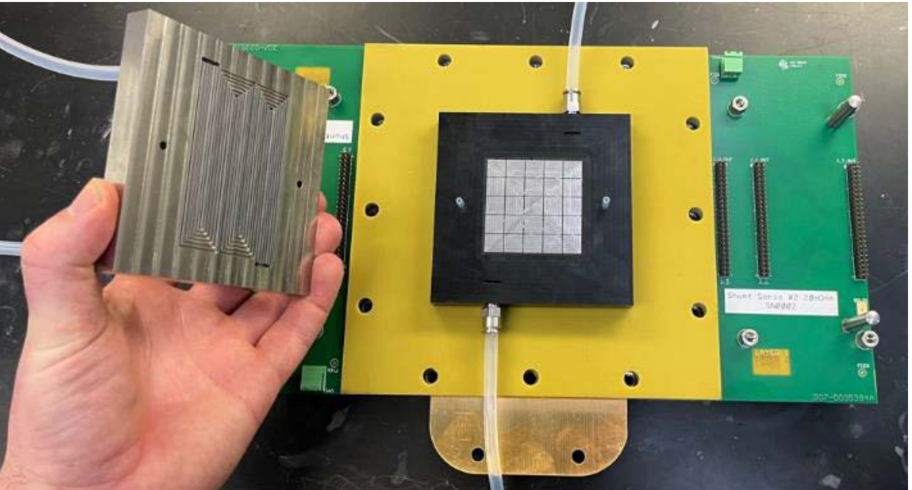
Flow Field: + 6 channel serpentine
+ segmented at anode

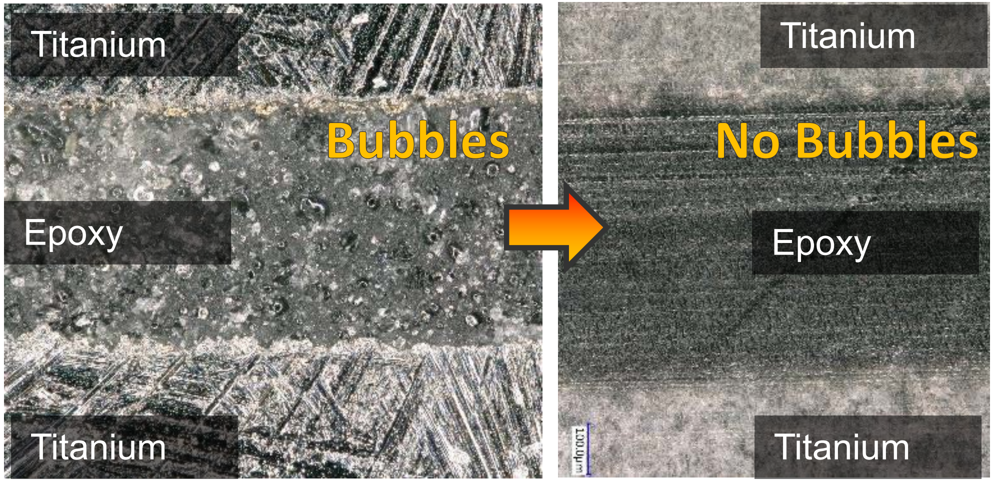
Data Collection

- Spring-loaded through-plane current sensing
- Max. Current Density: 15 A/cm^2
- Two current ranges at a time possible
- Current/Voltage sampling rate: ~10 points / s
- Amplification at cell for low noise operation

Flow-field Fabrication

- Bubbles cause Ti spreading shorts between segments during machining
- Vacuum supported gluing mitigates bubble formation





Accomplishments - Segmented Cell Development

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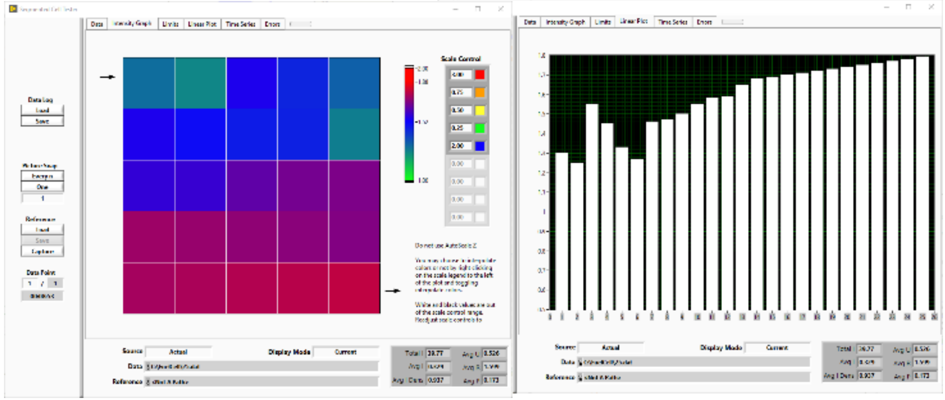
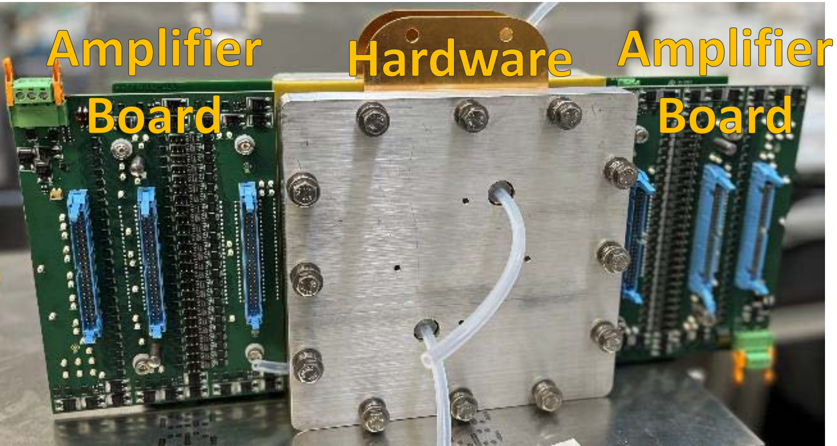
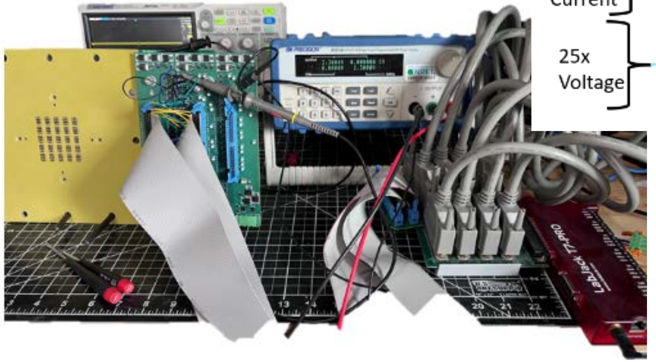
NREL Data Acquisition Features:

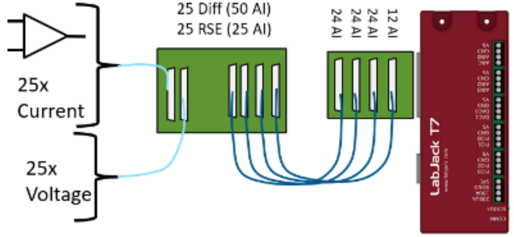
- LabVIEW software
- On-line 2-D and down-the-channel data representation
- Reference cell comparison option
- Calibration file for shunt and amplifier offsets
- Noise levels of ≤ 6mA/segment and 0.5mV
- Current Sense Amplifier Selectable Gain of 1-128x
- Voltage Sense 16bit ADC
- Temperature measurements under development

Software Development : 2D Heat Map & Down-the-Channel









Data-Acquisition Schematic and verification

Responses to Reviewer Comments

- Project progress was presented during 2022 AMR, but it has not been peer reviewed at that time.
- Below please find responses to reviewer comments from 2021 AMR

Comments: Several comments about lack of innovation and little evidence that the state of the art will be advanced (beyond what industry already knows).

Response: The membrane thickness imaging technique is totally novel. We have a patent and a TCF project with an equipment manufacturer to commercialize the technique. The state of the art in the industry, with the exception of a very few companies, is manual inspection of cell materials prior to MEA fabrication and stacking. All of our work directions and goals are based on inputs from our industry partners defining challenges that they have. Almost all of our recent HFTO FOA projects include QC development (efforts requested and defined by the industrial prime on the project), and this project provides the foundational basis of capability and development for those projects, which are very narrow and specific to the partner's manufacturing specifics.

Comments: Several comments regarding applicability of the work to hydrogen generation.

Response: For the last four or so years, we have included effort in this project to understand how to transition methods developed for fuel cells to PEM electrolysis MEAs, and where the methods are not applicable or where new needs are identified (e.g., related to PTLs), we have pursued new method development. As an example, PEM electrolysis MEAs currently use much thicker membranes than those for fuel cells. Our original implementation of the membrane thickness imaging technique worked well for thinner fuel cell membranes, but not for thicker membranes. We then made advancements to the technique, using either different detector hardware or different image processing techniques, to extend the method to thicker membranes relevant for PEM electrolysis.

Comments: Several comments regarding the need to understand the impact of manufacturing defects on not only performance, but durability.

Response: We whole-heartedly agree, and this has been our approach to the defect studies for many years. For fuel cell defect studies, we utilize drive cycle and/or accelerated stress testing, integrated with novel spatial testing. For PEM electrolysis, we have been working for a couple years now to expand our, especially spatial, testing methods to enable these same kinds of studies. In this presentation, we report on novel advancements relative to this goal.

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 co-organize the International QC series of meetings with NRC-C and Fraunhofer ISE, as a continuing forum to confirm barriers and needs and discuss collaborative actions
- We 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
- Coordinate development activities with M2FCT and H2NEW consortia
- Continue to develop non-X-ray-based areal uniformity measurement methods for all MEA fabrication steps
- Evaluate X-ray methods for multilayer MEA structures and highly light absorbing materials (e.g., PTLs)
- Study the effects of relevant defects and layer irregularities (e.g., thickness, porosity variations) on cell performance and lifetime
  - Focus on priority materials and cell structures in M2FCT and H2NEW
  - Apply specialized spatial in situ hardware and methods for LTE defect testing
- Continue to develop and apply predictive models to assist in the understanding of impacts of defects

Summary

- Relevance
  - Manufacturing and Quality Control R&D topics: 1) continue to garner domestic and international industry interest, 2) clearly support Hydrogen EarthShot and BIL goals
- Approach and Collaborations
  - Continued detailed partnership, collaboration, and information exchange with industry partners on QC priorities
  - Efforts will support the scale-up activities within the H2NEW and M2FCT consortia by providing foundational method and capability development
- Accomplishments
  - Finalized design of a mechanically stabilized fixture for chromatic confocal probes
    - Potential for in-line, single-point, high-rate measurement of electrode thickness and porosity
  - Investigated configuration requirements to acquire the optical transmission for highly absorbing 3-layer CCMs
  - Promising results from UV-VIS-NIR spectroscopy study opens path to monitoring of free radical scavengers
  - Followed up on the engagement with vehicle OEMs to gather defect and failure data
  - Predicted that even a micron thickness variation may markedly impact FC performance
  - Finalized development of the 25 cm^2 PEM LTE segmented cell
- Tech Transfer
  - Leveraging HFTO investments for many industry-focused projects including two new CRADA projects
  - Numerous new proposals and concept papers submitted by companies in partnership with NREL's QC team

Key Contributors

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- Bryan Miller
- Wanjun Dang
- James Brock
- Lindsey Welch
- Daniel Moghtader
- Guido Bender
- Hunter Simonson
- Ellis Klein
- Robin Rice
- Adam Weber (LBNL)
- Claire Arthurs (LBNL)
- Ahmet Kusoglu (LBNL)

Thank You

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Technical Backup and Additional Information

Technology Transfer Activities

- *New CRADA project with Chemours to investigate quality control methods for advance membranes used in FC applications*
- *New CRADA project with Fortescue Future Industries to demonstrate and, if needed, develop quality control techniques for PEM and AEM membranes and electrodes*
- HFTO Electrolysis Manufacturing FOA projects with 3M (P197) and Nel (P198)
- Advent Technologies loaned NREL's XRF system for in-line monitoring of Pt loading
- Industry collaborative projects completed
  - SPP project with Lubrizol
- New record of invention entitled "Inspection of surface quality in porous transport layers"

Italics: new projects in FY2023

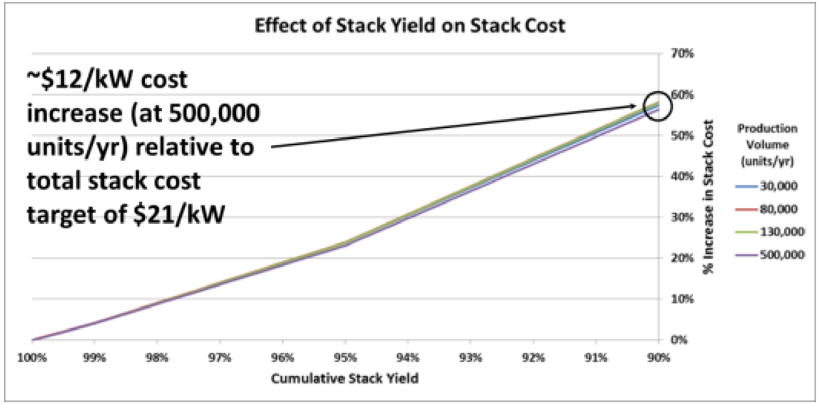
Publications and Presentations

- Brian G. Green, et. al., “A new simultaneous membrane thickness and catalyst loading measurement for fuel cell proton-exchange assemblies by IR transmission.” Journal of Power Sources https://doi.org/10.1016/j.jpowsour.2022.232601
- A. Yan, et. al., “Towards Deep Computer Vision for In-Line Defect Detection in Polymer Electrolyte Membrane Fuel Cell Materials.” Engineering Archive, https://doi.org/10.31224/2493

Progress toward DOE Targets or Milestones

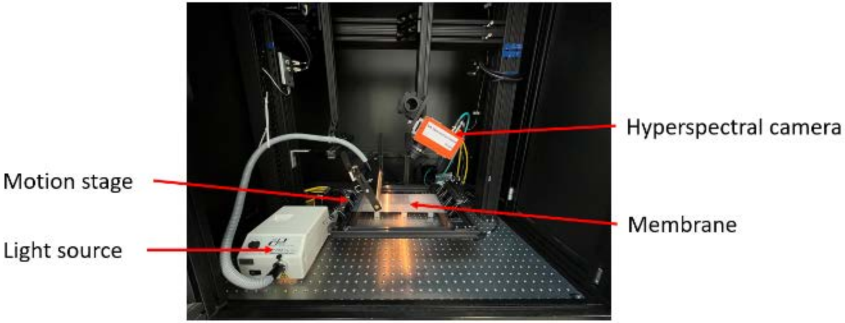
- This project has the goal of assisting industry in accelerating the scale-up of MEA and stack materials, toward program cost and timeline targets
  - Quality inspection is a critical challenge for Manufacturing R&D, per the MYRD&D plan, and as validated in the series of International workshops on QC
- The project is focused on addressing these needs for high priority applications such as water electrolyzers and fuel cells for heavy-duty applications, via industry partnerships
- Based on our analysis (right) using the Strategic Analysis Inc cost model (for LDVs), reduced levels of yield can have a significant impact on stack cost
  - Thus, quality tools and methods are needed to ensure high yield

Barriers	Target
E: Lack of Improved Methods of Final Inspection of MEAs	$20/kW (2025) at 100,000 stacks/yr
H: Low Levels of Quality Control	

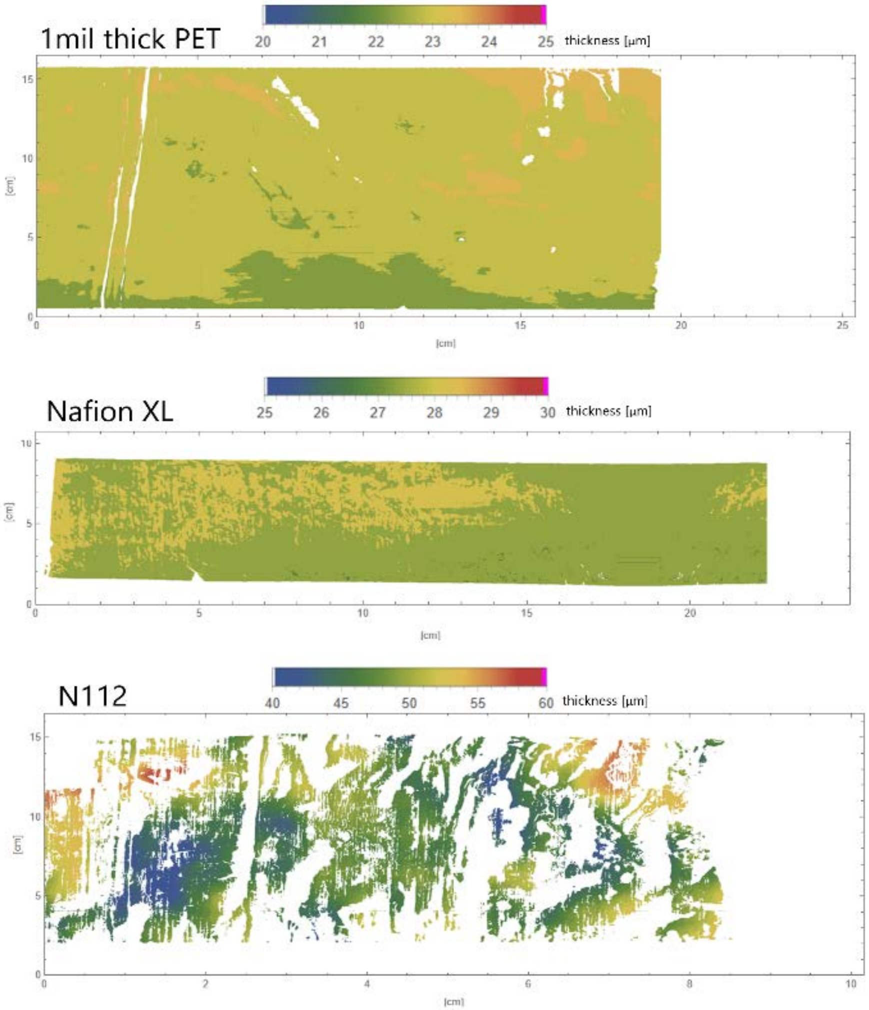


Accomplishments & progress

- NREL hosted an intern, Jingyang Yan, PhD candidate from Prof. Xian Du's group at UMass, Amherst.
  - NSF funded the internship
- Internship focused on thickness mapping technology for fuel cell and electrolyzer membranes using near-infra-red (NIR) hyperspectral imaging
  - in the past only visible light cameras were tested to map thickness of PEMs
- The effort allowed us to evaluate advantages and disadvantages of the NIR sensor as compared to visible light counterparts

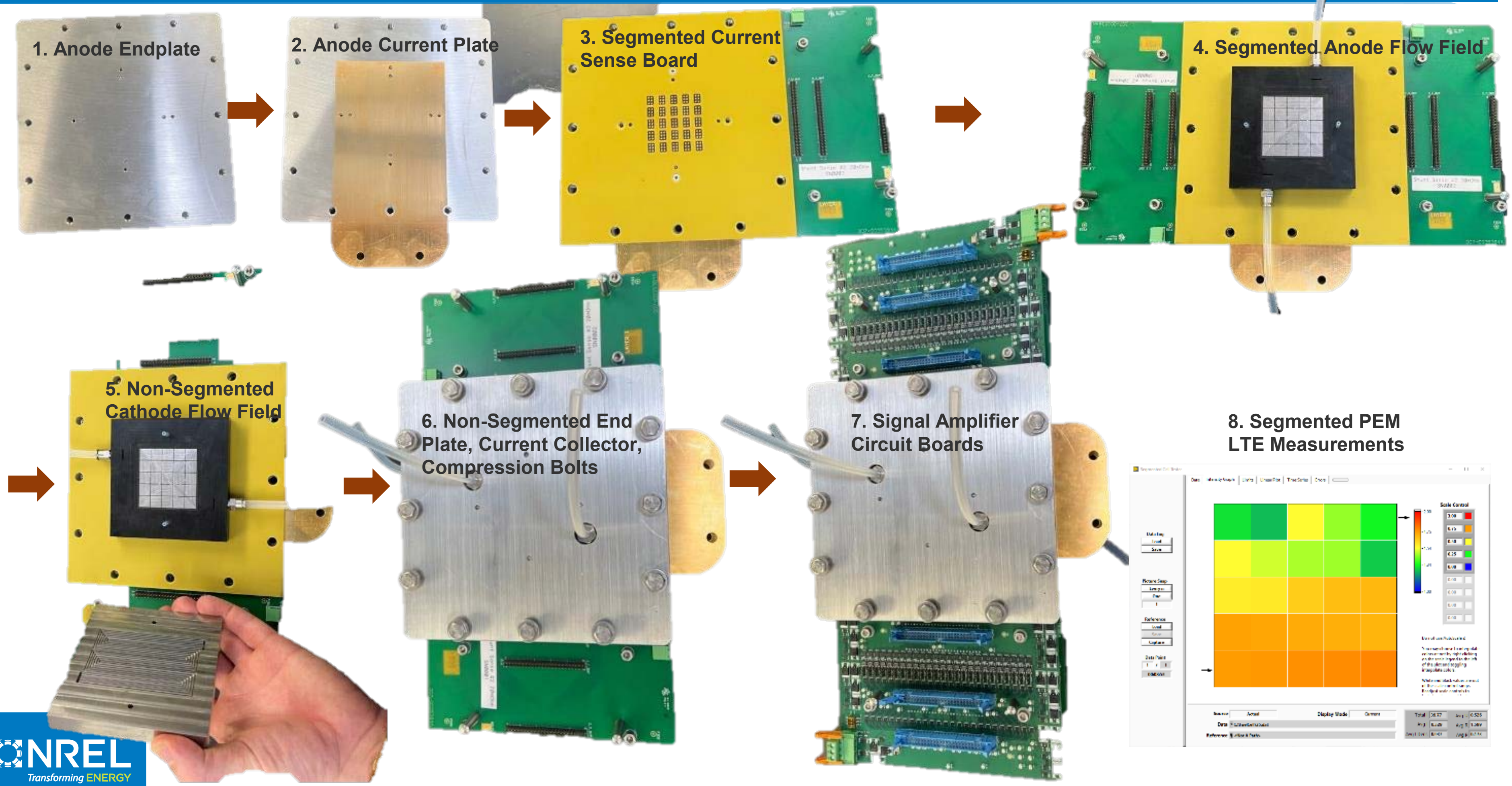


The experimental setup with NIR hyperspectral camera



Thickness maps obtained for three specimens

LTE segmented Cell Assembly – System Ready for Testing

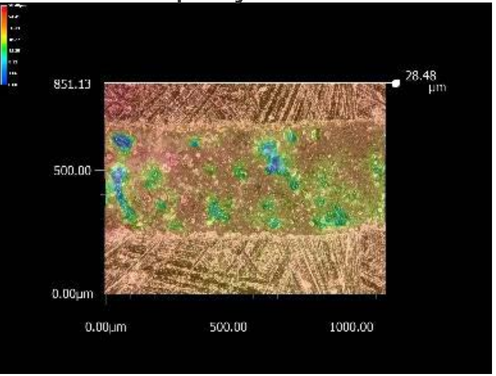


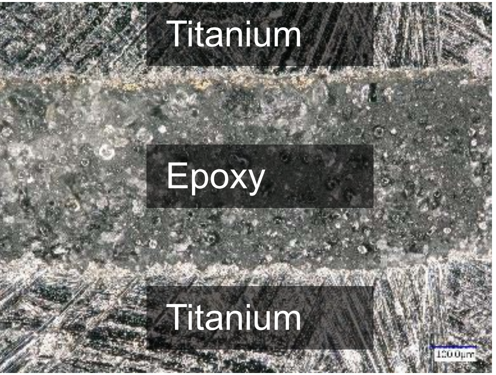
NREL
Transforming ENERGY

LTE segmented Cell Assembly – Flow Field Fabrication

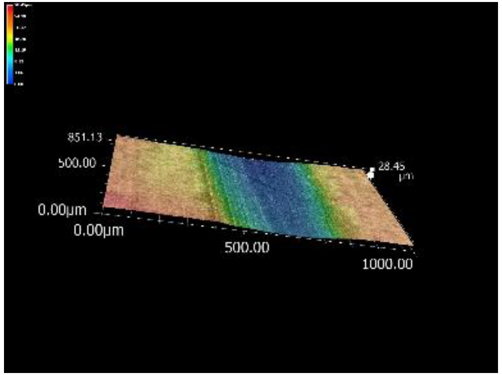
NREL Transforming ENERGY

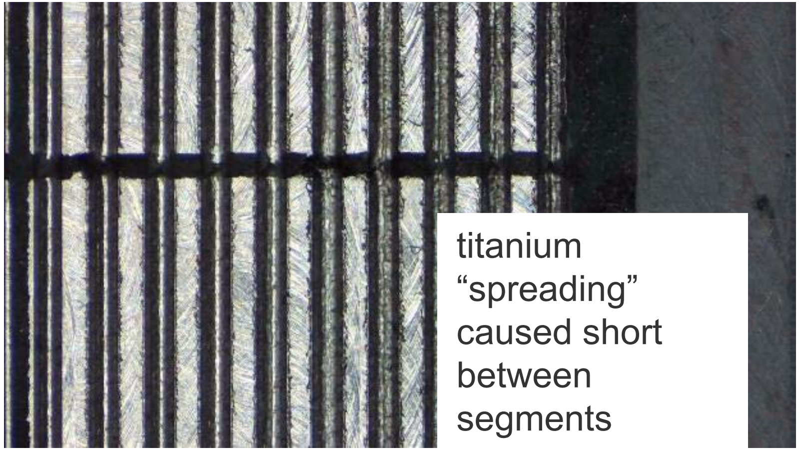
Old Epoxy Procedure





Updated Epoxy Procedure





Improved vacuum deposition method developed and implemented for fabricating segmented epoxy. Presence of bubbles in epoxy can cause segment shorts when machining Titanium, not seen in graphite fuel cell segmented flow field fabrication.