FC326

NIKOLA

DURABLE MEAS FOR HD FC TRUCKS

JOHN SLACK

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DOE Hydrogen Program
2022 Annual Merit Review and Peer Evaluation Meeting
Award DE-EE0008820

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PROJECT GOALS

1. **Fabricate, characterize, and evaluate** membrane electrode assemblies (MEAs) with novel catalyst layer structures to improve performance and durability.
2. **Minimize Pt/Sulfonate interaction** to unlock catalyst activity for identified durable catalysts (*e.g.* dissolution resistant shape-controlled alloys or another down-selected catalyst)
3. **Achieve high catalyst-layer oxygen diffusivity** by engineering large homogeneous pores in the CL and reduce Knudsen O_2 transport resistance by decreasing ionomer clustering on Pt surfaces

This will be accomplished using:

A “Nanocapsule” electrode structure: A repeating core/shell electrosprayed nanoparticle in the hundreds-of-nanometer diameter range which separates ionomer and Pt to maximize activity while allowing ionic transport

The outcomes of this project, if successful, will allow for better utilization of highly active and/or highly durable catalysts and the bridging of the activity gap between RDE and MEA.

OVERVIEW

Timeline & Budget

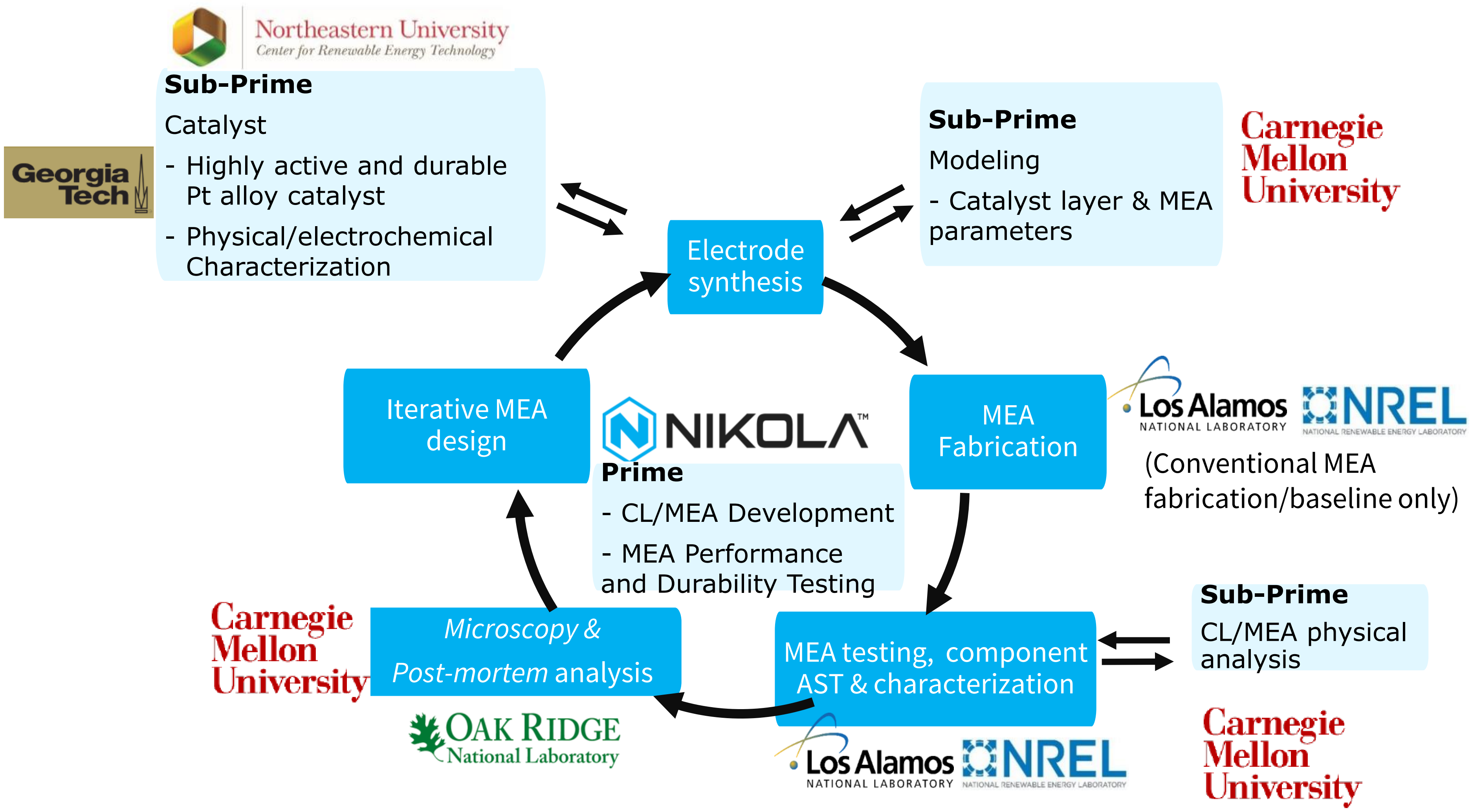
- ❖ Project Start: 10/1/2021
- ❖ Project End: 10/1/2024
- ❖ Total project budget: $2,125,000
  - Total Recipient Share: $425,000
  - Total Federal Share: $1,700,000
- ➢ DOE funds spent*: $ $392,317
- ➢ Cost Share Funds Spent*:$101,599
- ➢ Total funds Spent*: $493,916

* As of 4/25/2022

Partners

- ❖ Nikola Corporation, Project Lead
- ❖ Carnegie Mellon University
- ❖ Northeastern University
- ❖ Georgia Institute of Technology

COLLABORATION & COORDINATION



Targets and Status: Budget Period 1

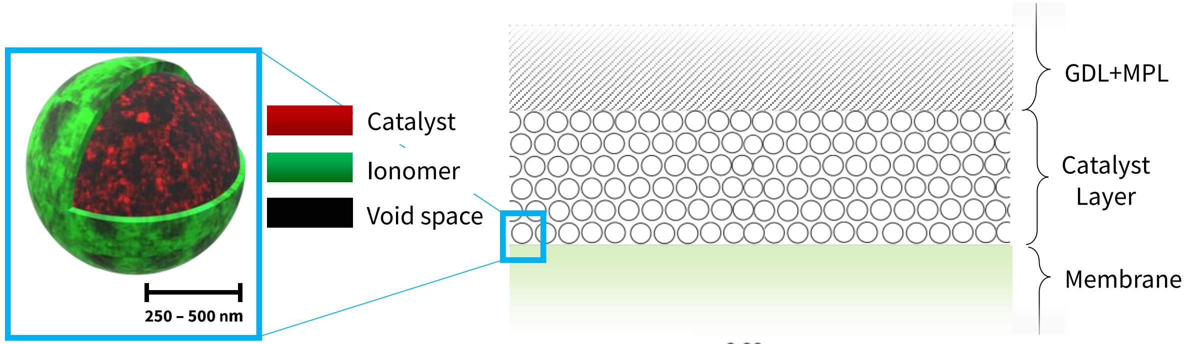
RELEVANCE

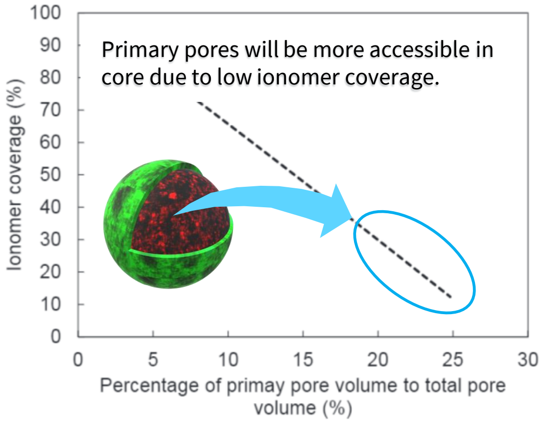
Task Number		Task or Subtask	Milestone Type/number: Milestone Description	Anticipated Month (from Start of the Project)
Task 1.1.1	✓	MEA component down-selection	• M 1.1.1: down-select commercial catalyst • M1.1.2: down-select membrane	M6, M9
Task 1.1.2	✓	Nikola CL optimization and fabrication	• M 1.1.3: Nikola CL optimization with commercial catalyst • M 1.1.4 : MEA Benchmarking Baseline Definition	M12
Task 1.2	✓	Development of cuboctahedral PtCo/C catalyst for Nikola CL	• M 1.2.1: Metal deposition optimization for cuboctahedral PtCo/C • M 1.2.3: fabrication and delivery of 6g of catalyst	M6, M9
Task 1.3	✗	Development of IBAD Pt/M-M/C catalyst for Nikola CL	• M 1.3.1: Metal deposition optimization for IBAD Pt/M-M/C • Milestone 1.3.3: Perform IBAD catalyst degradation analysis and delivery of 6g of catalyst	M8, M12
Task 1.4	✓	Modeling and analysis of Nikola CL	• M 1.4.1 Modeling of MEA CL • M1.4.2: structural analysis of Nikola CL and MEA	M6, M12
Task 1	⌛	Nikola CL MEA fabrication and performance (≤50cm^2 MEA active area)	• **Go/No-Go 1:** Nikola CL MEAs will be fabricated with commercial catalyst • MEA -Performance ≥ 350 mA/cm^2 at 0.8V, 200 kPa$_{ab}$, 80 °C , 0.3 mg/cm^2 PGM total	M12

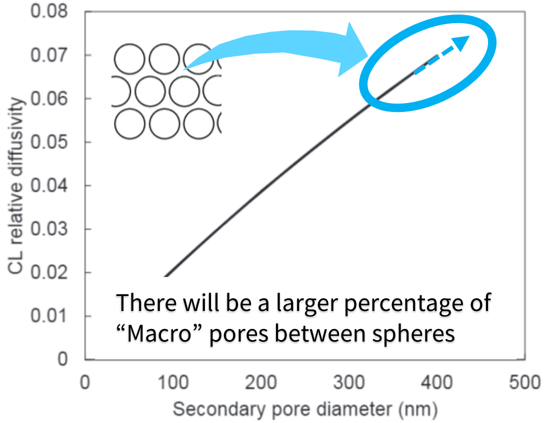
The Nanocapsule Catalyst Layer
APPROACH

Purposefully heterogeneous Ionomer & Catalyst within the catalyst layer

- **Optimized local I/C:** may reduce sulfonate poisoning while still providing conductivity
- **Leverages non-zero conductivity:** of protons across carbon and platinum surfaces[1,2]
- **Tune I/C in the shell:** minimum required for protonic transport from membrane to CL/MPL edge
- **Tune I/C in the core:** minimum required for protonic transport from shell to core to reduce confinement effects

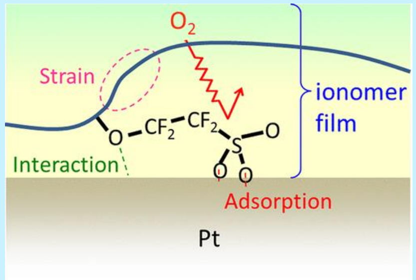






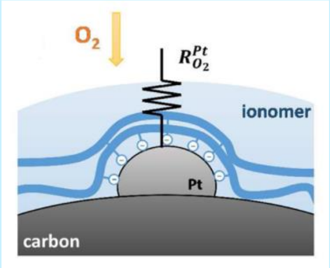
Separate ionomer and catalyst to:

Increase available Pt surface free of SO_3^-



Proposed mechanism for catalyst poisoning via ionomer adsorption.[1]

Improve oxygen concentration at Pt surface



Adsorbed ionomer can increase oxygen gas transport resistance.[3]

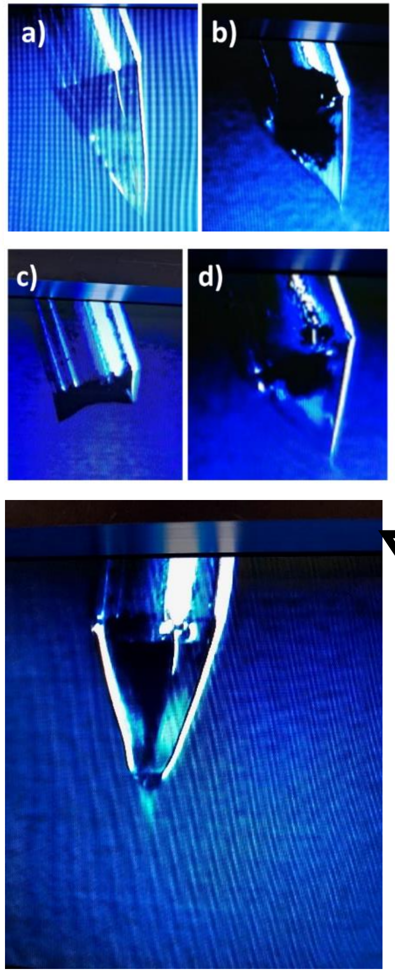
[1] A. Kongkanand, *U.S. DOE Annual Merit Review FC144*, **2018**
[2] A. Kongkanand, *U.S. DOE Annual Merit Review FC144*, **2019**
[3] K. Takahashi et al. *Journal of The Electrochemical Society*, 163, F1182-F1188, **2016**

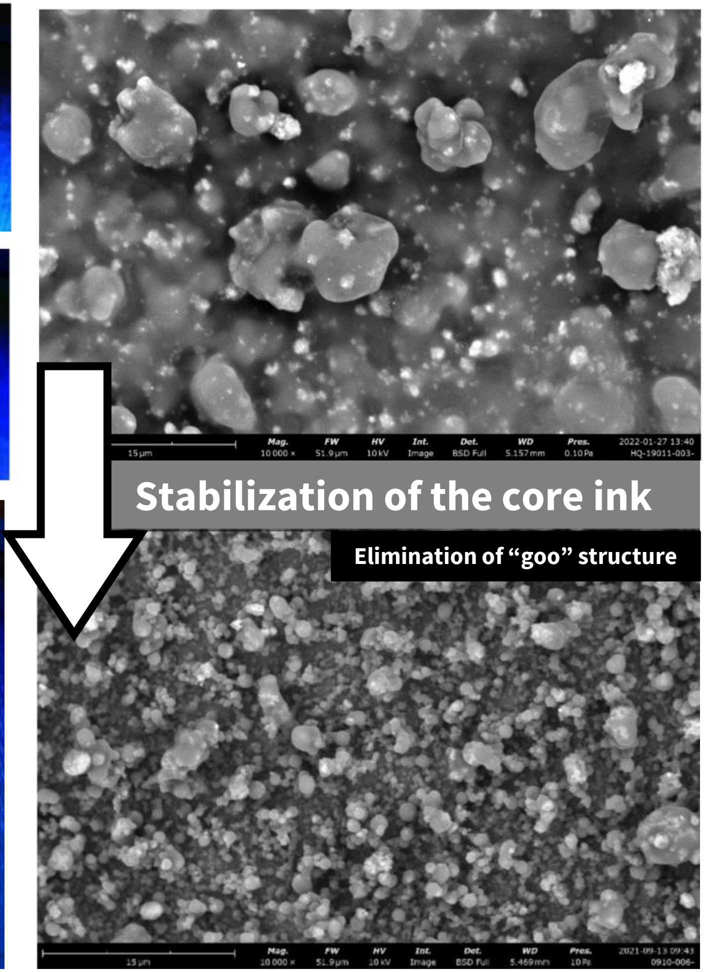
Nanocapsule catalyst layer optimization

ACCOMPLISHMENTS & PROGRESS (TASK 1.1)

Co-axial needle emitter attempts & results

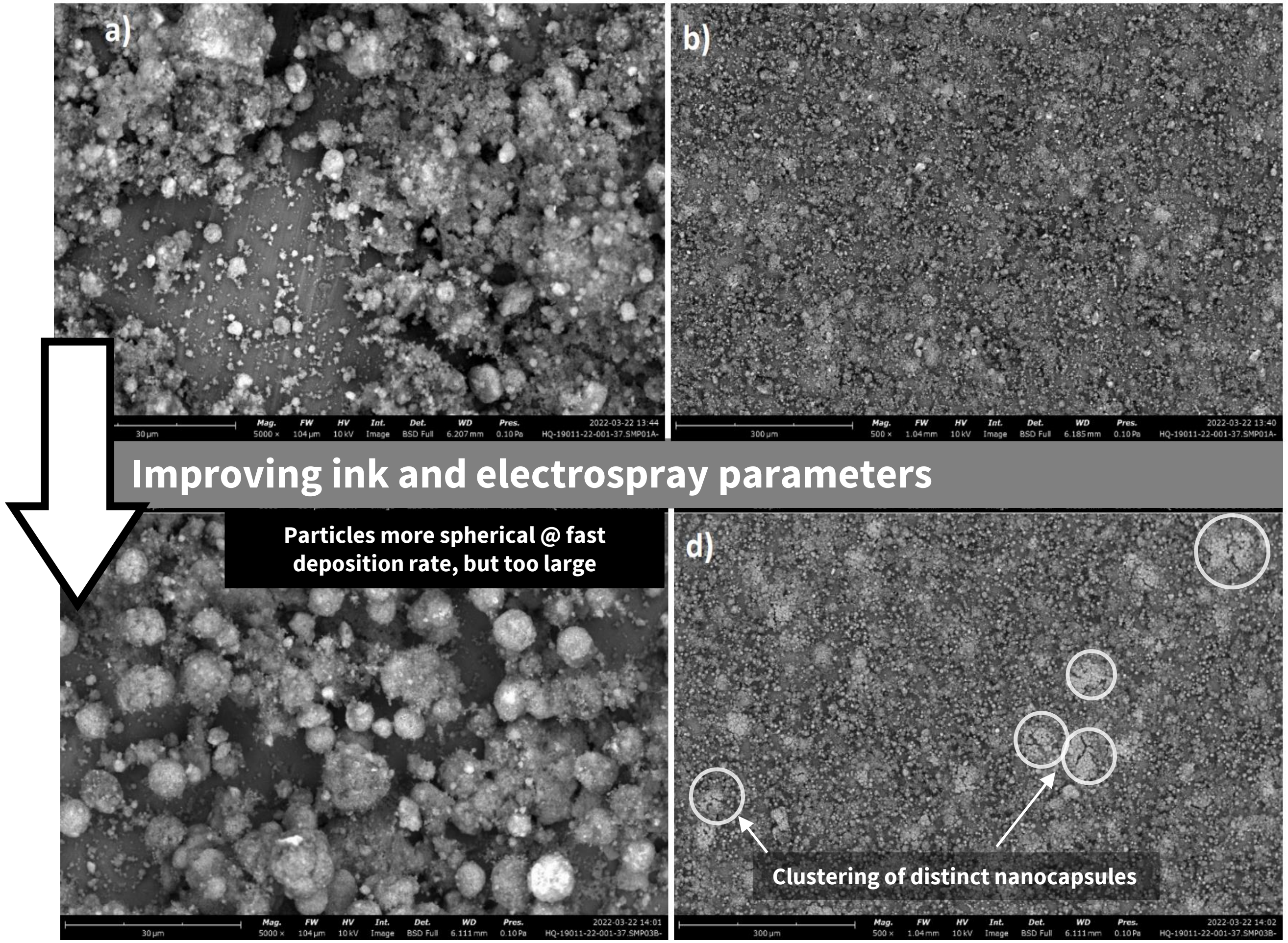
Summary: Sensitive to changes in ink & slower fabrication





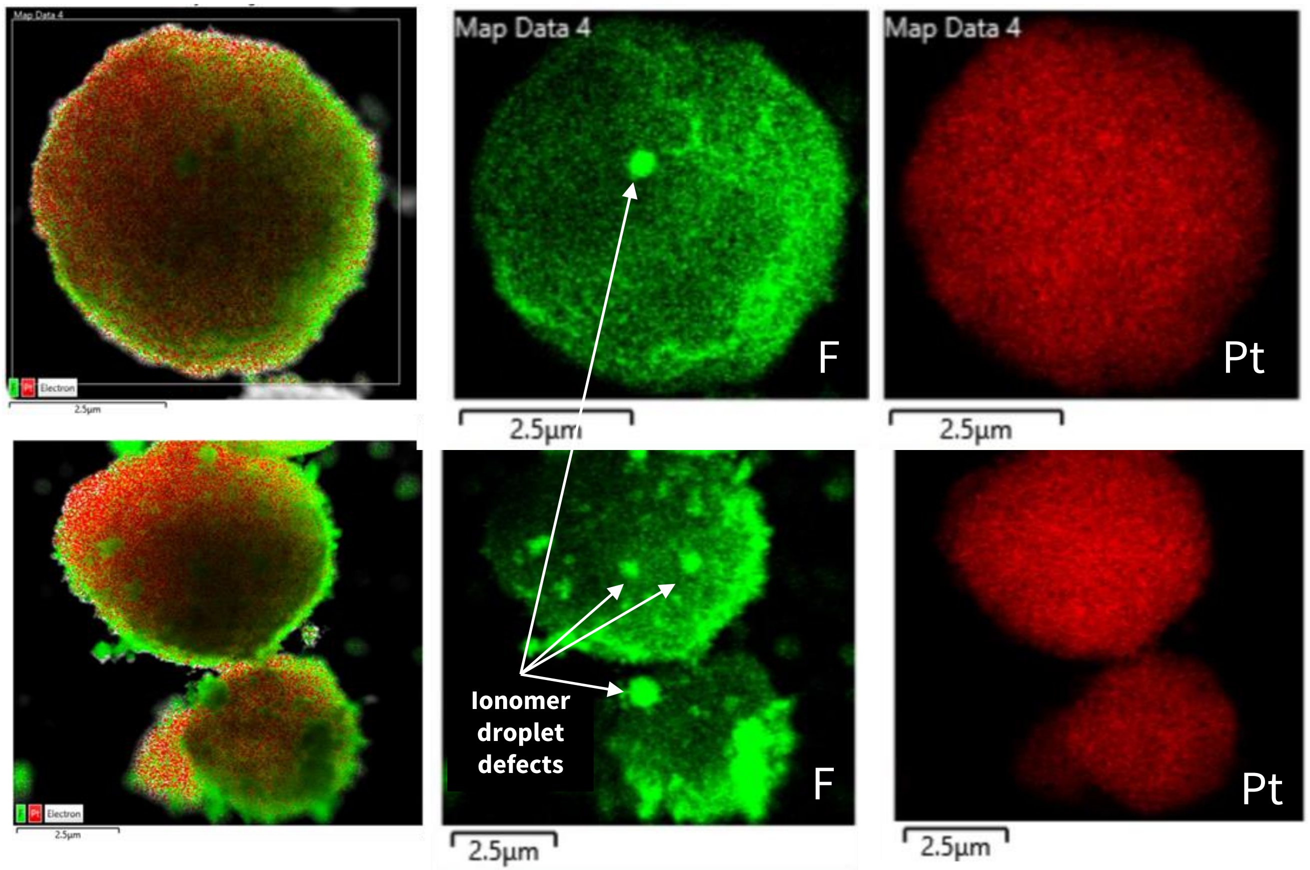
Single needle emitter results

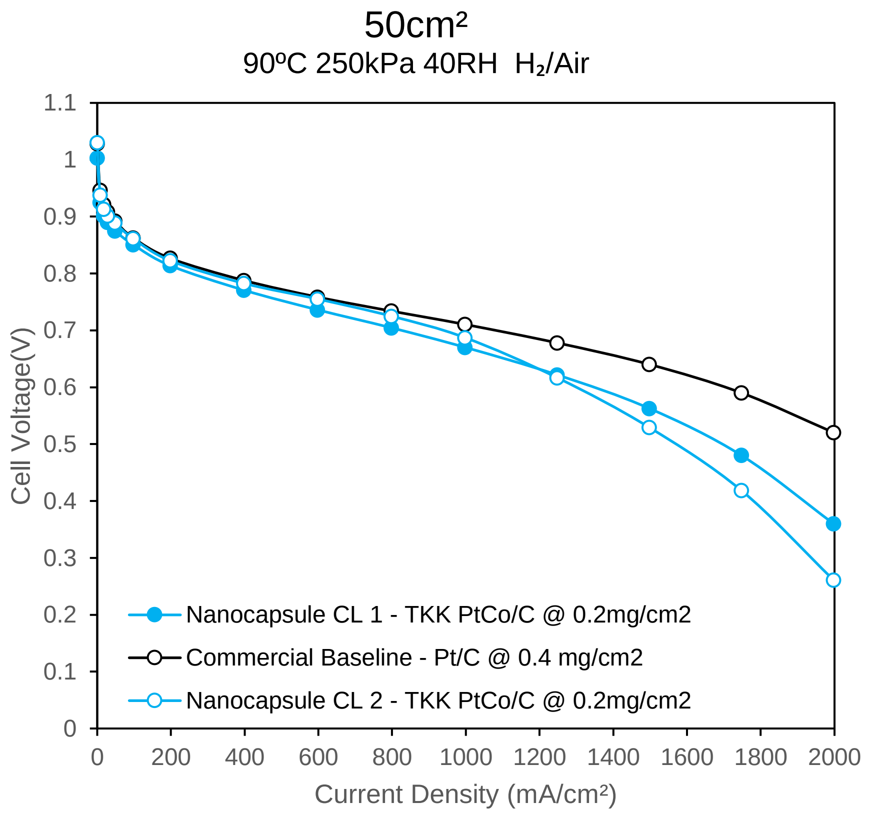
Summary: More robust & up to 10x faster fabrication



Physical and Electrochemical Analysis

ACCOMPLISHMENTS & PROGRESS (TASK 1.1)





SEM & STEM/EDS Analysis Summary

- Average particle Circularity: 92% ± 2% (4πArea/perimeter^2)
- Average Nanocapsule diameter: 5 µm ± 3 µm
- Nanocapsule ionomer-shell thickness: 200-500nm
- Typical radial ionomer profile: Step-function

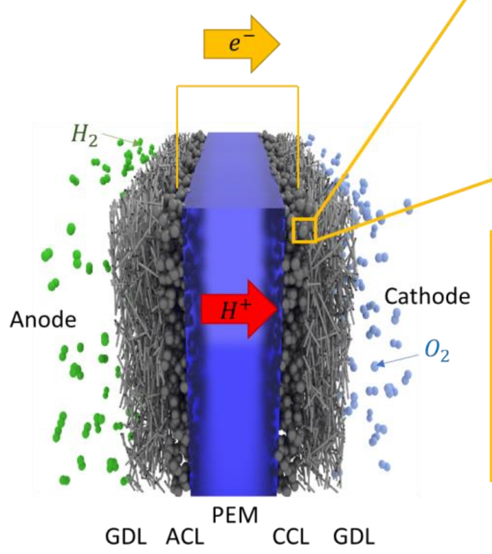
Electrochemical Analysis Summary

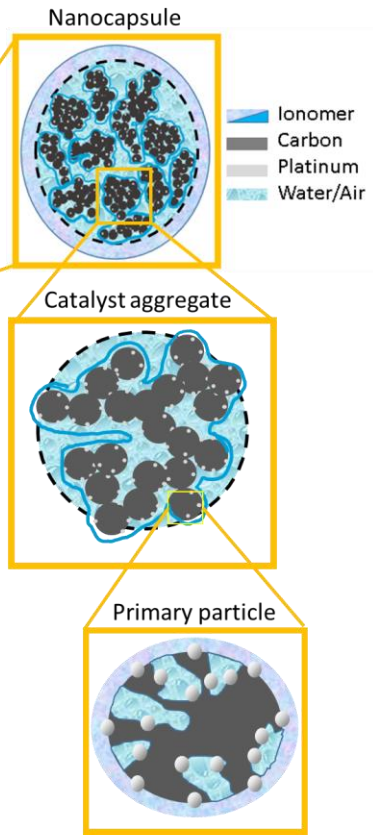
- On the path to surpassing BP1 Go/No-Go: (Present: 335 mA/cm^2. Target: 350 mA/cm^2)
- Transport losses with current structure
- Not yet achieved activity enhancement

Multi-scale modeling

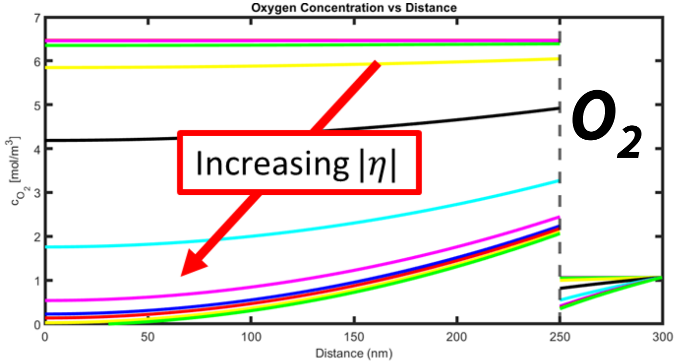
ACCOMPLISHMENTS & PROGRESS (TASK 1.4)

Four key length-scales to modeling the nanocapsule cathode performance in a fuel cell

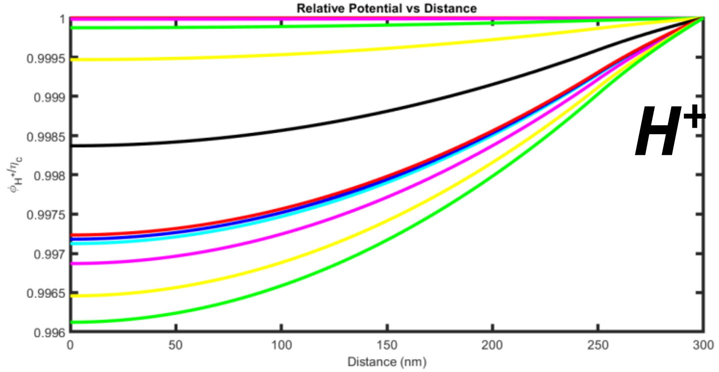


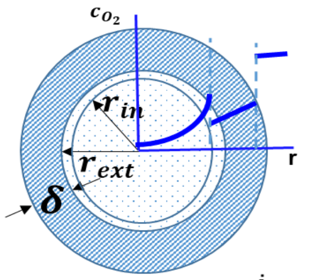


Radial distribution of O_2 concentration



Radial distribution of proton-phase electric potential





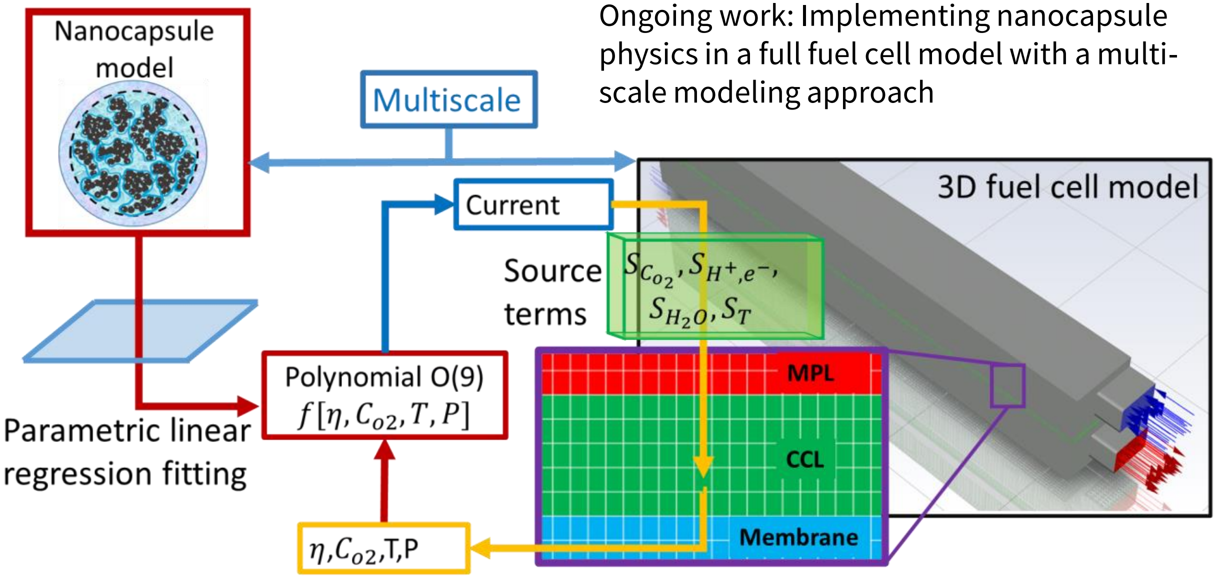
$$(\nabla \cdot D^{eff} \nabla c) = -\frac{j}{4F}$$
$$(\nabla \cdot \sigma_{H^+} \nabla \phi_{H^+}) = -j$$

Numerical nanocapsule model

- Oxygen, proton, and liquid water transport
- Separate ORR kinetics and local Pt O_2 transport for ionomer contact or not
- Separate solubility in ionomer and water domains

Multi-scale Nanocapsule-in-Fuel Cell Modeling

Ongoing work: Implementing nanocapsule physics in a full fuel cell model with a multi-scale modeling approach



Multi-scale modeling

ACCOMPLISHMENTS & PROGRESS (TASK 1.4)

Initial application of model framework applied to key design parameters:

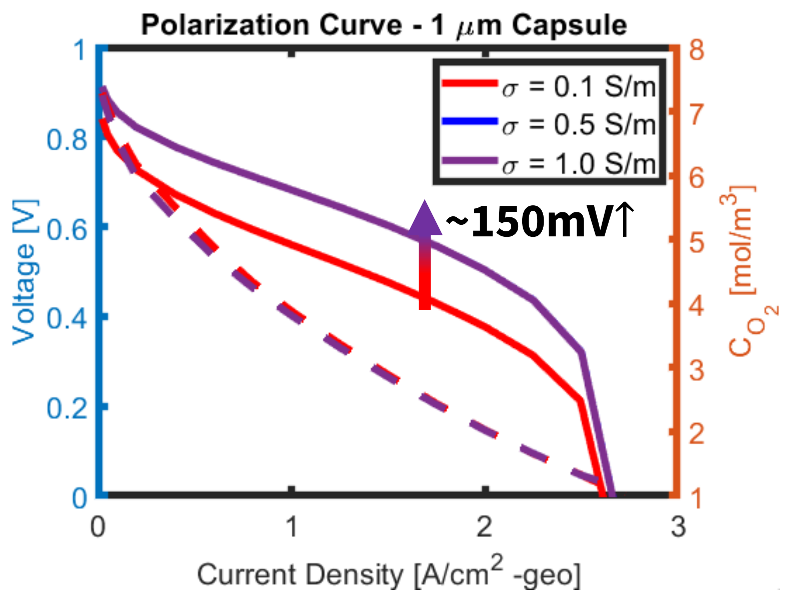
- Diameter of the nanocapsule
- Conductivity in the core

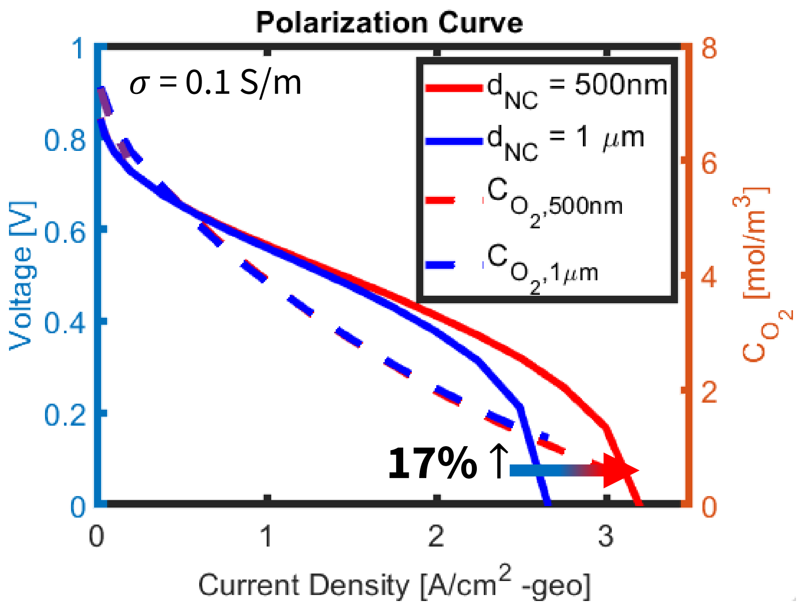
Core conductivity ranging from 1/10th of conventional catalyst layers to typical conductivity (1 S/m):

- A 1 µm diameter nanocapsule requires an internal core conductivity of approximately 0.5 S/m for maximum performance

Comparison of a 0.5 and 1 µm diameter nanocapsules:

- Significant improvement in mass transport limited performance when reducing to 0.5 µm due to reduced internal diffusion length scales





Multi-scale modeling

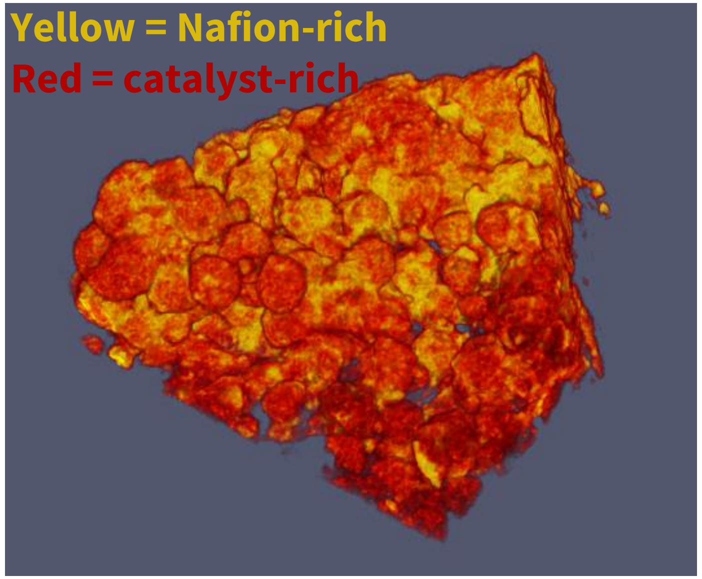
ACCOMPLISHMENTS & PROGRESS (TASK 1.4)

Cesium-stained ionomer for X-ray contrast and 3D ionomer mapping

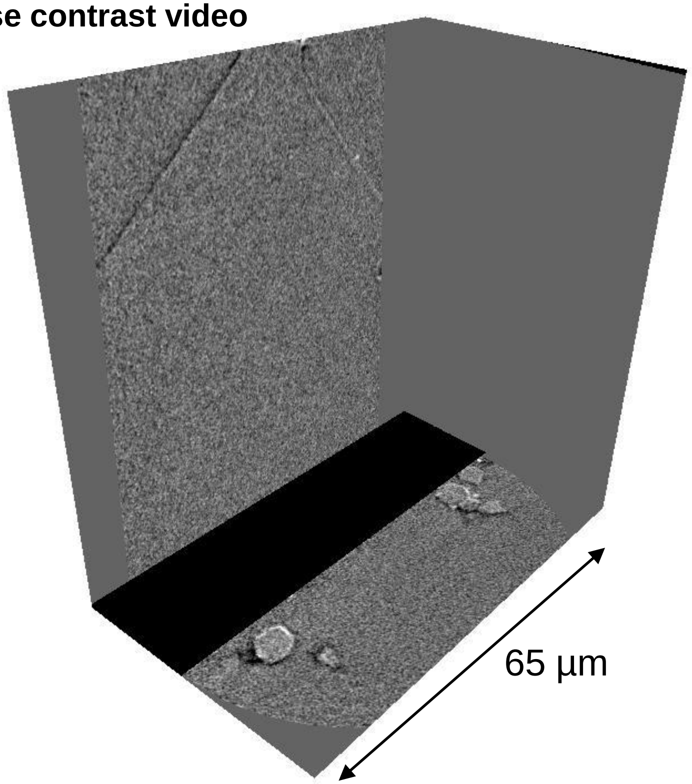
Phase contrast scan shows higher contrast of ionomer layer around catalyst-filled spherical particle agglomerates (e.g., nanocapsules)

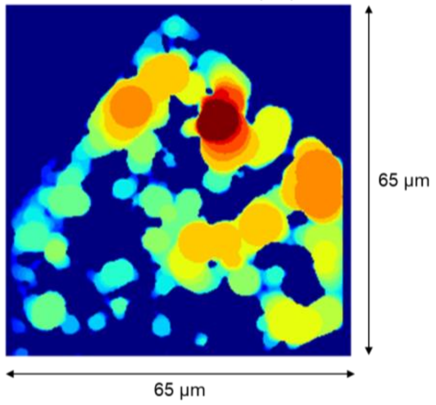
Ionomer films have a thickness of ~500 nm that partially disappears where multiple particles aggregate together

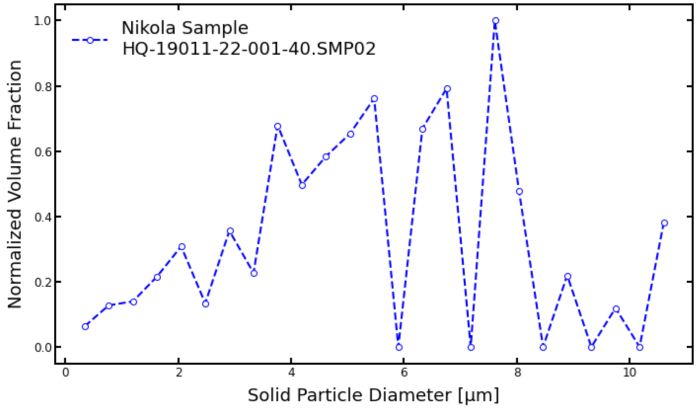
Size distribution analysis of nanocapsules shows a range of 4-8 µm



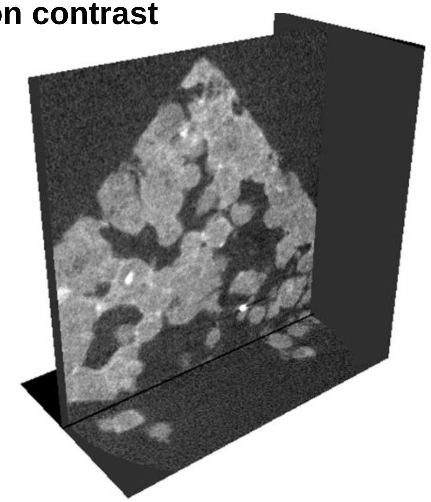
Phase contrast video

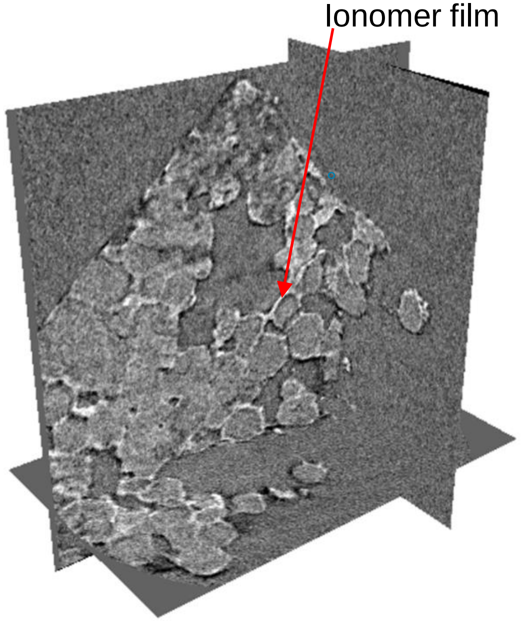






Cesium-ion stained ionomer and absorption contrast



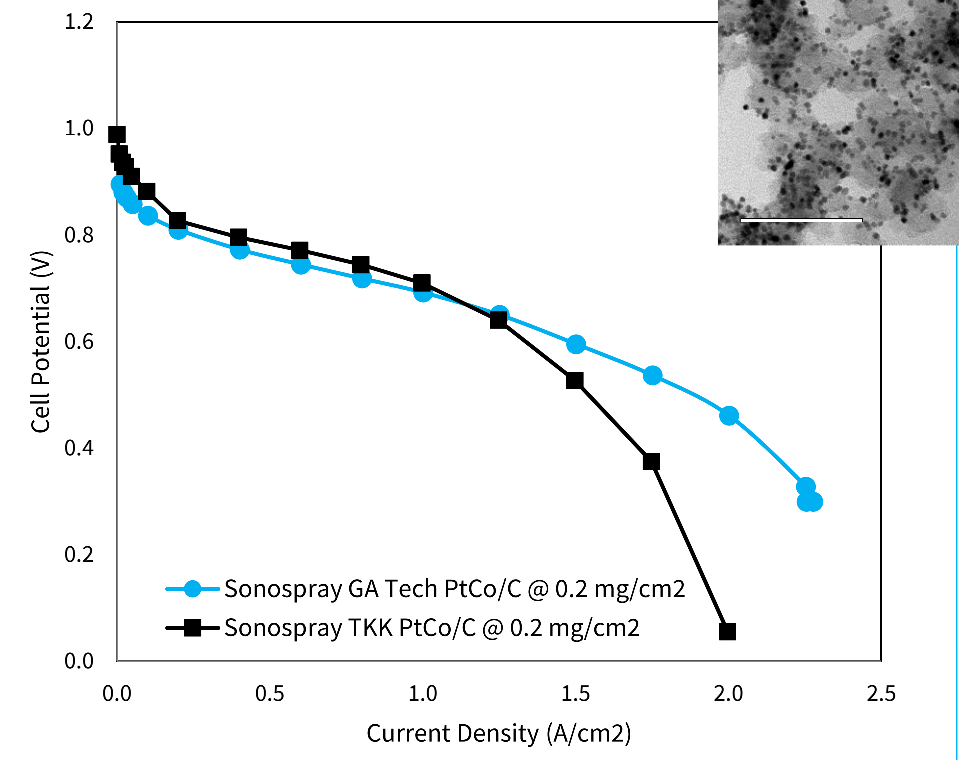


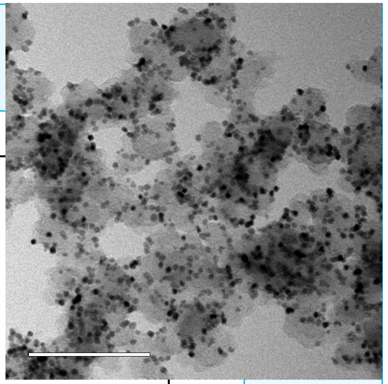
Experimental Catalyst Development & Analysis

ACCOMPLISHMENTS & PROGRESS (TASKS 1.2 & 1.3)

Georgia Tech

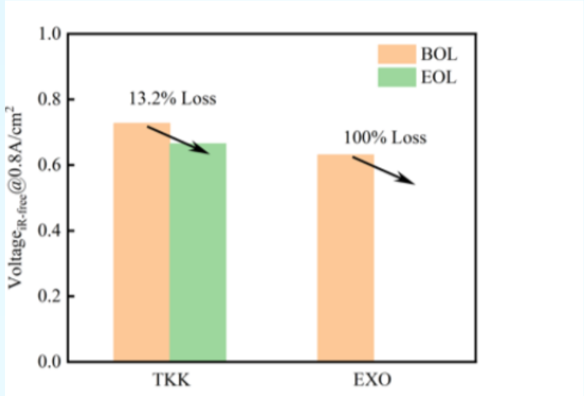
MEA Data from NREL

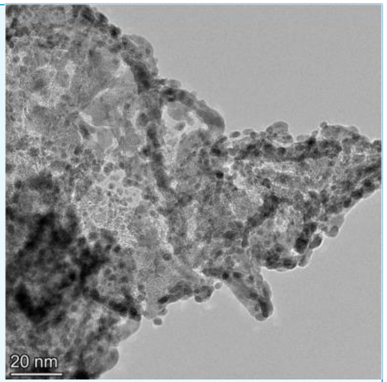




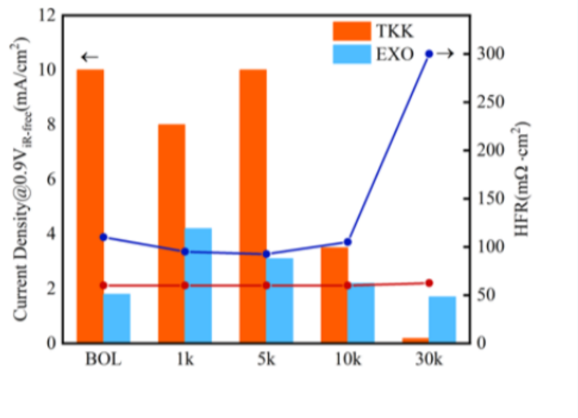
Loading: 0.2 mg_{Pt}/cm^2, pressure: 150 kPa, temperature: 80, RH: 100%, I/C: 0.3

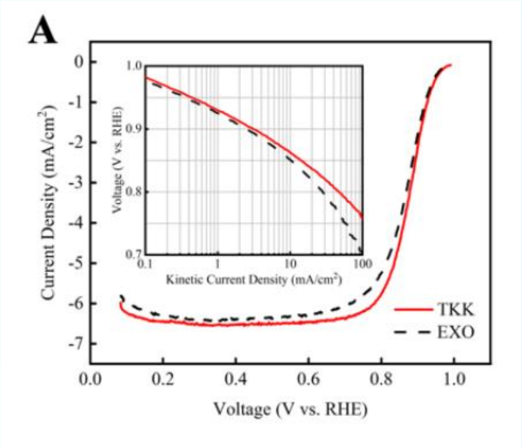
Northeastern University





Neither performance nor durability matches baseline.





REMAINING CHALLENGES & BARRIERS

Challenge	Resolution
Nanocapsule size too large ***Ionomer droplets on surface of nanocapsule*** ***Nanocapsule size polydispersity***	• Explore the following properties to solve: • Solvent choice, I/C ratio, solids concentration, conductive additives, polymeric additives, viscosity modifiers, flow rate, emitter/collector distance, voltage, temperature of the environment, RH, carbon support type
Scale up to full-size MEA	• Fabricate onto large platform using optimized strategy from sub-scale. • Employ strategies to increase deposition rate (i.e., single-phase emitter: already under development)

YEAR 1 MILESTONES AND PROGRESS

SUMMARY

Challenges overcome:

- Overcome instability issues with inks
- Achieved core/shell structure
- Produced highly spherical particles
- Step-function core & shell radial ionomer profile
- Increased production rate

PROGRESS

Milestone	Period	Progress
Nanocapsule CL fabrication/ physical analysis/ electrochemical analysis iteration process underway	Q2	Complete ✓
Development of cuboctahedral PtCo/C catalyst for Nikola CL	Q4	Complete ✓
Development of IBAD Pt/M-M/C catalyst for Nikola CL	Q4	Incomplete ✗
Modeling and analysis of Nikola CL (Nano-CT, Multi-scale model) underway	Q4	Complete ✓
Go/No-Go 1: MEA -Performance ≥ 300 mA/cm^2 at 0.8V	Q4	On track ⌛

PROPOSED FUTURE WORK

Budget Period 2 Iterative Plan:

Nanocapsule Electrode Optimization

Step	Locations
1. **Fabricate** two each of several electrode types (e.g., changing fabrication parameters) per week. Collect SEMs of every electrode structure made.	NIKOLA™
2. **Screen** these MEAs (+2 baseline MEAs) for BOL performance in a short stack • Polarization data, ECSA, mass activity 3. **Comprehensively Test** the best MEA of the short stack per week in a single cell platform • Confirm stack data + GTR, CL ionic resistance, sulfonate coverage, air/O_2 gain	NIKOLA™ + NREL NATIONAL RENEWABLE ENERGY LABORATORY
4. **Physically Analyze** the electrode structure of the week's best performing MEA using STEM/EDS • Send the single best performing electrode structure per month to CMU & ORNL for more comprehensive analyses including nano-CT	Carnegie Mellon University OAK RIDGE National Laboratory

TECHNICAL BACKUP & ADDITIONAL INFORMATION

TECHNOLOGY TRANSFER ACTIVITIES

Patents

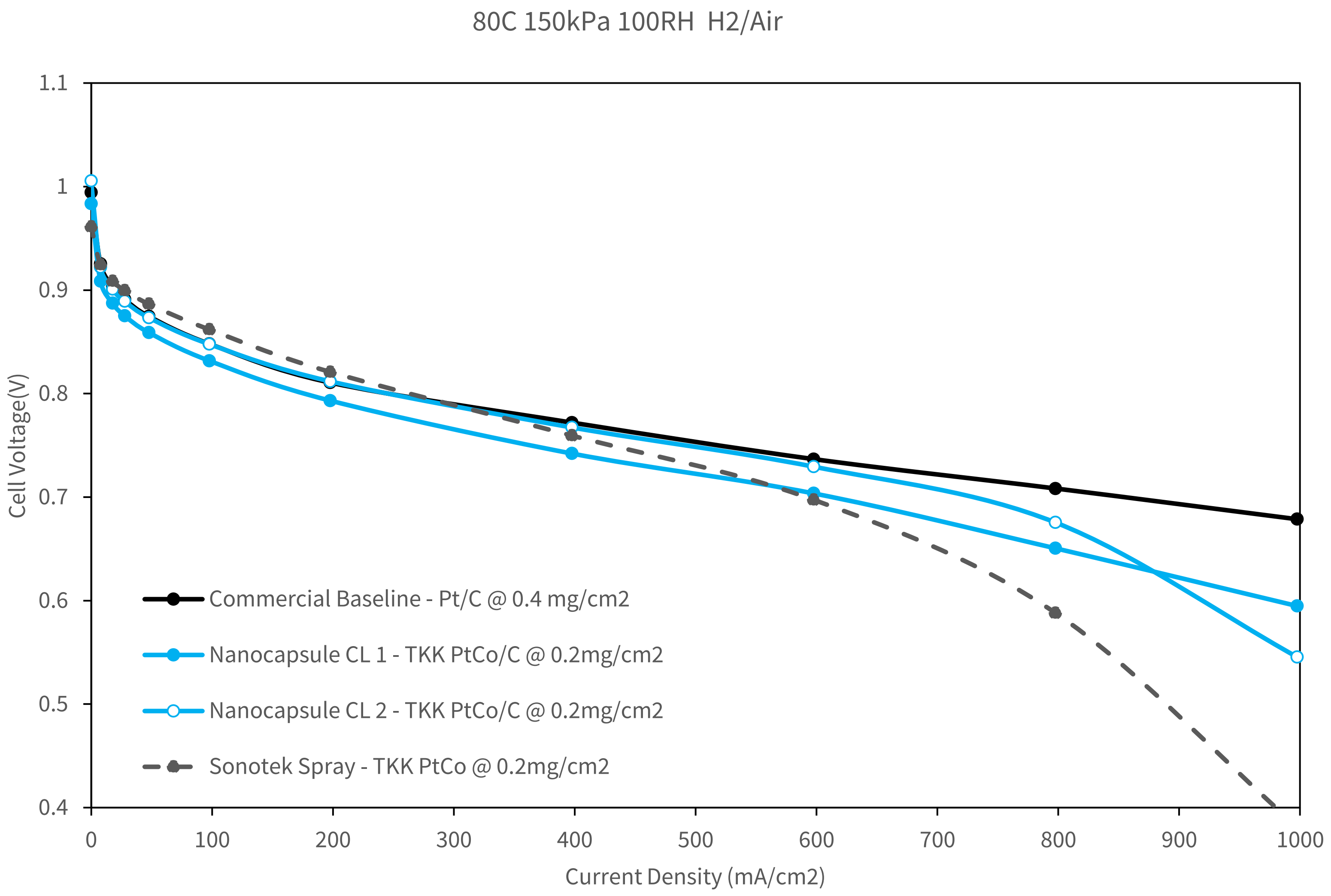
Methods of Making catalyst layers of membrane electrode assembly comprising structured units. Patent No.: US 11,283,083 B2 2022. (This IP was submitted prior to DOE funding).

Tech-to-market activities

Commercialization of catalyst layer technology is anticipated if proposed advances are realized

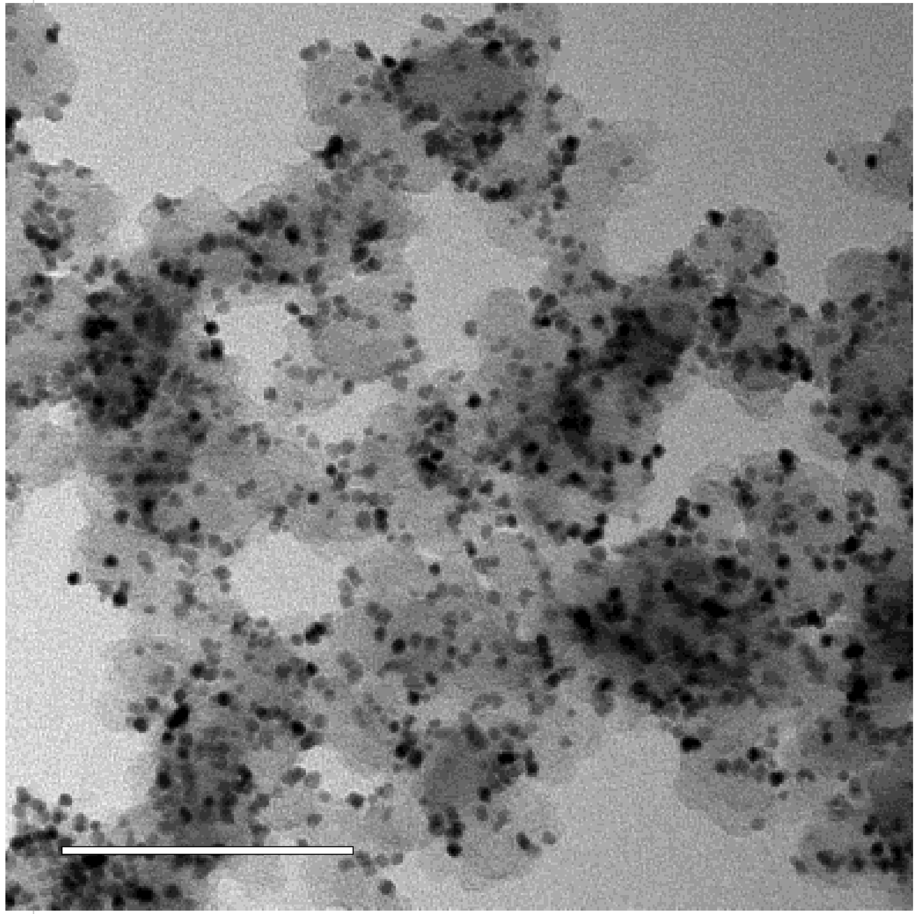
Future/Additional Funding: n/a

Cell performance at 80 C 100RH



Georgia Tech PtCo Truncated Octahedral Catalyst Synthesis

Georgia Tech has shipped 6g of Catalyst to Nikola

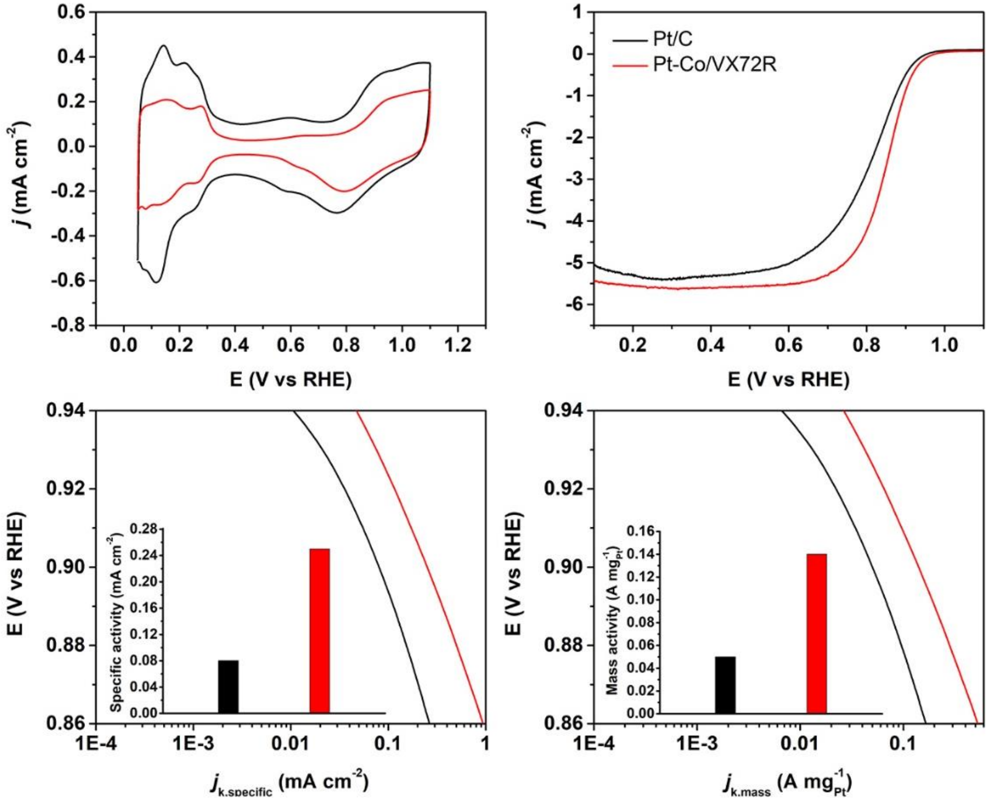


- 30wt% Pt loaded PtCo nanocrystals with a uniform size of **5 nm** were homogeneously dispersed on carbon support (Vulcan XC72-R)
- The nanocrystals feature a **truncated octahedral shape**

Standard Synthesis (from Georgia Tech):

$Pt(acac)_2$: 150 mg, 0.38 mmol
$Co(acac)_2$: 150 mg, 0.58 mmol
Benzoic acid: 1300 mg, 10.66 mmol
Carbon: 100 mg, 8.33 mmol
DMF: 150 mL, 1.94 mol
160 ˚C 12 h

Georgia Tech PtCo Truncated Octahedral Catalyst Synthesis



ORR Activity in 0.05M H_2SO_4

- **0.05 M H_2SO_4 solution**

- **Cyclic Voltammetry**
  - **Scanning Rate:** 50 mV s^{-1}
  - **Potential Range:** 0.05-1.1 V

- **Linear Sweep Voltammetry**
  - **Rotating Speed:** 1600 rpm
  - **Scanning Rate:** 10 mV s^{-1}
  - **Potential Range:** 0.1-1.1 V

Catalyst	ECSA (m^2 g^{-1})	Specific Activity (mA cm^{-2})	Mass Activity@0.9V (A mg^{-1})
Pt-Co/VX72R	**56**	**0.25**	**0.14**
Commercial Pt/C	64	0.08	0.05