Advanced Electrode Manufacturing to Enable Low Cost PEM Electrolysis

Project ID: TA036

PI: Christopher Capuano

Overview

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Advanced Electrode Manufacturing to Enable Low Cost PEM Electrolysis

PI: Christopher Capuano,
Nel Hydrogen

Barriers

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

Timeline and Budget

- Project Start Date: July 1, 2019
- Project End Date: June 30, 2021
- Total Project Budget: $1.9M
  - Total Recipient Share: $500K
  - Total Federal Share: $1.4M
  - Total DOE Funds Spent*: $404,451

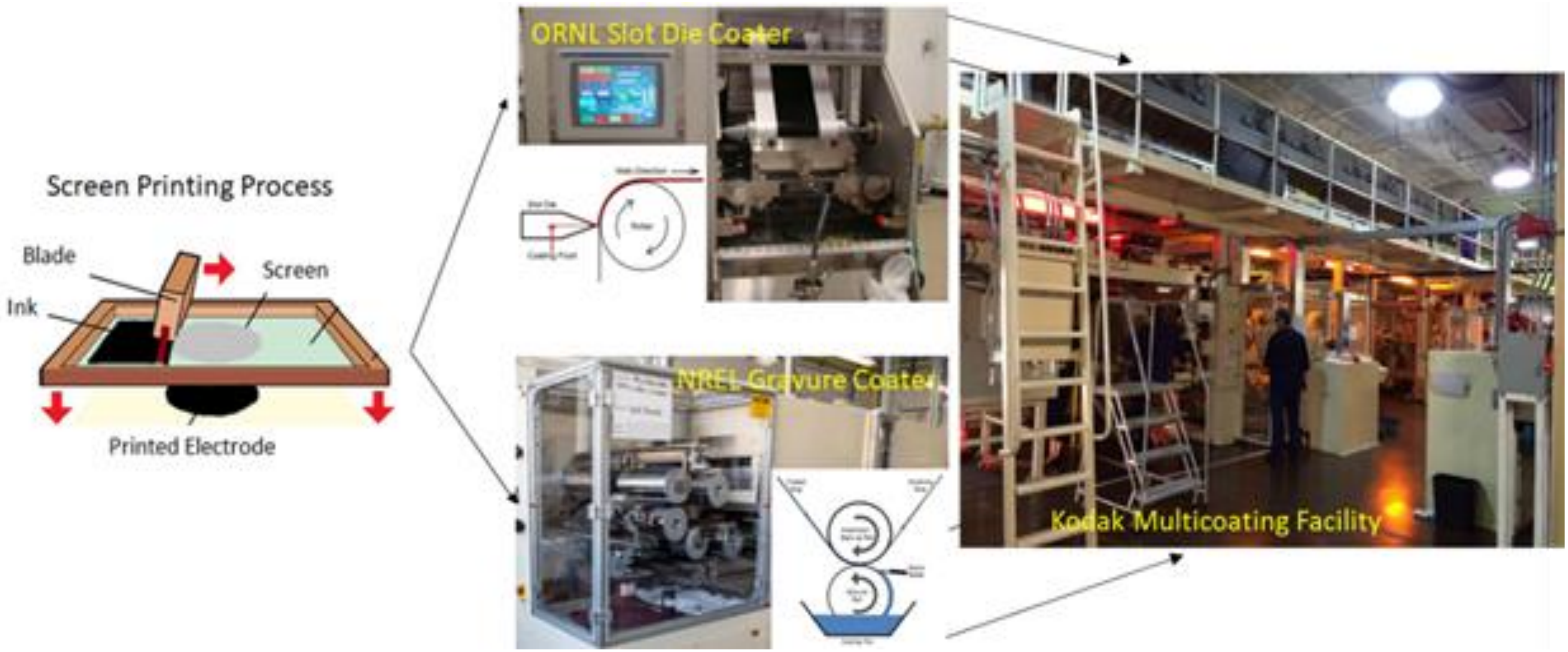
* As of 04/30/2020

Partners

NREL: Mike Ulsh
ORNL: David Wood
GM: Craig Gittleman
Kodak: Dan O'Corr

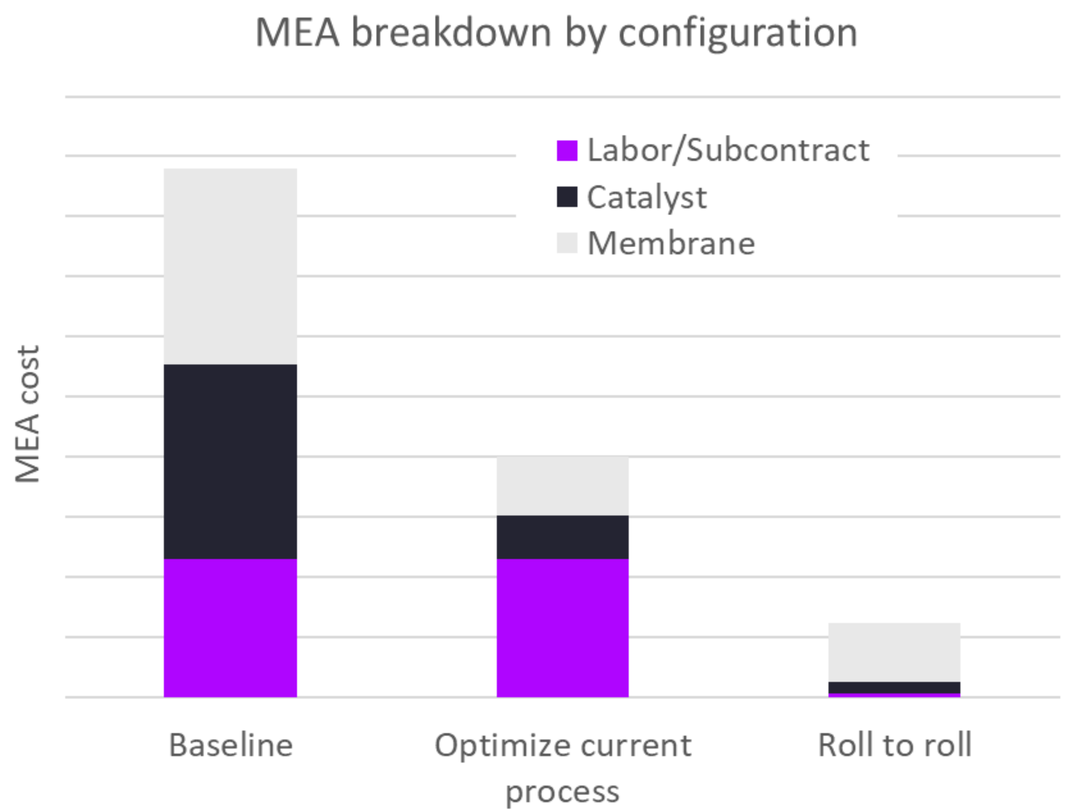
Relevance

- The overall outcome of this project will be development of coating inks and R2R coating process parameters specific to ultra-low loaded catalysts.



Relevance – Cost

- Roll to roll provides significantly larger potential for cost reduction than process optimization alone



Approach: Summary

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Goals

Establish R2R manufacturing of membrane electrode assembly components for PEM water electrolyzer through:

1. Improving the manufacturing speed rate by 100 times
2. Reducing the total PGM catalyst loading by 75%
3. Reducing the labor cost by 10 times
4. Retaining the durability similar to baseline

The success of this project depends on knowledge transfer of the learnings between the partner institutions and integration of the developments

Approach: Innovations

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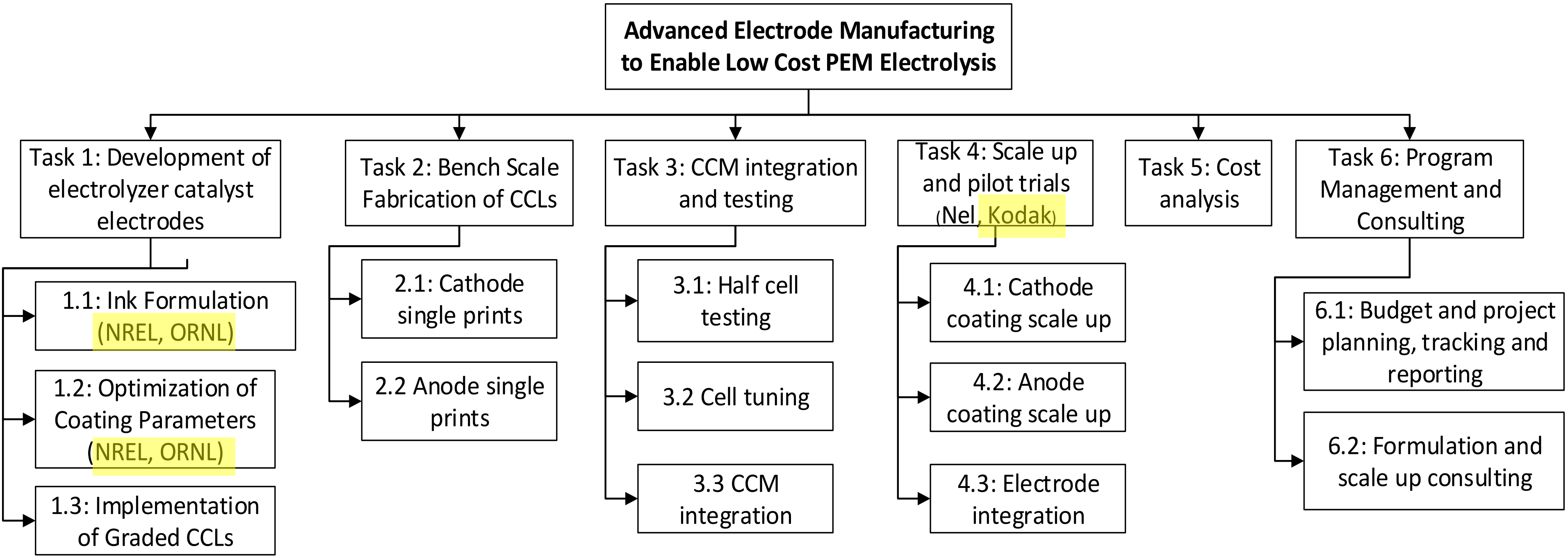
Specification	Proton State of the Art	Proton Lab Demonstrated	Specific Project Target
CCM manufacturing	S2S	S2S	R2R
PGM catalyst loading, mg/cm^2	3	0.5	0.7
Membrane cost, % of baseline cell	40%	17%*	23%
Labor cost, % of baseline cell	27%	N/A*	2%
Total CCM cost, $ /kW	$250/kW	$70/kW*	$90/kW
Durability, mV/1000 hr	<5	<5	<5
Manufacturing Readiness Level (MRL)	9	2	5

S2S = Sheet-to-Sheet
R2R = Roll-to Roll
*Estimated at high volume production

Approach: Task Outline



Tasks led by Proton/Nel unless noted/highlighted

Accomplishments: Budget Period 1 Milestones

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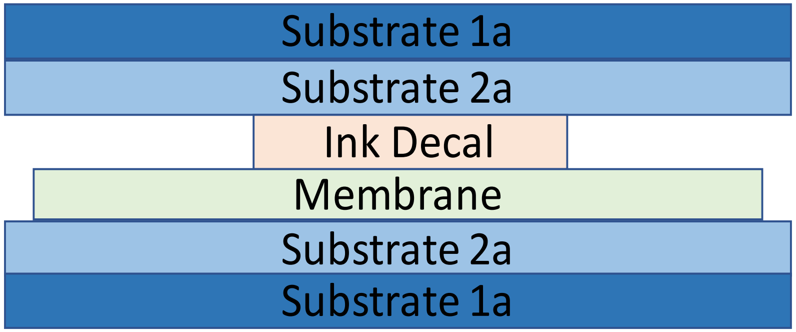
Milestone #	Project Milestones	Type	Milestone Completion Date (Project Quarter)		
			Planned	Actual	Percent Complete
M1.1.1	Develop ink formulation for slot die and gravure printing with no visible voids and good adhesion.	Milestone	9/30/19	9/30/2019	100%
M2.2.1	Identify key coating parameters and optimize small-scale trial runs	Milestone	12/31/19	12/31/2019	100%
M1.2.1	Determine feasibility of reaching catalyst loading targets through demonstration of +/- 10%	Milestone	3/31/20	3/31/2020	100%
G/NG 3.1	Validate target total PGM loadings of <0.7 mg/cm^2 & benchmark performance	Go/No-Go	6/30/20	4/30/2020	100%

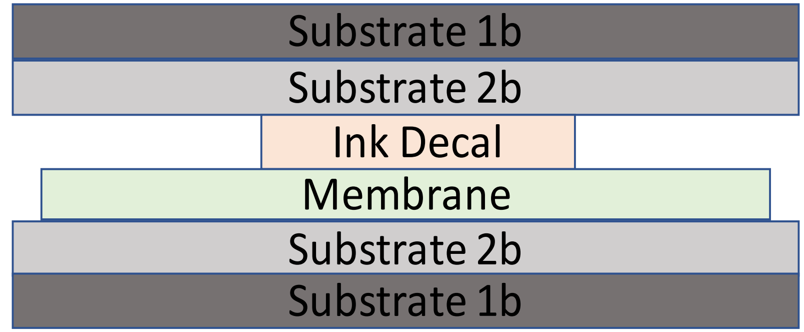
OAK RIDGE National Laboratory NREL NATIONAL RENEWABLE ENERGY LABORATORY GM KODAK

Accomplishments to Date - Lamination

(a)



(b)

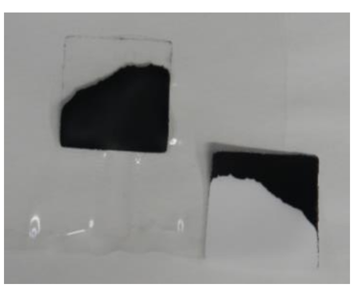


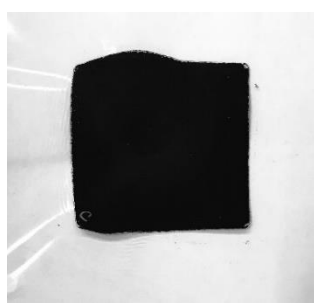
(a) Previous press setup with the decal and membrane positioned between baseline substrate

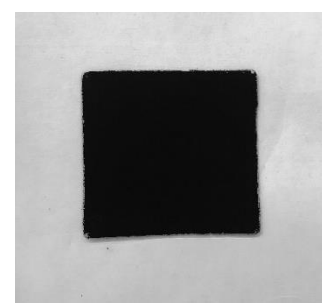
- Short press time (<5 minutes) but required high pressure for transfer
- Temperature adjusted with I/C ratio
- Unable to transfer with lowest I/C ratios even at highest desired temperature
  - All conditions with this setup led to no transfer, partial transfer, or distorted CCM

(b) Current press setup with the decal and membrane positioned between alternate press substrate

- 8x lower pressure, similar temp, longer time
  - Improved heat and load distribution due to softer backing material
  - Less thermal and load stress
  - Transferred decals with no distortion



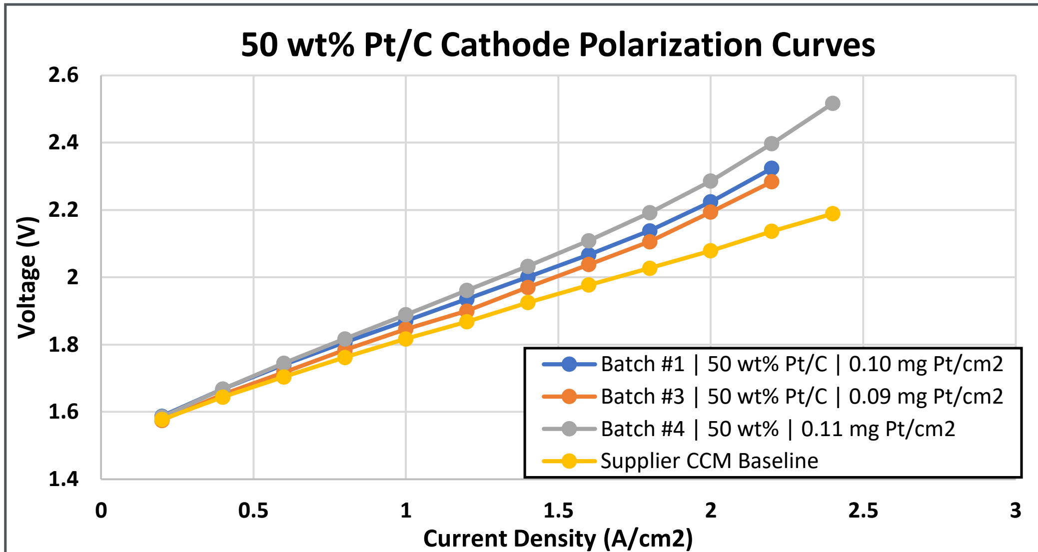


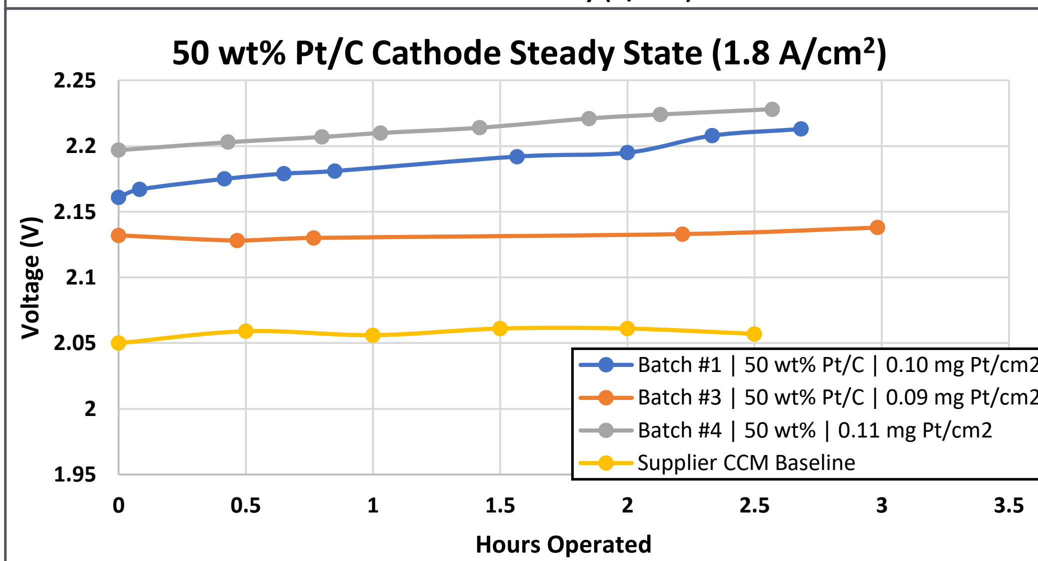


Accomplishments to Date – HER Catalysts (Platinum/Carbon 50 wt%)

- HER catalysts tested in 25 cm^2 platform, at 50°C and ambient pressure
  - Batch #1: Highest I/C
  - Batch #3: Lowest I/C
  - Batch #4: Medium I/C
- Successful in achieving consistent loading with change in ink composition
- No clear trend with increase in I/C ratio on performance
- Batch #3 achieved best performance at 1/10th loading of baseline

HER ink composition established with successful achievement of 0.1 mg/cm^2

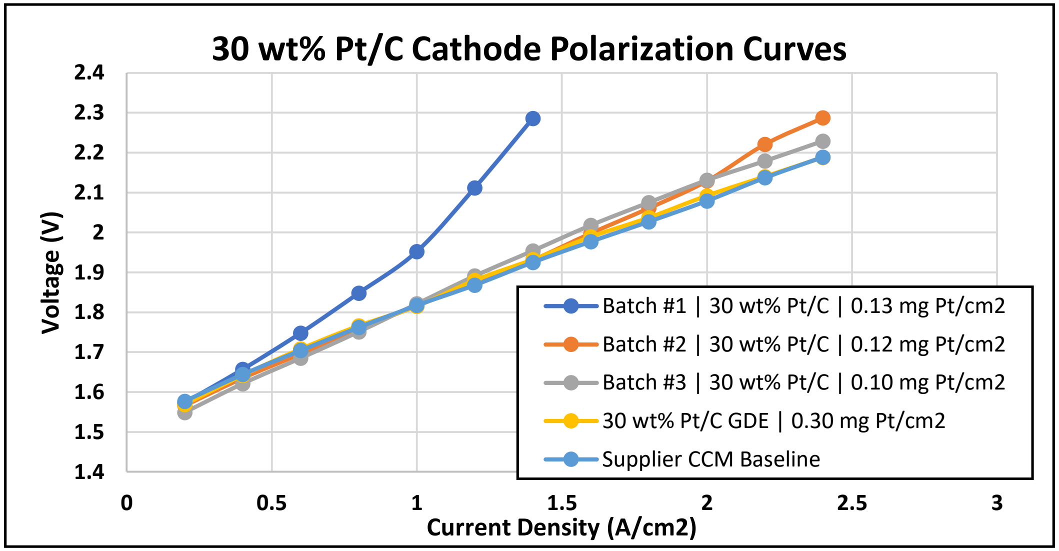


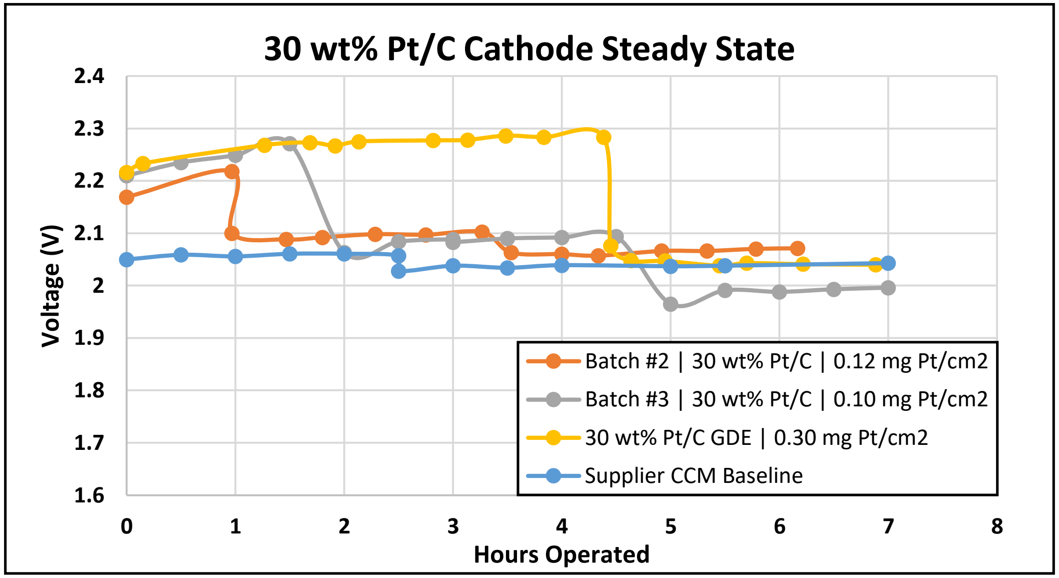


Accomplishments to Date – HER Catalysts (Platinum/Carbon 30 wt%)

- HER catalysts tested in 25 cm^2 platform, at 50°C and ambient pressure
  - Batch #1: Highest I/C
  - Batch #3: Lowest I/C
  - Batch #4: Medium I/C
- Successful in achieving consistent loading with change in ink composition
- No clear trend with increase in I/C ratio on performance
- Batch #3 achieved best performance
- Experimental batches more sensitive to higher current densities than baseline

HER catalyst down-selected with performance exceeding baseline at target loadings of 0.1 mg/cm^2

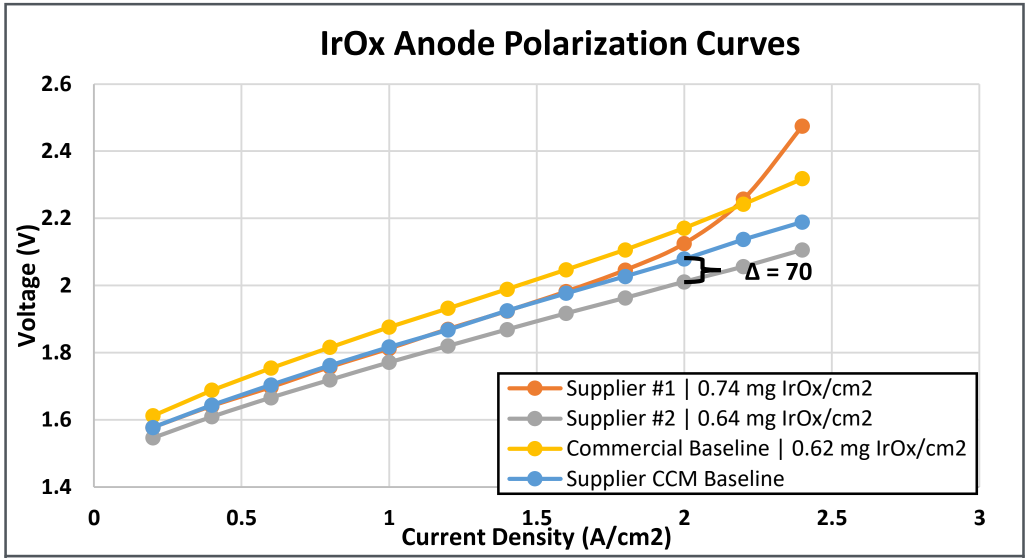


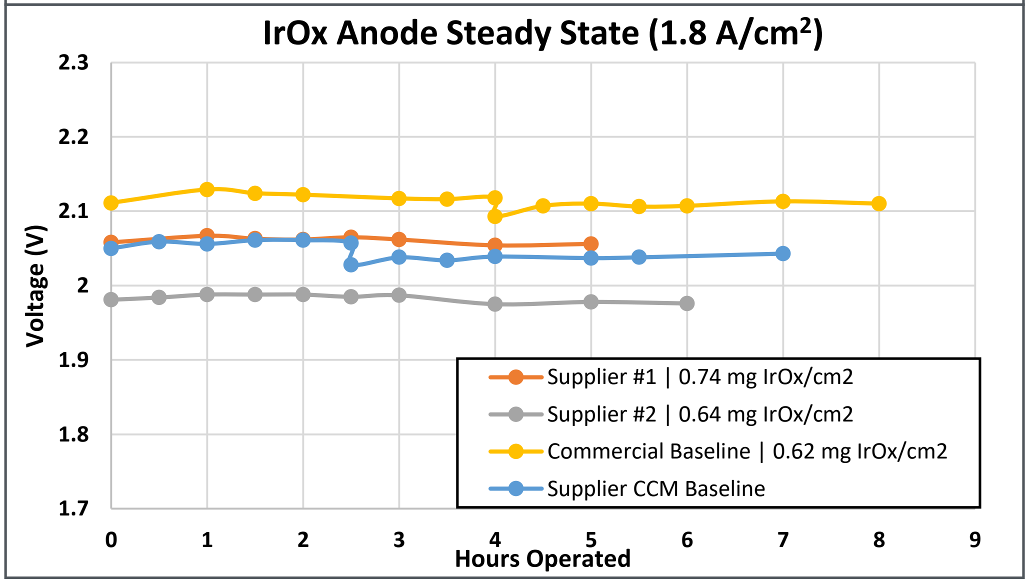


Accomplishments to Date – OER Catalysts

- OER catalysts tested in 25 cm^2 platform, at 50^{o}C and ambient pressure
- Both Supplier #1 and #2 performed better than the commercially available baseline
- Supplier #1 and Supplier #2 remained stable for duration of steady state
- Supplier #2 showed approximately 70 mV improvement at 2.0 A/cm^2 and during steady state vs. the Supplier CCM Baseline

OER catalyst down-selected with performance from supplier #2 exceeding baseline at target loadings of 0.6 mg/cm^2





Accomplishments to Date – Recombination Layer

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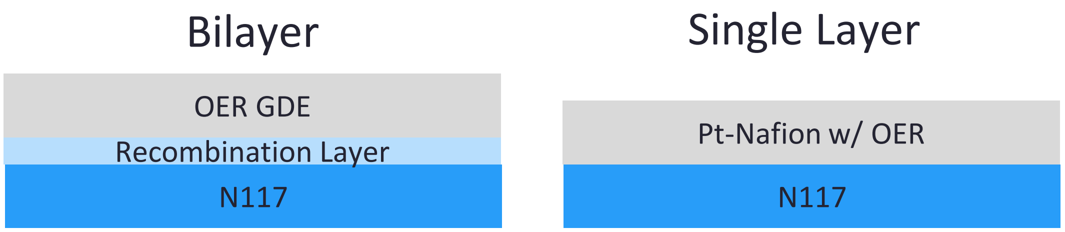
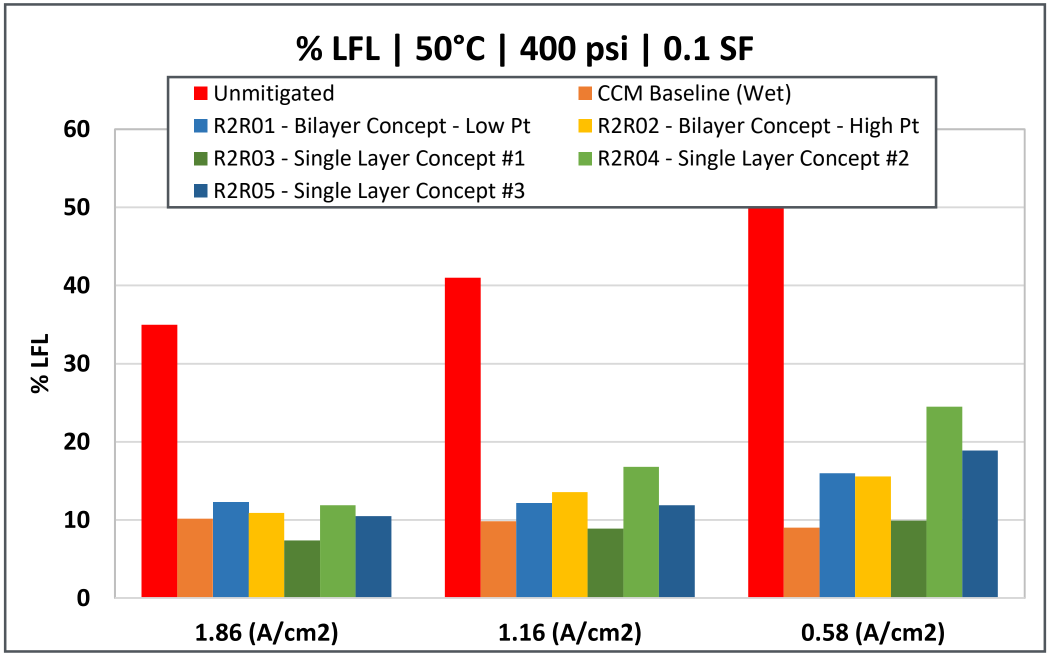
- Tested on a 0.1 SF platform, at 1.8 A/cm^2 50°C and 400 psi
- Both recombination layer concepts were successful in mitigating hydrogen crossover when benchmarked against unmitigated baseline
- Difficulty achieving repeatable performance with single layer concept – more work is necessary to optimize concept





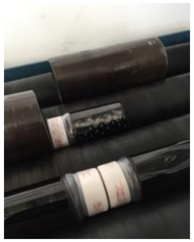
Collaborations:

Partners	Project Roles

National Renewable Energy Lab	• Specialize in advanced manufacturing
	• Focusing on gravure coating within R2R project
	    • Ink mixing method
	    • Ink formulation
	    • Gravure coating parameters

Oak Ridge National Lab	• Specialize in advanced materials • Focus on slot die coating and characterization of catalyst inks within R2R project
General Motors	• Expertise in coating, catalyst, and high volume manufacture
Kodak	• Facilities and equipment for proof of concept at pilot scale

Collaboration:
Effect of Mixing Method



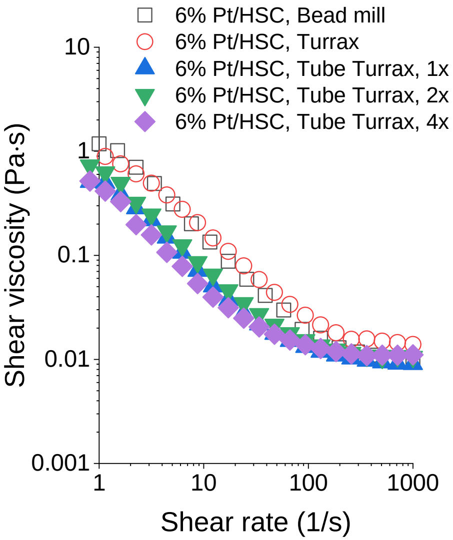
Ball mill
(Small batch, ~6 mL)

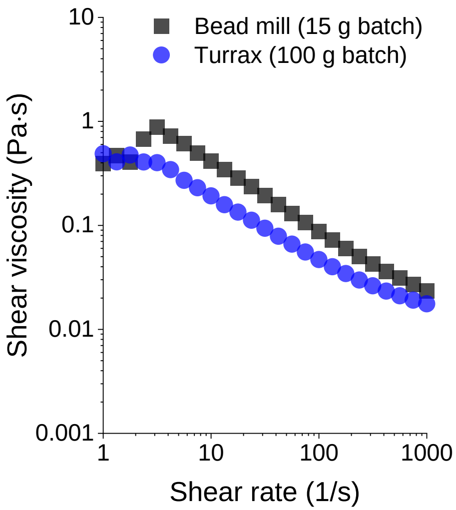
Tube Turrax
(Medium batch, < 15 mL)

Turrax
(Medium - Large batch, 20 - 100s mL)

- **The bead mill** – used in lab scale
  - a need for large amount of beads may be problematic for high volume ink processing
  - difficulty in saving the ink on the beads
- **Turrax mixers** – suitable for high volume dispersion, and Kodak, which will be contributing to the upscaling study, uses a similar mechanism

- Similar rheology can be achieved with changing ink formulation methods
- Confirmed no big changes of the liter-level ink dispersion





Collaboration:
Ink Formulation – Solvent Ratio

NREL
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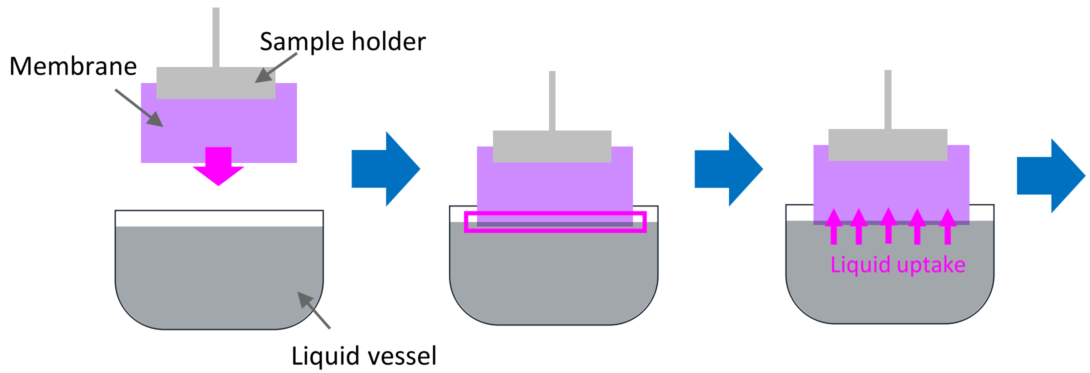
Force Tensiometer – KRÜSS K100

Solvent Uptake - Measurement Procedure

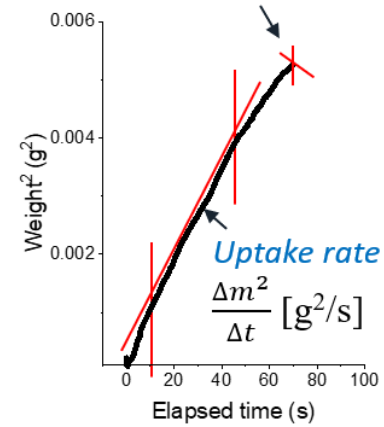


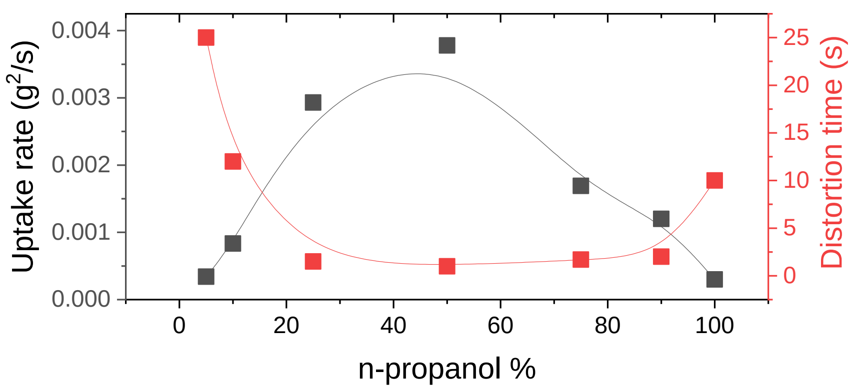
Sample approaches to liquid surface

Auto detection of the specimen

Measurement starts

Distortion point (measurement stops)





- NREL developed novel uptake measurement method using the force tensiometer
- By using solvent ratio conditions with relatively low uptake rate and slow distortion, better mechanical stability of the membrane can be secured when performing direct coatings

Collaboration:
Coating Development of Half CCMs

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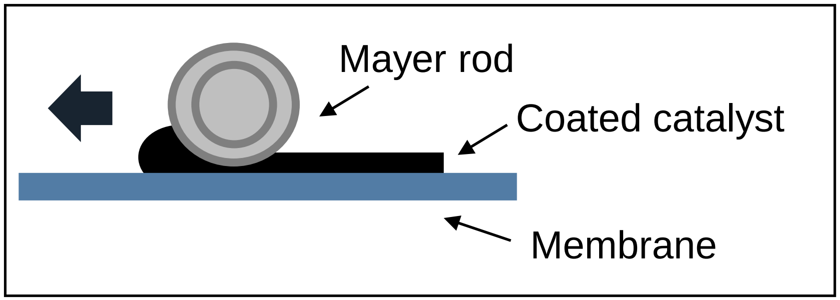
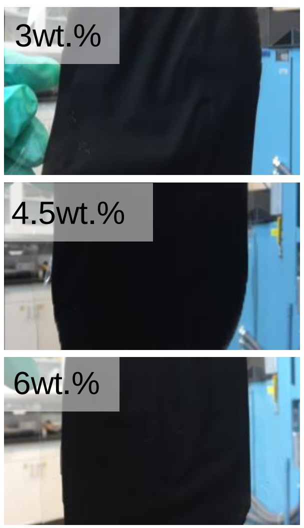
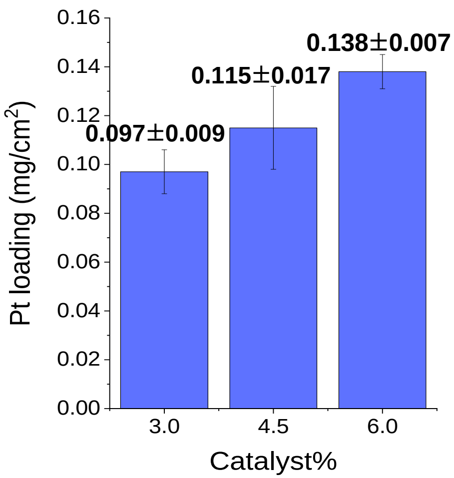
Lab Scale → Gravure Coating

Mayer rod coating

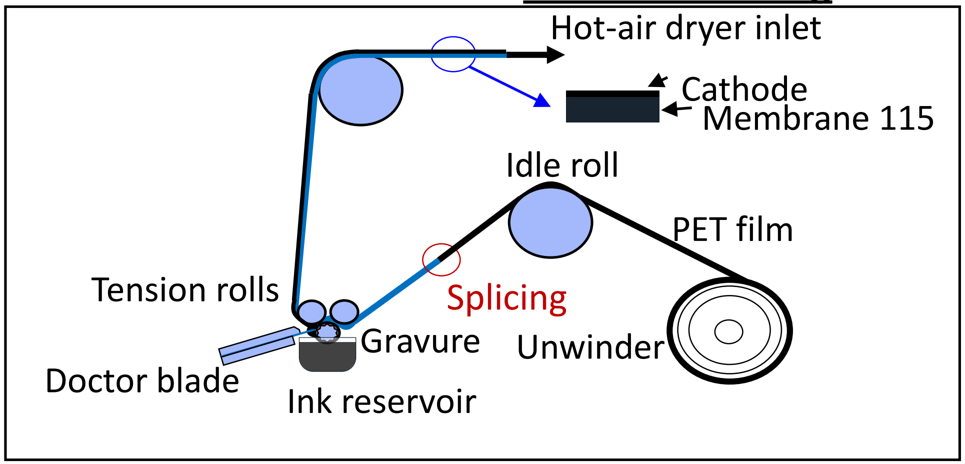




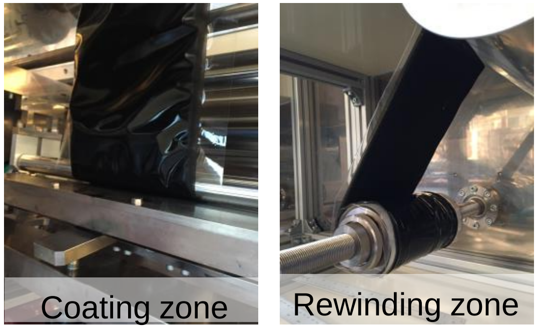


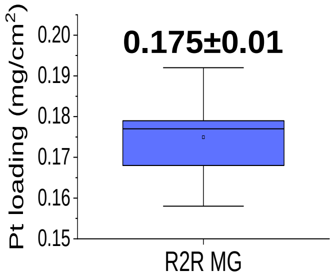
- Direct coating of catalyst inks onto membrane was successfully performed.
- Achieved the target loading (0.1 mg/cm^2) and target uniformity (+/- 10%).

Gravure coating



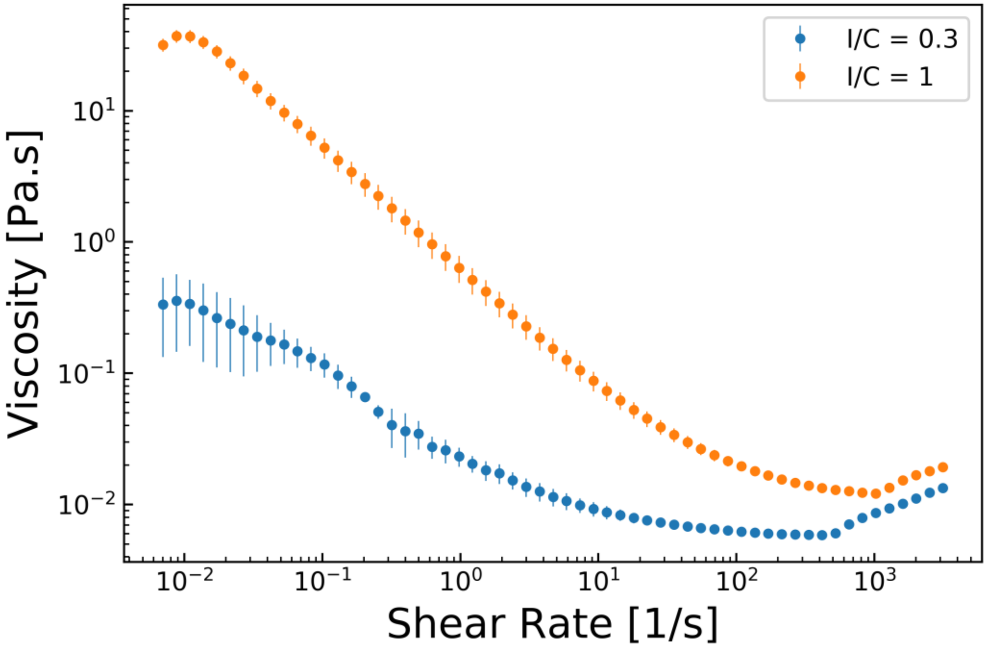
- R2R gravure coating was successfully performed.
- The result shows the possibility of continuous production of CCMs along with achieving a coating uniformity of 10% or less that will enable low-cost PEM electrolysis.





Collaboration: Rheology of Catalyst Inks vs. I/C Ratio

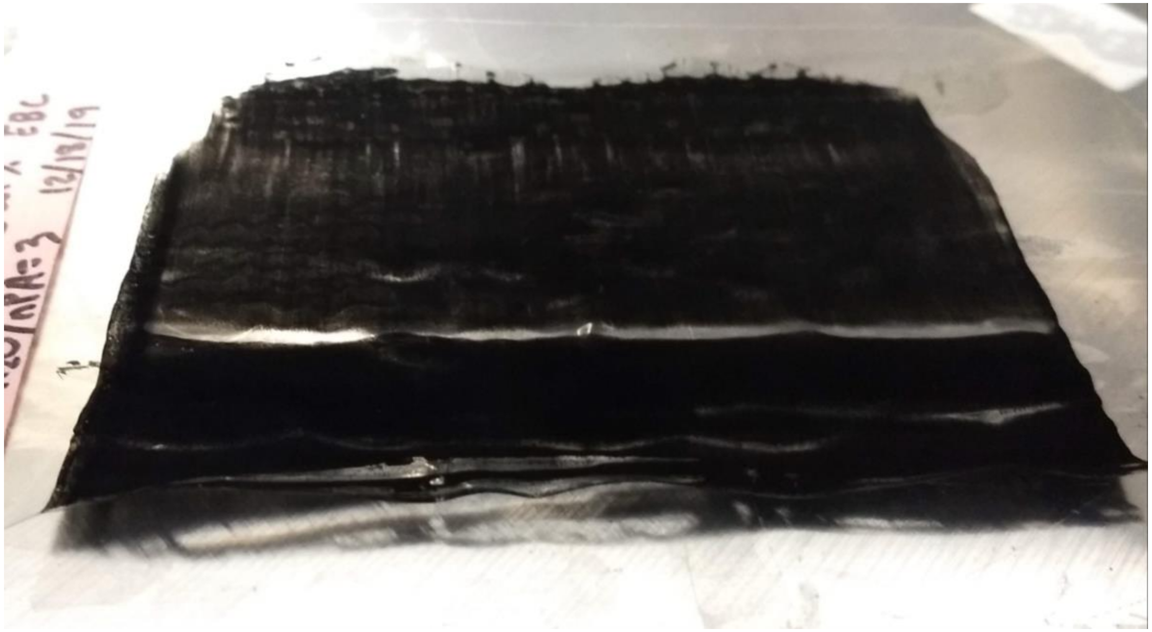
- Both catalyst inks are shear thinning as expected due to the presence of catalyst particles
- I/C = 0.3 is less viscous than I/C = 1 because it has less ionomer and thus more solvent.



Collaboration:
Doctor Blade Coating

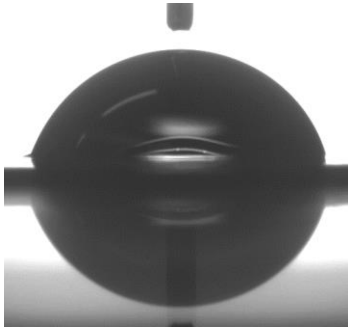
- Significant membrane wrinkling
- Slurry viscosity too low because slurry spread beyond doctor blade boundaries

6 wt.% Pt/C
I/C = 0.3
H_2O/nPA = 3

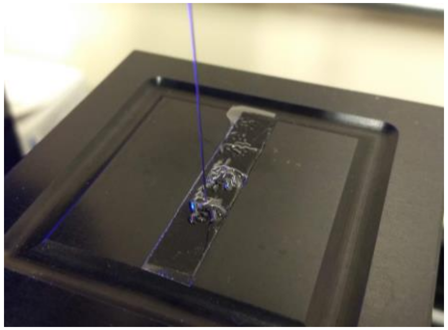


Collaboration:
Static Contact Angle Measurements

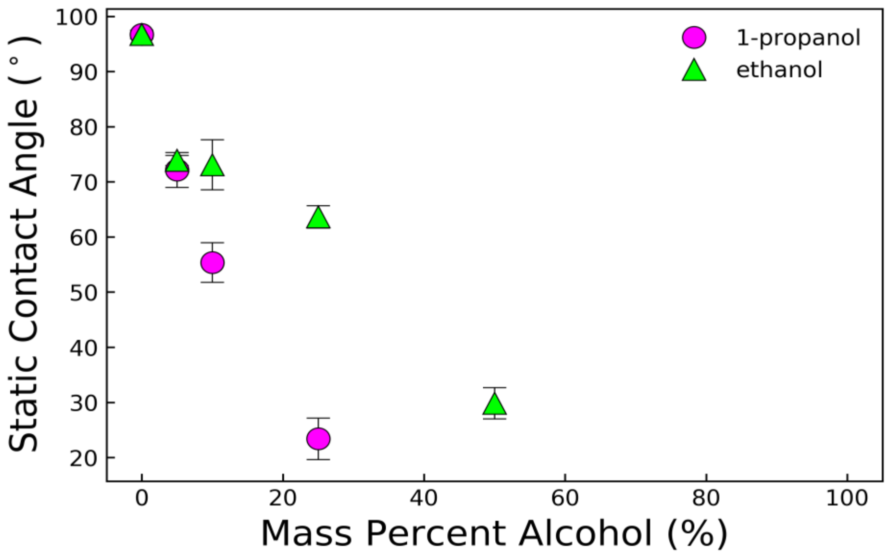
- Sessile drop static contact angle technique on a tensiometer
- **Substrate**: Strip of Aquivion E98-09S membrane adhered to a solid platform with double-sided carbon tape
- **Solvent**: water and alcohol mixtures
- 5 droplets of 5 µL measured for each solvent system



contact angle image
5:95 wt.% ethanol:water



Membrane wrinkling with
> 25 wt.% alcohol



- Best contact angle (< 90° but minimize alcohol content to minimize deformation)
  - 1-propanol: 5-10 wt.% alcohol
  - Ethanol: 5-25 wt.% alcohol
- Future work
  - Contact angle measurements with catalyst ink slurries with "best" solvent mixtures
  - Doctor blade coatings with low alcohol content

Challenges and Barriers

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- Work on high volume manufacturing has been predominantly focused on supported fuel cell catalysts
  - The inclusion of OER, oxide catalysts into an electrode ink that can be scaled and used in roll-to-roll manufacture is a new area of research
  - Leveraging characterization capabilities from the National Labs and expertise in MEA manufacture from GM and Kodak should help to minimize this risk.
- The incorporation of membrane treatment needs to be included and it is uncertain how this will be translated to a continuous manufacturing process
  - Work is happening in parallel to address this manufacturing question early in the bench-scale development
  - Support from the labs and industry experts are continuously developing fabrication techniques for a full CCM that is translatable to high volume manufacturing equipment
- Electrolysis MEAs developed with previous bench methods do not scale well to large active area, high throughput manufacturing
  - The bench-scale techniques of Mayer rod, gravure, and slot die have all shown good translation to roll-to-roll manufacture for fuel cell MEAs
  - GM to provide background experience in scale and volume coatings

Proposed Future Work:

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- Efforts are ongoing to down-select HER catalysts
  - Expect to complete by June 30th, 2020
  - OER catalysts identified for balance of program work
- Membrane down-select in progress and expected to be completed by August '20
  - Several candidates showing promise
  - Final selection based on cost and durability
- Recombination strategy has shown feasibility, with focus shifting to manufacturability
- Ink compositions are being assessed for optimal solvent concentration to minimize wrinkling
- All down-selected and optimized components will be scaled-up and assessed in >1000 hr tests before moving to roll-to-roll

Summary

- Significant progress made in identification of HER and OER catalysts for transition to roll-to-roll processing
  - Combined loading target of 0.7 mg/cm^2 was achieved
- Understanding of I:C ratios of ink and solvent content continues to improve
  - Earlier work showing significant distortion of membrane during coating has been slowly resolved through ink and process mods.
- Work planned will quickly transition to full MEA durability testing
  - ≥1000 hr testing in 100 cm^2 cell stack to verify integrated assembly