Enabling Low-Cost PEM Electrolysis at Scale Through Optimization of Transport Components and Electrode Interfaces

Project ID: P198

PI: Christopher Capuano

Nel Hydrogen

June 8th, 2022

DOE Hydrogen Program

2022 Annual Merit Review and Peer Evaluation Meeting

DOE Project Award #DE-EE0009238

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Overview

Enabling Low-Cost PEM Electrolysis at Scale Through Optimization of Transport Components and Electrode Interfaces

PI: Christopher Capuano, Nel Hydrogen

Timeline and Budget

- Project Start Date: 10/01/2020
- Project End Date: 9/30/2023
- Total Project Budget: $5.5M
  - Total Recipient Share: $1.1M
  - Total Federal Share: $4.4M
  - Total DOE Funds Spent*: $601K
- * As of 03/31/2022 – Does not include National Labs

Barriers

- Hydrogen Production:
  - F. Capital Cost
  - G. System Efficiency and Electricity Cost

Partners

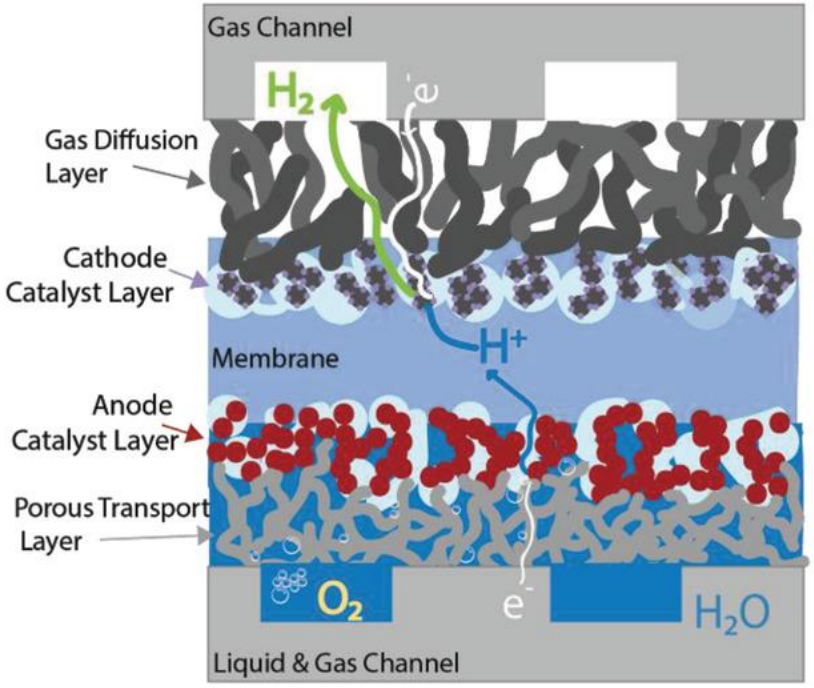
NREL: Mike Ulsh
ORNL: David Wood
DeNora: Andrew Smeltz
UCI: Iryna Zenyuk

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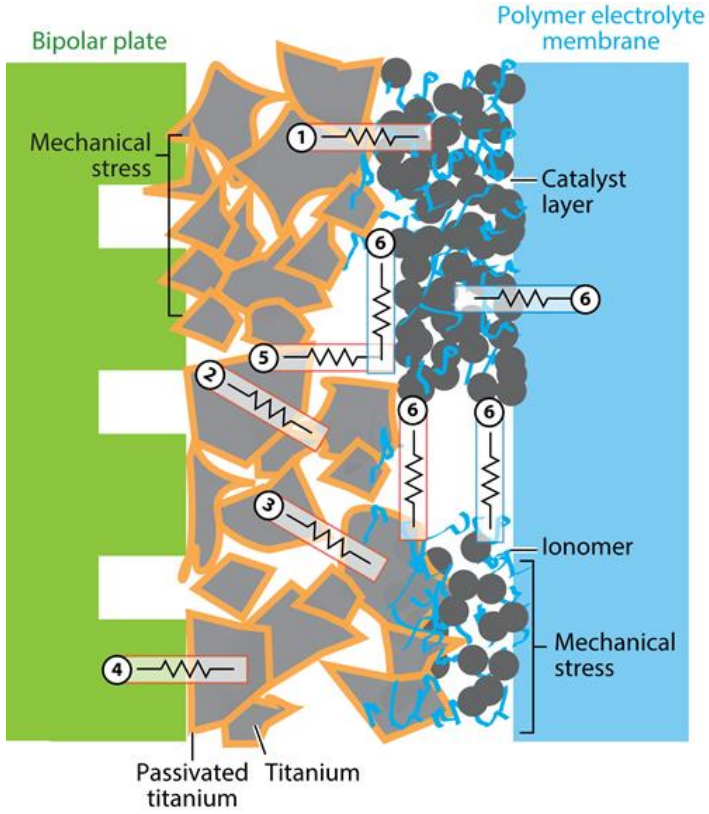
Relevance

- **The goal of the proposed project is to develop an integrated porous transport layer enables good fluid management while providing a uniform interface to the catalyst and membrane.**

- The PTL serves many purposes:
  - Distributes water flow across the cell
  - Allows oxygen gas to be removed from the anode
  - Provides electrical contact between the anode and current collector
  - Provides mechanical support for the membrane



X. Peng et al. *Advanced Science* (2021), *8*, 21, p 2102950

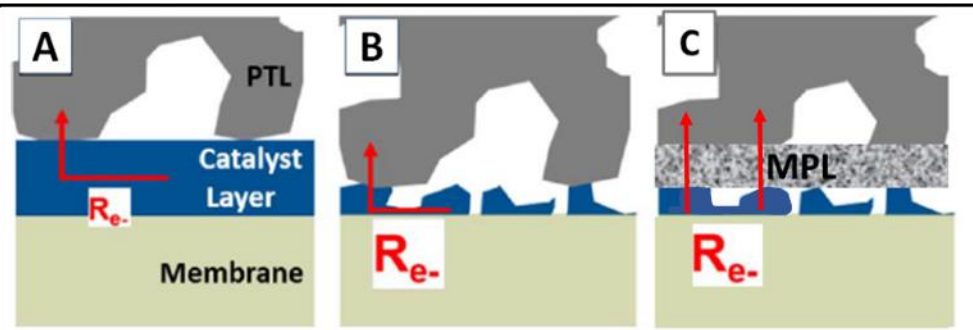


Ayers K, et al. 2019. *Annu. Rev. Chem. Biomol. Eng.* 10:219–39

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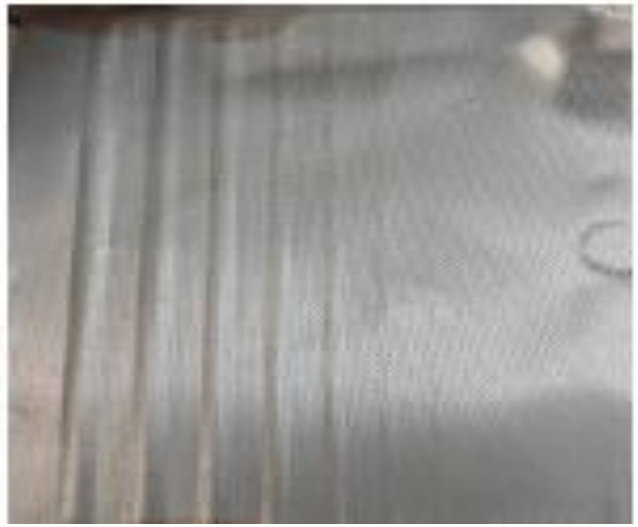
Relevance – Issues Addressed

- Available PTL materials are adapted from other industries and not optimized for electrolysis
- Porosity must be balanced with contact area
  - An added microporous layer provides a closer packed pore structure immediately adjacent to the catalyst layer
- Porosity also must be balanced with mechanical strength to support hydrogen pressure
  - The PTL must span the flow channels without deformation
  - A variable channel width fixture shows the deformation of insufficiently robust materials (right).



A – Thick electrode catalyst layer has lower resistance to the porous transport layer
B – Thinner electrode catalyst has very high resistance to porous transport layer
C – Microporous layer effectively contacts catalyst layer

Gasteiger Bernt M, Siebel A, Gasteiger HA. 2018. *J. Electrochem. Soc.* 165(5):F305–14



Deformation of PTL in spanwise test

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Project Goals

The project outcomes will be the development of an optimized porous transport layer designed for an electrolyzer system, upscaled to a manufacturing level. This PTL will enable integration of advanced membrane electrode assemblies, in order to advance PEM electrolyzers towards the DOE cost goals of $2/kg.

Goals

- Develop a microporous layer (MPL) for metallic PTLs compatible with liquid flow
- Optimize the PTL/MPL thicknesses for effective interfacial contact
- Develop conductive coatings on the MPL/PTL which are stable up to 2 V
- Demonstrate stable performance of the optimized component in electrolysis cells
- Demonstrate baseline efficiency and stability with low anode catalyst loadings
- Develop inspection process and qualifications for manufacturing process
- Quantify the cost savings enabled towards the DOE production target of $2/kg

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Approach: Schedule

Table 1: Gantt chart of tasks		Q 1	Q2	Q3	Q4	Q5	Q6	Q7	Q8	Q9	Q10	Q11	Q12
Task 1: MPL Prototyping and Characterization													
1.1	Parameter Range Testing	■	■	■									
1.2	Physical Testing		■	■	■								
1.3	MPL and PTL CT characterization		■	■	■	■	■	■	■	■			
1.4	Model predictions	■	■	■	■	■	■	■	■	■	■	■	■
1.5	Design down selection					■	■	■	■	■	■	■	■
1.6	Quantify batch-to-batch variability					■	■	■	■	■	■	■	
1.7	Model testing of morphological variability						■	■	■	■	■	■	■
Task 2: Manufacturing Development to Scale													
2.1	Baseline PTL manufacturing	■	■										
2.2	Initial manufacturing feasibility study			■	■								
2.3	Down-selection and manufacturing of PTLs for small-scale cell testing					■	■	■	■	■			
2.4	Manufacturing Scale Up								■	■			
Task 3: Quality Control Method Development													
3.1	Baseline PTL analysis	■	■	■									
3.2	Method development for MPL/PTL			■	■	■	■	■	■	■	■		
3.3	Scale method development					■	■	■	■	■			
3.4	Method verification									■	■	■	■
Task 4: Surface Treatment													
4.1	Coating Parameter Development	■	■	■	■	■	■						
4.2	Coating Characterization	■	■	■	■	■	■	■	■	■	■		
4.3	Coating selection and scale up								■	■	■		
Task 5: Cell Integration													
5.1	25 cm2 Bench Testing	■	■	■									
5.2	Small Scale Hardware				■	■	■	■					
5.3	Large Scale Hardware								■	■	■	■	■

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Approach: Milestones

Milestone List			
Area of Development	**Milestone**	**Due Date**	**Status**
Model Predictions	Complete initial model for predicting PTL transport properties, based on 3D simulations.	10/31/2021	Complete
Parameter Range Testing	The PTL specification framework will be developed with key component parameters identified and initial ranges established.	1/31/2022	Complete
Baseline PTL analysis	Demonstrate PTL inspection feasibility using in- line quality control methods.	1/31/2022	Complete
25 cm^2 Bench Testing	Demonstrate the initial prototype PTL/MPL samples meet performance baseline PTL as measured as electrical efficiency, degradation rate, and assessment of water flow restriction.	1/31/2022	Complete
Initial manufacturing feasibility study	Show translation of structures developed at prototype level to manufacturing facility based on demonstration of equivalent performance. Operating parameters to meet or exceed are 2.15 V at 58°C, and 1.8 A/cm^2, based on an N117 MEA with a degradation rate of 5µV/hr.	4/30/2022	Complete
Small Scale Hardware	Operationally test manufactured PTL/MPL parts in 100 cm^2 cell stack at 2.2 A/cm^2, 400 psi, and 58°C for 1000 hrs with a degradation rate of ≤ 5 µv/hr. OER catalyst loadings will be reduced by 50% versus the baseline MEA loadings.	10/31/2022	In-Progress
	Demonstrate MPL structure meeting performance requirements with catalyst activity of 2000 mA/mg vs. baseline of 800 mA/mg at 2.1V.	10/31/2022	Pending

- All milestones completed to date
- Activities in progress for next goals
  - Improved catalyst utilization
  - Scale-up in stack active area and cell count
- Go-No/Go Milestone for BP1

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Approach: Task Outline

➢ The project will consist of 5 primary tasks:

I. MPL prototyping and characterization: optimization of the MPL thickness, particle size, and porosity (**ORNL/UCI**);

II. Manufacturing development to scale: translation of optimized structures to high-volume manufacturing processes (**De Nora**);

III. Quality control method development for high-speed inspection of large areas of precision components (**NREL**);

IV. Surface treatment: Deposition of surface layers for conductivity (**Nel, De Nora**);

V. Cell integration: accelerated testing, and process scale up to full active area cells with performance validation vs. baseline and resulting cost analysis (**Nel**).

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Collaborations

Partners	Project Roles
Oak Ridge National Lab	➢ Specialize in rapid prototyping of PTLs ➢ Develop optimal part design specifications at the ORNL Manufacturing Demonstration Facility (MDF)
De Nora	➢ Integration of prototype PTL design specifications at scale ➢ Develop manufacturing process for high volume fabrication
University of California, Irvine	➢ Imaging of water/gas transport through the PTL ➢ Modeling of interface between PTL and catalyst layer
National Renewable Energy Lab	➢ Line inspection methods for maintaining quality control

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Accomplishments: Oak Ridge National Laboratory

Wafer Manufacturing and Characterization

- Pure titanium wafers printed on Arcam electron beam melting (EBM) system
- XCT scans were performed on wafers to determine porosity



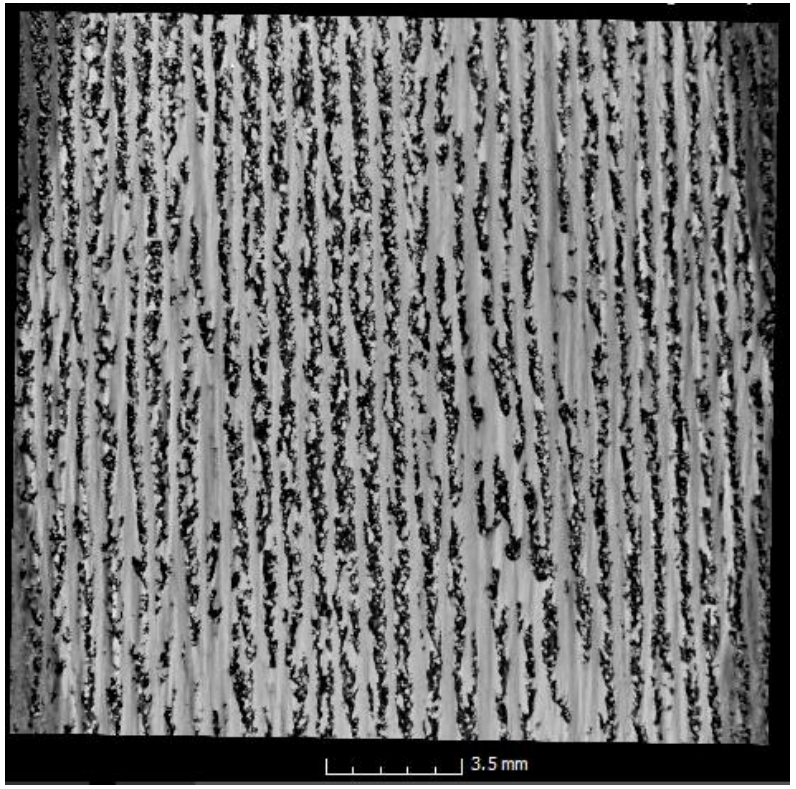
Arcam Q10

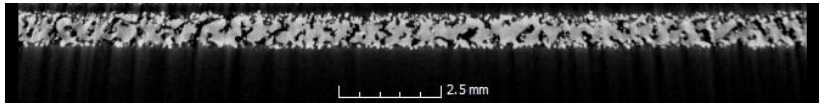


Wafer Build 75 mm Tall

XCT XZ Section

BD





XCT XY Section

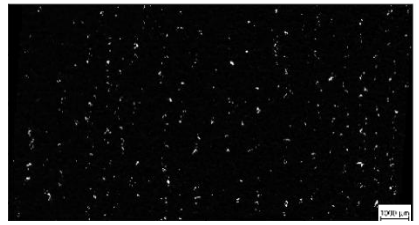
Accomplishments: Oak Ridge National Laboratory

Preliminary Process Parameter Effects

- Focus offset (FO), current, and overlap have the largest Effect on porosity
  - Densities estimations from thresholding transmission images (XCT scans done on later builds)
  - FO and current influence printability as well
  - Number of lines and line spacing were varied in later builds and compared to thickness and porosity using XCT

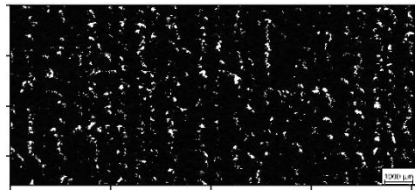
20211030 Build and Optical Densities					
Set	FO	Spot Time	Overlap	Current	Density (%)
1	0	0.4	0.6	5	99.43
6	25	0.4	0.6	5	96.66
8	15	0.4	0.6	5	98.04
9	15	0.5	0.6	5	98.49
10	15	0.4	0.6	3	93.62
20211108 Build and Optical Densities					
Set	FO	Spot Time	Overlap	Current	Density (%)
7	15	0.4	0.6	5	95.54
8	15	0.4	0.3	5	87.81

Set 1



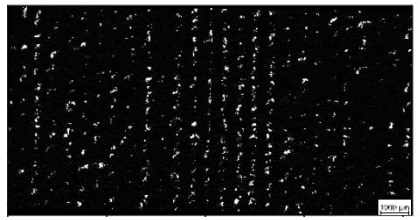
Density ~ 99.43

Set 6



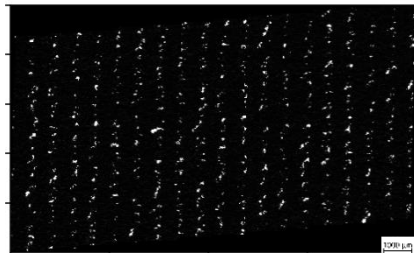
Density ~ 96.66

Set 8



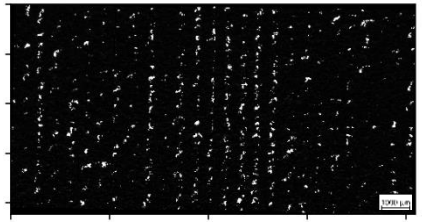
Density ~ 98.04

Set 9



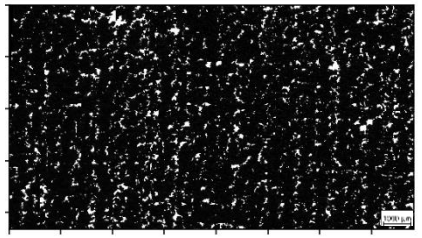
Density ~ 98.49

Set 8



Density ~ 98.04

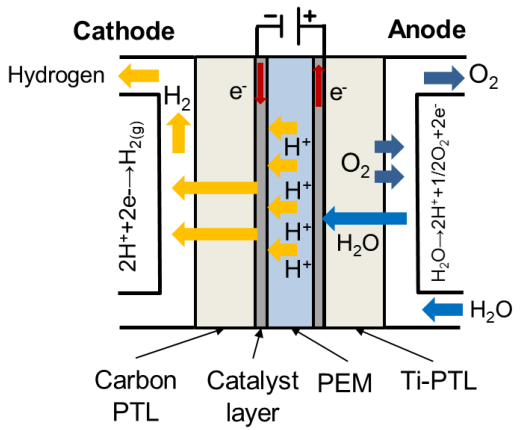
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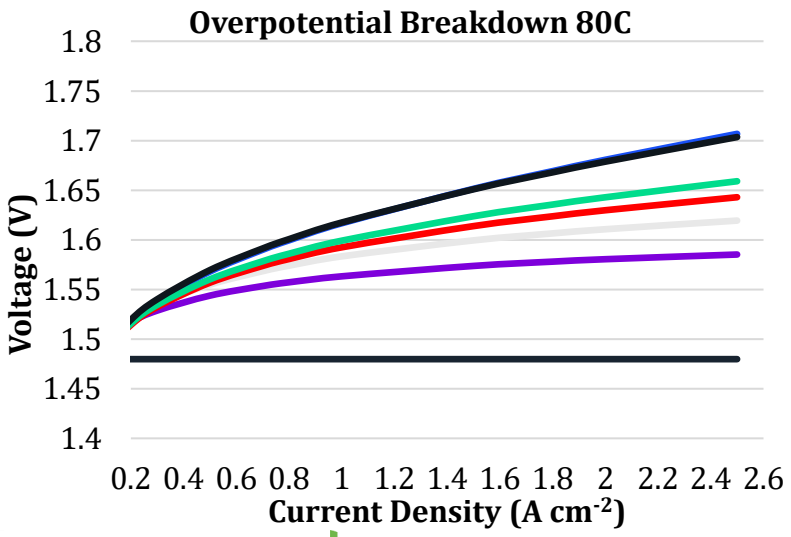


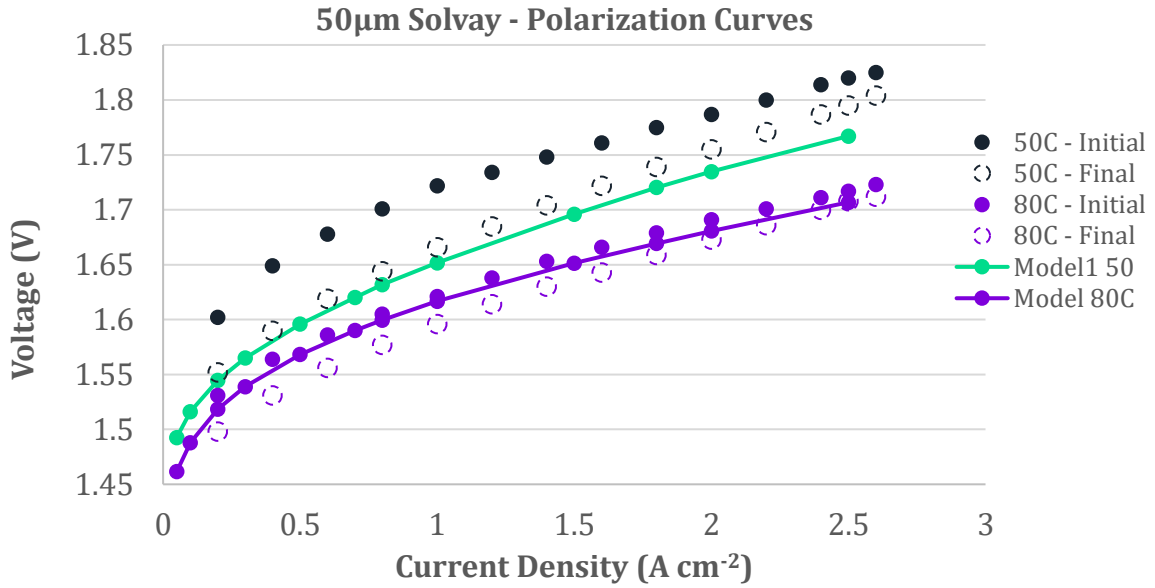
Density ~ 93.62

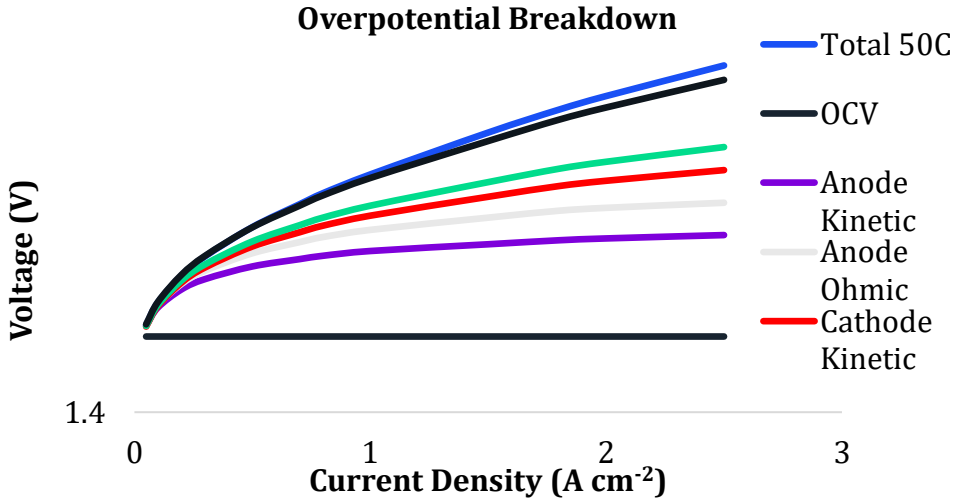
Accomplishments: University of California, Irvine

- 2D steady-state, cross-section PEWE model developed (Comsol)
  - OER with Tafel equation, currently no oxygen coverage
- Model validated with experimental data for 50 and 80C
- Overpotential analysis carried out to investigate PEWE losses
  - Anode kinetics show the highest loss with thin membranes
  - Membrane Ohmic is the second largest overpotential
  - At 50°C Ohmic overpotential increases to 71 mV from 44 mV at 80°C at 2.5 A/cm^2





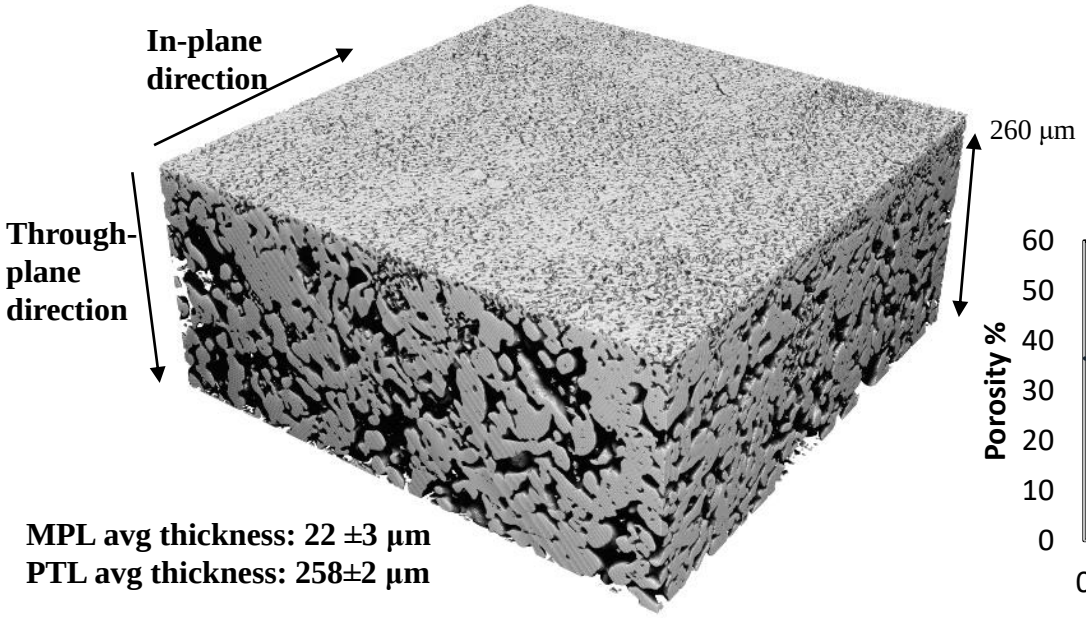




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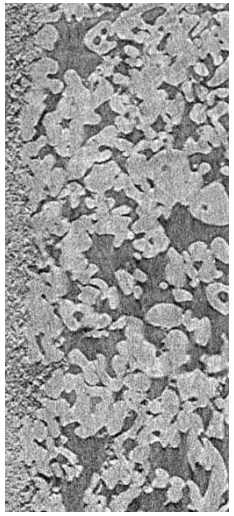
Accomplishments: University of California, Irvine

- X-ray computed tomography (CT) imaging and high throughput data analysis for samples quality control (QC):
  - Automated algorithms for porosity, tortuosity, pore-size distribution
  - 3D visualization
  - X-ray CT (1 µm resolution) resolves MPL & PTL
- Example of process on project sample

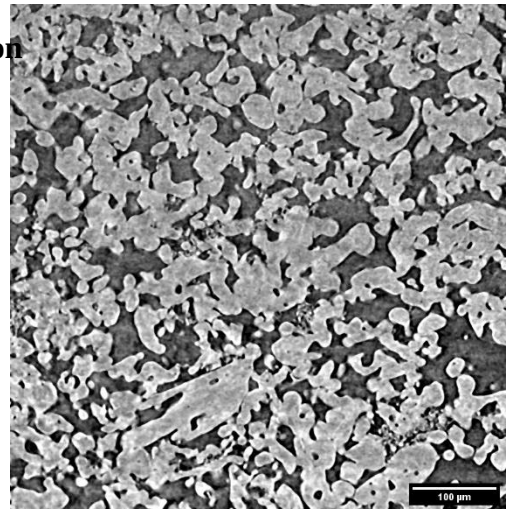


MPL avg thickness: 22 ±3 µm
PTL avg thickness: 258±2 µm

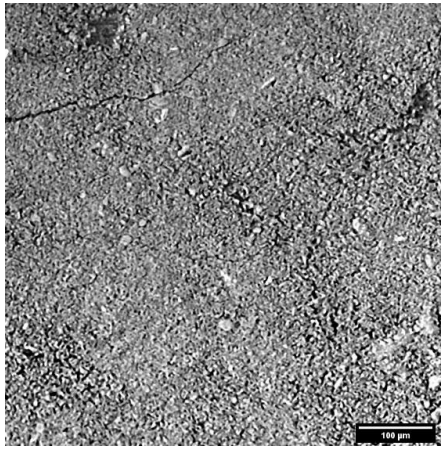
Side cross section



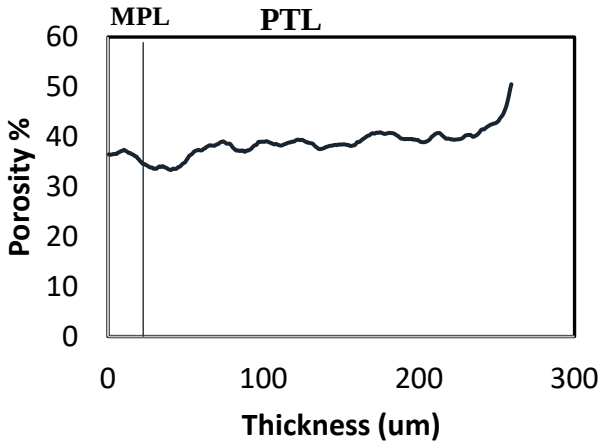
PTL cross section (in-plane)



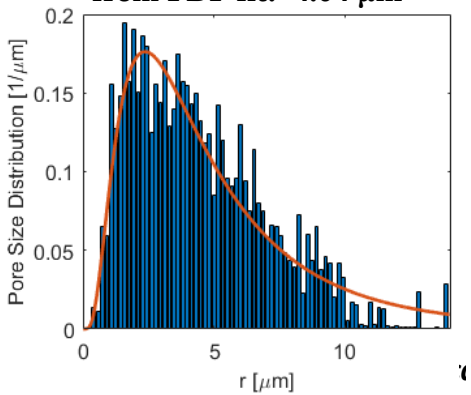
MPL cross section (in-plane)



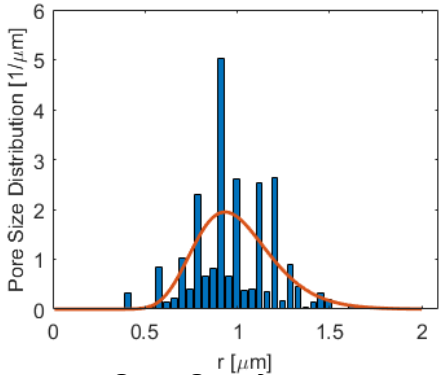
MPL average porosity: 36.7 ±0.4
PTL average porosity: 38.8 ±1.9



PTL average pore radius from PDF fit: 4.04 µm



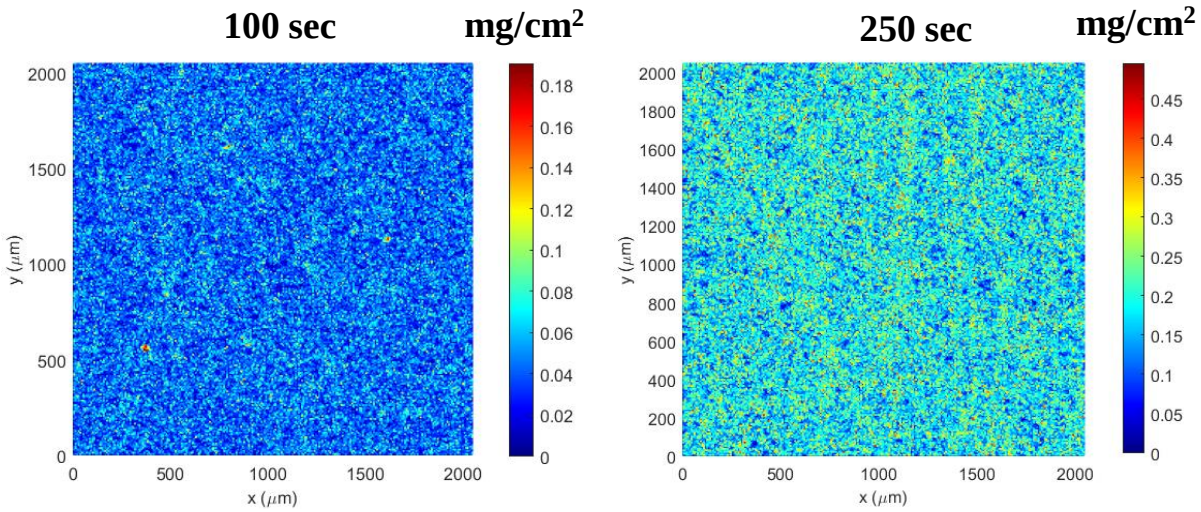
MPL average pore radius from PDF fit: 0.97 µm



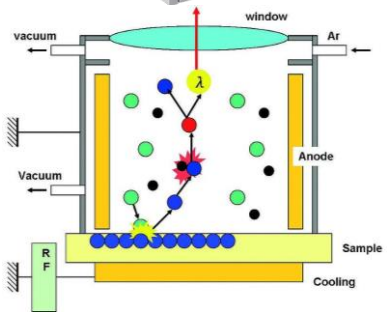
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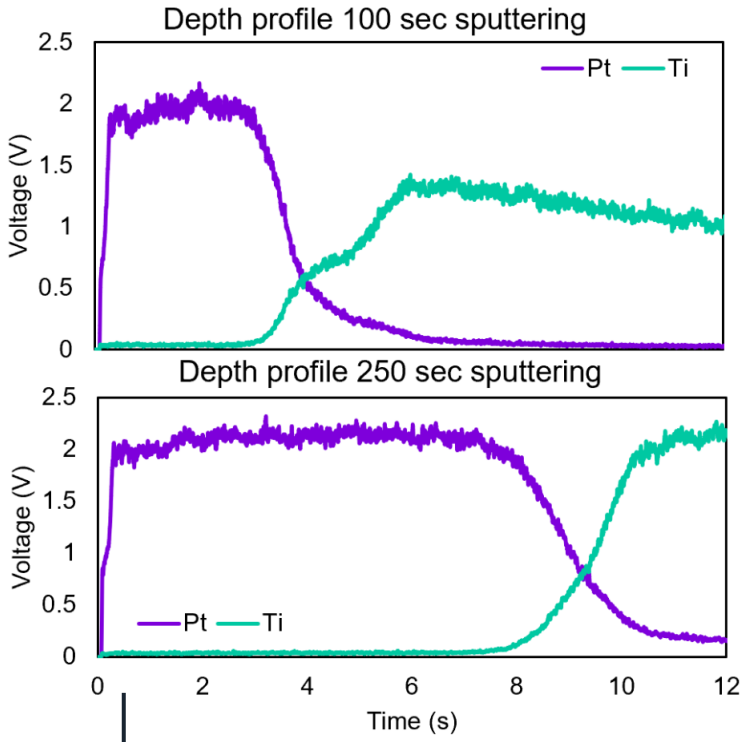
Accomplishments: University of California, Irvine

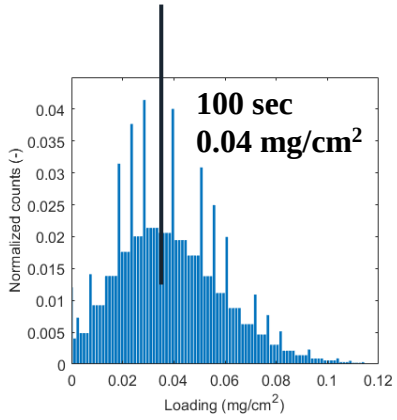
- X-ray fluorescence (XRF) is used to obtain loading distribution for Pt on PTLs:
  - For 100 sec of sputtering time average loading = 0.04 mg/cm^2, for 250 sec = 0.15 mg/cm^2
  - Calibration curves being built to correlate sputtering time to Pt thickness and loading
- GD-OES is used to obtain thickness of Pt deposition; time is correlated to thickness
  - 100 sec vs 250 sec showed 3x loading and 3x thickness

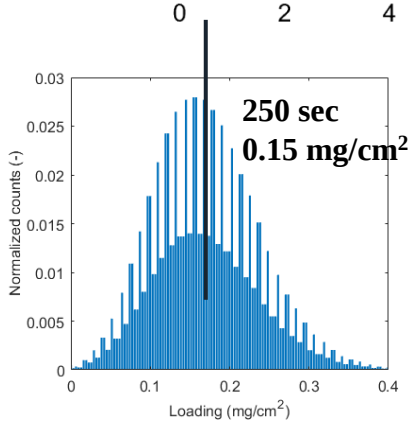




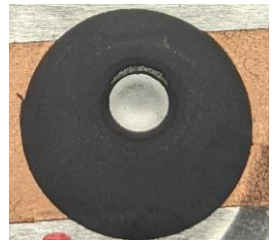








GD OES crater

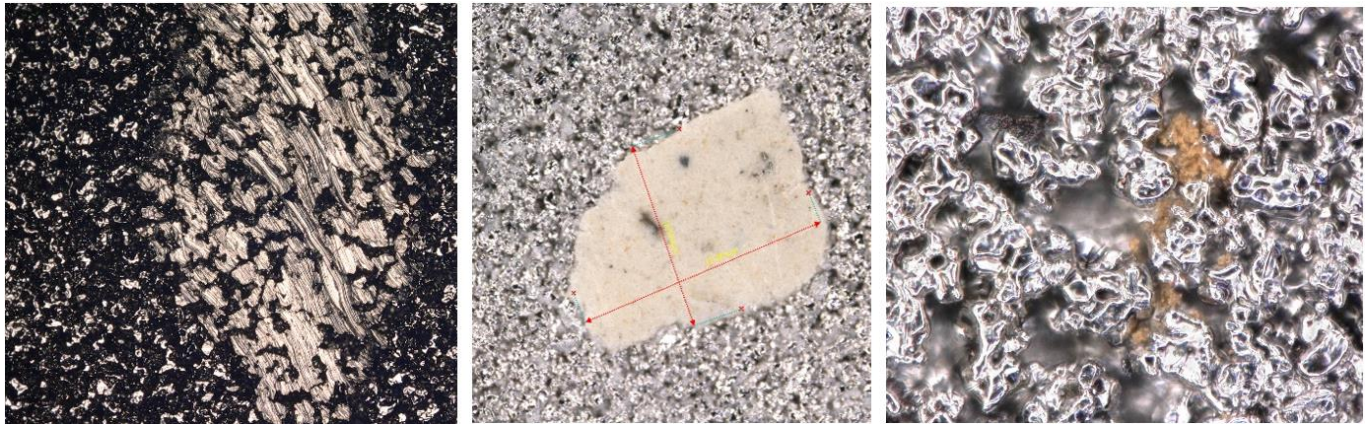


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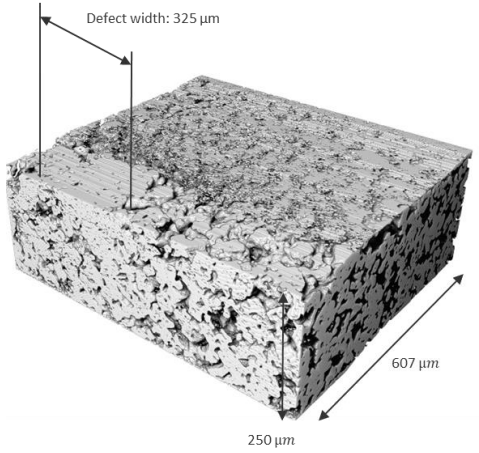
Accomplishments: National Renewable Energy Laboratory

QC methods for baseline PTLs

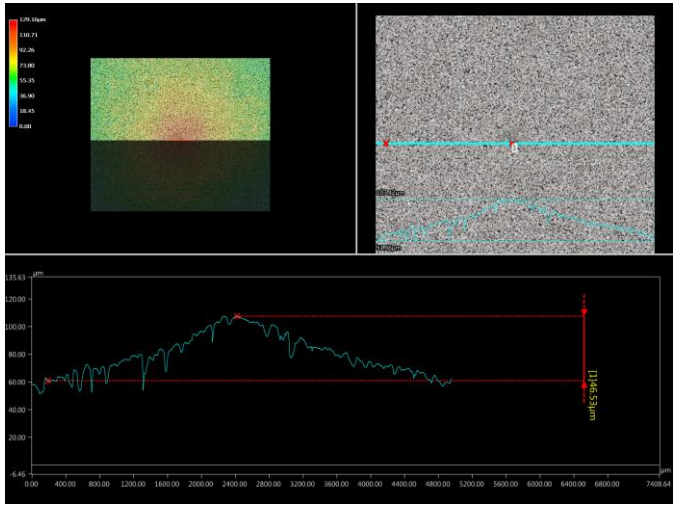
- Microscopy and CT analysis for a series of PTL defects:
  - Large cracks, shiny spots, indentations, dark spots and discolorations.
- Summary with conclusions on automatic detection and potential severity (critical vs. benign) of the inhomogeneities were drawn.



Example defects in PTLs: shiny spot (left), embedded foreign object (center) and brown color contamination (right).



CT reconstruction of a so-called black spot



Surface topography around an indentation defect



Example of cross-sectional micrograph, which was used to obtain local porosity values.

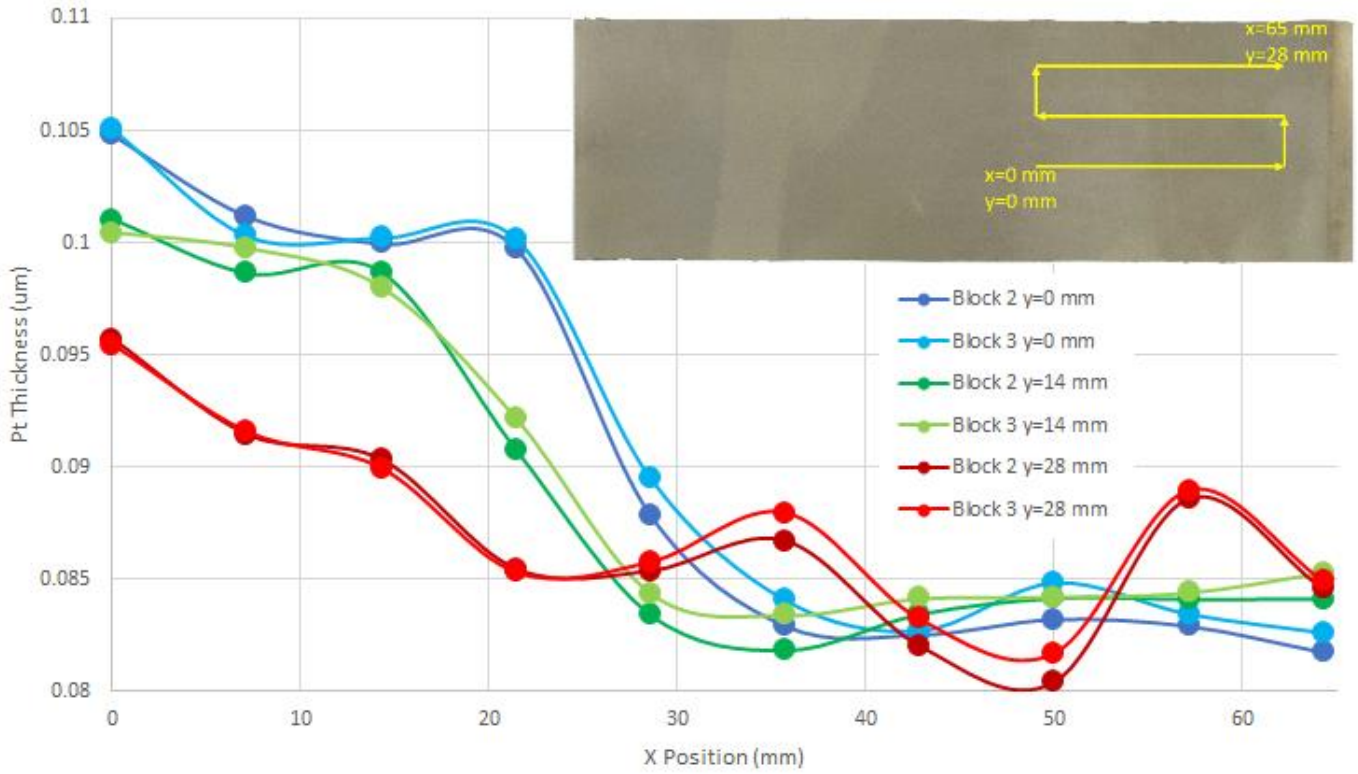
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Accomplishments: National Renewable Energy Laboratory

QC method evaluation for PTL with Pt coating

- Performed Pt thickness measurements using a benchtop XRF apparatus
  - Three repeats were acquired to estimate the experimental noise
  - Three 65mm long profiles, 10 spots per profile
- Obtained results:
  - High repeatability was observed with variations less than 0.8% for a spot size of about 1mm.
  - Pt thickness was found to be position dependent in a range from 82 to 105 nm with the relative coefficient of variance equaled 9% over the entire area tested.
- XRF was found to be a robust technique for estimation of Pt coating thickness. In-line implementation of the technique is possible.

Small range XRF measurement to determine Pt coating thickness as a function of position, X.



Statistical results

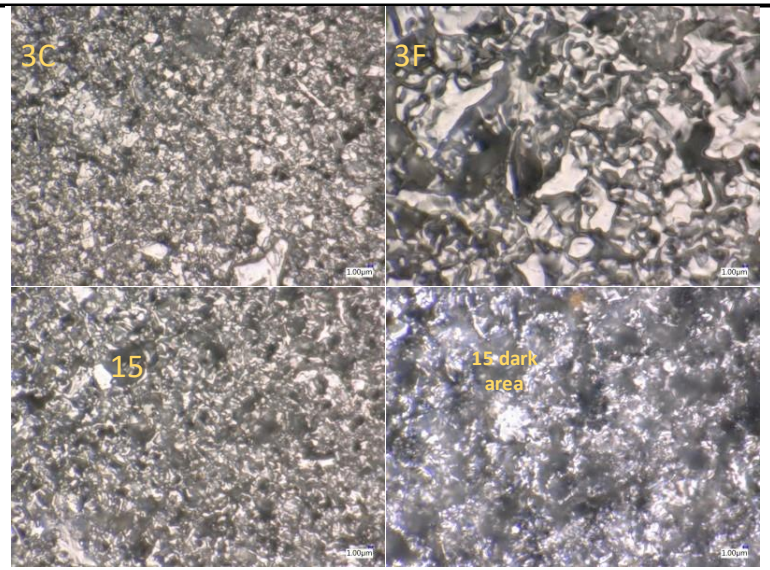
Average thickness	**89 nm**
Standard deviation	**8 nm (8%)**
Repeatability	**0.7 nm (0.8%)**

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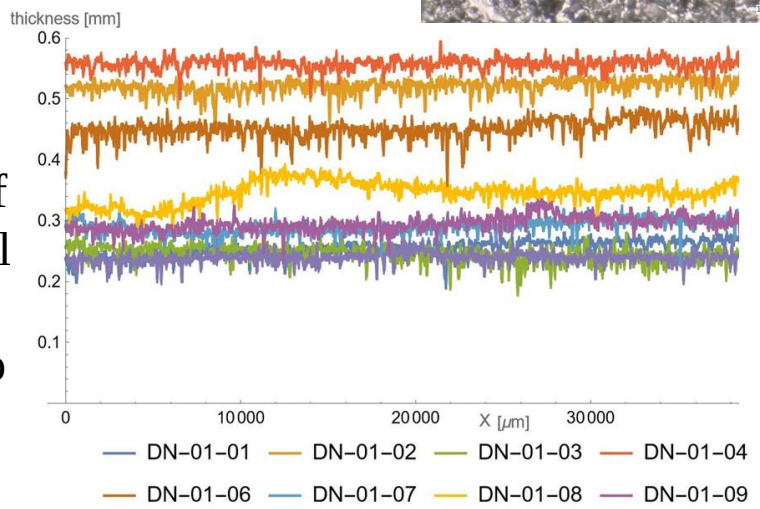
Accomplishments: National Renewable Energy Laboratory

QC methods evaluation for PTLs with MPL

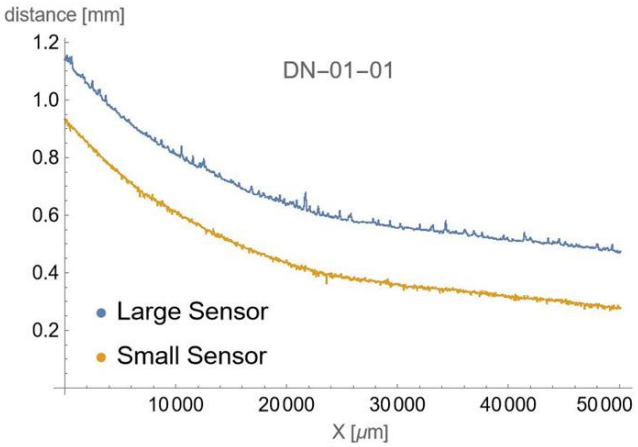
- Analyzed thickness, curvature, and surface roughness profiles of selected PTLs with and without MPL layers
  - Chromatic confocal probes for geometrical characterization
  - Optical microscopy to visualize MPL particles
- Both methods can potentially be used for high-throughput in-line motoring when material under test is in motion.
- The surface roughness acquired was a function of the material (MPL vs. PTL) and the sensor spatial resolution, or "spot size".
- The thickness standard deviation was observed to be 4-5 larger for the initial 3D printed parts as compared to the baseline PTL material.



Comparison of micrographs of three different MPL samples.



Thickness profiles obtained using chromatic confocal probes



Example of distance profiles showing plate curvature over 50mm long distance

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Accomplishments: De Nora

De Nora PTLs

Sample ID	Production Measurements						Sample Size	Quantity	Comments
	Layer #1 - Macro-Porous Layer			Layer #2 - Microporous Layer					
	Thickness (mm)	Particle size (um)	Porosity (%)	Thickness (um)	Particle size (um)	Porosity (%)			
DN-00-00	0.25								Single Layer PTL
DN-00-3-C	0.25								
DN-00-3-F	0.25								
DN-00-15	0.494								
DN-01-01	0.25						7in x 7in	1	
DN-01-02	0.5						7in x 7in	1	
DN-01-03	0.25						7in x 7in	1	
DN-01-04	0.5						7in x 7in	1	
DN-01-05	0.25						7in x 7in	1	
DN-01-06	0.5						7in x 7in	1	
DN-01-07	0.25						7in x 7in	1	
DN-01-08	0.25						7in x 7in	1	
DN-01-09	0.25						7in x 7in	1	

13 samples from DeNora have been received
1 sample is a baseline PTL with no MPL

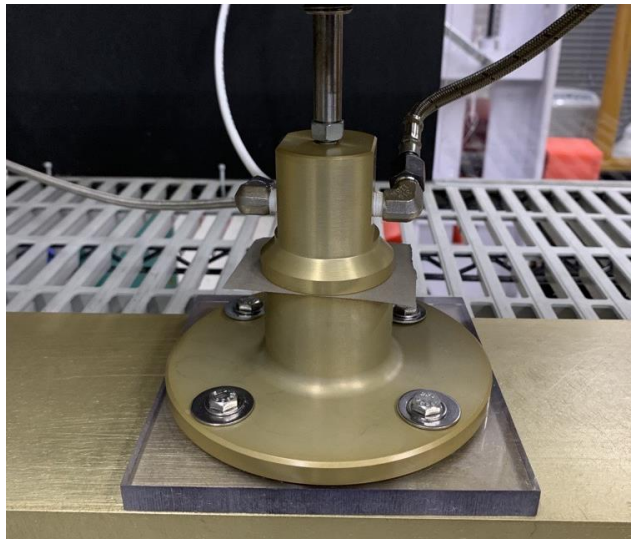
PTL Specifications

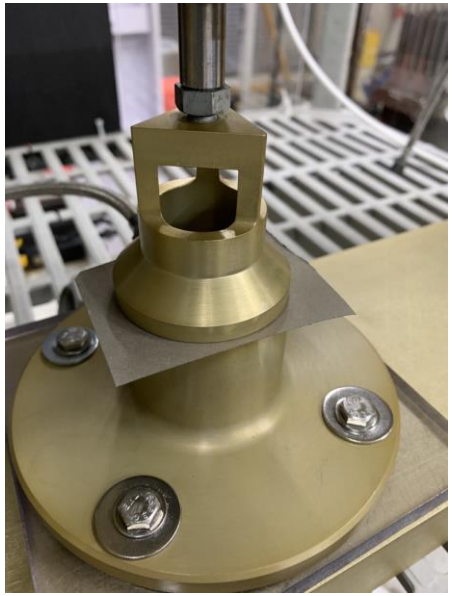
Property		Test	Acceptable Range
Composition		XRF	ASTM B265 Grade 2 Composition
Appearance		Visual Examination	
Surface Contamination		Visual Examination	
Density		(Measured Sample Density/Theoretical Density of Titanium)*100	
Bubble Point	Initial	Pressure at which first bubble appears through PTL	
	Full	Pressure at which bubbles appear to pass through the majority of the PTL area	
Tensile Strength		ASTM E8	
Surface Rougness		Material Inspection	
Flatness		Lay sample on a flat surface and measure the change in height	
Porosity		Visual Examination. Correlation with Bubble Point Testing	
Thickness		Micrometer measurement across PTL	
Resistance to Water Flow		Sample placed on a vacuum flask. Water is filled in the funnel above the sample and a vacuum is turned on. Stopwatch used to measure how long it takes for water to pass through the PTL.	
Spanwise Strength		PTL placed in a fixture that has channels with increasing width. N2 is applied to the plate at a pressure of 780 psi for at least 30hrs. Sample is then visually inspected.	
Resistivity		PTL is placed between Au-plated Cu, which is placed between terminal plates, which is placed between insulating plates. The setup is then placed in a mechanical press to 350 psi. 25A current is applied to the setup and the voltage is measured.	
Particle Size		SEM or XRD	
Pore Size		SEM and Image Analysis	

Framework for PTL specifications completed
Final acceptance criteria still pending testing

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Accomplishments: Nel







Gas Flow Testing

Determines the resistance to gas flow through the PTL sample

Sample is secured in a fixture where N_2 gas can be applied to one side of the sample and gas flow through the PTL is measured

Water Flow Through Testing

Water flow rate through the PTL/MPL sample

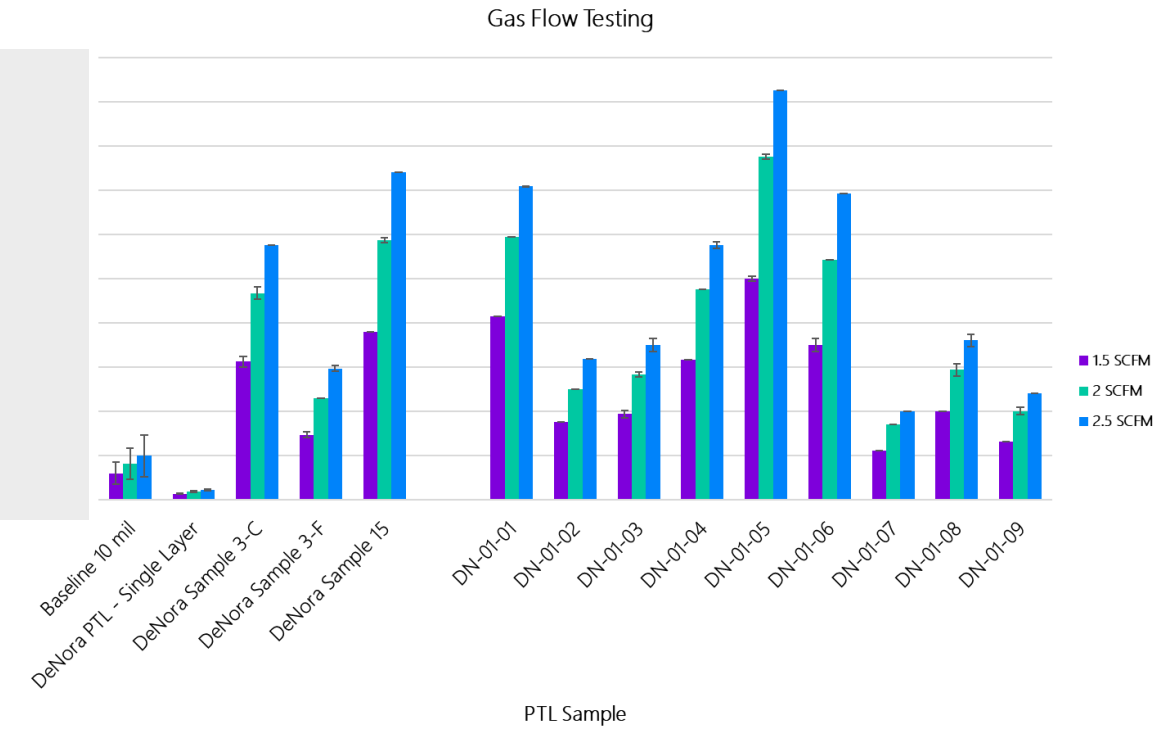
- Slower flow rate -> Risk of water starvation
- Too high of a flow rate -> Possible large porosity could lead to poor membrane support

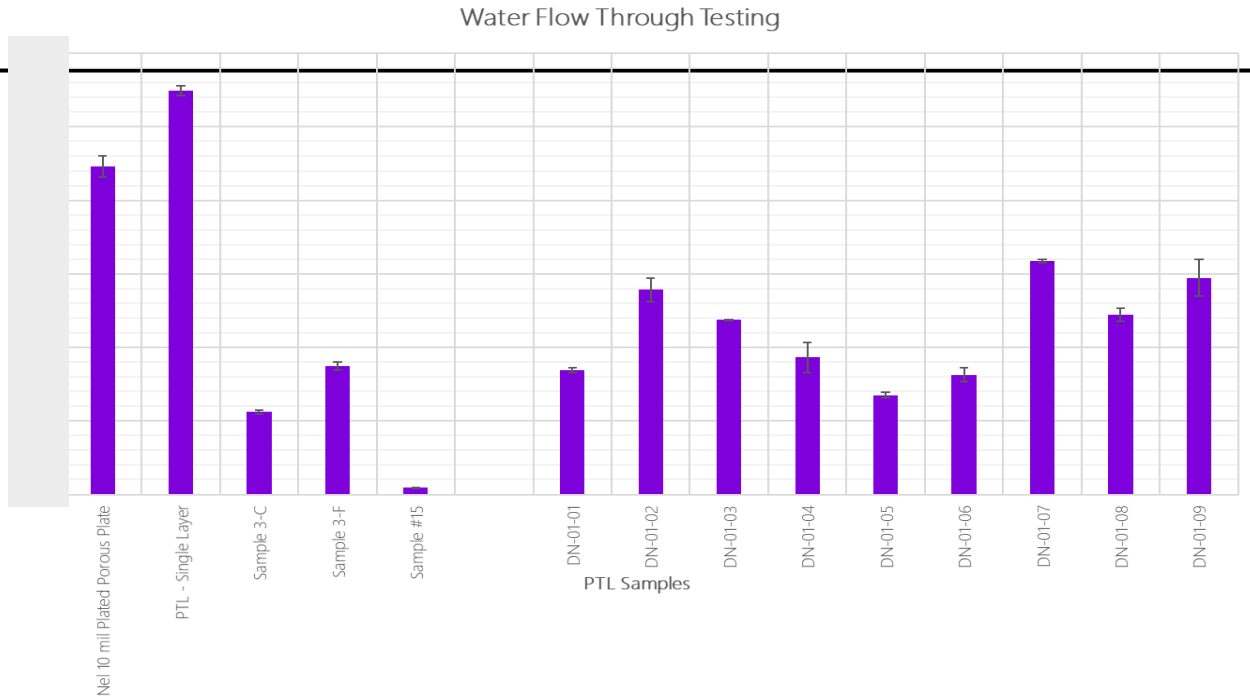
Spanwise Strength Testing

Understand the general strength of the PTL under compressive force

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Accomplishments: Nel

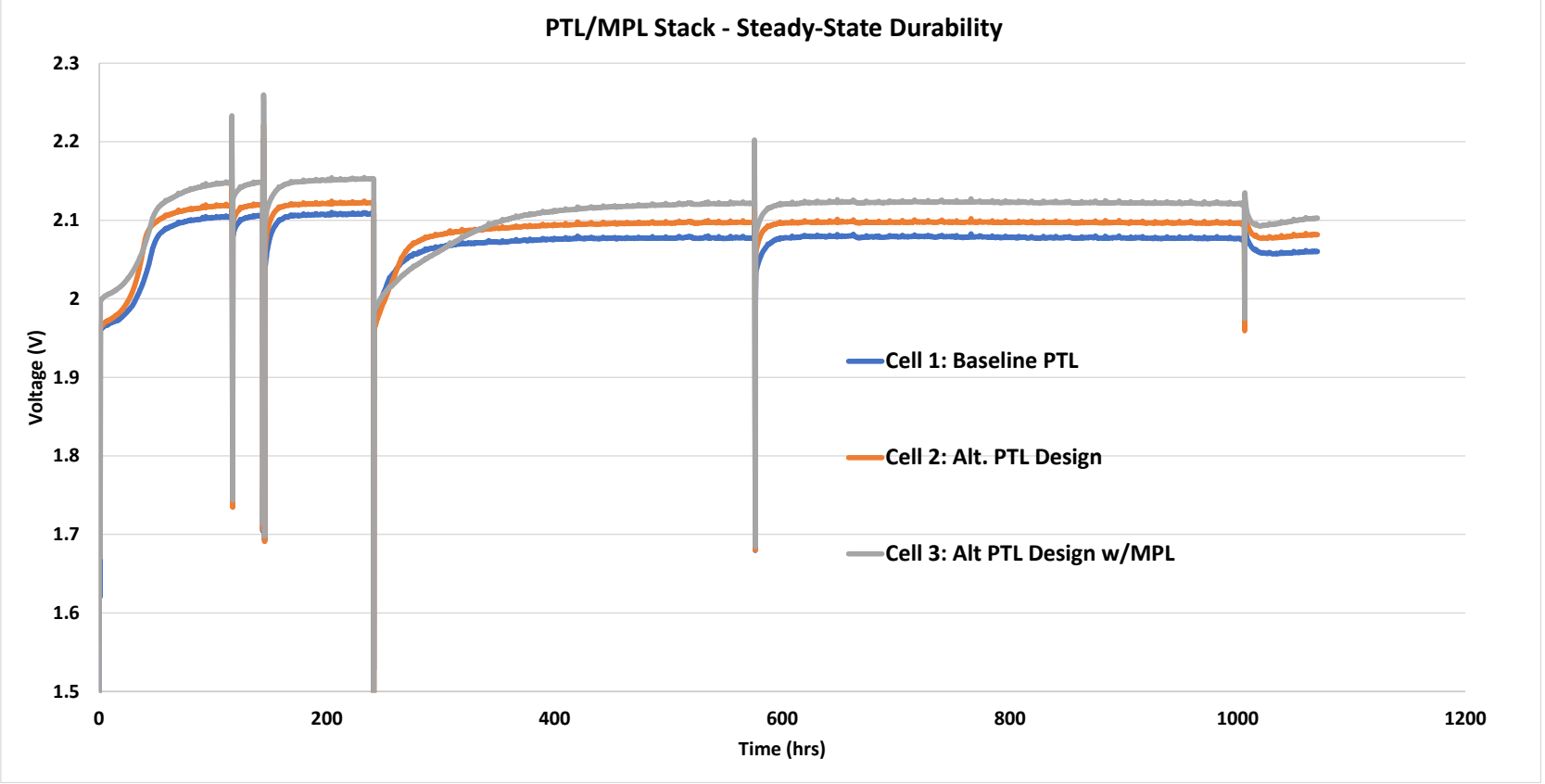




- Data collected to date
- Using to establish correlation to in-cell testing
  - Will aide in development of final inspection criteria
  - Predictive testing to narrow operational testing

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Accomplishments: Nel



- Test conducted at 1.8 A/cm^2, 50°C, 400psi
- 28cm^2 active area

- Steady-state testing of early samples to look for gross performance differences
  - Baseline
  - Alternate PTL
  - Alternate PTL/MPL
- Degradation rate collected from 600 – 1000 hrs:
  - Cell 1: -19.0 µV/hr
  - Cell 2: -7.8 µV/hr
  - Cell 3: -11.3 µV/hr
- Early testing shows addition of MPL does not negatively change rate of degradation
- Work on-going to optimize structure

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Challenges and Barriers

Potential Risks	Possible Mitigation Approach
Higher density MPL restriction degrades performance over time	Leverage CT imaging and flow model to quantify surface flow and design margin
Optimal prototype structures developed within project are not manufacturable by DeNora	Leverage prototyping and modeling effort to understand full range of acceptable parameters for manufacturing
Base PTL does not have sufficient mechanical strength	Work with supplier on higher density materials; apply DeNora processes to alternate qualified PTL
Alternate PGM coatings at 10% loading target degrade over time	Explore post-processing to assist adhesion; increase loadings while staying below baseline

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Future Work:

- Continue with evaluation of PTL/MPL samples to narrow the configurations for down-select
- Develop specification table with parameters for optimal PTL/MPL composition
- Refine list of inspection methods for use as QC techniques in production
- Scale-up form factor of PTL/MPL to show translation from smaller sample sets
- Scale-up cell stack active area testing and demonstrate enabling capability of the MPL to reduce OER catalyst loadings

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Summary

- Work has developed a model with convergence to data collected in-cell
  - Will assist with predictive work for new configurations of PTL/MPL being explored
- QC measurement techniques used at NREL for R2R and other processes have shown translation to inspection of PTL sheets
- Framework developed for specification criteria used to define final PTL/MPL configurations
- Successful fabrication and test of PTL/MPL samples
  - 1000hrs completed with stable operation
  - Will look at optimizing structure to narrow performance gap
- Hardware is ordered and scale-up testing is in progress
  - Demonstrate translation to scale and enabling ability of the MPL to lower OER electrode loadings

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Technical Back-up and Additional Information

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Technology Transfer Activities

- None planned at this time in the project.

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