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# Advanced Manufacturing Processes for Gigawatt-Scale Proton Exchange Membrane Water Electrolyzers

May 7th, 2024

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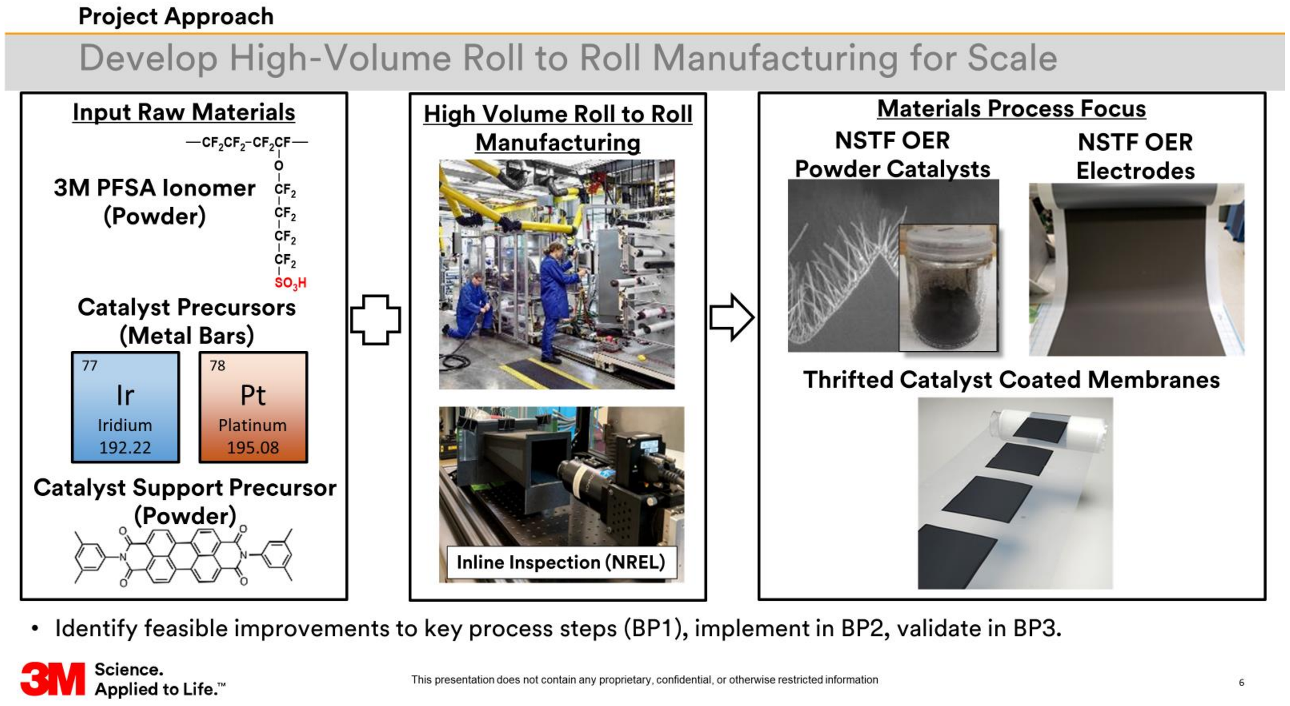
Project P197

DE-EE0009237

# Project Modified for Better Alignment

## Previous Approach: Develop High Volume Processes for OER Catalysts, Electrode and Thrifted CCMs




- Previous scope: process development for OER catalyst, OER electrodes, and thrifted CCMs.
- Project modification warranted due to 2022 3M announcement to exit PFAS manufacturing.
- Proposed modifications:
  1. Remove electrode and thrifted CCM process development from project scope. Electrode material development remains in scope.
  2. Add new subcontractor to produce ~100kW worth of CCM to enable stack durability evaluation of final up-scaled project catalyst
  3. 9 month no-cost extension to enable adaptations.
- NCE approved; scope modifications pending.

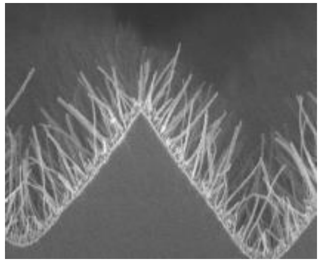
# Develop Processes for Validated Gigawatt-Scale PEM Electrolysis Catalyst

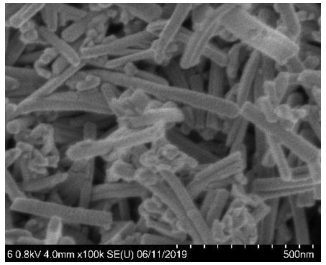
## Project Goals

1. **Develop manufacturing processes** for advanced PEM water electrolysis OER catalysts suitable for GW/year scale.
2. **Scale up** developed processes to GW/year scale and **produce** 100g of OER catalyst (~2 MW equivalent).
3. **Assess and validate** resultant high-volume catalyst against efficiency, durability, power density, and low Ir content metrics in megawatt-capable stacks relevant for GW/year deployment scale.

## Project Component

**NSTF 78wt% OER Powder Catalyst**



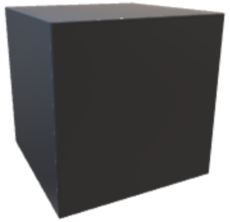




## 1 GW/yr Scale

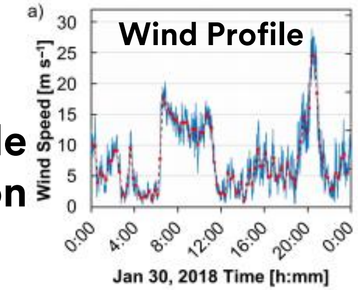
2g/m$^2$, 35kW/m$^2$

**Conversion of 77kg Iridium To Catalyst Powder**



**Cube, 13.6 cm per side**

**Assess: Variable Renewable Integration**




**Validate: Plug Power MW-Capable Stack**
(100kW test)

# Project Addresses Key Cost and Manufacturing Barriers

## Timeline

**Project Start:** 1/1/2021
**Project End:** 6/30/2025*
*Includes no-cost extensions

## Barriers

**F. Capital Cost**
**K. Manufacturing**

## Budget

**Total DOE Project Value:** $6.476MM*
**Total Funding Spent:** $5.092MM*
**Cost Share Percentage:** 26.48%
*Includes DOE, contractor cost share and FFRDC funds as of 3/5/2024

## Technical Targets

| | |
|---|---|
| **Catalyst Fab. Rate*:** | 3.75GW/year |
| **Catalyst Produced*:** | 100 g |
| **Cell Voltage at $2A/cm^2$:** | 1.725V |
| **Electrode Loading:** | $0.2\ mg_{Ir}/cm^2$ |
| **Decay Rate**:** | 4 μV/hr |
| **Stack Power Density:** | $7.6\ W/cm^2$ |

*Based on $3.45W/cm^2$. **Under project wind load profile.

## Partners

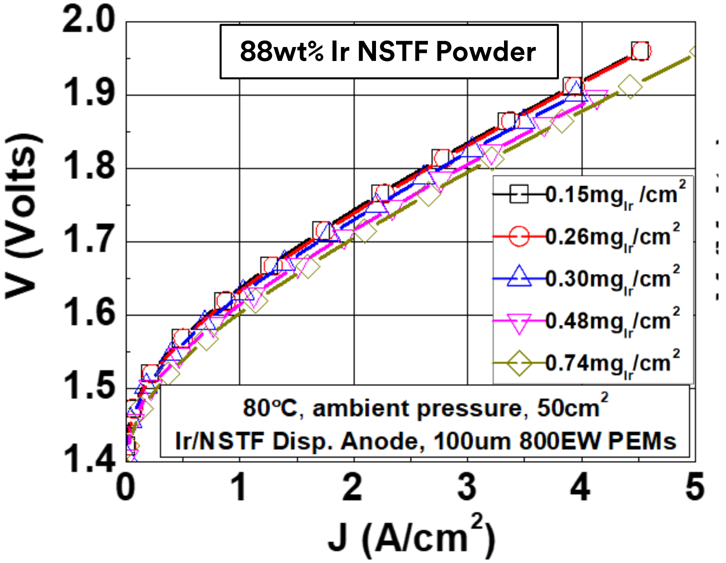
Giner (N. Macauley)
Plug Power (C. Mittelsteadt, Z. Green)
NREL (P. Rupnowski, E. Padgett, S. Mauger)
ORNL (D. Cullen)

# Stringent Material and Processes Requirements for GW/year Scale

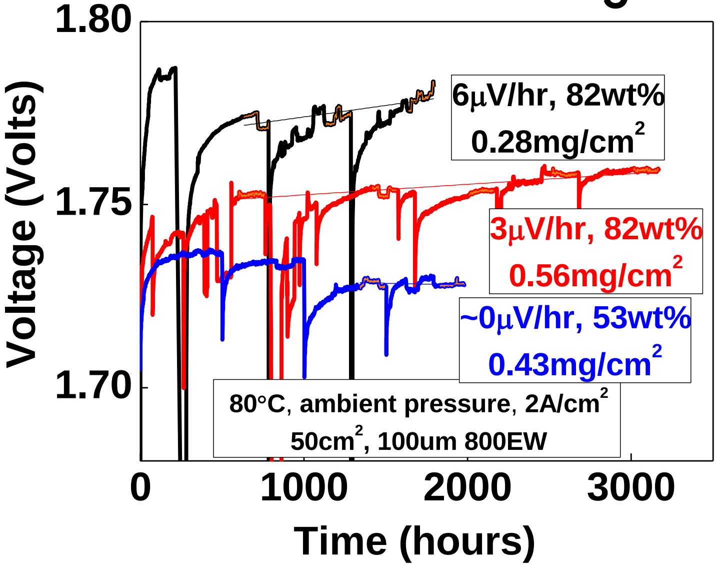
- **<u>Industry Needs for PEM Electrolysis At Scale *(Gigawatts per year)*</u>**
  1. Advanced catalysts which meet efficiency, durability, **<u>and</u>** power requirements ***<u>with low Ir content</u>***.
  2. High volume capacity and reduced manufacturing costs (lower CapEx).
  3. High uniformity, reliability, reproducibility, and quality.

**Project addresses industry needs by development of advanced fabrication processes capable of producing reproducible and uniform state-of-the-art OER catalysts at GW/year scale.**

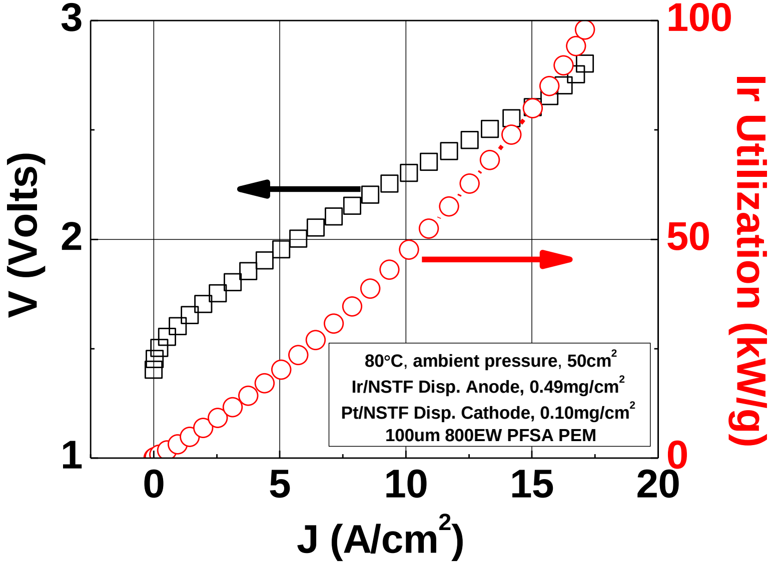
### High Performance with Ultra-Low Ir Loadings




### High Durability at State-of-the-Art Ir Loadings




### High Power Capability



# Project Performance and Durability Targets Achieved

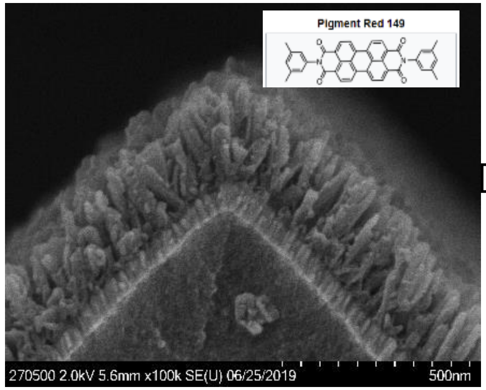
| Project Target | Target Value | '24 Status | |
|---|---|---|---|
| Catalyst Fabrication Rate (GW/year) | ≥ 3.75 | 1.5[1] | Mfg. Targets |
| Catalyst Produced at Target Rate (g) | ≥ 100 | Not started (BP3) | |
| Cell Voltage @ $2A/cm^2$ | ≤ 1.725[3] | 1.721[2] | |
| Electrode Loading ($mg/cm^2$) | ≤ 0.20[3] | 0.19[2] | Perf., Durability, Loading |
| Decay Rate (µV/hr) | ≤ 4[3] | 4[2] | |
| Stack Power Density ($W/cm^2$) | ≥ 7.6 | Not started (BP2) | At-Scale Validation |

1. Fabrication rates based on demonstrated rates extrapolated to one machine/year per process step, and are based on process model incorporating capacity factors.
2. Laboratory CCM with $0.19mg/cm^2$ 78wt% Ir/NSTF scale-up phase powder OER catalyst/electrode, $0.08mg/cm^2$ Pt/NSTF dispersed catalyst/electrode, 3M 800EW 100 micron membrane. $50cm^2$ cell, 80˚C, $2A/cm^2$. Wind VRE Protocol.
3. Project target assessed with $50cm^2$ cell, 80˚C, $2A/cm^2$, 3M 800EW 100 micron membrane, project wind variable renewable energy (VRE) protocol. Performance and durability milestones in stack derated to 1.735V and 5µV/hr.

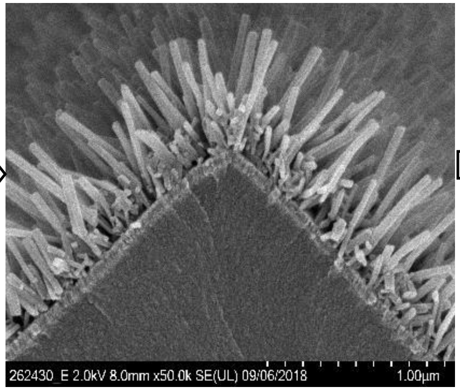
- Catalyst manufacturing steps demonstrated at equivalent to ≥ 1.5GW/year.
  - One step is overall rate-limiting, but good recent progress suggests imminent improvement.
- Performance and durability targets exceeded at target loading ($0.20mg_{Ir}/cm^2$) using scale-up catalyst process.
  - Ultimate project target requires demonstration with production-scale materials made at project target rate (BP3).
- Stack validation testing at Giner continuing; Plug Power ramping now.

# 2 of 4 Process Steps Demonstrated in Excess of Project Target Rate

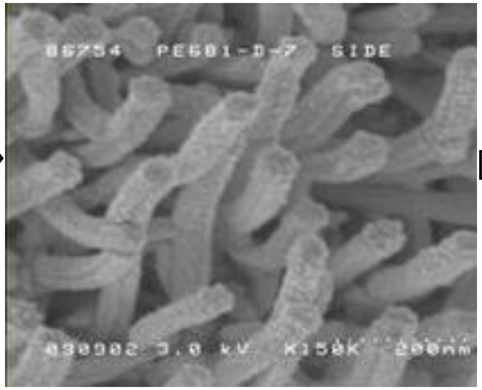
**1. Precursor Deposition**
**1.5 GW/year**



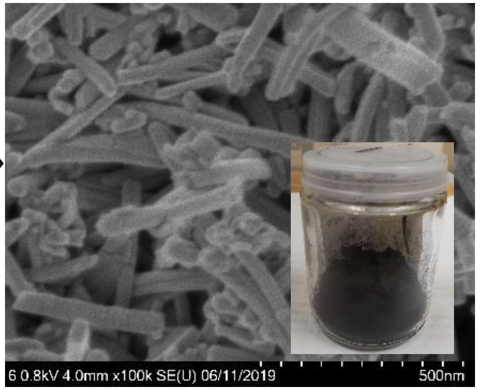

**2. Support Growth**
**> 3.75GW/year**




**3. Catalyst Deposition**
**2.5GW/year**



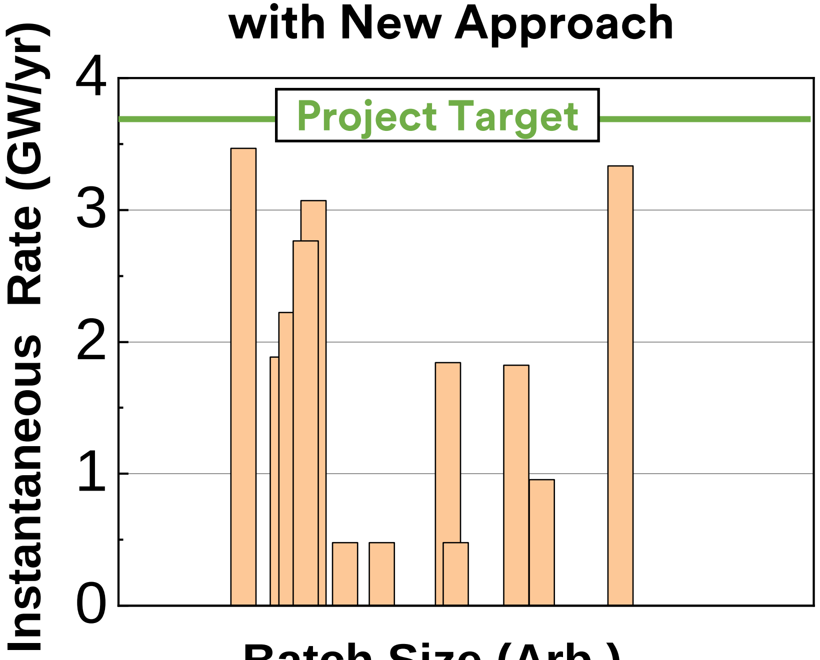
**4. Catalyst Powder**
**> 3.75GW/year**



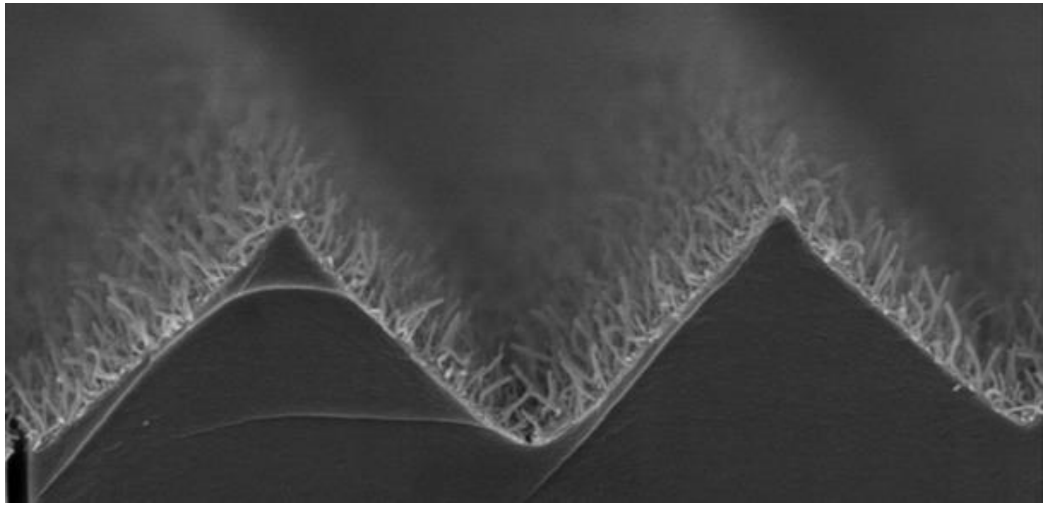

- Overall catalyst process consists of 4 high level process steps.
- Key process focus of budget period 2 has been design and implementation of new precursor and catalyst deposition steps (1 and 3) to enable achievement of project targets.
- 3 of 4 process steps demonstrated above BP2 GNG (2GW/yr) and 2 above project target rate (3.75GW/year) .
  - Precursor deposition requires further development to achieve targets, but is rapidly improving.

# New Precursor Deposition Approach Enables High Rates

**High Precursor Deposition Rates Achieved with New Approach**



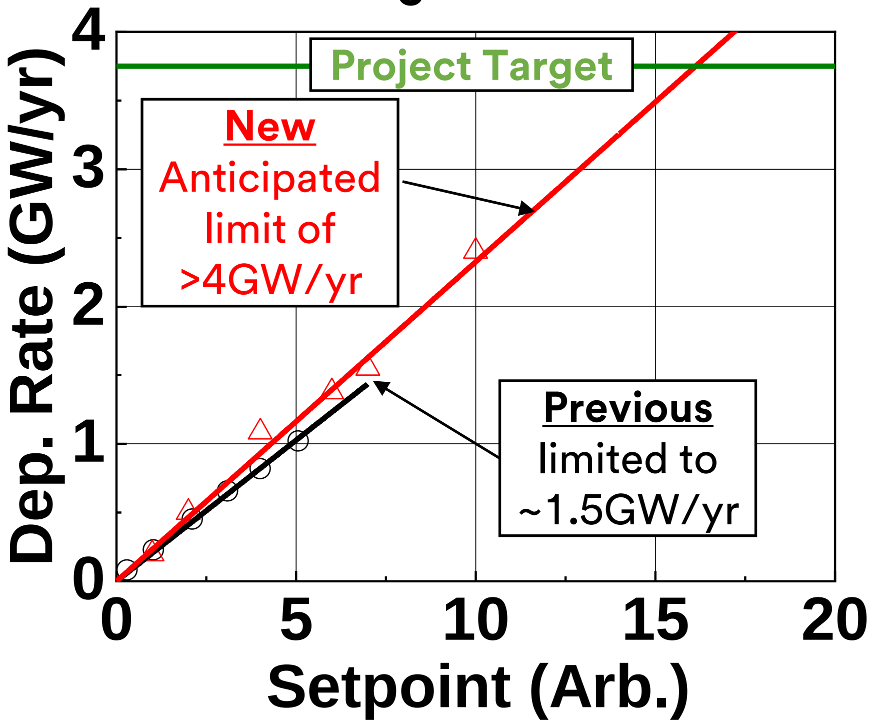

**Whisker Supports Generated from Precursor Films Produced at 2 GW/year Equivalent Rate**



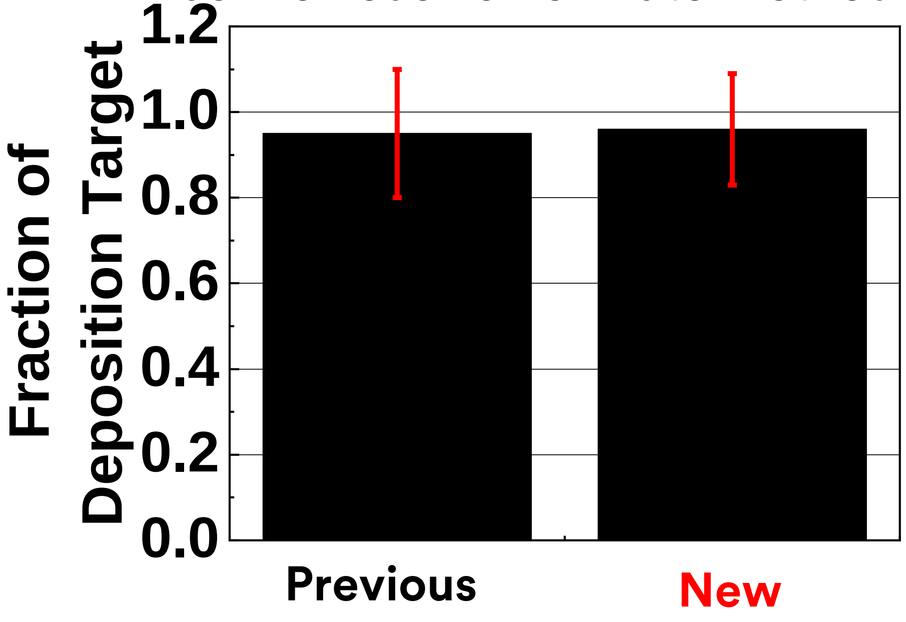
- New precursor deposition approach enables high-rate manufacturing (in excess of 3 GW/year, 3x baseline), but does not yet meet batch size requirements.
- Fabrication rate and batch size depends strongly on process conditions.
- High-rate precursor films convert into support whiskers with typical morphology and performance.
- Current focus: optimize conditions to enable increased batch sizes towards the final project target.

# New Catalyst Deposition Also High Rate Capable

## New Ir Deposition Approach Achieves High Rates



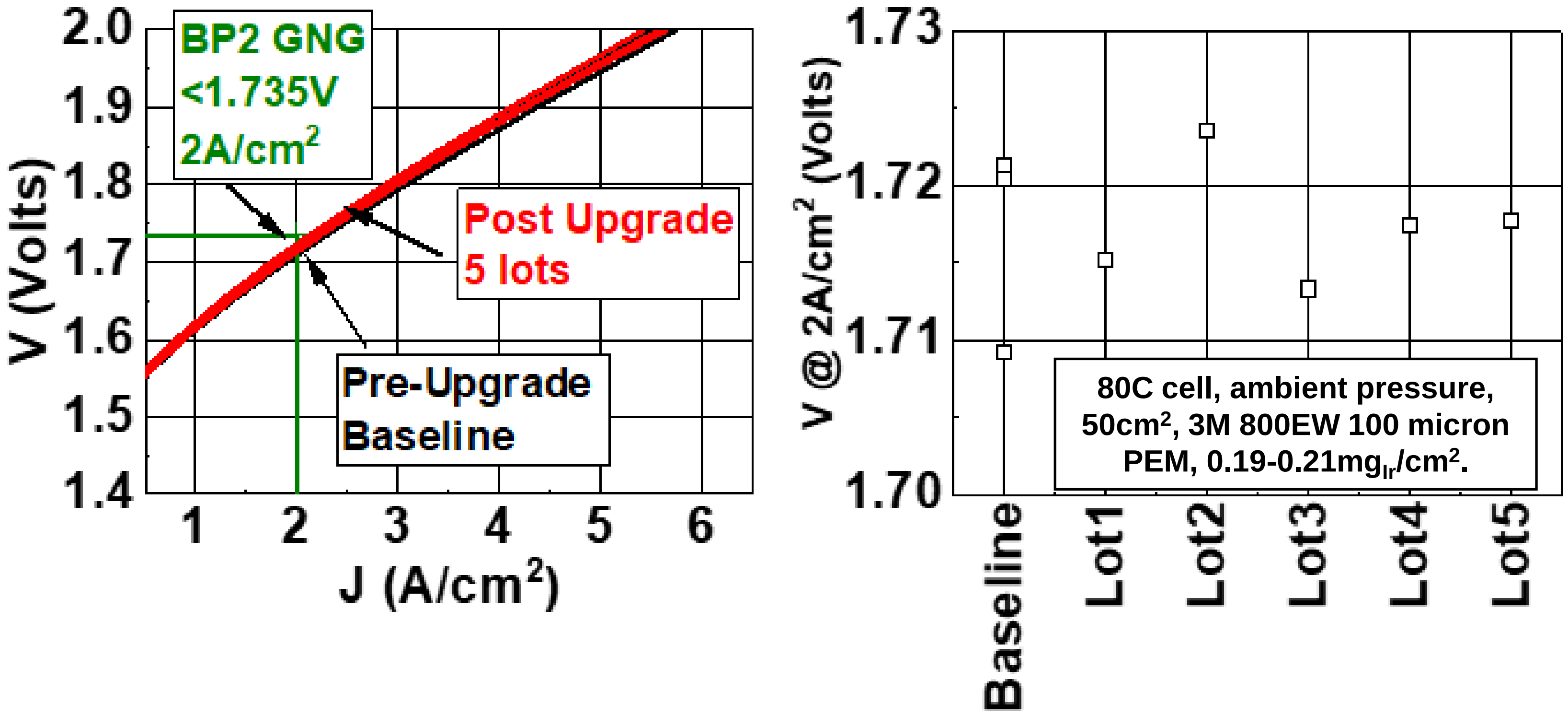

## New High-Rate Approach Has Similar Uniformity as Previous Lower-Rate Method




- New catalyst deposition approach demonstrated at > 2x rate of previous source, and with similar uniformity.
- No known barriers to achieving 3.75 GW/year project target – plan to demonstrate in BP3.

# Scale-up Catalyst Meets Performance Targets

## PEMWE Cell Tests of Baseline and Post-Upgrade Catalyst Lots




- 5 lots of project 78wt% catalyst generated after deposition approach upgrades.
- Performances largely match or exceed performance of pre-upgrade catalyst and meet project performance and loading targets.

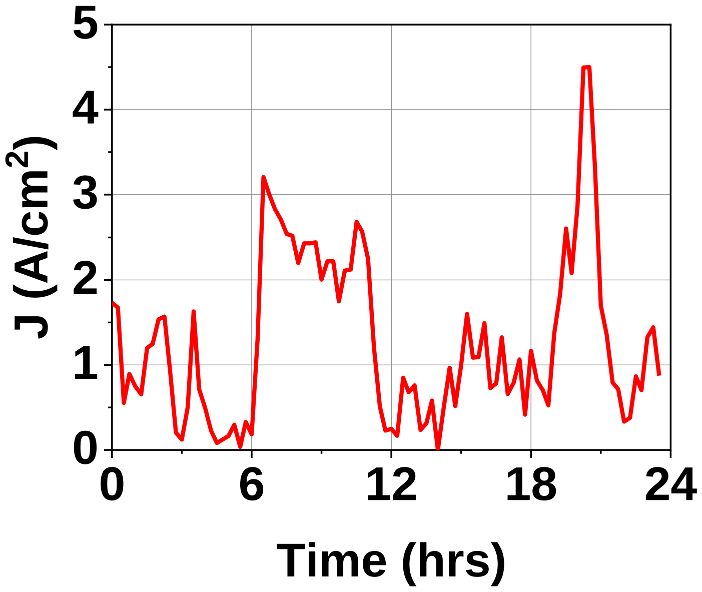
# Project Materials Assessed Against Three Accelerated Stress Tests

## 1. Project Durability Target Protocol

**Variable Renewable Energy ("VRE")**

500hrs x (24hr wind simulation, 0-4.5A/cm²)

Diag. pol. curves every 100 hours



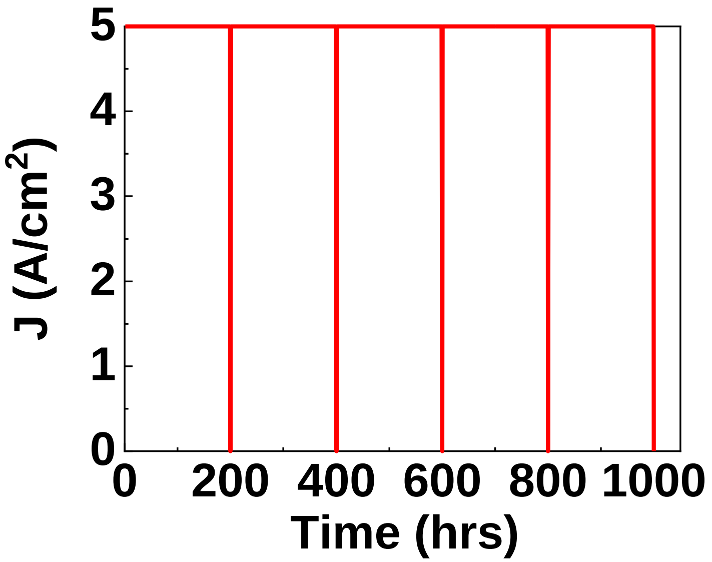

- Simulates direct integration to wind turbine; from Alia et al. (2019), scaled to 4.5A/cm².

## 2. High Power Steady State

**Diagnostic/Comparative**

1000+ hours at 5A/cm²

Diag. pol. curves every 200 hours



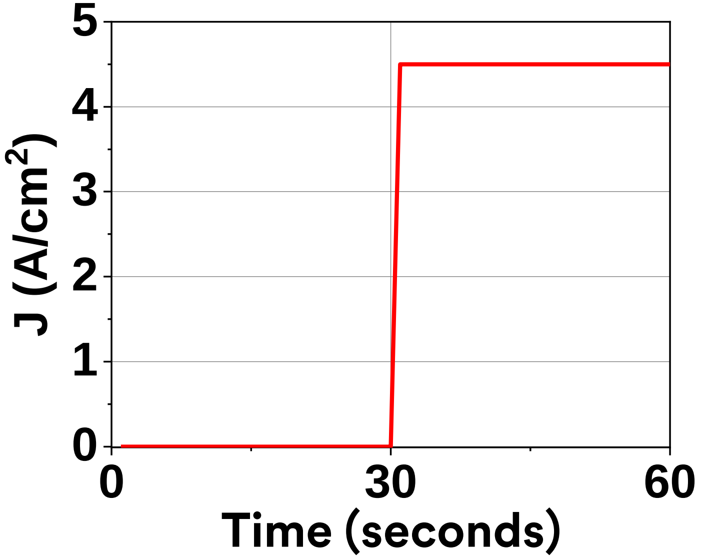

- Very limited cycling.
- >2x higher average power.

## 3. Highly Accelerated Stress Test

**Rapid On/Off Cycling ("HAST")**

30k cycles (30s 4.5A/cm², 30s off); 500hrs

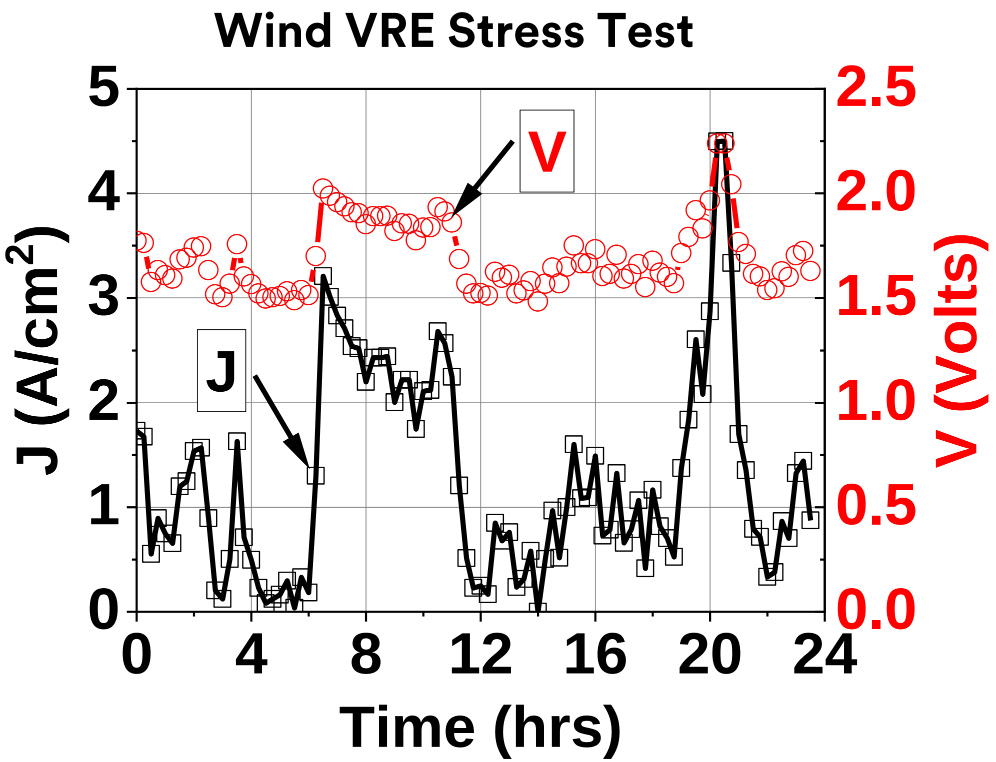
Diag. pol. curves every 100 hours

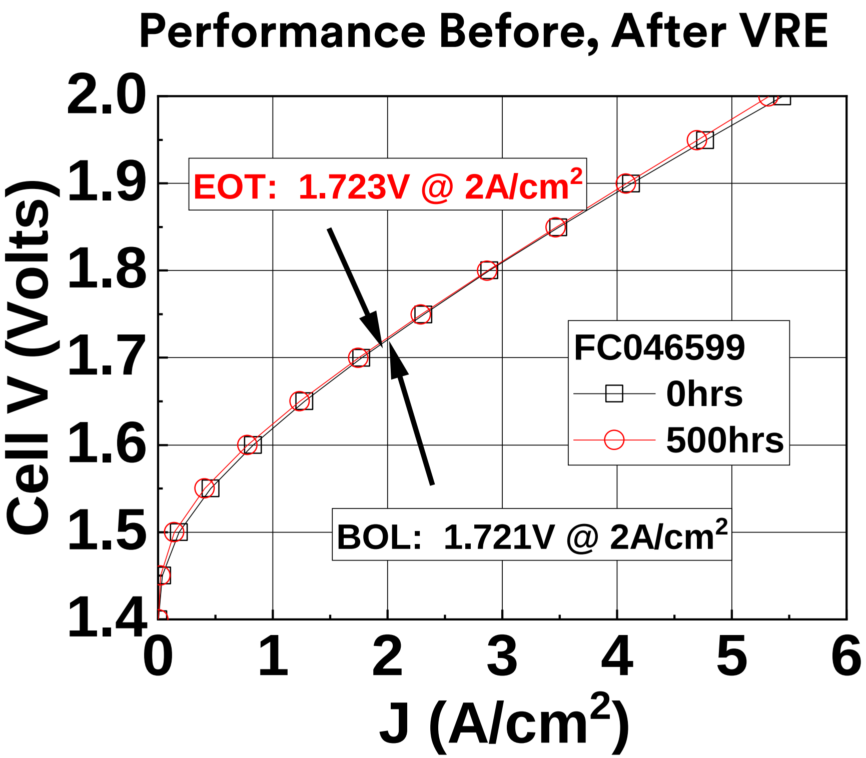



- (Ideally) enable larger degradation and elucidate differences more quickly.

**Common conditions (3M): 80˚C cell, ambient pressure, 75mL/min, 50cm² cells**

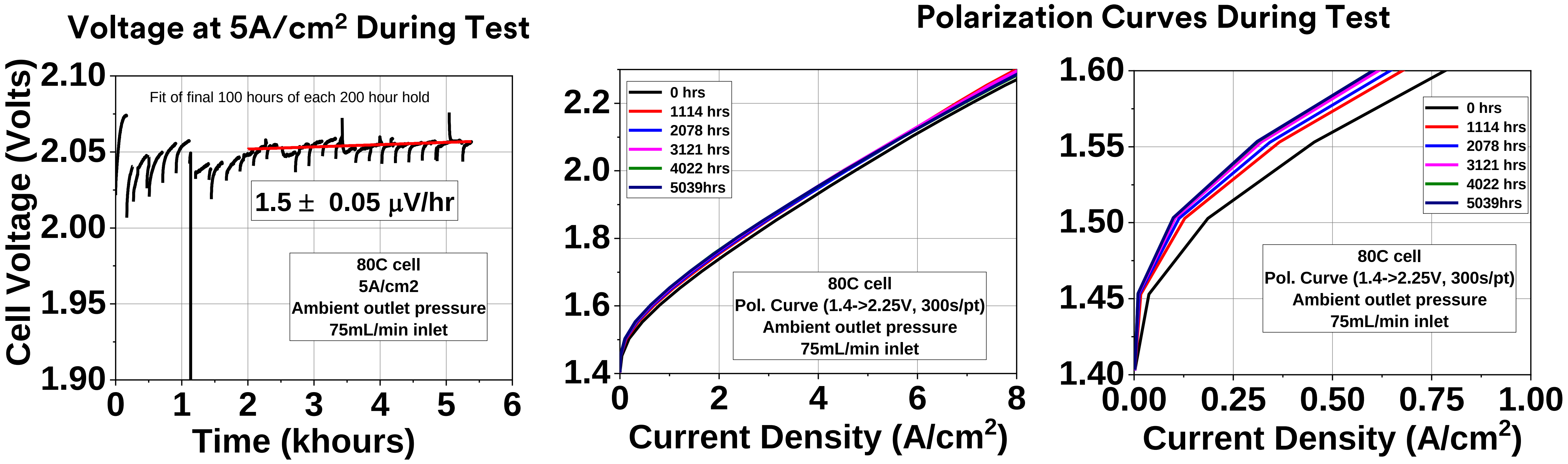
# Scale-up Phase Catalyst Meets Performance, Durability Targets




- Post-upgrade catalyst lot tested for durability under project wind VRE durability protocol.
- 2mV voltage increase at 2A/cm$^2$ over 500 hour test → 4μV/hr.
- Scale-up catalyst achieves project performance, durability, and loading targets.

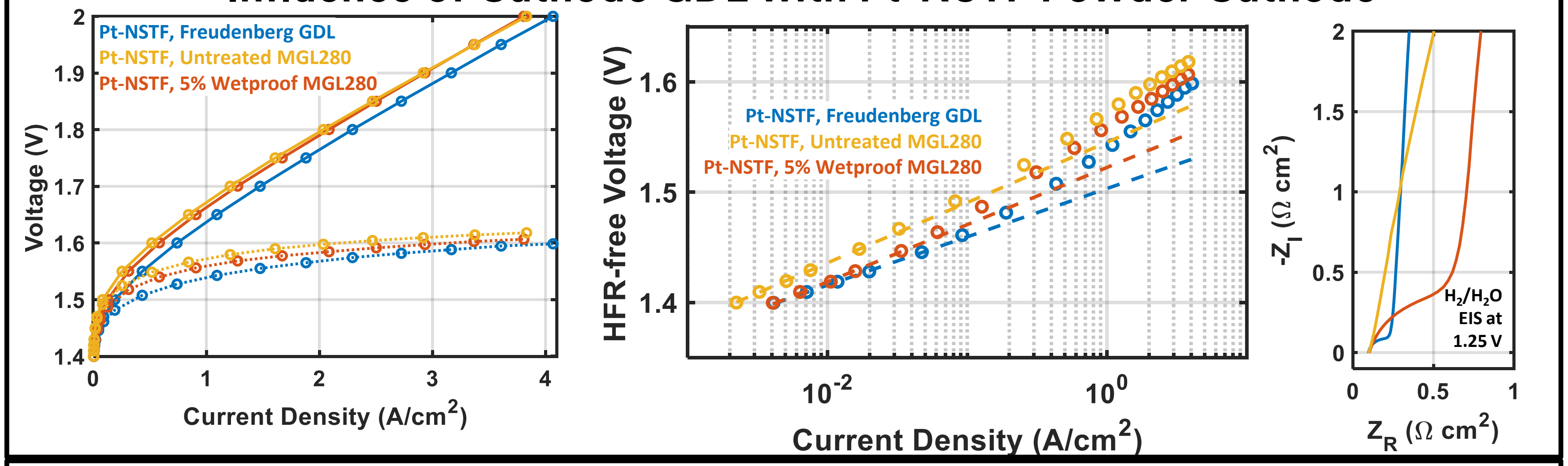
# Catalyst Lifetime Test – High Stability for 5300 hours at 5A/cm$^2$



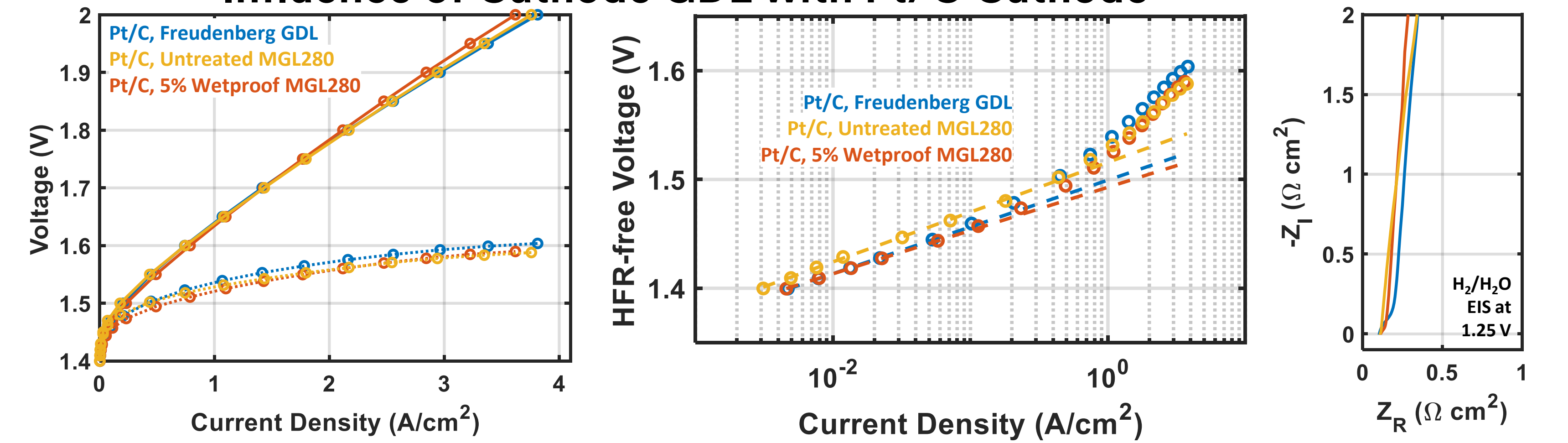

- 5A/cm$^2$ steady state lifetime test conducted with project 78wt% catalyst at 0.25mg$_{Ir}$/cm$^2$ loading.
- Steady state decay rate at 5A/cm$^2$ over final 3300 hours of test: 1.5 μV/hr.
- Polarization curves reveal primary loss was OER kinetics; no significant changes in transport or other loss modes.
- Minimal HFR change over course of test – 87 (at BOT) to 85 mOhm-cm$^2$ (EOT) (see backup).
- Test halted due to station priority – cell/MEA still healthy at end.

# Significant Influence of Cathode on NSTF Anode Activity, Utilization

## Influence of Cathode GDL with Pt-NSTF Powder Cathode




## Influence of Cathode GDL with Pt/C Cathode

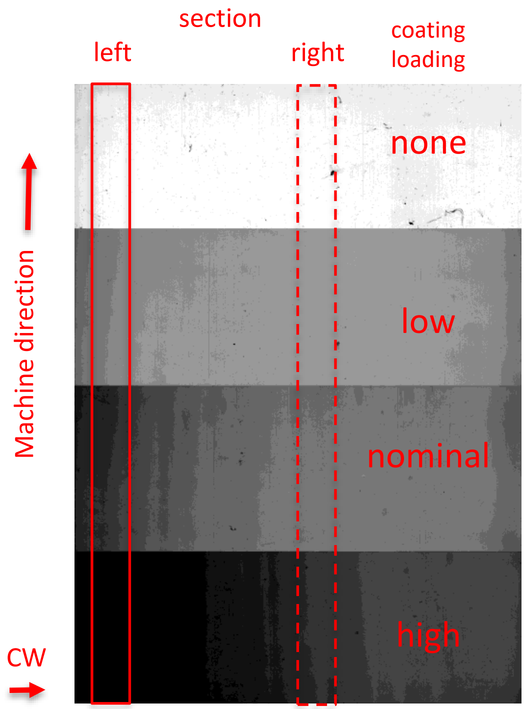



- **Objective:** Improve Ir-NSTF anode catalyst utilization.
- Both cathode GDL and cathode catalyst influence anode activity, utilization.
- Cells with Pt-NSTF cathode can yield better performance (lower HFR) than with Pt/C cathode, but are more sensitive to cathode GDL properties.
  - Hydrophilicity of cathode?
- Studies continue to elucidate mechanism.

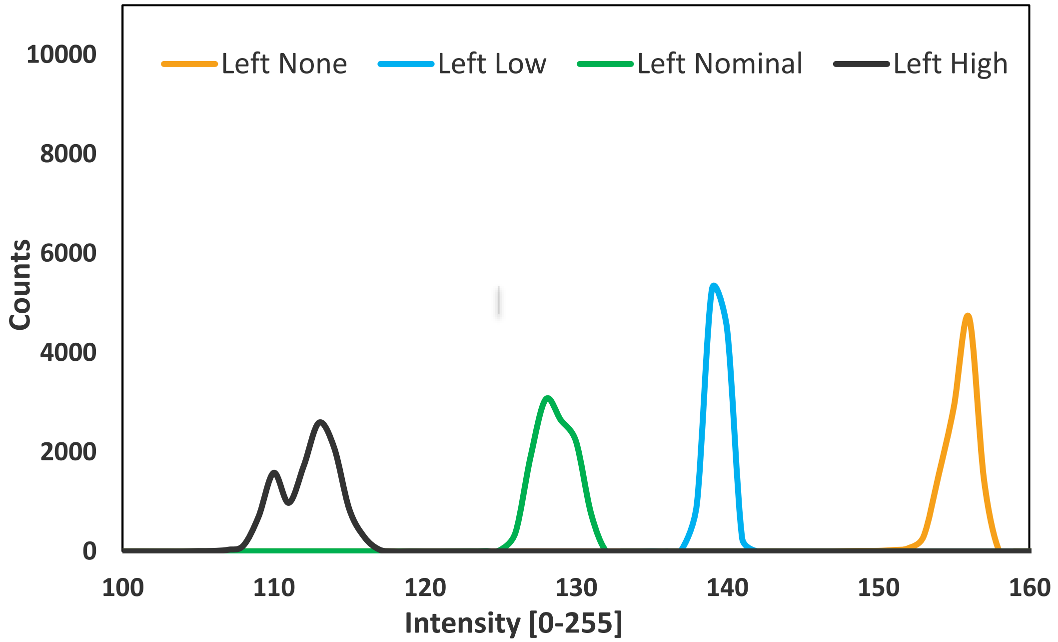
**80C, 5cm². Anode: Ir-NSTF 0.25mg/cm². Cathode: x. PEM: Nafion 115**

# Quality Control Tool Development for Catalyst Precursor

## White Line Transmittance Maps Of Precursor Films with Different Loadings




## Transmittance Intensity vs. Precursor Loading




- NREL is developing QC tool towards potential in-line thickness mapping of deposited precursor films.
- White light transmittance detects differences in precursor films with no coating and 50, 100, and 150% loadings.
- Correlation with actual thicknesses in progress.

# Ir-NSTF Successfully Integrated with Nafion Electrode Ionomer

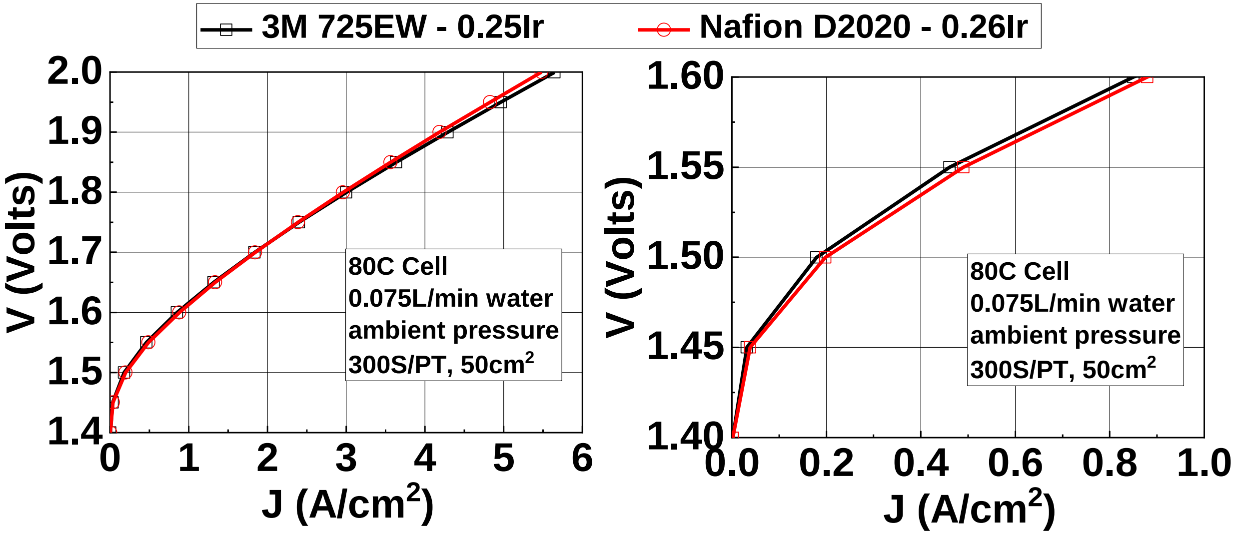
## Formulation Dev. Enables High Quality Electrodes

Decreasing $H_2O$ Content (50% $H_2O$ to 10% $H_2O$)


*3M Ir-NSTF, Nafion D2020 ionomer, 20 wt% catalyst, 3M substrate*


Coating is highly uniform to 10s of microns with no mud-cracking.

## Performance with 3M 725EW vs. Nafion D2020




- **Motivation:** Confirm Ir-NSTF is compatible with Nafion D2020 ionomer.
  1. High quality coatings achieved via solvent development.
  2. Minimal performance impact of switch from 3M 725EW PFSA to Nafion (1000EW).
- Next steps: Assess durability.

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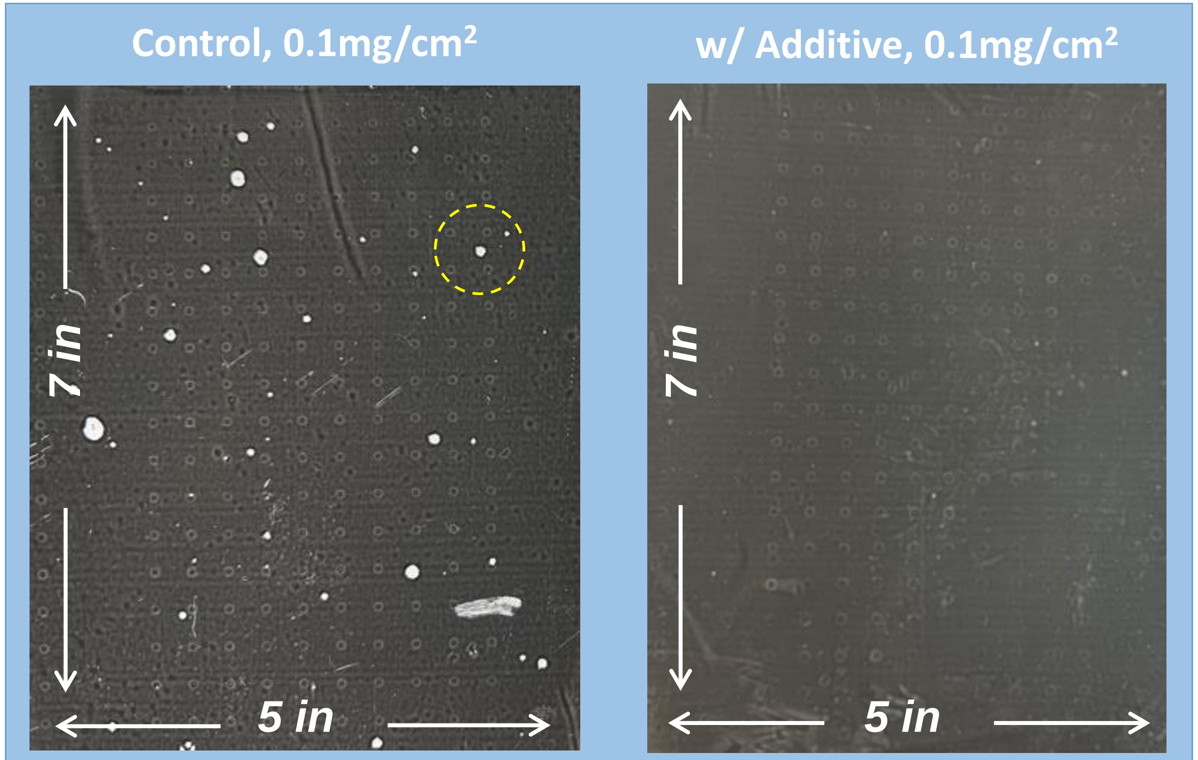
NREL Transforming ENERGY

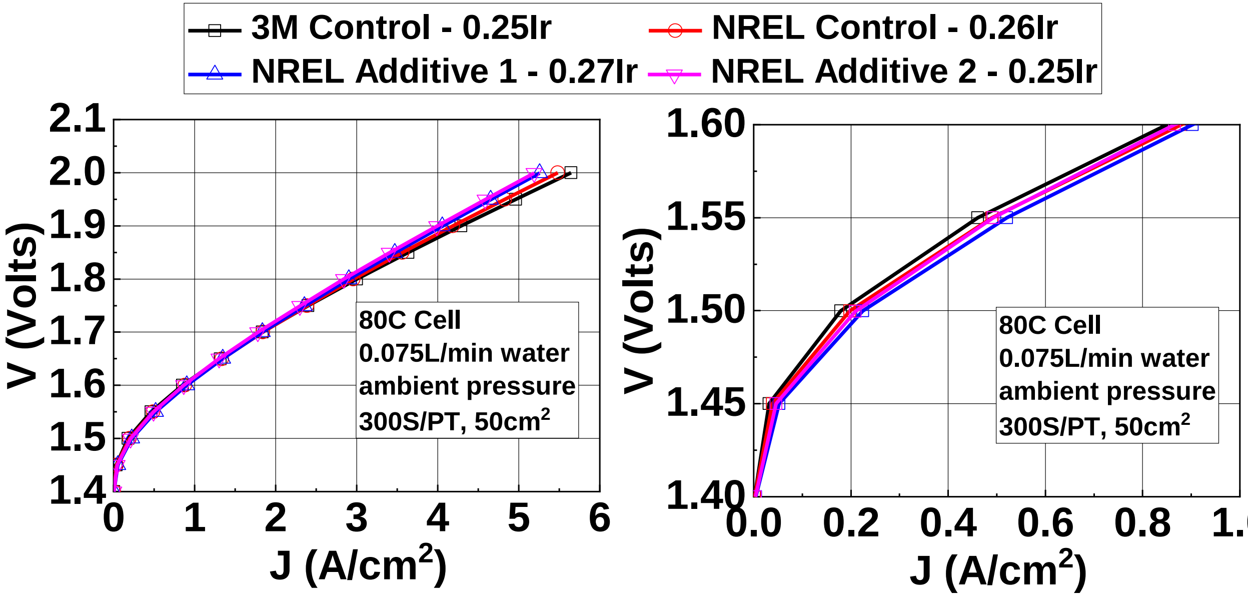
# Electrode Additives Improve Uniformity with Low Performance Impact

## Additives Greatly Improve Electrode Uniformity with Ultra-low Ir-NSTF loadings (~0.1 $mg_{Ir}/cm^2$)




*3M Ir-NSTF, Nafion D2020 ionomer, 3M substrate*

## Cell Performance of Coatings with Additives




- **Motivation:** Significant defects at low loadings may negatively impact performance and durability.
- Electrode additive yields similar performance as controls with moderate Ir loadings.
- Next steps: Assess durability and performance at ultra-low loadings.

# Team Spans Industry and National Laboratories

## Collaborations

- **3M - Component Process Development, Durability, Inspection Development**
  - A. Steinbach (PI), G. Thoma, F. Sun, Y. Liu, M. Kurkowski, J. Phipps, A. Goethel, S. Myers, T. Shefelbine, J. Bender, J. Varjabedian, K. Jochem, C. Rock, C. Foley, L. Allen-Olson
- **National Renewable Energy Laboratory – Electrode and QC Development; Durability**
  - P. Rupnowski, S. Mauger, E. Padgett, C. Baez-Cotto, I. Berry
- **Giner, Inc. – Sub-scale Component Performance Validation; Durability**
  - N. Bencomo, S. Ding
- **Oak Ridge National Laboratory– Microscopy of Developmental and Durability Components**
  - D. Cullen, S. Reeves
- **Plug Power – Performance Validation, Stack Testing**
  - C. Mittelsteadt, Z. Green

- Acknowledgements to H. Dukes, J. Drumm, I. Stuyvenberg, D. Gilbert, D. Tarnowski

## Response to Reviewers' Comments

- This project was not reviewed last year.

# Project Challenges Are Addressable

| Remaining Challenges | Future Work |
|---|---|
| 1 of 4 catalyst process steps (precursor deposition) has not achieved the BP2 target. | Process optimization to improve batch size. |
| 2 of 4 process steps have not achieved project final target. | Catalyst deposition is likely feasible to achieve project target – simple modification required.<br>Precursor deposition – rate limiting factor identified; implement identified improvement pathways. |
| 3M exited from PFAS manufacturing; impacts project electrode, thrifted CCM technologies.<br>3M to Exit PFAS Manufacturing by the End of 2025 - Dec 20, 2022 | Modification in progress<br>• Electrode, thrifted CCM process development de-emphasized.<br>• CCMs for stack testing to be made at 3rd party.<br>• No-cost extension |
| Stack power density target not yet demonstrated. | Anticipate demonstrating in Giner short stacks in ‘24; Plug Power stack in BP3. |

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

# Summary

- Project catalyst process development has advanced; goals achievable.
  - 2 of 4 steps demonstrated above project targets
  - New catalyst metal deposition approach yields significant improvement; target believed feasible.
  - New catalyst precursor deposition approach enables high-rates exceeding 3 GW/year, but further work needed to achieve requisite batch sizes.

- Project catalyst/electrodes are highly durable under variety of aggressive protocols
  - Scale-up catalyst meets project performance and durability targets at $0.2 mg_{Ir}/cm^2$
  - Low decay rates demonstrated at $5 A/cm^2$ for > 5000 hours with $0.25 mg_{Ir}/cm^2$

- Improved understanding of factors influencing anode catalyst utilization in low loading electrodes.
  - Significant and unanticipated influence of cathode catalyst/electrode and GDL.

- Project Ir/NSTF catalyst powder successfully integrated with Nafion ionomer – high quality electrodes with expected performance.

# Safety Planning and Culture

## Project was required to submit a hydrogen safety plan

- 3M provided a revised plan based on feedback; further revision requested and is in progress.

## Management of Incidents and Near Misses

- 3M policy requires all incidents or near misses to be reported, documented, investigated with a team, and communicated to employees.
- Incidents at specific severity requires specific investigation tools and communications, globally
- Bench marking is performed for companies who have similar incidents to ensure the risk is mitigated within 3M

## Best Practices/Lessons Learned

- Involve EHS early
- Lessons learned are presented to campus though a safety committee which allows EHS to cascade and implement changes where needed in current processes

## Prioritization of Safety: Risk Identification, Hazard Analysis, Risk Management through Mitigation

- Risk Identification:
  - Maintenance Conscious Engineering (MCE): Equipment installations are thoroughly reviewed by Maintenance and Engineering through design, installing, and the equipment and process and maintaining equipment post installation.
- Hazard Analysis, Risk Management and Mitigation:
  - Management of Change (MOC): Formal documentation addressing scope of work, required for all changes at 3M. Includes categorization of risk, identifying continuous improvement for all programs involved in the proposed change, and determining what is needed for risk mitigation. Review and approval is needed for all changes that are not like-for-like.
  - MOC process involves experts from a technical standpoint, researchers, EHS, Engineering, and topic experts for a collaborative approach.