



Roll-to-roll Advanced Materials Manufacturing Lab Collaboration

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National Renewable Energy Lab Lead: Michael Ulsh

Oak Ridge National Lab Lead: Georgios Polyzos

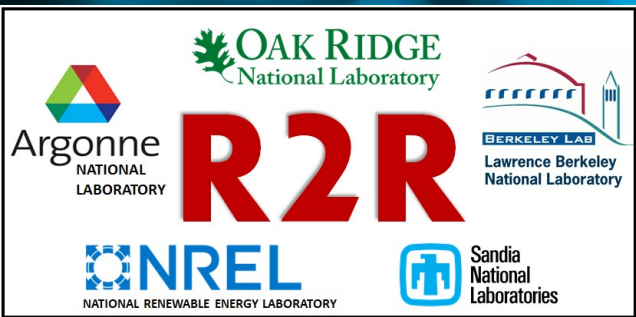
Sandia National Lab Lead: Randy Schunk

June 10, 2021

DOE Hydrogen Program

2021 Annual Merit Review and Peer Evaluation Meeting

Project ID #TA007



Project Goal

Per HFTO guidance, we have combined two separately funded activities of the R2R Multi-lab Collaboration in this presentation. Where appropriate, and due to slide count constraints, we will include information for both sub-activities on one slide.

AMO Fuel Cell Core Lab Activity

- Project Goal: Leverage the focus areas within the overall AMO activity on multilayer coating, electrospinning, and metrology to advance process understanding and explore novel structures for fuel cell MEAs

HFTO R2R Activity

- Project Goal: Leveraging the extensive capabilities within the 5 labs, identify and complete “key deliverable” projects defined by four key U.S. industry participants to directly address electrolyzer and fuel cell MEA scale-up challenges

Overview

Barriers

Barrier	Target
A. Lack of high-volume MEA processes	\$20/kW (2025) at 100,000 stacks/yr
H. Low levels of quality control	

Collaboration Partners: ORNL (lead), ANL, LBNL, NREL, SNL

Timeline and Budget

AMO Core Lab Activity

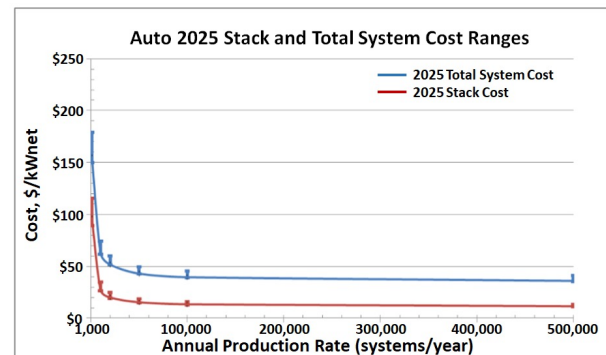
- Project Start Date: 4/1/2016
- FY20 AMO Funding: \$3M
- FY20 HFTO Funding: \$150K for Nel Hydrogen CRADA
- FY21 AMO Funding: \$3M

HFTO R2R Activity

- Project Start Date: 3/5/2020
- FY20 HFTO Funding: \$850K
- Industry Participants:
 - Electrolysis: Plug (formerly Giner ELX), Nel
 - Fuel cell: GM, Plug Power

Relevance

- R2R is the only manufacturing process platform that will meet cost and volume targets for MEAs.
- R2R enables conversion of multiple dissimilar materials into a multi-layer cell.
- All DOE-sponsored cost analyses for high-volume production of MEAs/cells assume R2R processing.
- Addressing DOE Goals:
 - Supporting manufacturing scale-up challenges 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 greenhouse gas emissions and enable clean energy power and fuel infrastructures in the U.S.



SA Inc. Mass Production Cost Estimation of Direct H₂ PEM Fuel Cell Systems for Transportation Applications: 2017 Update

Task 1: Membrane Electrode Assemblies

- 1.2 Develop processes for direct coating of electrodes on membranes or gas diffusion media.
- 1.3 Develop continuous MEA manufacturing processes that increase throughput and efficiency and decrease complexity and waste.

HFTO MYRD&D Plan - Manufacturing R&D

Approach

AMO Fuel Cell Core Lab Activity

- Leverage key focus areas and capabilities within the overall AMO activity to advance MEA processing understandings
 - Multilayer coating
 - Electrospinning
 - Metrology
 - Advanced characterization for inks and electrodes
 - Device testing
- Focus in FY21 on novel multilayer and electrospun electrode structures

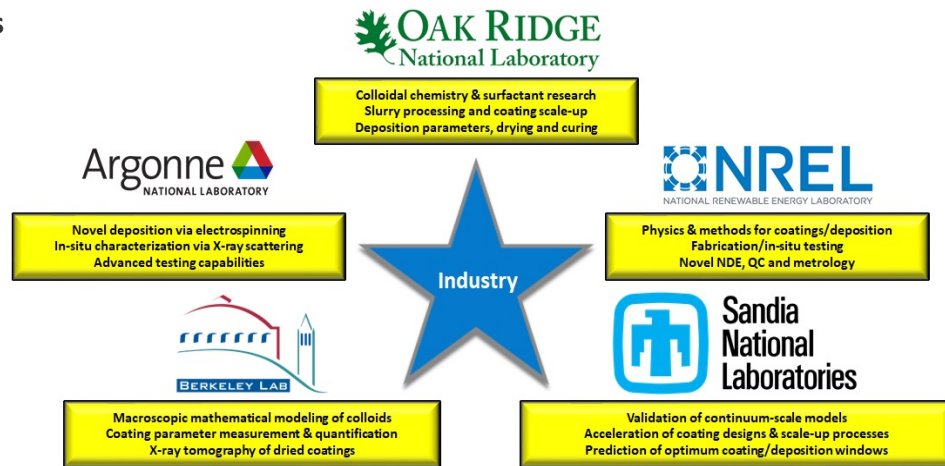
HFTO R2R Activity

- Address HFTO guidance to ensure relevance to industry MEA scale-up
- Work with four industry partners to define key deliverable projects
- Focused lab teams collaborate to complete projects, with input from partners
- Transfer learnings and results to partners

Approach

Overall, the multi-lab collaboration leverages extensive capabilities at the five labs, and leverages broad industry partnerships

- Broad range of research task areas focused on roll-to-roll manufacturing
 - Coating consolidation modeling (LBNL, SNL)
 - Particle-scale ink modeling (LBNL)
 - Coating flow modeling (SNL)
 - Colloids and rheology studies (NREL, ORNL)
 - Ink characterization: USAXS, SANS (ANL, ORNL)
 - Coating Methodologies
 - Multilayer coating studies (NREL, ORNL)
 - Electrospinning (NREL, ANL)
 - Electrode characterization: Xray Spectroscopy, XCT, Electron Microscopy, XRF (ANL, ORNL, NREL)
 - MEA fabrication and testing (NREL)
 - QC development (NREL)
- Task areas will include continued research on fuel cell and low-temperature electrolysis materials



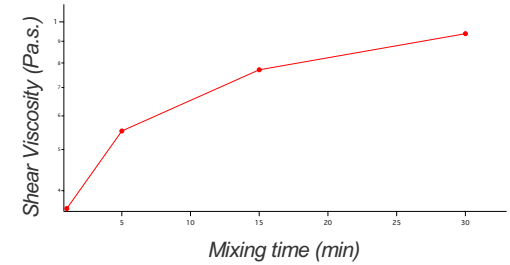
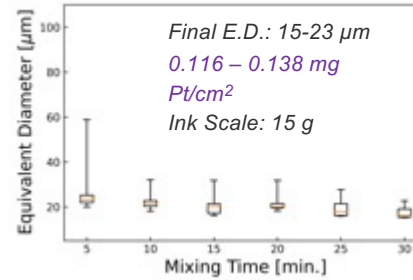
AMO Core Lab Activity

Accomplishments and Progress:

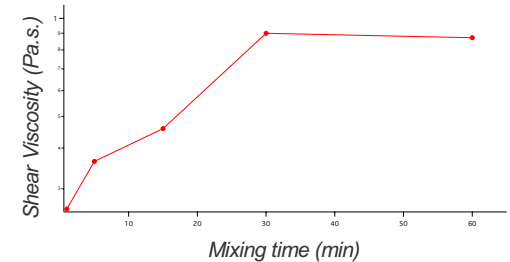
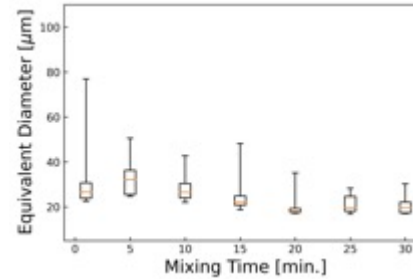
Determining Effectiveness of Scalable Mixing Methods

- There is a need to determine effectiveness of scalable mixing methods and their impact of performance and durability
- Ball milling results in most reduction in size but is slow
- Determined that rheology correlates with mixing and can be used to determine mixing times
- Rotor stator mixing found to be dependent on mixing element geometry and speed
- Work continuing to determine performance and durability impacts

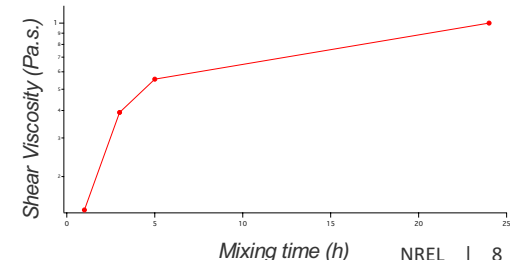
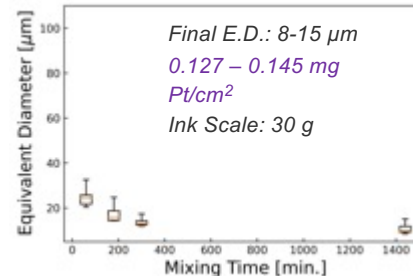
Tube Rotor Stator (6k rpm)



Rotor-Stator (10k rpm)



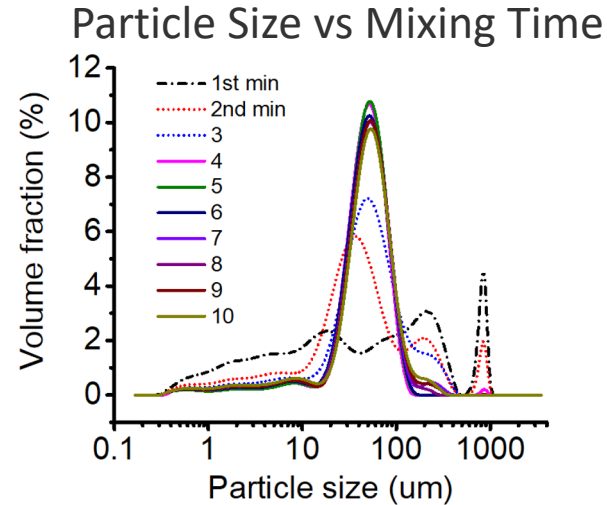
Ball Mill (20 speed units)



Accomplishments and Progress:

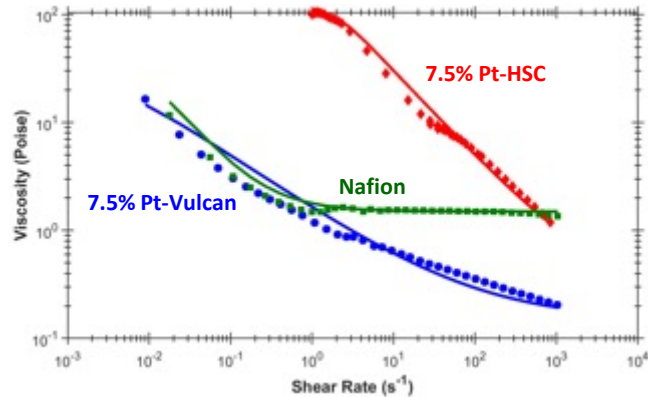
Building Portable Mixing Dynamics Experimental Setup

- Computer-controllable
 - Customized homogenizer
 - Peristaltic pump
 - Chiller
 - Sample stage
- Measurement
 - Temperature
 - Particle size analyzer
 - Microscope flow cell
 - More to come...
- Allows consistent sample production and analysis in conjunction with instruments in different locations
- Collects data about component-level interactions, needed for development of mixing models and initial conditions for coating drying models
- Effort funded by AMO

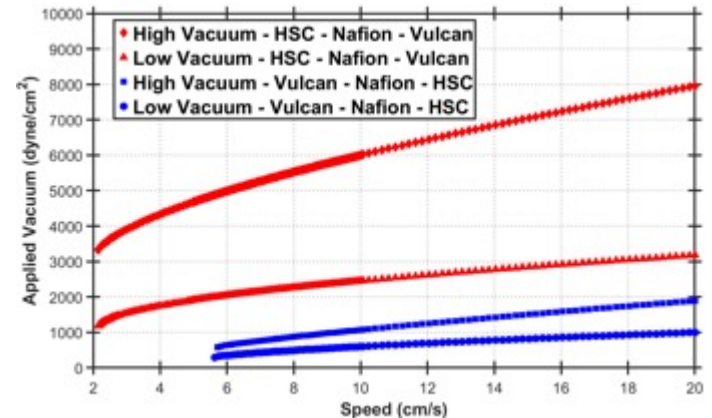
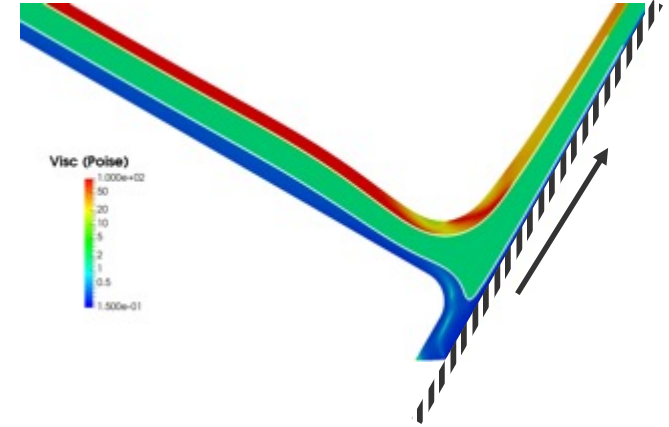


Accomplishments and Progress: Continuing Development of Coating Flow Models

Rheological Model



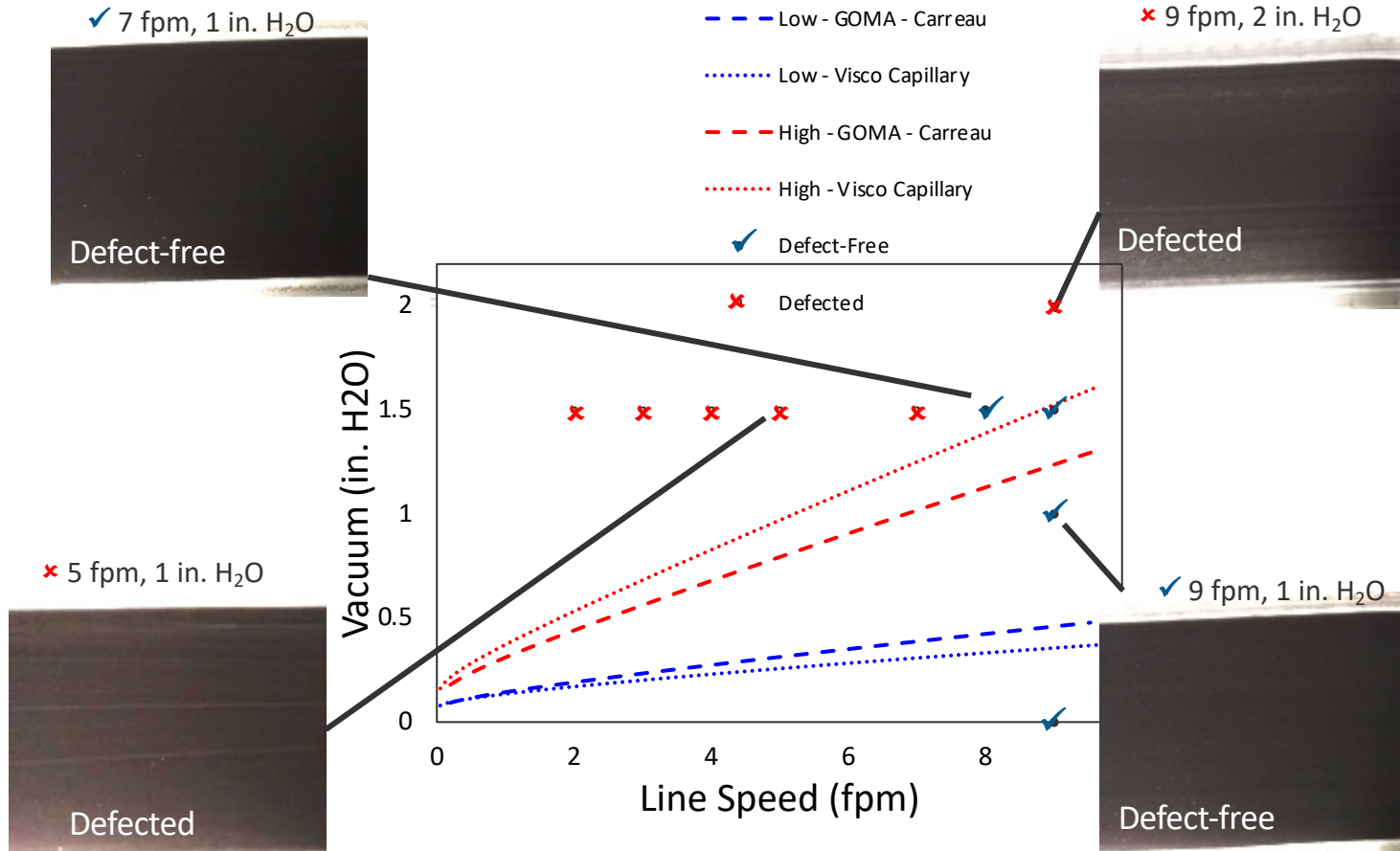
- Coating flow modeling approach:
 - Slide Die - Steady two-dimensional finite element model – Goma 6.0
 - Slot Die –
 - Two-dimensional finite element method – Goma 6.0 (Sandia/UNM)
 - One-dimensional viscocapillary – Saint Gobain
- Rheological models account for shear-thinning behavior of catalyst inks and ionomer dispersion
- Predict operability limits of vacuum pressure versus line speed
- Ongoing work: Stability analysis of multilayer flow



Accomplishments and Progress:

Experimental Validation of Slot-Die Coating Window Predictions

- R2R slot-die coating of PEMFC cathode ink
- The defect-free coating window from two models accurately predicts the experimental coating window for inks with **low concentrations**.
- Predictions decreased ink and substrate waste by eliminating the need to hunt for a set of coating parameters that result in a stable, defect-free coating.
- Continuing to develop model to better predict high viscosity inks



Accomplishments and Progress: Slide Flow Studies

Top Layer 25% Water/75% IPA

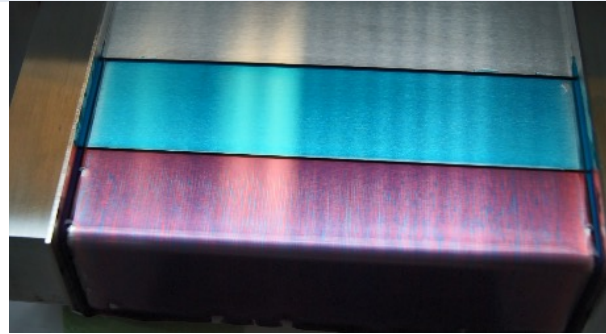
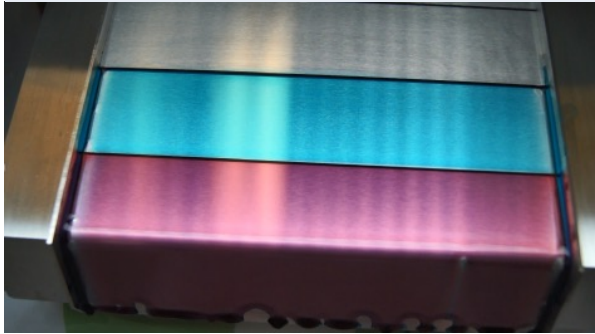
75% Water/25% IPA

75% Water/25% IPA

Bottom Layer 75% Water/25% IPA

75% Water/25% IPA

25% Water/75% IPA

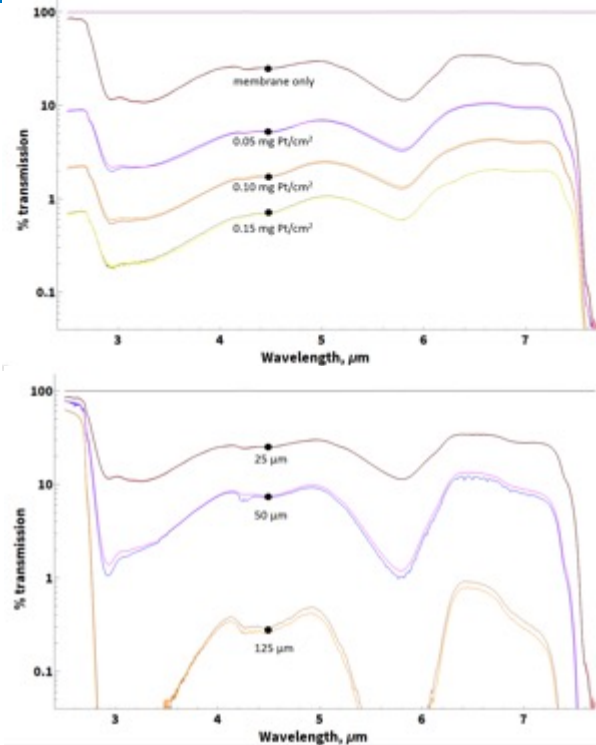


- Conducted slide flow trials with polymer solutions in water/isopropanol to simulate catalyst layers or ionomer dispersion
- Experiments designed to determine proper layering of fluids
- Results show that top layer must have lower surface tension than bottom layer
- These results are being used to guide catalyst ink formulation for multilayer catalyst layer coating trials

Accomplishments and Progress

Continued development of spectrometry-based metrology for multilayer (CCM) structures

- Performed IR spectroscopy of membranes and half-CCMs using benchtop device
 - NRE211, NRE212, N115 membranes
 - NRE211 membranes with 0.05, 0.1, and 0.15 mg Pt/cm² electrode loadings
- Spectra from 3-7 μm very sensitive to membrane thickness and catalyst loading
- Testing of additional membranes and electrode loadings ongoing
- Could lead to in-line method to simultaneously measure thickness and loading



IR spectra showing sensitivity to CCM catalyst loading (top) and membrane thickness (bottom)

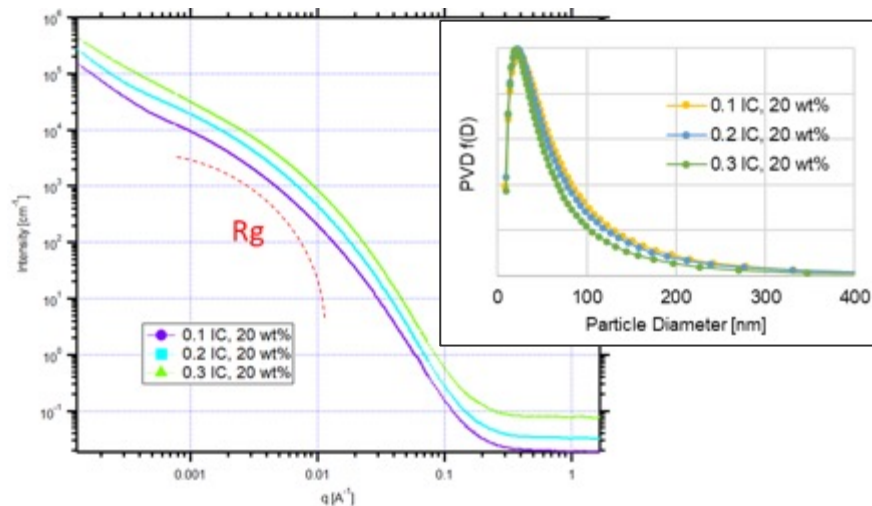
HFTO R2R Activity

Accomplishments and Progress:

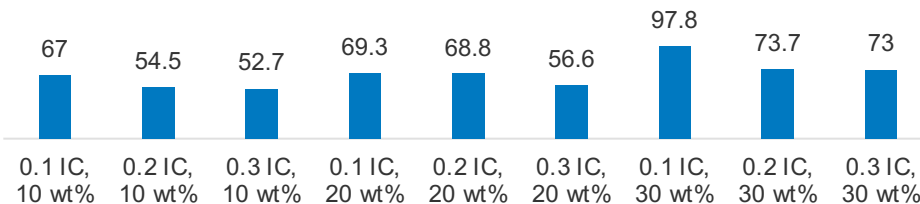
Working with Nel to Determine Impact of Ink Formulation on Catalyst Agglomeration

- Goal: Study relationships between ink formulation, electrode morphology, and performance for an IrOx-based anode
 - ✓ Study impact of I:C, catalyst solids %, and solvent on fabrication properties and performance
 - ✓ Rod coating on decals to send to Nel for in situ testing (phase 2 would include R2R coating/drying)
- Brief overall summary of status
 - ✓ NREL established a matrix of 10 ink compositions, varying ionomer to catalyst ratio, wt% catalyst, and ionomer type. 10 electrodes deposited on PTFE were made by NREL by blade coating
 - ✓ ANL characterized these inks and electrodes by X-ray scattering and Nano-CT
 - ✓ Catalyst layers will be sent to Nel for in situ testing in the coming months

USAXS profiles and particle volume distribution for IrOx Electrodes



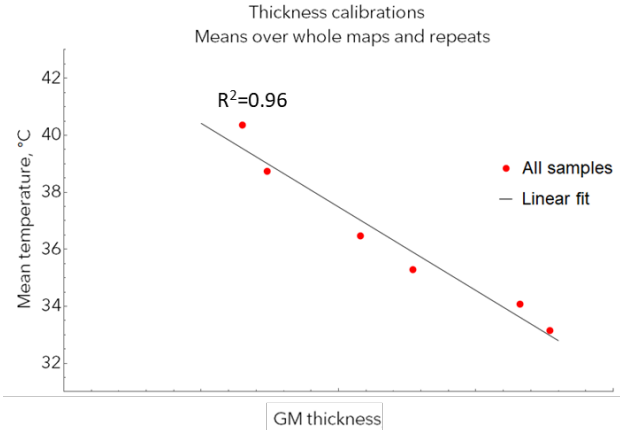
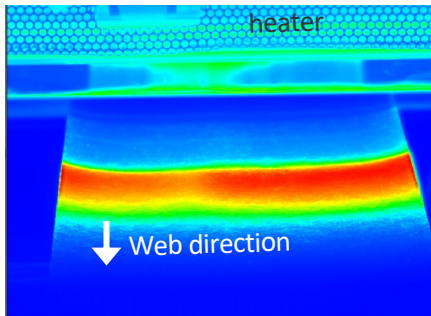
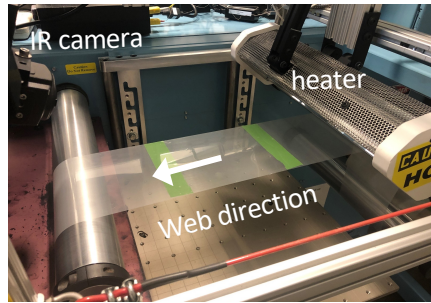
Radius of Gyration of Primary Aggregates [nm]



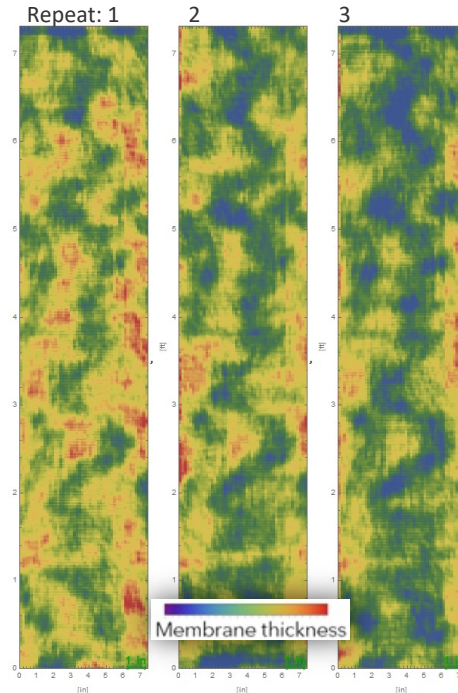
Membrane diagnostics by active thermal scanning for half-MEA roll materials fabricated by General Motors

Objectives: Based on 6 roll specimens provided by GM determine signal sensitivity to membrane thickness for the web-line platform. Evaluate the effect of resolution (spot size) on measurement error

Key results: a) First web-line experiment was performed and analyzed; b) Based on measured temperature, NREL was able to properly order the samples by thickness. The thickness order was then confirmed by GM; c) Calibration curve is of high R^2 ; d) Repeatability values are below 6%.



Experimental setup (left top) and a typical thermography frame (left bottom). Correlation curve between the thermal response and the actual membrane thickness (top).



resolution	repeatability [%]
0.25 in	5.3
0.5 in	5.2
1 in	5.1
2 in	4.9
4 in	4.6
8 in	2.5

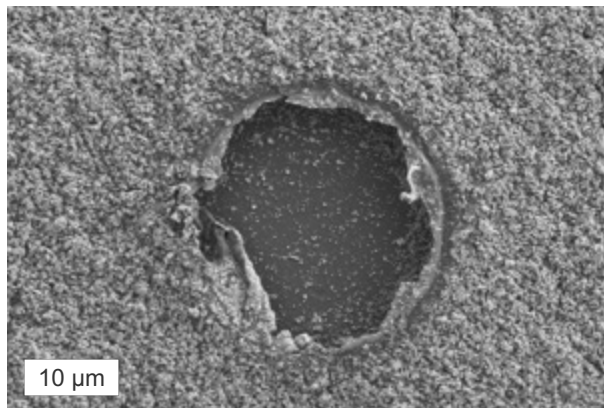
Membrane thickness map for the same specimen from 3 thermal scans (left). Calculated overall repeatability from all 6 specimen rolls as a function of spot size (top).

Accomplishments and Progress: Determining Effect of Time Between Mixing and Coating on Defects

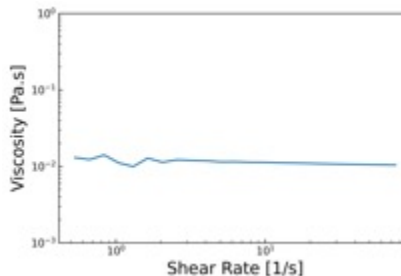
Goal: Determine impacts of coating and drying conditions on defect formation in R2R coated CCMs



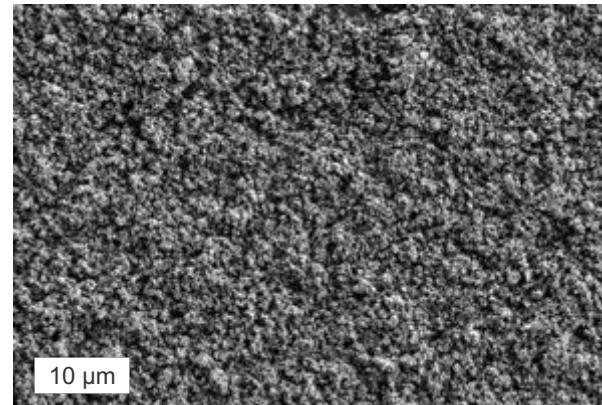
Applying ink immediately after high-shear mixing results in bubbles from the foaming during mixing ending up in the coating, creating hole defects in the film.



Direct membrane coating with a slot die



Low-velocity stirring overnight after high-shear mixing allows the ink to degas and results in a mostly defect-free coating.

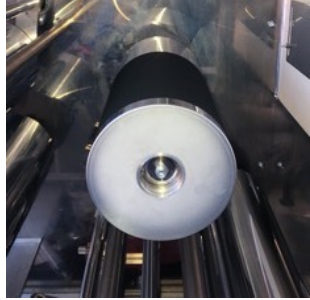


Anode ink is Newtonian.
Ink will still be coatable with higher viscosity from higher solids loading.

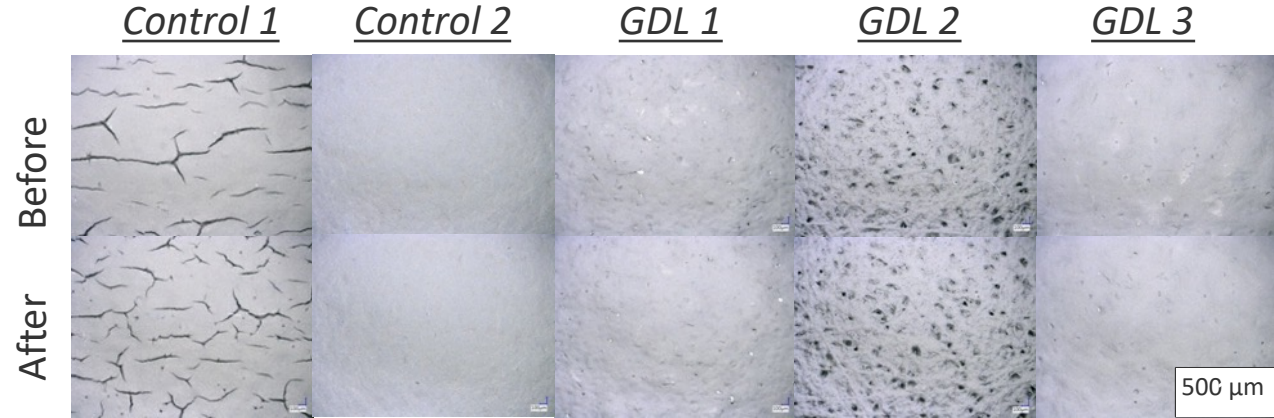
Accomplishments and Progress: Assessment of GDL Materials

Goal: Assessment of the suitability of US-manufactured flexible gas diffusion media

Wrap Testing



Sample sheets wrapped around a 6" roller in R2R coater to determine impact on MPL cracking

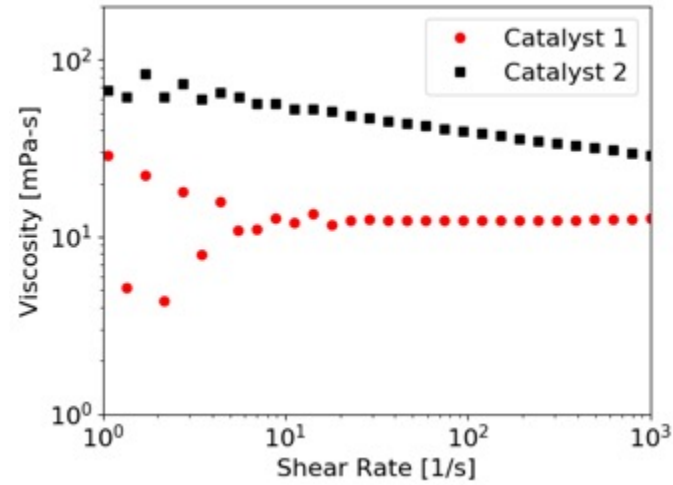
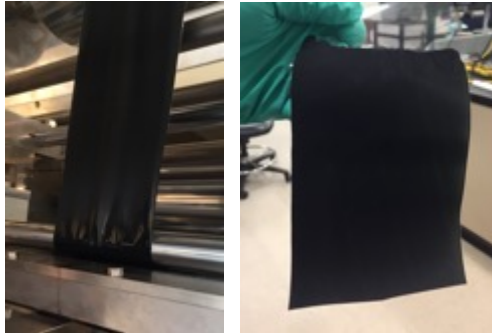
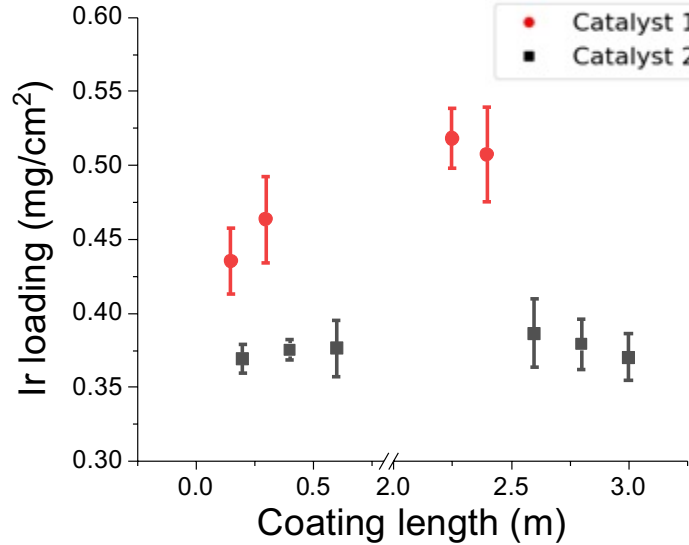


- Variety of MPL structures/ available
- MPL of a control sample cracked during wrapping
- US-manufactured MPLs did not crack under wrapping around 6" roller
- Future work: wrap testing with smaller bend radius, electron microscopy, in-line defect detection

NeI CRADA

**“Roll to Roll Manufacturing of
Electrolysis Electrodes for Low
Cost Hydrogen Production”**

Accomplishments and Progress: Development of Direct Coating Methodology



- Developed methodology for direct gravure coating on to membrane
- Higher viscosity leads to lower ink transfer/Ir loading
- Half CCMs sent to Nel for in situ testing
- Assisting Nel with scaling up process to pilot scale

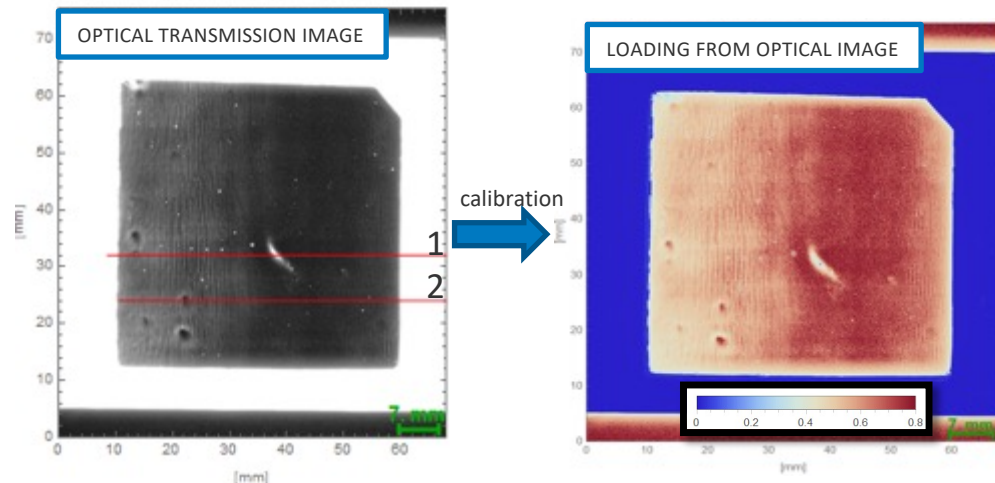
Accomplishments and Progress: Development of Optical Transmission Loading Measurement Methodology

Objective: Evaluate the optical transmission as method for the high-resolution imaging of the loading of Ir-oxide coatings.

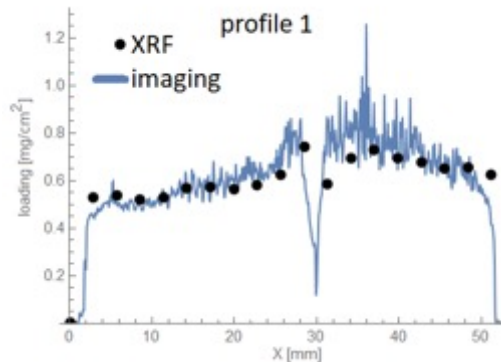
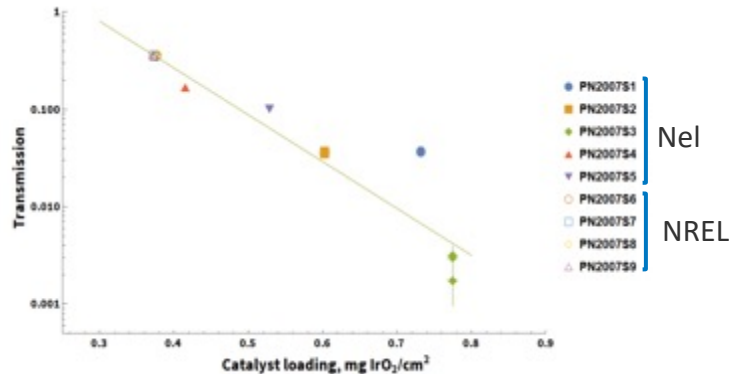
Methods: 1) **sample-scale correlation analysis** - Study based on 9 anode specimens fabricate by Nel and NREL. Average values were analyzed here. 2) **local correlation analysis based on two anode specimens**. Total of 80 local XRF loading measurements were obtained along two lines on each sample. XRF measurement spots were then aligned spatially with the corresponding transmission images to obtain the transmission-to-loading correlation parameters. Finally, the high-resolution loading maps of the two anodes were obtained.

Results: Good correlation exists between Ir oxide loading and transmission values. This method has a potential to become a valuable loading characterization technique for in-line monitoring of fabrication processes of PEMWE anodes.

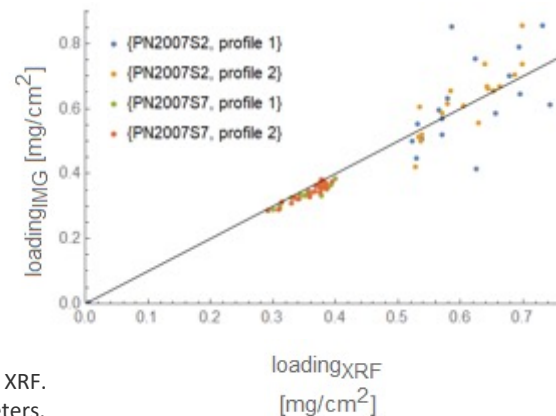
LOCAL CORRELATION ANALYSIS



SAMPLE-SCALE CORRELATION ANALYSIS



Loading at 40 spots per sample were obtained using XRF. This allowed to obtain the image calibration parameters.



Accomplishments and Progress:

Response to Previous Year* Reviewers' Comments**

“manufacturing...should normally be left up to industry”

The combination of unique equipment and techniques of this collaboration does not exist in industry, which enables us to develop a more fundamental understanding of topics than industry.

“investment was made with minimal industrial involvement”

“project needs to add an automotive original equipment manufacturer (OEM) to help guide priorities”

We have worked to engage industry more this year with the HFTO-funds that are being leveraged to specifically address research topics defined by industry input.

“Understanding the structure, property, and performance issues as a function of the drying rate and solvents used would be a better use of effort”

The project has topic areas focused on drying and ink formulation, which include experimental and theoretical work.

* Repeated FY2019 responses, per HFTO guidance.

** Responses to comments on main AMO fuel cell core lab project. HFTO R2R Activity was not previously reviewed.

Collaboration and Coordination

Laboratory	Contributors	Tasks
ORNL	Claus Daniel, David Wood, Erin Creel, Georgios Polyzos, Jaswinder Sharma, Kelsey Grady, Yarom Polsky	Colloids, rheology studies, multilayer slot coating studies, electrode characterization, ink characterization
NREL	Mike Ulsh, Scott Mauger, Janghoon Park, Min Wang, Jason Pfielsticker, Sunil Khandavalli, Sadia Kabir, Peter Rupnowski, Brian Green, Ying-Ying Chen	Colloids, rheology studies, multilayer slide coating studies, MEA fabrication and testing, electrode characterization, QC development, electrospinning
ANL	Greg Krumdick, Debbie Myers, Jae Hyung Park, Firat Cetinbas, Yuepeng Zhang, Erik Dahl	Ink characterization, electrode characterization, electrospinning
LBNL	Vince Battaglia, Kenny Higa, Zhi Huang, Bei Fan, Buyi Zhang, Ravi Prasher	Particle-scale ink and consolidation modeling
SNL	Randy Schunk, Kristianto Tjiptowidjojo, Nelson Bell	Flow and consolidation modeling

Also collaborating with Colorado School of Mines and University of Massachusetts, Amherst for x-ray photoelectron spectroscopy and extensional rheology, respectively

Proposed Future Work

AMO Core Lab Activity

- Overall R2R multi-lab collaboration will need to renew or repropose to extend the activity past FY2021
- Assuming continuation of activity
 - Continue to leverage application agnostic process development work toward fuel cell and electrolysis MEA challenges
 - Study novel structures, especially to address durability
 - Coordinate with HFTO Consortia
- Complete Nel CRADA effort

HFTO R2R Activity

- Complete current key deliverable projects and transfer learnings to industry partners
- Get feedback from industry on project concept and continuation
- Pursue continued funding with HFTO

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

Summary

AMO Core Lab Activity

- Continued development of coating models for catalyst layers and MEAs
- Quantifying applicability of mixing methods for mass production
- Developing full-area QC inspection techniques
- Determined effects of carrier polymer concentration on spinnability and electrochemical performance

HFTO R2R Activity

- Characterizing impacts of web handling on GDL materials
- Developing methodologies for full-area measurement of membrane thickness
- Determining impacts of process conditions in R2R-coated catalyst layers
- Exploring impacts of catalyst ink formulation on catalyst layer structure and performance



Thank You

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Technical Backup and Additional Information

Technology Transfer Activities

AMO Core Lab Activity

- Nel CRADA project
- Ad hoc collaboration with industry partners and equipment providers
- Formation of new Industry Engagement Panel, including fuel cell and electrolysis companies
 - Ensure industry relevance
 - Obtain feedback on current and planning technical directions and work efforts
- Publications and presentations

HFTO R2R Activity

- Direct transfer of project results to four industry partners

Progress toward DOE Targets or Milestones

- These activities have the goal of assisting industry in accelerating the scale-up of MEA and stack materials, toward program cost, volume and timeline targets
- The work efforts will be focused on addressing these needs for high priority applications such as water electrolyzers and fuel cells for heavy-duty applications

Publications and Presentations

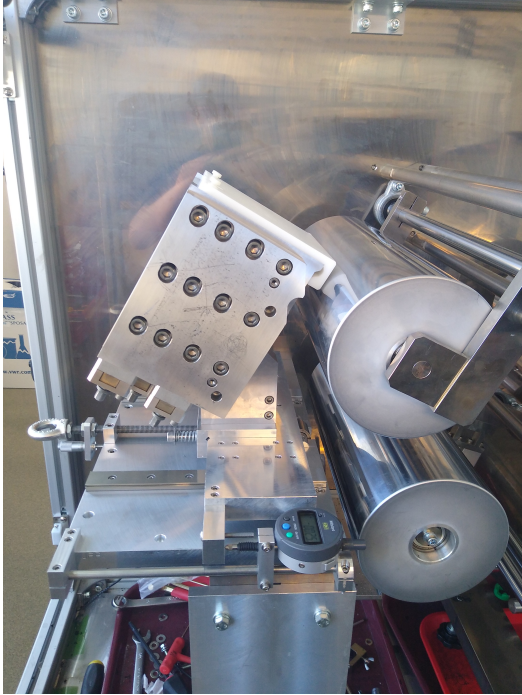
Publications

- Kabir, van Cleve, Khandavalli, Medina, Pylypenko, Mauger, Ulsh, and Neyerlin. "Toward Optimizing Electrospun Nanofiber Fuel Cell Catalyst Layers: Microstructure and Pt Accessibility." *ACS Applied Energy Materials*, 2021. <https://doi.org/10.1021/acsaem.0c03073>.
- Khandavalli, Sharma-Nene, Kabir, Sur, Rothstein, Neyerlin, Mauger, Ulsh. "Towards Optimizing Electrospun Nanofiber Fuel Cell Catalyst Layers: Polymer-Particle Interactions and Spinnability." *ACS Applied Polymer Materials*, 2021. doi.org/10.1021/acsapm.0c01354
- Tjiptowidjojo, Park, Mauger, Ulsh, Schunk, "Process Model for Multilayer Slide Coating of Polymer Electrolyte Membrane Fuel Cells." accepted in *J. Coating Technology Research*
- Mauger, Wang, Cetinbas, Dzara, Park, Myers, Ahluwalia, Pylypenko, Hu, Litster, Neyerlin, Ulsh. "Development of High-Performance Roll-to-Roll-Coated Gas-Diffusion-Electrode-Based Fuel Cells." submitted to *Journal of Power Sources*

Presentations

- Mauger, Wang, Cetinbas, Dzara, Park, Van Cleve, Hu, Litster, Ahluwalia, Pylypenko, Myers, Neyerlin, Ulsh. "Influence of Ink Formulation and Drying Conditions on Ionomer Distribution in High-Performance Roll-to-Roll-Coated Gas-Diffusion Electrodes" *2020 Prime Meeting*, MA2020-02 2218
- Park, Pfeilsticker, Tjiptowidjojo, Schunk, Mauger, Ulsh. "An Experimental Investigation of Multilayer Flow in a Slide Die Coating Process" *20th International Coating Science and Technology Symposium*. 2020
- Creel, Wang, Sharma, Polizos, Grady, Tjiptowidjojo, Schunk, Ulsh, Mauger, Wood. "Exploring Roll-to-Roll Slot-Die Coating Parameters for Concentrated Electrocatalyst Layer Inks for PEM Fuel Cells." *20th Annual ISCST Symposium*.
- Creel, Wang, Sharma, Polizos, Grady, Tjiptowidjojo, Schunk, Ulsh, Mauger, Wood. "Roll-to-Roll Slot-Die Coating of Concentrated Electrocatalyst Layer Inks for PEM Fuel Cells," ORNL's Center for Nanophase Materials Sciences Annual User Meeting.
- Creel, Wang, Sharma, Polizos, Grady, Tjiptowidjojo, Schunk, Ulsh, Mauger, Wood. "Roll-to-Roll Slot-Die Coating of Concentrated Electrocatalyst Layer Slurries for PEM Fuel Cells." Fall 2020 ACS Meeting.
- Tjiptowidjojo, Park, Mauger, Ulsh, Schunk. "Multilayer Slide Coating Model for Manufacturing of PEMFC." *20th International Coating Science and Technology Symposium*. 2020

Accomplishments and Progress: Slide Die Installed in R2R Coater

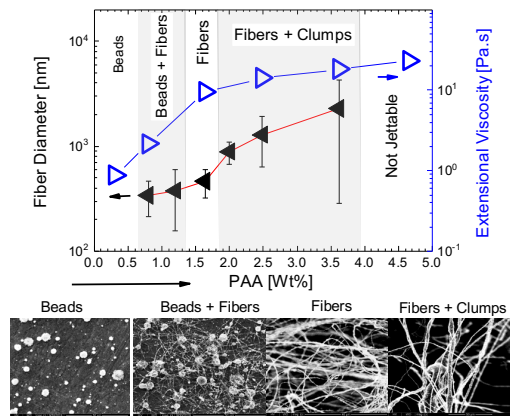
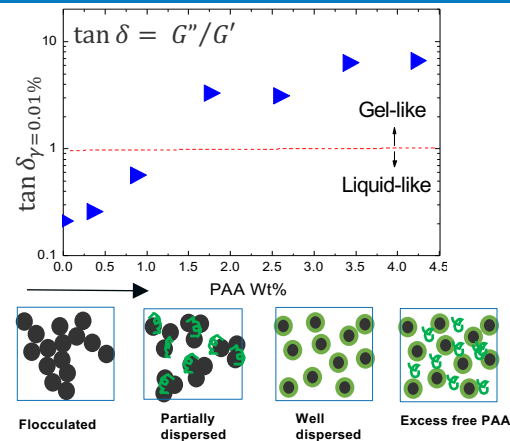


- Multilayer slide die mounted in R2R coater
- Installation has been delayed due to late delivery, need for reworking, hoist being built to lift die (die weighs ~100 lbs), and modifications to coating machine to mount die
- Coating work will begin on two-layer coatings of fuel cell catalyst layers

Accomplishments and Progress:

Effect of carrier polymer concentration on electrospinnability and nanofiber electrode fabrication

- Elucidated carrier polymer (polyacrylic acid, PAA) interaction with catalyst, and the effect of PAA concentration on ink structure, processing, and performance.
- PAA stabilizes the catalyst agglomerates but above a critical concentration becomes excess free polymer that simply increased inks' viscosity.
- Increasing PAA wt% enhanced inks' extensional viscosities that promoted fiber formation, but concurrent increase in shear viscosities led to defective fibers at large PAA wt%
- Maximum electrochemical performance was found at optimal PAA wt% where the agglomerates are minimal and there is low concentration of excess PAA in the ink/bulk.
- On going studies exploring impacts of ionomer chemistry and solvent systems on spinnability. Also investigating carrier-polymer-free solutions.



Accomplishments and Progress:

Determining Effectiveness of Scalable Mixing Methods

- Many studies prepare catalyst ink using ultrasonic mixing, which is not scalable
- There is a need to determine effectiveness of scalable mixing methods and their impact of performance and durability
- Performing image analysis to characterize level of break up of large catalyst agglomerates

Tube Rotor-Stator (High Shear)



Rotor-Stator (High Shear)



Ball Mill

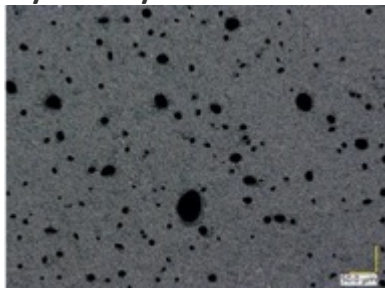


Ink (g)	15	110	30
Speed	6,000 rpm	6,000 – 20,000 rpm	20 speed units
Time	1 – 30 min	1 – 30 min	1 – 24 h

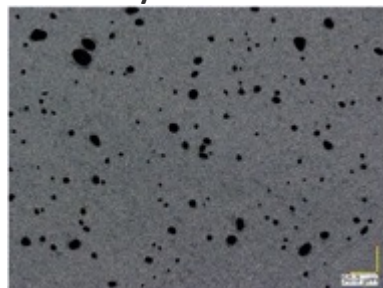
Catalyst Layer Particle Size Analysis



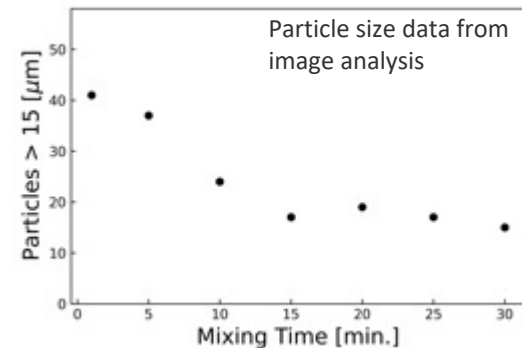
5 min



15 min

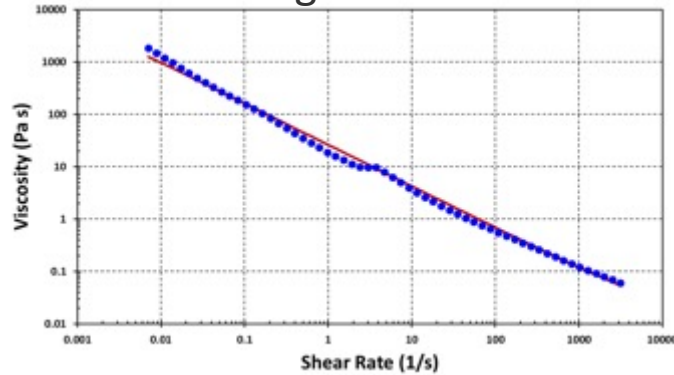


30 min



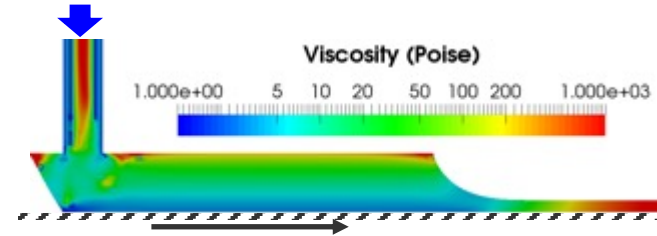
Accomplishments and Progress: Continuing Development of Pt/C in Slot Coating Model

Rheological Model

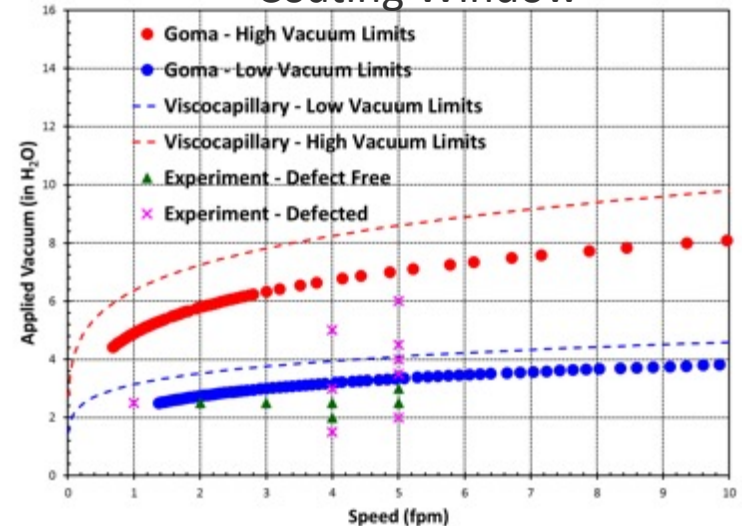


- Coating flow modeling approaches:
 - Two-dimensional finite element method – Goma 6.0 (Sandia/UNM)
 - One-dimensional viscocapillary – Saint Gobain
- Rheological models account for shear-thinning behavior of Pt/C catalyst ink
- Flow models predict operability limits of vacuum pressure versus line speed and compare with experimental observation
- Ongoing work: Resolve discrepancy between predictions and observations

Flow Model



Coating Window

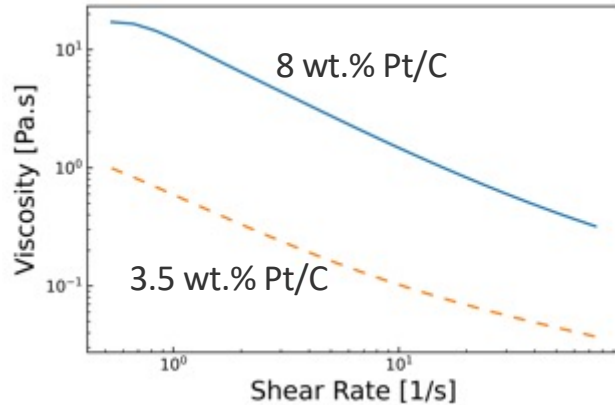


Accomplishments and Progress:

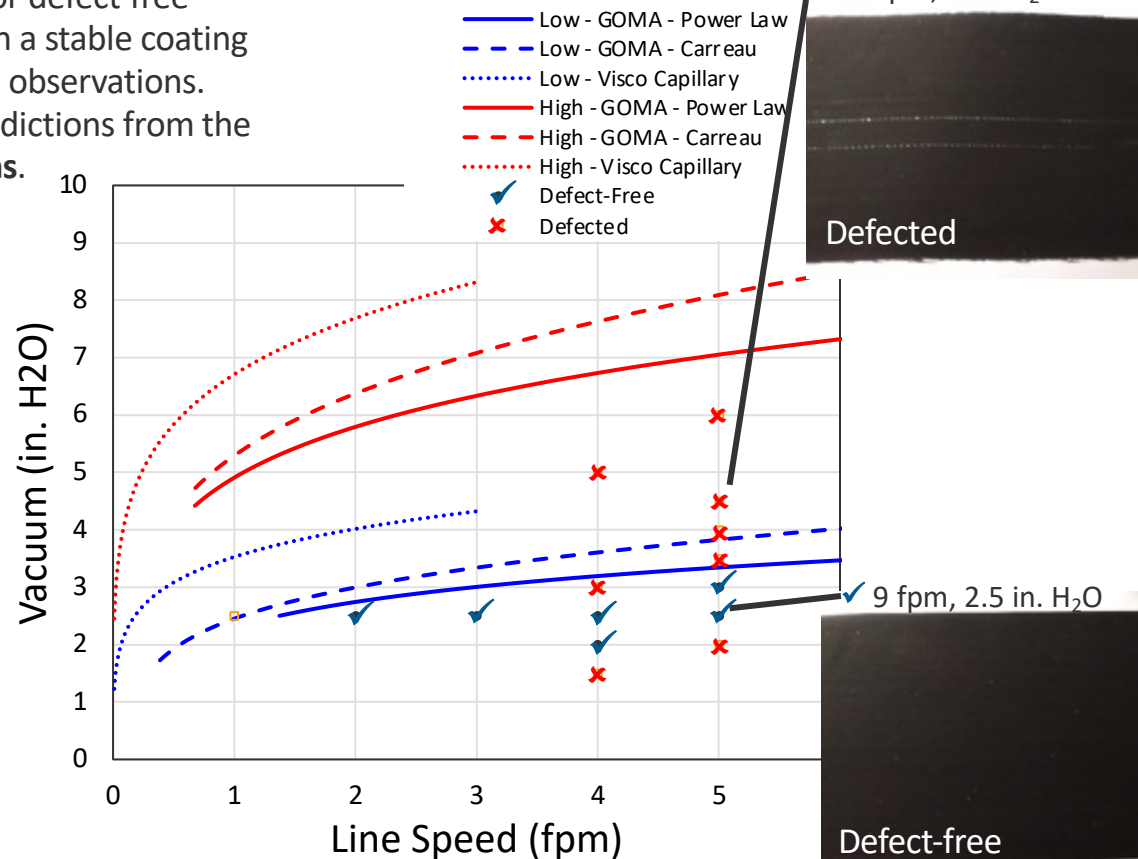
Slot-Die Coating Window Predictions Less Accurate at High Concentrations

8 wt.% Pt/HSC PEMFC cathode GDE, Wet thickness = 30 μm , Gap = 150 μm

- Despite overprediction of vacuum needed for defect-free coating, models helped researchers establish a stable coating quickly due to predictions being close to the observations.
- Working to improve the coating window predictions from the two models for inks with **high concentrations**.

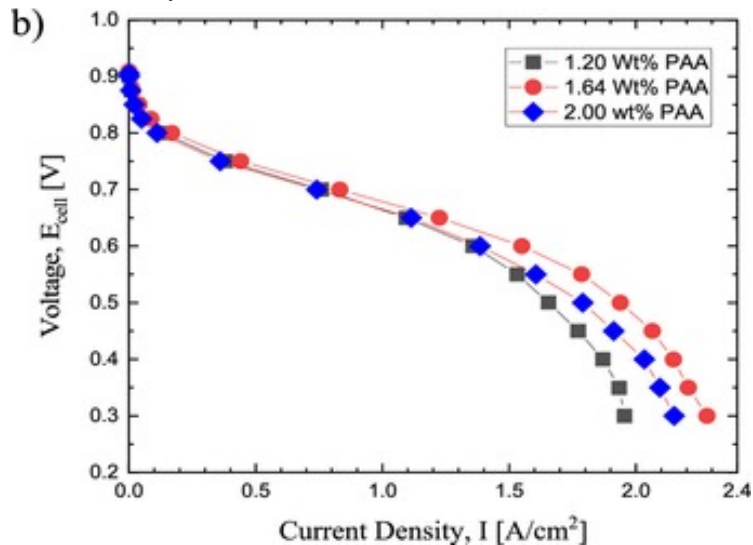


- Improving fit with measurement of ink contact angle on substrate and ink surface tension
- High ink concentration save drying energy budget and CapEx on oven length.

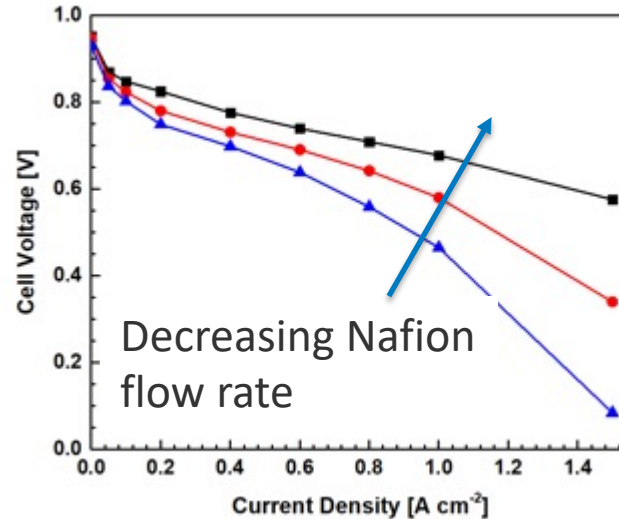


Accomplishments and Progress: Relating Processing to Performance

Impact of Carrier Polymer Concentration in Electrospun Nanofibers



Impact of Nafion flow rate in dual-slot-coated catalyst layers

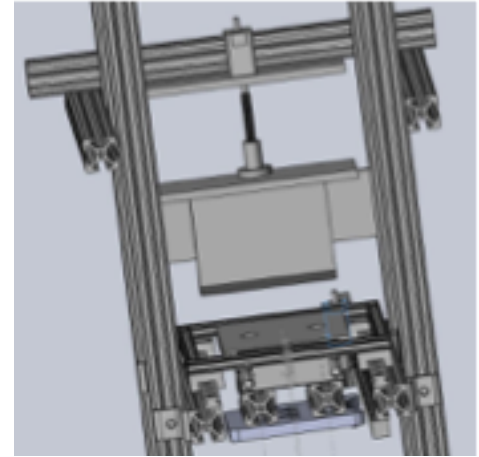
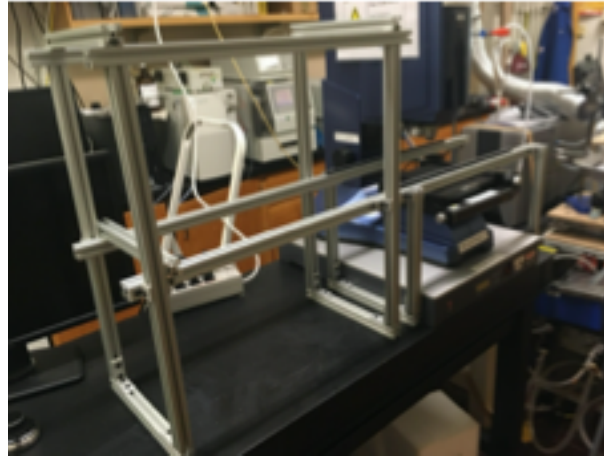


- Conducting in situ electrochemical testing of to understand impacts of processing conditions on performance
- Results used to refine process conditions and improve performance

Accomplishments and Progress:

Drying dynamics experimental setup

- Computer-controlled
 - Coating
 - Heating
 - Sample delivery
- Observation
 - Confocal microscopy
 - X-ray radiography
 - More to come...
- Portable, modular units intended to simulate industrial processing conditions at instrument-compatible scales
- Collects data about component-level behavior during drying, needed for development of coating drying models
- Effort funded by AMO and VTO



Tech Transfer - FCTO-funded R2R Activity

- \$850K total funding to the five labs to assist industry with MEA scale-up
- Initial workplan developed by labs to maximize leveraging of AMO-funded R2R process work
- Workplan revised per the inputs for four industry participants (GM, Plug Power, Nel/Proton, Giner)
 - Ensured effort will address specific relevant scale-up needs for PEM FC & EC MEAs
 - Prioritized work and developed key deliverables for first year of effort
 - Will provide ongoing guidance and feedback as work progresses
- Revised workplan approved by industry participants and FCTO
- Major task areas:
 - Model development (particle-level ink model, coating flow model, consolidation model)
 - Ink and colloid studies and characterization
 - Coating studies and electrode characterization
 - Real-time quality method development (coatings and inks)
 - In situ testing to validate the impact of ink and process parameter studies
- Lab roles: same as for overall AMO effort