Energy Materials Network
U.S. Department of Energy

HydroGEN
Advanced Water Splitting Materials

HydroGEN: Low-Temperature Electrolysis (LTE) and LTE/Hybrid Supernode

G. Bender and H.N. Dinh

Presenter: Guido Bender, NREL

Date: 5/20/2020

Venue: 2020 DOE Annual Merit Review

Project ID # P148C

NREL National Renewable Energy Laboratory · Berkeley Lab · Sandia National Laboratories · INL Idaho National Laboratory · Lawrence Livermore National Laboratory · SRNL

Advanced Water-Splitting Materials (AWSM)

Relevance, Overall Objective, and Impact

**AWSM Consortium
6 Core Labs:**

NREL NATIONAL RENEWABLE ENERGY LABORATORY

BERKELEY LAB

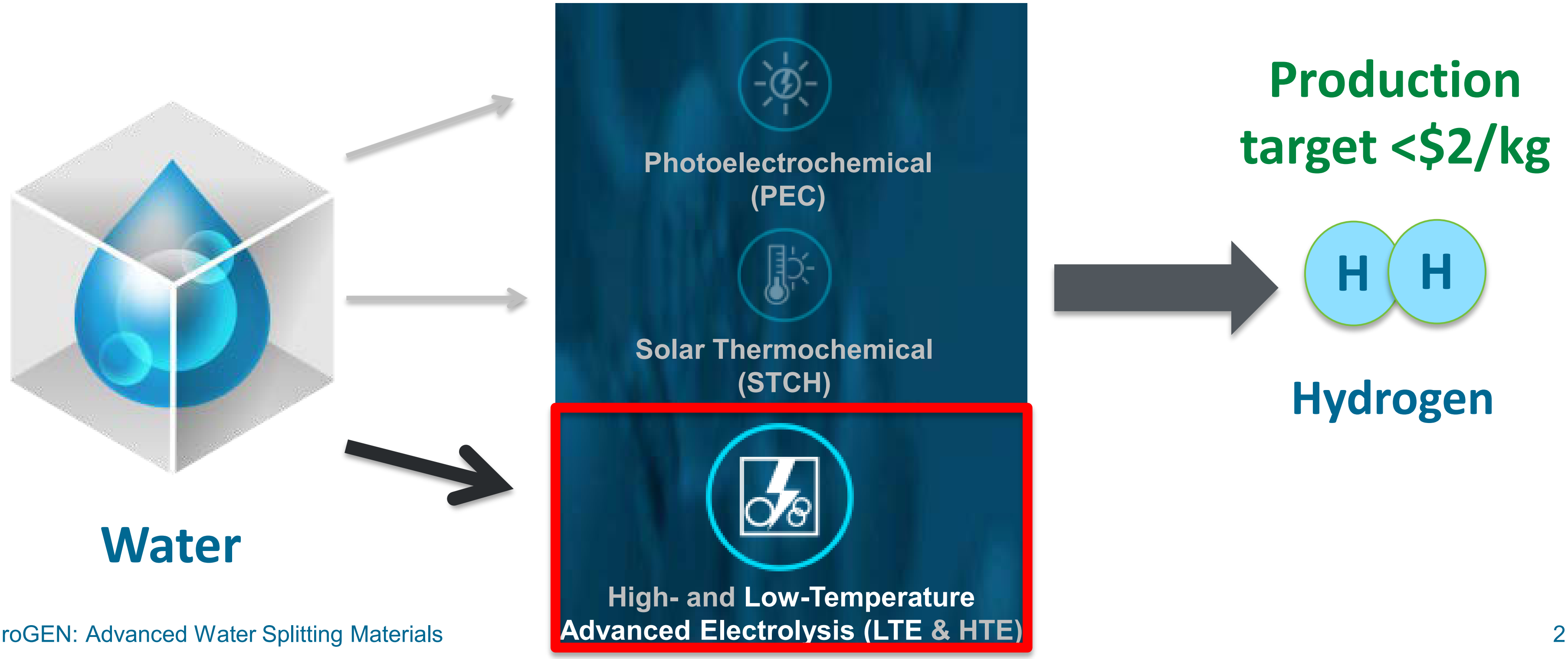
Sandia National Laboratories

INL Idaho National Laboratory

Lawrence Livermore National Laboratory

SRNL

Accelerating R&D of innovative materials critical to advanced water splitting technologies for clean, sustainable & low cost H_2 production, including:



Water

Photoelectrochemical (PEC)

Solar Thermochemical (STCH)

High- and Low-Temperature Advanced Electrolysis (LTE & HTE)

Production target <$2/kg

Hydrogen

Overview - LTE Technology Relevance / Impact

PEM

- Gas Crossover
- Membranes
- Catalyst Materials
- Catalyst Loading
- PTL Materials

AEM

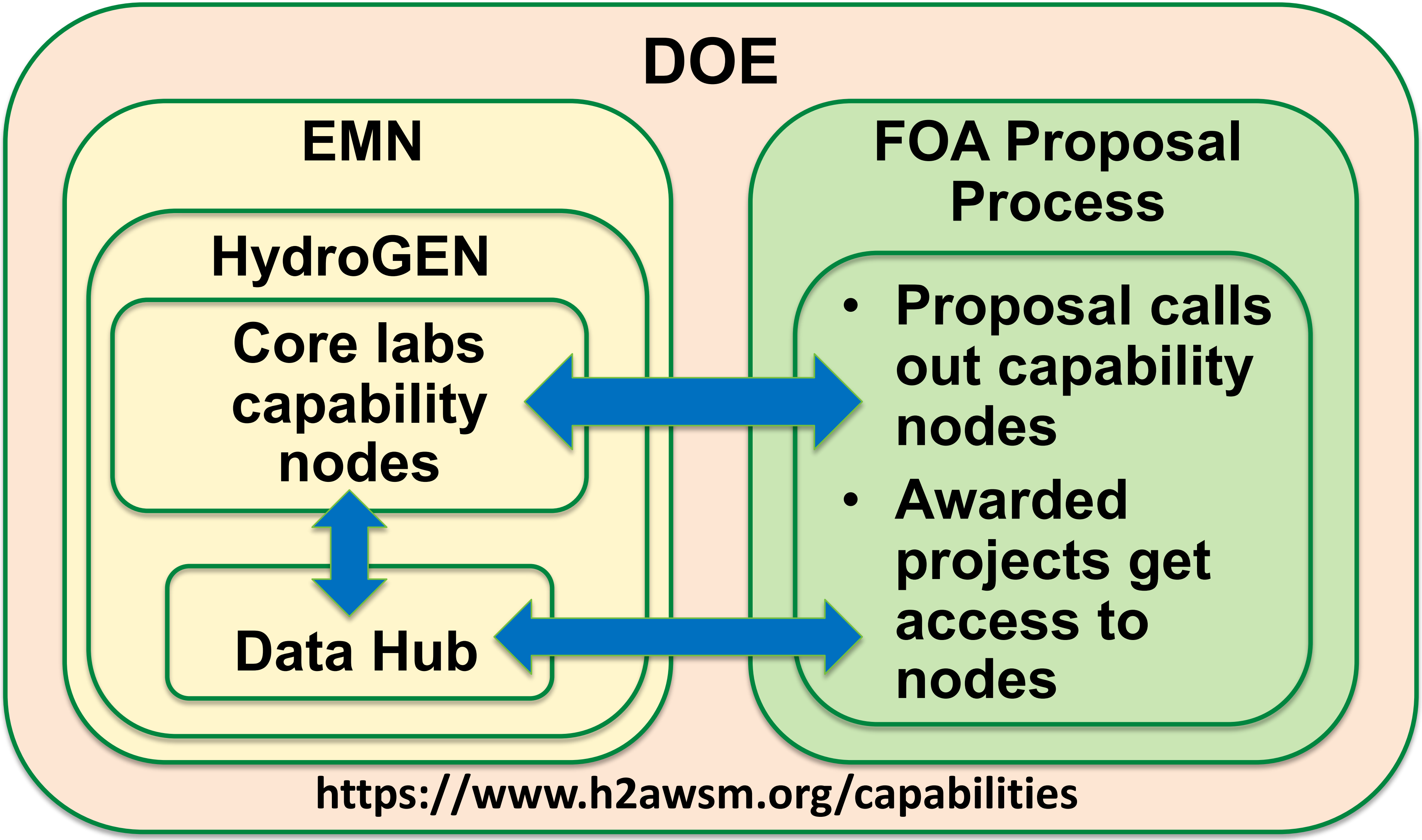
- Membranes
- Catalyst
- Ionomer
- Electrolyte feed required?
- BOP Materials

Common Barriers

- Material Integration
- Material Cost
- Understanding Interfaces and Interactions

BOP – Balance of Plant
PTL – Porous Transport Layers

Approach – HydroGEN EMN



DOE

EMN

HydroGEN

Core labs capability nodes

Data Hub

FOA Proposal Process

- Proposal calls out capability nodes
- Awarded projects get access to nodes

https://www.h2awsm.org/capabilities

Approach – HydroGEN EMN

Low Temperature Electrolysis (LTE)
- Proton Exchange Membrane (PEM)
- Alkaline Exchange Membrane (AEM)

Barriers
- Cost
- Efficiency
- Durability

LTE Node Labs

NREL
NATIONAL RENEWABLE ENERGY LABORATORY

BERKELEY LAB

Sandia
National
Laboratories

Lawrence Livermore
National Laboratory

Support through:

Personnel
Equipment
Expertise
Capability
Materials
Data

LTE FOA Projects

nel

Argonne
NATIONAL LABORATORY

Northeastern University
Center for Renewable Energy Technology

Los Alamos
NATIONAL LABORATORY (2)

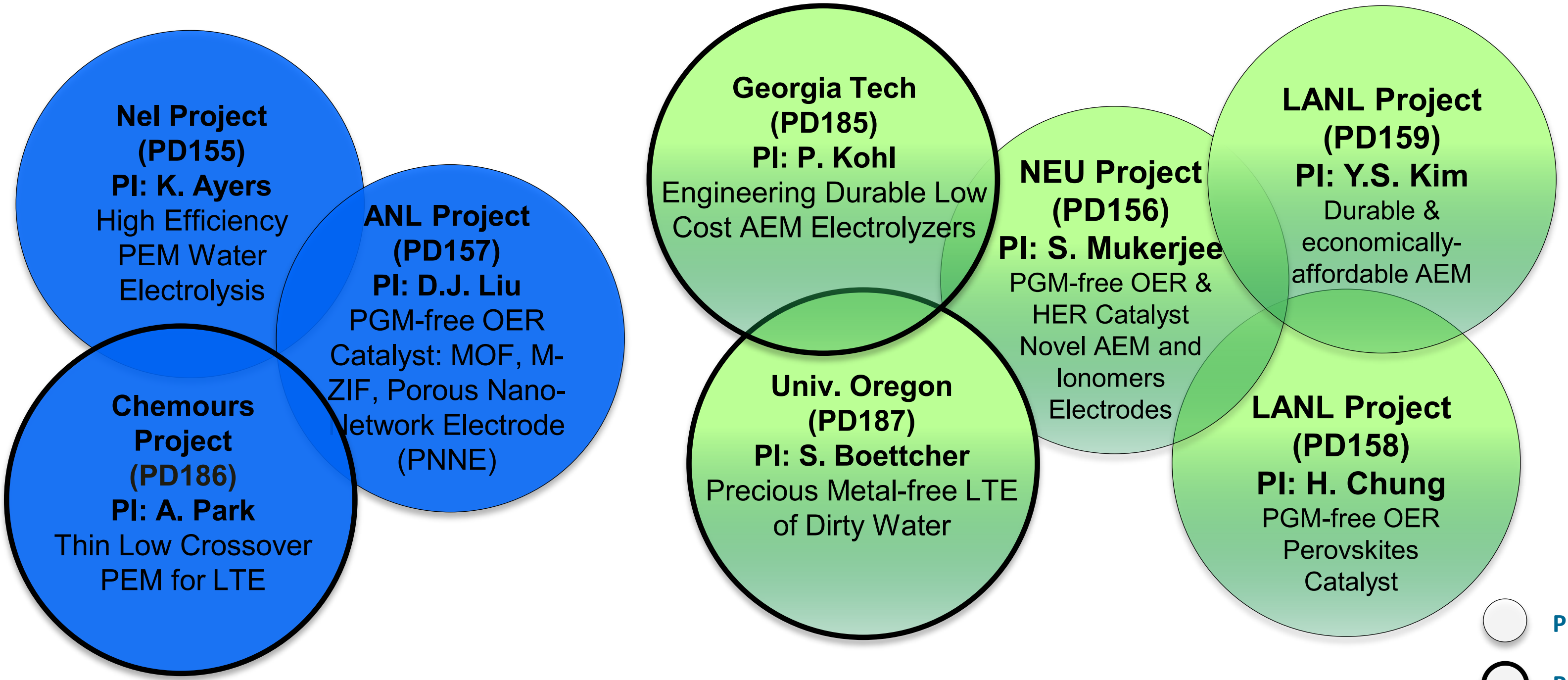
Georgia Institute of Technology

O

Chemours™

UNIVERSITY OF
OREGON

Accomplishments and Progress:
3x Seedling Projects Added to LTE Activities



Nel Project (PD155)
PI: K. Ayers
High Efficiency PEM Water Electrolysis

ANL Project (PD157)
PI: D.J. Liu
PGM-free OER Catalyst: MOF, M-ZIF, Porous Nano-Network Electrode (PNNE)

Chemours Project (PD186)
PI: A. Park
Thin Low Crossover PEM for LTE

Georgia Tech (PD185)
PI: P. Kohl
Engineering Durable Low Cost AEM Electrolyzers

NEU Project (PD156)
PI: S. Mukerjee
PGM-free OER & HER Catalyst Novel AEM and Ionomers Electrodes

LANL Project (PD159)
PI: Y.S. Kim
Durable & economically-affordable AEM

Univ. Oregon (PD187)
PI: S. Boettcher
Precious Metal-free LTE of Dirty Water

LANL Project (PD158)
PI: H. Chung
PGM-free OER Perovskites Catalyst

○ **Projects added FY'18**

◯ **Projects added FY'20**

Supernodes	**A. Weber** **T. Ogitsu**	**Understanding OER Across pH Ranges Through Multiscale, Multi-Theory Modeling**	**Discussed in PEC, P148A**
	H. Dinh **B. Pivovar**	**Linking LTE/Hybrid Materials to Electrode Properties to Performance**	**Discussed here in LTE, P148C**

PEM: Understanding and improving materials

AEM: Developing and understanding materials

Accomplishments and Progress:
Node Utilization for Project Support

49 nodes requests for LTE

19 nodes used by LTE projects

<u>Node Classification</u>

27x	Characterization
10x	Computation
7x	Material Synthesis
5x	Process and Manufacturing Scale-Up

Collaboration and Coordination - Node Utilization

FY'20 Projects

Lab	Node	LTE Super	Chem	UO	GT	Nel	ANL	NEU	LANL 1	LANL 2
NREL	Data Hub									
LLNL	Computational Materials Diagnostics and Optimization						✓			
LBNL	DFT and Ab Initio Calculations						✓		✓	
LBNL	Multiscale Modeling	✓	✓		✓	✓		✓		✓
SNL	LAMMPS							✓		
NREL	Novel Membrane Fabrication	✓		✓		✓		✓		
SNL	Separators for Hydrogen Production			✓					✓	✓
NREL	Multi-Comp. Ink Development, High-Throughput Fabrication, & Scaling	✓			✓	✓	✓	✓		

Computation

Processing & Scale Up

Material Synthesis

Collaboration and Coordination - Node Utilization

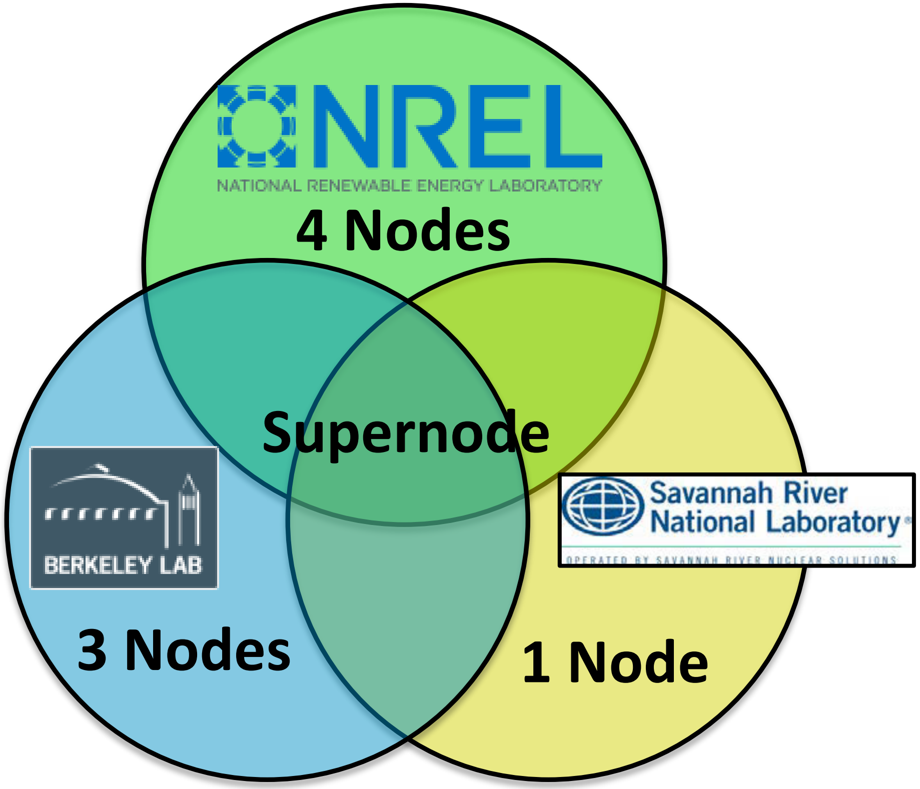
			FY’20 Projects							
Lab	**Node**	**LTE Super**	**Chem**	**UO**	**GT**	**Nel**	**ANL**	**NEU**	**LANL 1**	**LANL 2**
SNL	Advanced Electron Microscopy						✓			
NREL	Catalyst Synthesis, Ex situ Characterization & Standardization	✓				✓	✓			
LBNL	Ionomer Characterization and Understanding	✓	✓	✓		✓		✓		✓
NREL	In Situ Testing Capabilities	✓	✓	✓	✓	✓		✓	✓	✓
LBNL	Understanding Inks & Ionomer Disp.	✓		✓						
SNL	Near Ambient Pressure E-XPS								✓	
NREL	Surface Analysis Cluster Tool						✓		✓	
LBNL	Probing & Mitigating Corrosion					✓				
LBNL	PEC In Situ Testing using X-Rays								✓	
LBNL	Water Splitting Device Testing									✓
SRNL	Fabrication & Characterization of Electro-catalyst & Components for H2 Production	✓								

Characterization

Project Accomplishment
LTE Supernode

Supernode – Accelerate Science through Collaboration

LTE Supernode



4 Nodes

Supernode

3 Nodes

1 Node

Supernodes Objectives:

- Combine/integrate nodes to demonstrate value when connected (sum greater than combination of individual parts)
- Increase collaboration across core labs
- Provide core research for EMN labs, beyond just project support
- Phase 1 measurable objective: Confirm that ex-situ characterization approaches can be validated for their applicability to device performance and durability

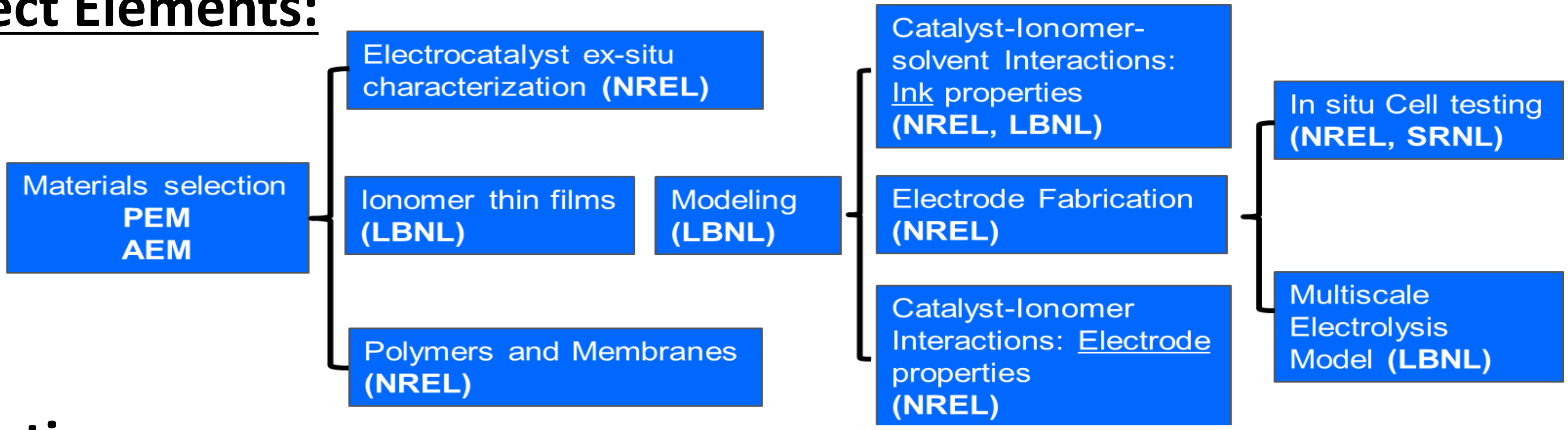
Linking LTE/Hybrid Materials to Electrode Properties to Performance

Supernode Goals

8 Nodes

Goals: Create true understanding between ex-situ and in-situ performance. Identify how material properties are linked to electrode properties and how these are linked to electrolyzer performance.

Project Elements:



Materials selection
PEM
AEM

Electrocatalyst ex-situ characterization **(NREL)**

Ionomer thin films **(LBNL)**

Modeling **(LBNL)**

Polymers and Membranes **(NREL)**

Catalyst-Ionomer-solvent Interactions: Ink properties **(NREL, LBNL)**

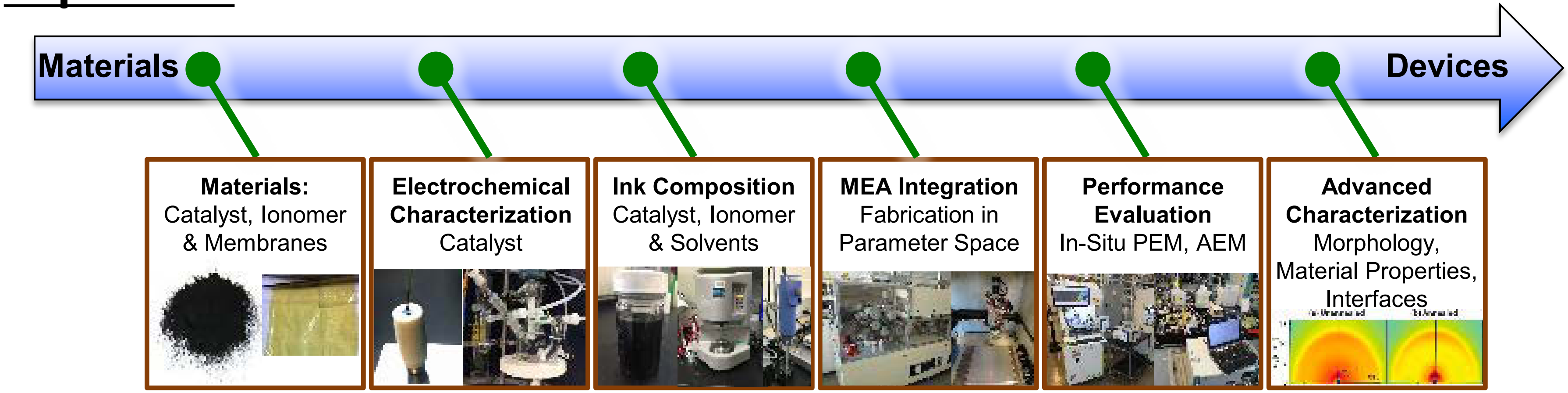
Electrode Fabrication **(NREL)**

Catalyst-Ionomer Interactions: Electrode properties **(NREL)**

In situ Cell testing **(NREL, SRNL)**

Multiscale Electrolysis Model **(LBNL)**

Expertise:



Materials: Catalyst, Ionomer & Membranes

Electrochemical Characterization Catalyst

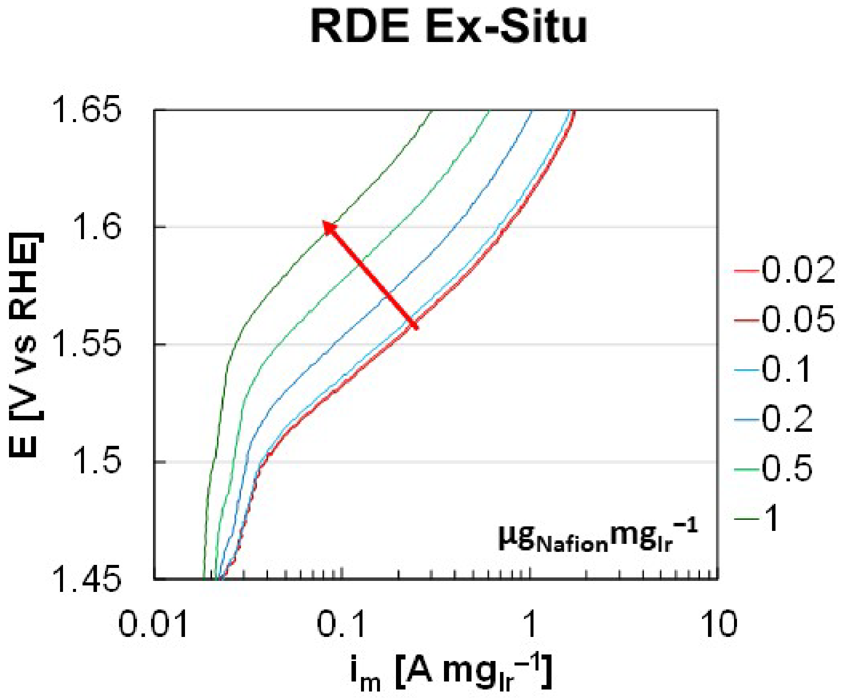
Ink Composition Catalyst, Ionomer & Solvents

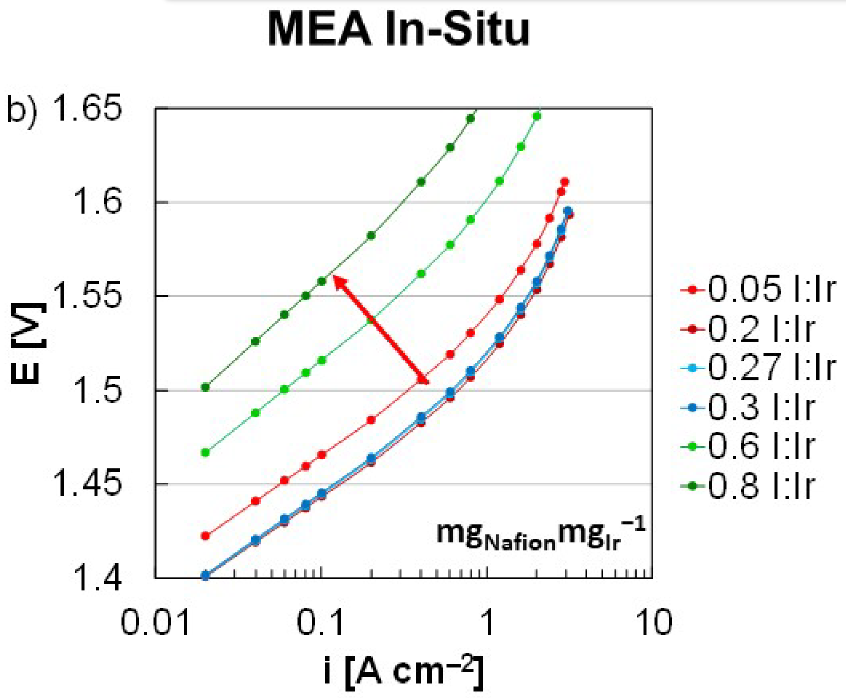
MEA Integration Fabrication in Parameter Space

Performance Evaluation In-Situ PEM, AEM

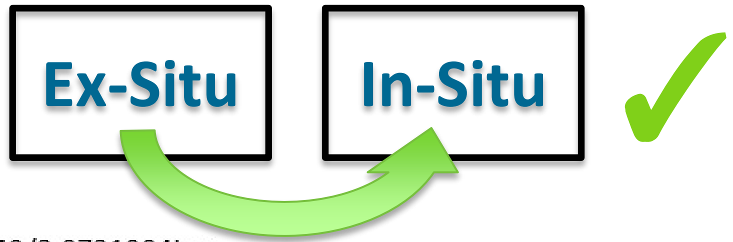
Advanced Characterization Morphology, Material Properties, Interfaces

Supernode Accomplishments: RDE/MEA Correlation





- **Correlation between RDE and MEA systems confirmed**
- **Same trend in RDE observed in MEA for effects of:**
  - **Catalyst loading**
  - **Ionomer content**
  - **Catalyst used**



Ex-Situ

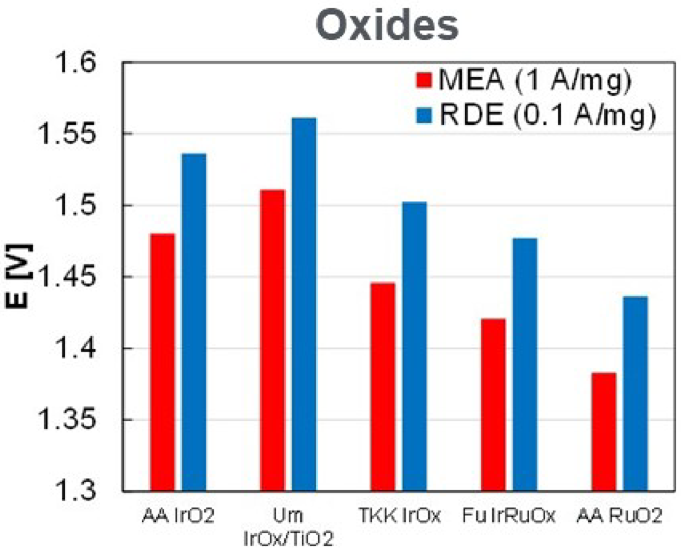
In-Situ

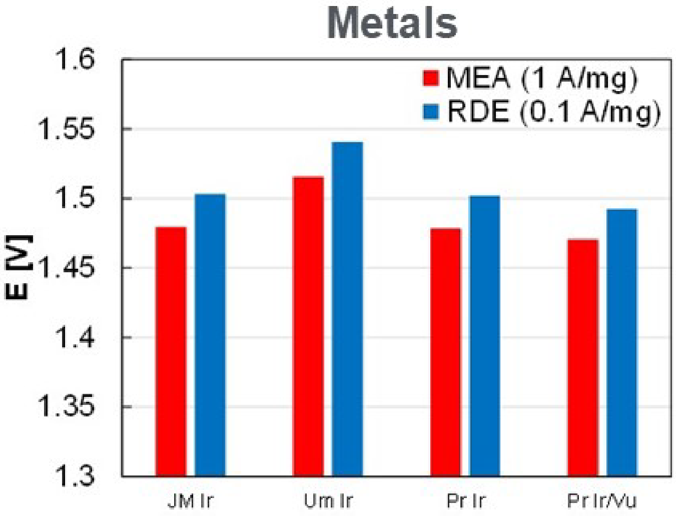
✓

S.M. Alia, G.C. Anderson, *J. Electrochem. Soc.*, **2019**, *166*(4), F282-F294. DOI:10.1149/2.0731904jes

S. M. Alia, S. Stariha and R. L. Borup, J. Electrochem. Soc., **2019**, 166(15), F1164. DOI: 10.1149/2.0231915jes

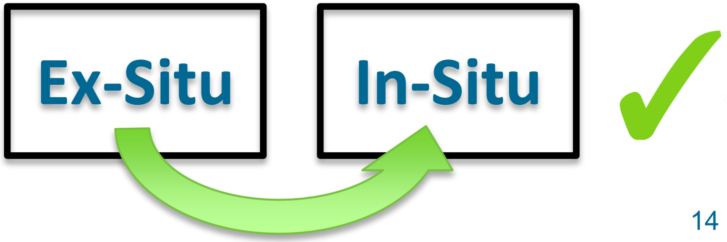
Supernode Accomplishments: Met Go/No Go Milestone by Correlating Ex- with In-situ Performance





		E^{MEA} [V]	ΔE [mV]	E^{RDE} [V]	ΔE [mV]	ΔE [%]
Alfa Aesar	IrO_2	1.480	–	1.536	–	–
Umicore	IrO_x/TiO_2	1.511	30.5	1.561	25.4	-16.7
TKK	IrO_x	1.446	-34.5	1.502	-33.6	-2.6
Furuya	$IrRuO_x$	1.421	-59.6	1.477	-58.8	-1.3
Alfa Aesar	RuO_2	1.383	-97.4	1.436	-99.5	2.2
Johnson Matthey	Ir	1.480	–	1.503	–	–
Umicore	Ir	1.516	36.0	1.541	37.4	3.9
Premetek	Ir	1.479	-1.1	1.502	-1.2	9.1
Premetek	Ir/Vu	1.471	-9.1	1.493	-10.6	16.5

LTE/Hybrid Supernode GNG: Demonstrate that the catalyst performance (overpotential in the kinetic region) measured via ex-situ RDE (at 0.1 A/mg) can be linked to in-situ MEA single cell performance (overpotential at 1 A/mg) within ± **20% for 5 commercial catalysts**. This success will demonstrate that **ex-situ RDE characterization**, which is simpler and quicker than in-situ MEA testing, **can be relevant and a good predictor of catalyst performance in the device**. As a result, the development of electrolysis material components can be accelerated.



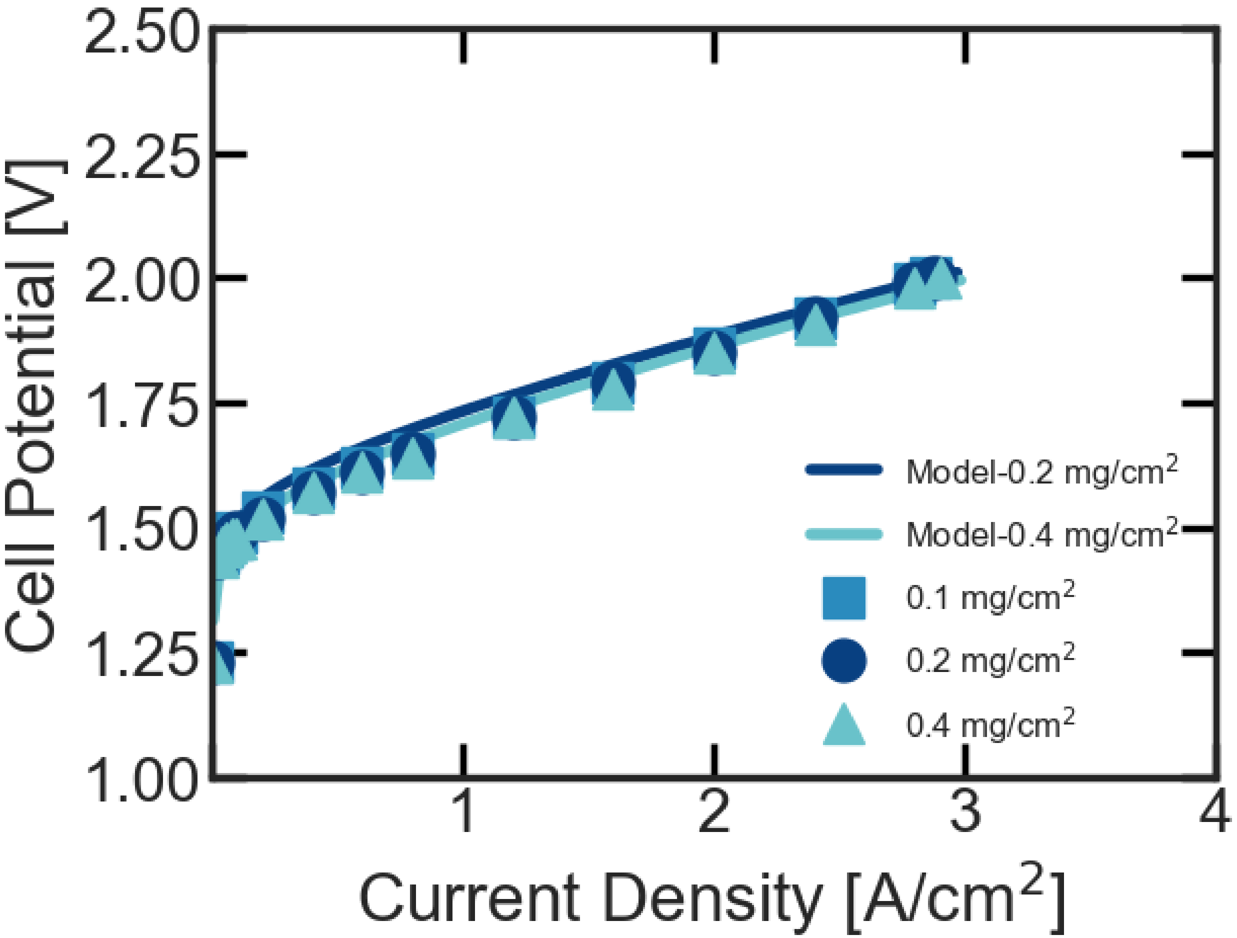
Ex-Situ

In-Situ

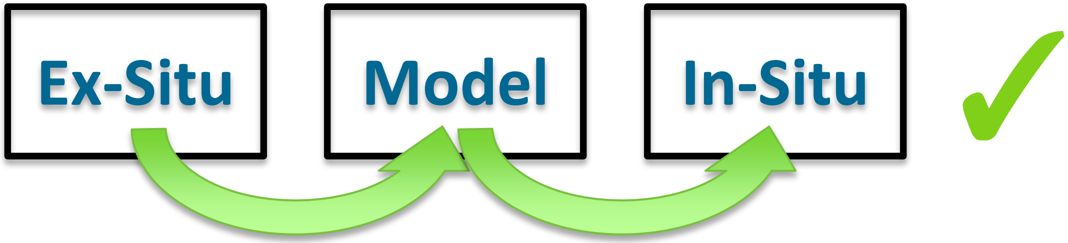
✓

S.M. Alia, M.-A. Ha, G.C. Anderson, C. Ngo, S. Pylypenko, R.E. Larsen, *J. Electrochem. Soc.*, **2019**, *166*(15), F1243-F1252. DOI:10.1149/2.0771915jes

Supernode Accomplishments: Multiscale Modeling Agrees with Experimental Data



- Kinetic results determined with *ex-situ* RDE were used as inputs into cell model
- Modeling results show good agreement with experimental *in-situ* results
- Minimal loading effect also reproduced by model



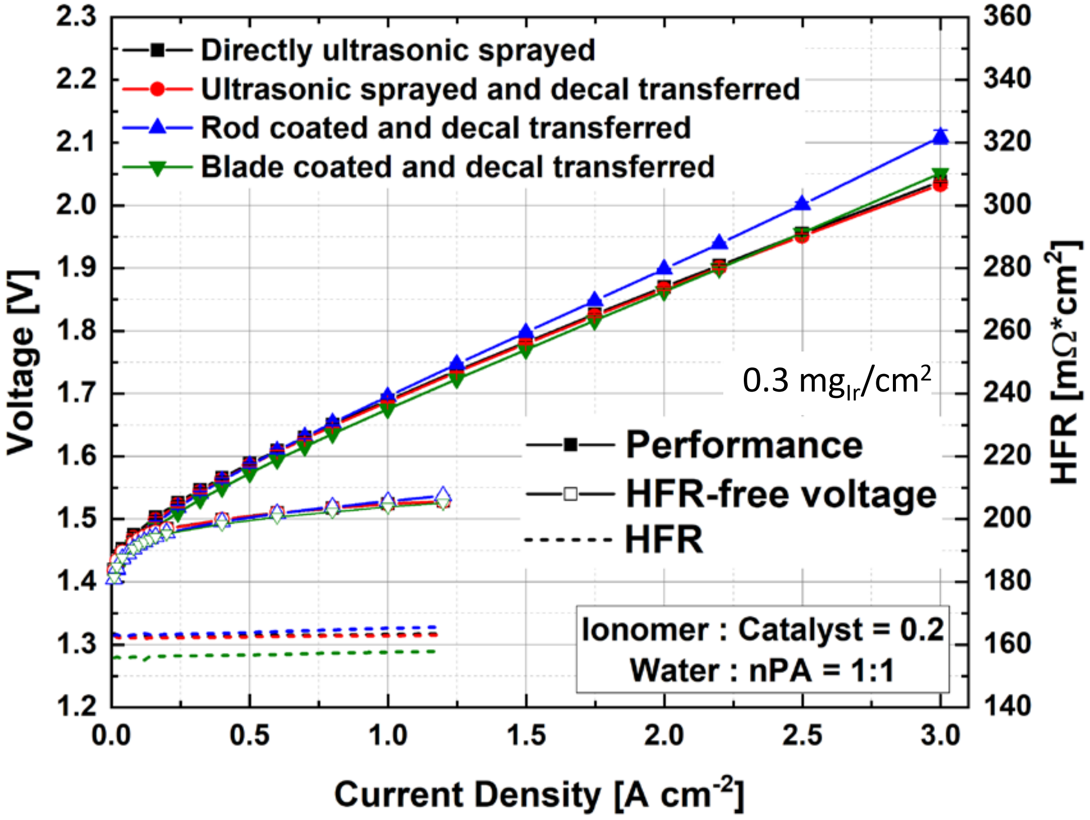
Ex-Situ

Model

In-Situ

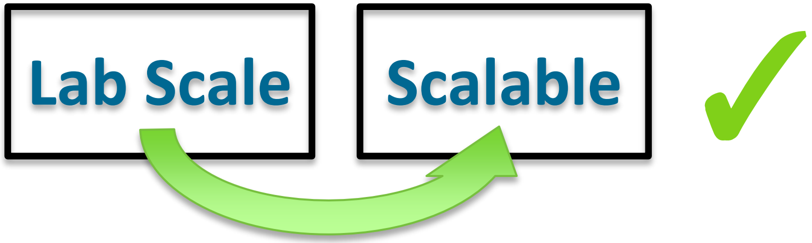
✓

Supernode Accomplishments: Doctor Blade and Mayer Rod Comparisons



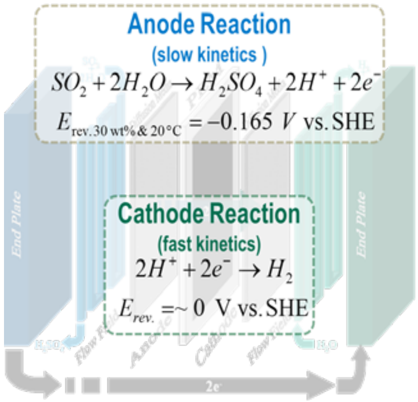
- Demonstrated a wide range of loading possible using scalable coating methods
- Decal transfer does not limit performance compared to directly sprayed electrodes
- Doctor blade coated electrode performs better than rod coated
- **Scalable coating methods (doctor blade) show comparable performance to lab-scale coatings (ultrasonic spray)**

- Ionomer : catalyst ratio from 0.1 to 0.3 did not significantly impact performance for blade- or rod-coated methods.
- Ink composition found to be less impactful at higher loading. Thicker catalyst layers may mask nonuniformities.

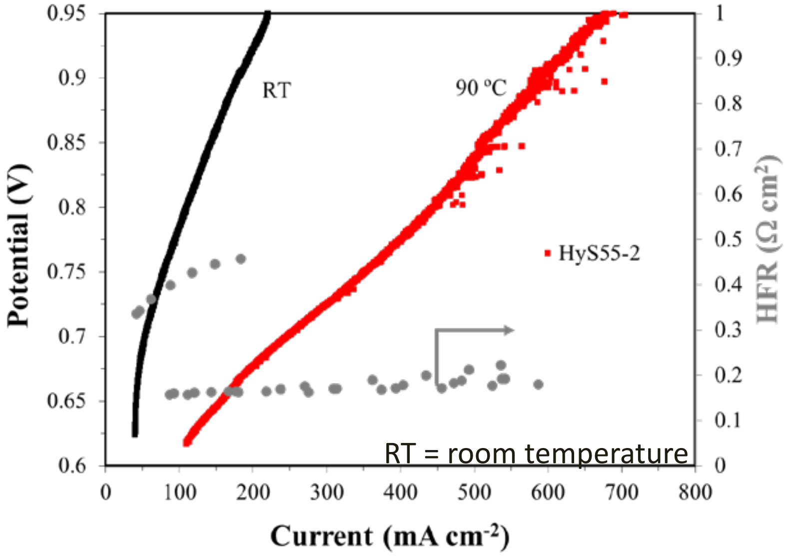


Supernode Accomplishments: Hybrid Cycle Correlating Ex- & In-situ Testing

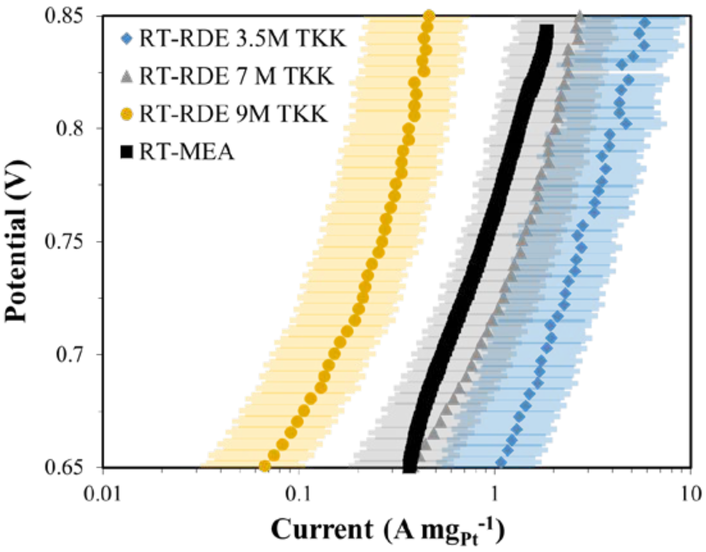
Hybrid Cycle



MEA testing



RDE vs MEA testing

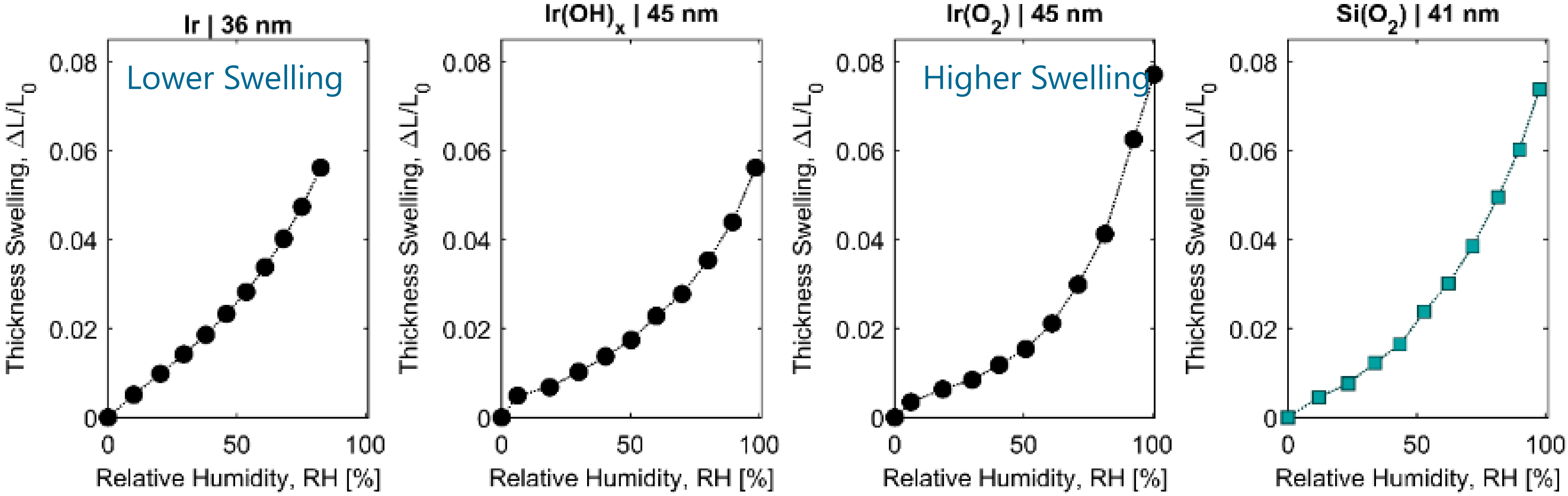


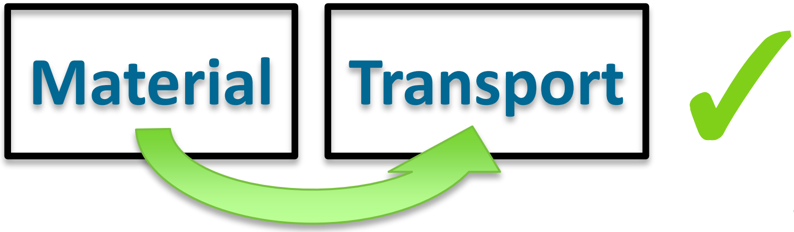
- Met Go/No Go milestone criteria of 50 % agreement between RDE and MEA testing (indicated by shaded area)
- Good agreement between in-situ and ex-situ measurements at acid concentration of 7 M
- Performance sensitivity to coating method and catalyst ink formulation is similar to that observed in fuel cells, but different from LTE
- Sensitivities differ between gas fed and liquid fed systems

Ex-Situ → **In-Situ** ✓

Supernode Accomplishments: Thin Film Morphology: GISAXS

- **Swelling behavior** of 40 nm Nafion film on substrates studied
- Si(O$_2$) $\approx$ Ir(O$_2$) [Bulk Oxide] > Ir(OH) [functionalized] $\gtrsim$ Ir [Metal]
  - Metal oxide has lower swelling but comparable structure
  - Functionalized OH lower swelling but no phase-separated structure
  - Ir(O$_2$) is most similar to Si, both structurally and hydration wise





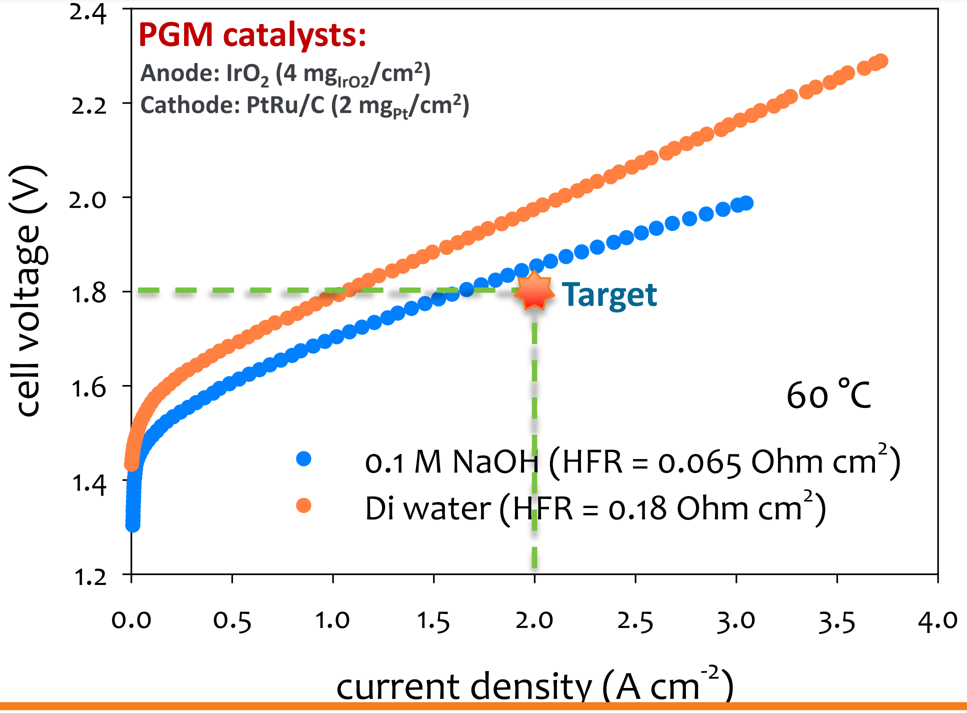
Project Accomplishment Summary Slides

5 Nodes

Scalable Elastomeric Membranes for Alkaline Water Electrolysis

Project Goal: Preparing durable and economically-affordable alkaline hydroxide conducting SES materials and demonstrating the high performance and durability in AEM-based water electrolysis

Highlight: The project team developed polystyrene based alkaline polymers that approach the 2020 target performance (2 A/cm² at 1.8 V) for AEM electrolyzer

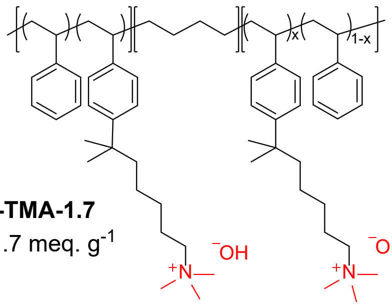


PGM catalysts:

Anode: IrO_2 (4 mg_{IrO2}/cm^2)

Cathode: PtRu/C (2 mg_{Pt}/cm^2)

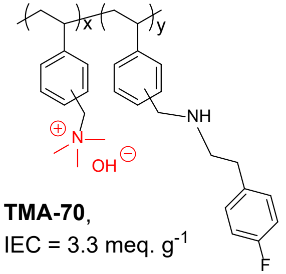
AEM (RPI)



SES25-TMA-1.7

IEC = 1.7 meq. g^{-1}

Ionomer (LANL)



TMA-70,

IEC = 3.3 meq. g^{-1}

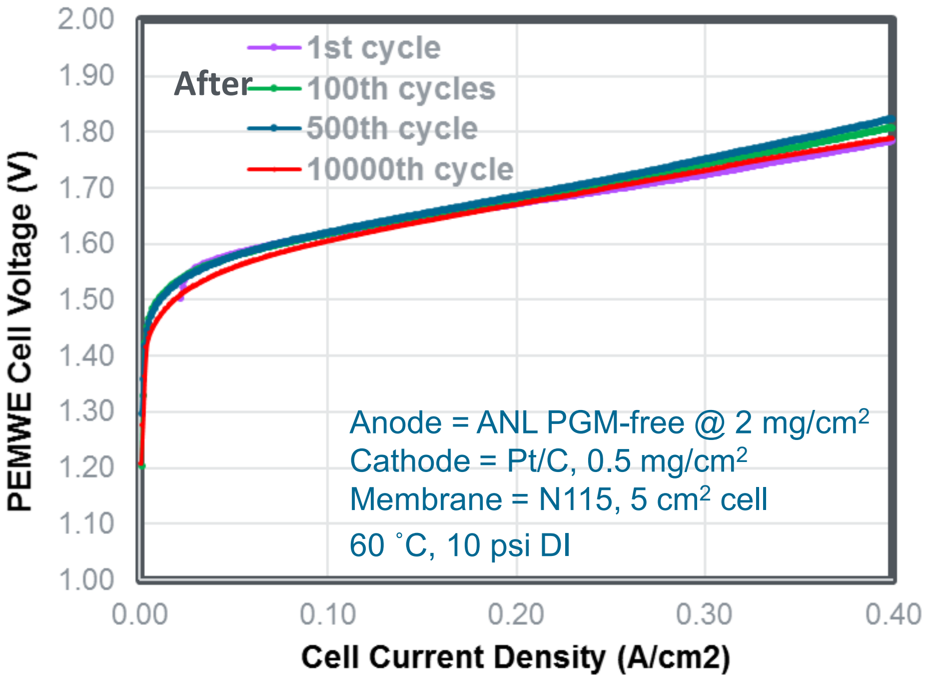
Y. Kim, PD159

PGM-free OER Catalysts for PEM Electrolyzer

5 Nodes

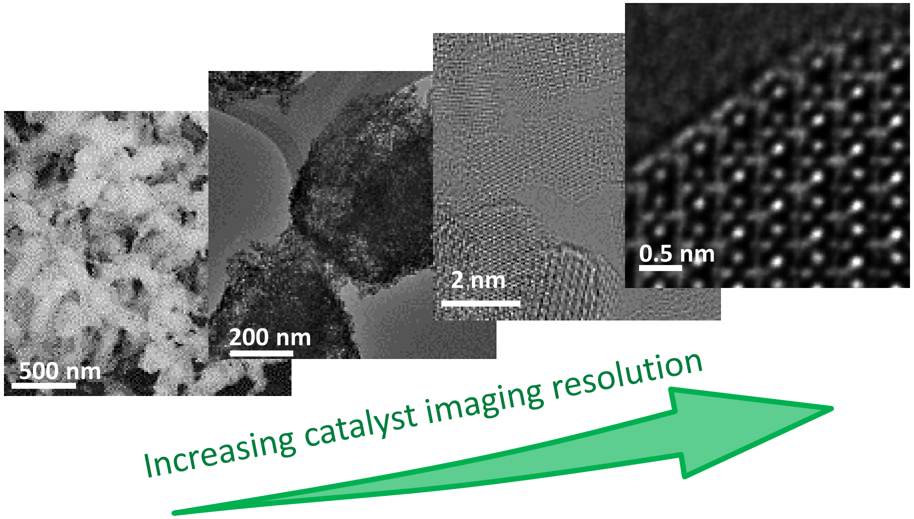
Project Goal: To develop platinum group metal-free (PGM-free) oxygen evolution reaction (OER) electro-catalysts as viable replacement for Ir in proton exchange membrane water electrolyzer (PEMWE)

Highlight: An ANL PGM-free OER catalyst demonstrated an unprecedented current density of 400 mA/cm^2 @ 1.8 V and stability over 10,000 voltage cycles in PEMWE.



Anode = ANL PGM-free @ 2 mg/cm^2
Cathode = Pt/C, 0.5 mg/cm^2
Membrane = N115, 5 cm^2 cell
60 ˚C, 10 psi DI

High resolution electron microscopy shows ANL PGM-free catalyst contains interconnected nanocrystallite aggregates with morphology similar to its MOF precursor



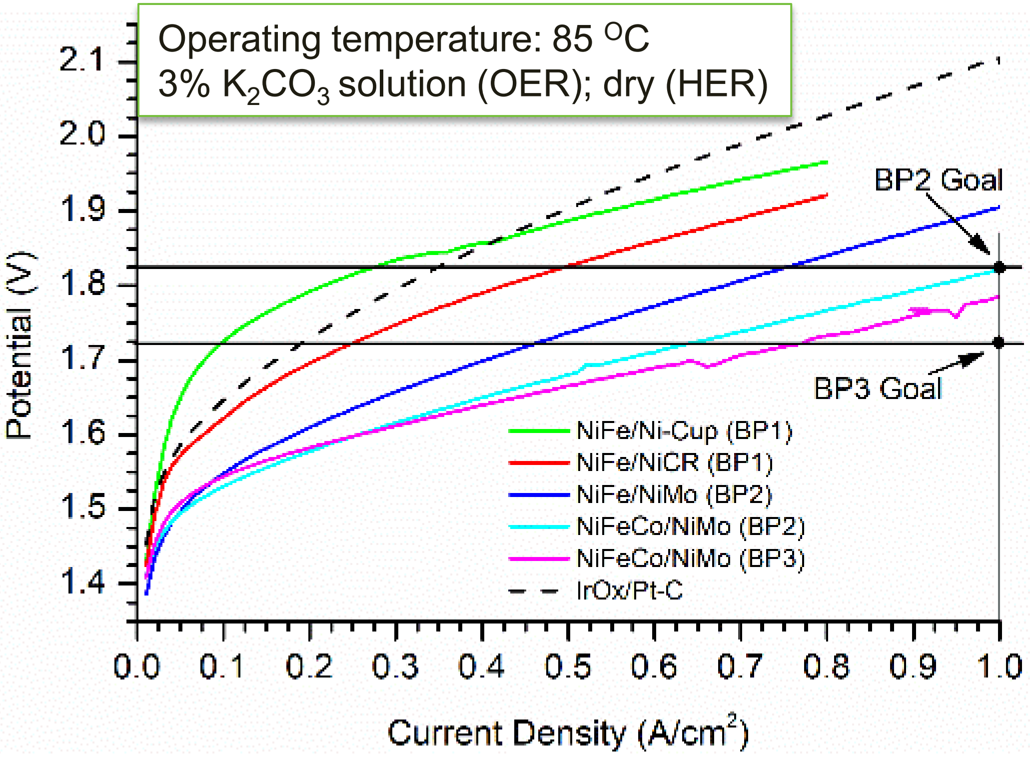
D-J Liu, PD157

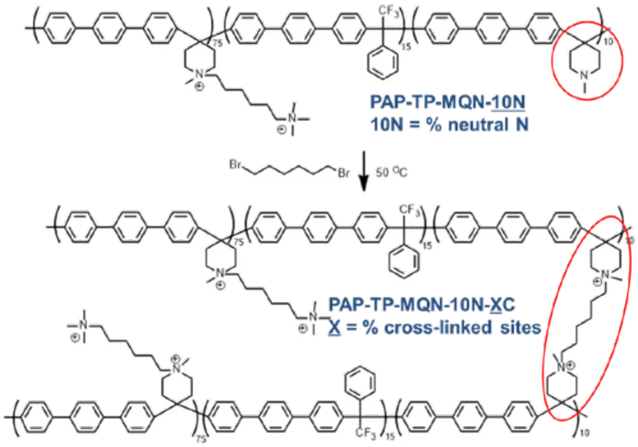
Novel PGM-free Catalysts for Alkaline HER and OER

Northeastern University | University of Delaware | Advent | Sandia National Laboratories | NREL National Renewable Energy Laboratory | Berkeley Lab | **3 Nodes**

Project Goal: Decrease the cost of hydrogen production via water electrolysis using high-performing PGM-free catalysts and a novel, temperature-stable anion exchange membrane.

Highlight: AEM electrolysis cell that achieved a potential of 1.78 V @ 1A/cm^2 with a net decay rate of 1 mV/hr measured over 65 hours. The end of project goal is 1.72 V @ 1 A/cm^2.





Crosslinking membranes improves mechanical stability with minimum loss of performance

10% Crosslinked:

IEC: 2.7 mequiv/g

Swelling @ 75^{O}C: 12%

ASR @ 80 ^{O}C: 0.65 Ω·cm^2

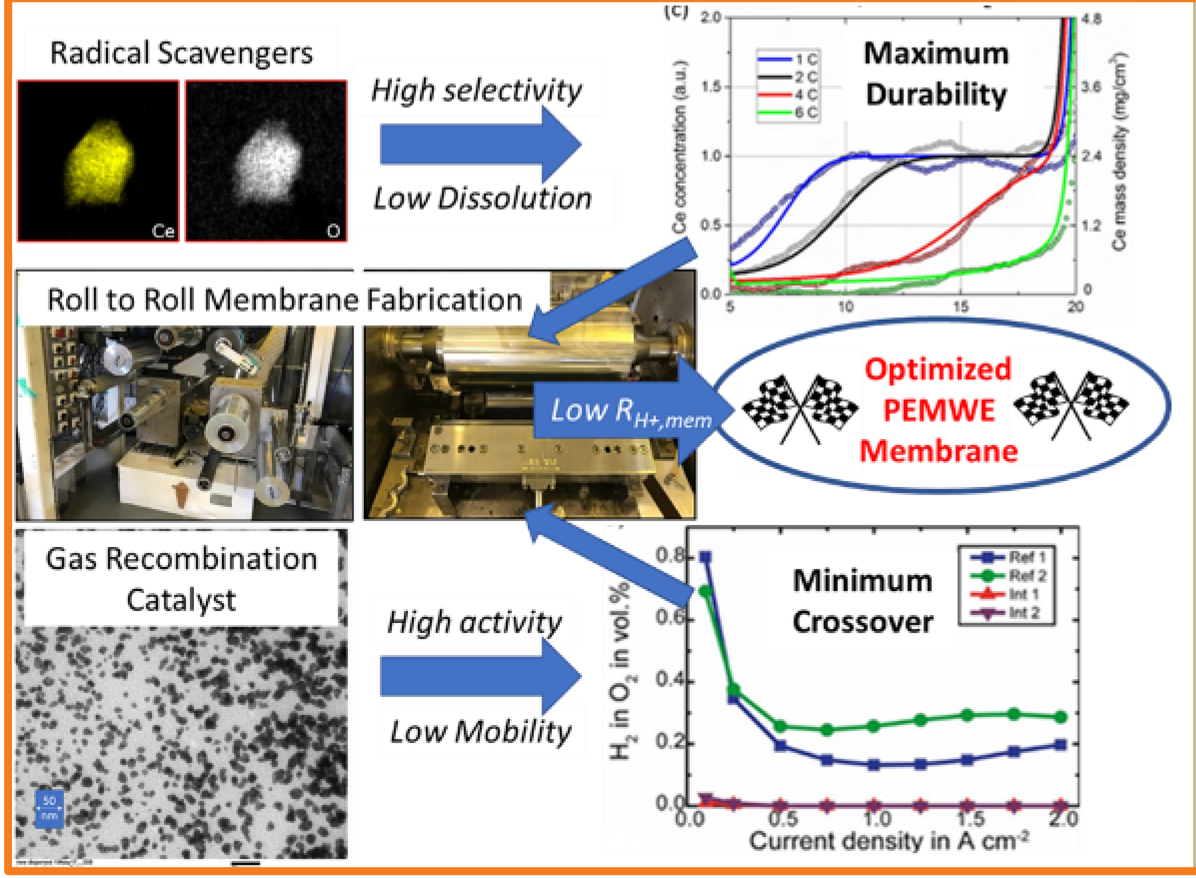
IEC loss after 1000hr in 90^{O}C KOH: ~8.13%

S. Mukerjee, P156

Performance and Durability Investigation of Thin, Low Crossover Proton Exchange Membranes for Water Electrolyzers

Chemours · Los Alamos National Laboratory | NREL National Renewable Energy Laboratory · Berkeley Lab | **3 Nodes**

Project Goal: **Developing thin membranes with low ohmic loss on roll-to-roll equipment for PEMWE systems, leveraging fundamental understanding of performance- and durability-enhancing additives to maximize efficiency and minimize cost of H_2.**



Highlight

State of the art PEMWE membranes are thick (>125 µm) and unreinforced with no stabilizing additives. This project intends to improve on state of the art by:

1. Engineering a thin, reinforced membrane on roll to roll scale
2. Remediating gas crossover with recombination catalyst
3. Preventing membrane degradation with immobile radical scavengers

A. Park, P186

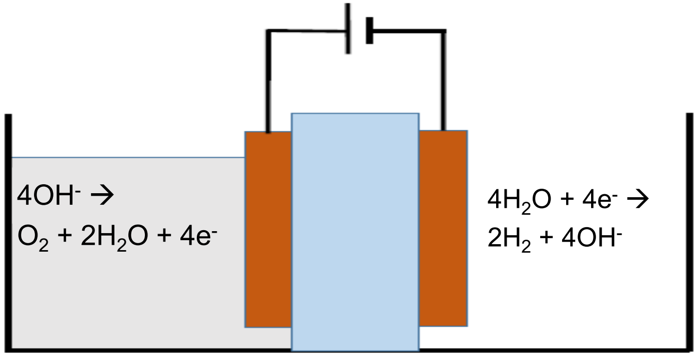
High-Performance AEM LTE with Advanced Membranes, Ionomers and PGM-Free Electrodes

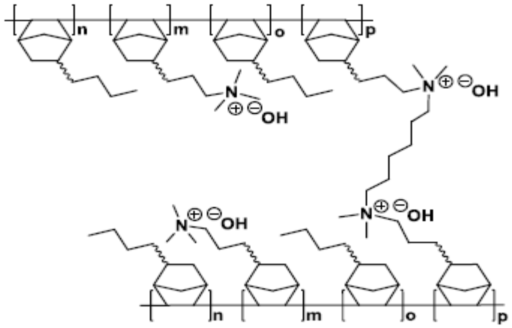
Georgia Institute of Technology · U of SC South Carolina · nel · PAJARITO POWDER FUEL CELL CATALYSTS · NREL NATIONAL RENEWABLE ENERGY LABORATORY · BERKELEY LAB

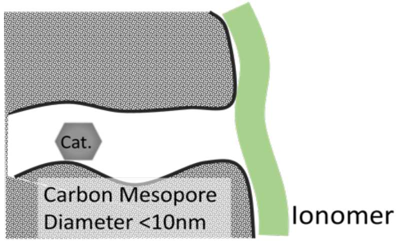
3

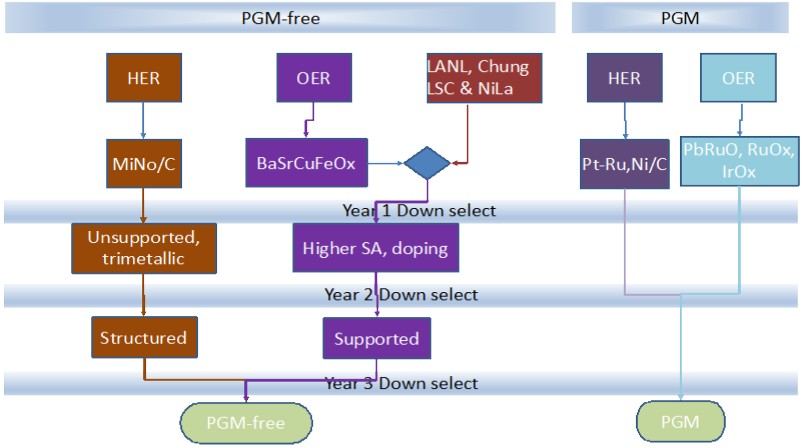
Project Goal: To enhance and combine state-of-the-art alkaline polymer electrolyzer components into one optimized membrane electrode assembly (MEA) system to achieve the DOE targets for low temperature electrolysis (LTE)

Highlight: Non-platinum group metal catalysts are combined with state-of-the-art anion conducting polymer membranes and ionomers to form high-performance MEAs









PGM-free

PGM

HER

OER

LANL, Chung LSC & NiLa

HER

OER

MiNo/C

BaSrCuFeOx

Pt-Ru,Ni/C

PbRuO, RuOx, IrOx

Year 1 Down select

Unsupported, trimetallic

Higher SA, doping

Year 2 Down select

Structured

Supported

Year 3 Down select

PGM-free

PGM

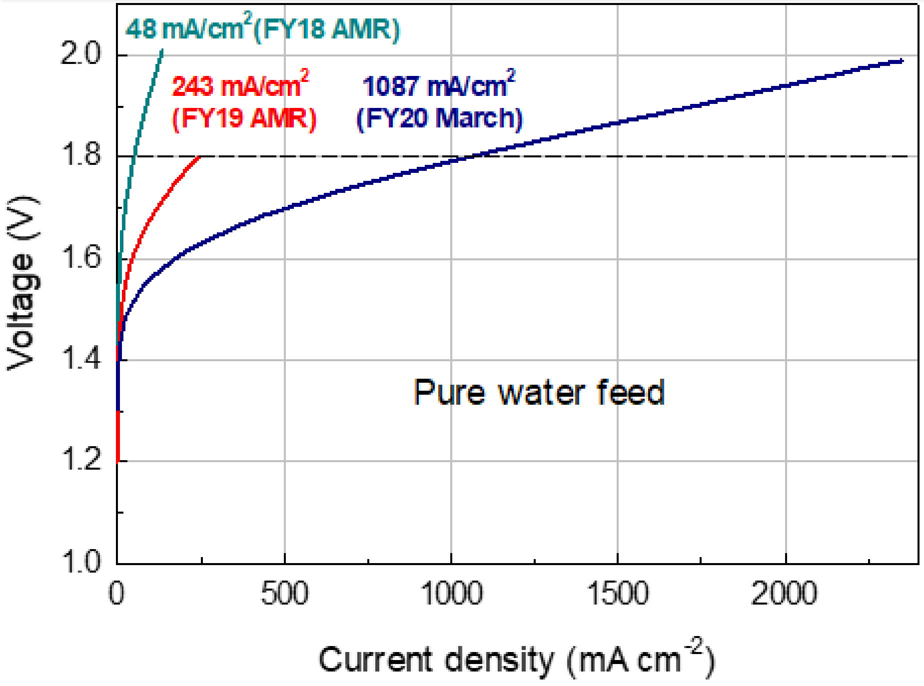
P. Kohl, PD185

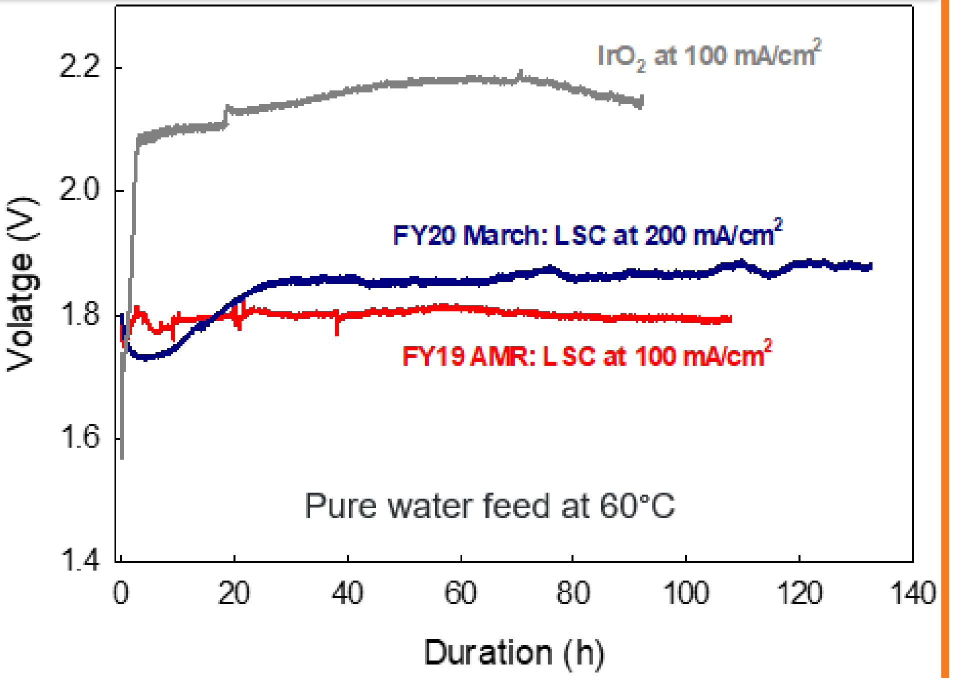
PGM-free OER Catalysts for Alkaline Water Electrolysis

Los Alamos NATIONAL LABORATORY · PAJARITO POWDER FUEL CELL CATALYSTS · Sandia National Laboratories · NREL NATIONAL RENEWABLE ENERGY LABORATORY · BERKELEY LAB · **5 Nodes**

Project Goal: Development of PGM-free Perovskite OER catalysts with high performance and durability in the alkaline solution-free pure water AEM water electrolyzer

Highlight: The project team achieved significant progress since FY18. It improved performance to 1.04 A/cm^2 at 1.8 V at 85˚C and slowed degradation rates to 0.2 mV/hr at 200 mA/cm^2 at 60˚C.





H. Chung, PD158

Hydrogen From Membrane Electrolysis of Dirty Water

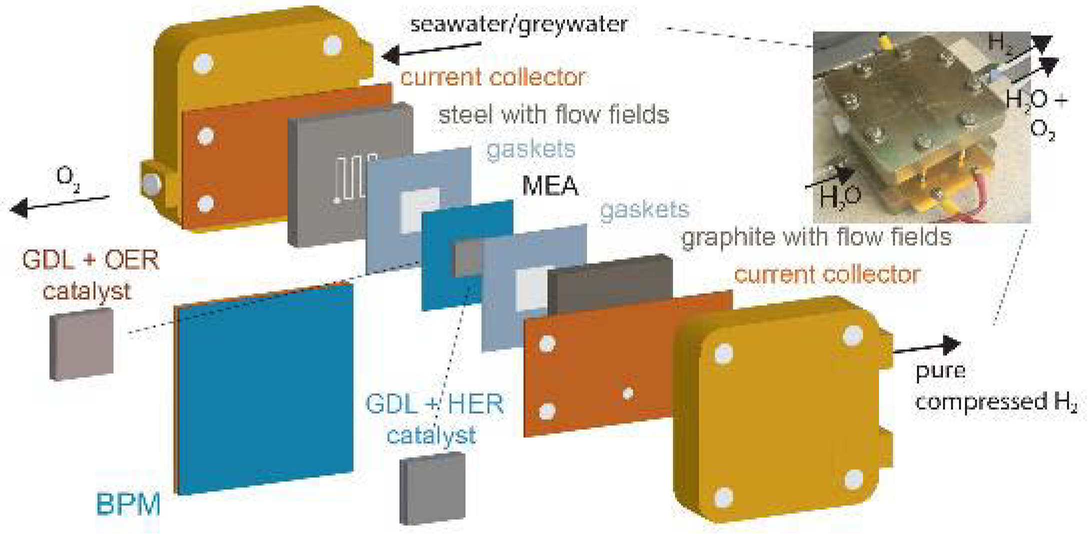
UNIVERSITY OF OREGON

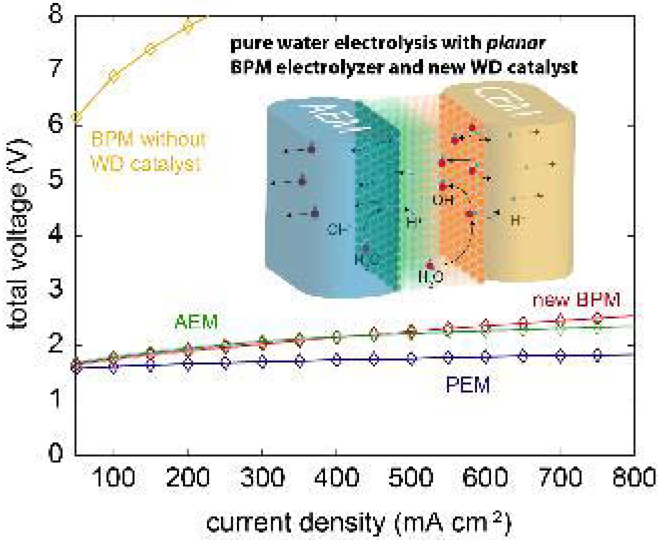
Sandia National Laboratories NREL NATIONAL RENEWABLE ENERGY LABORATORY BERKELEY LAB

6 Nodes

Project Goal: Develop a technical understanding of performance degradation of alkaline and bipolar membrane electrolyzers in pure and dirty water and engineer impurity tolerant systems.

Impact: Alkaline and bipolar membrane electrolysis systems enable PGM-free devices that may be more tolerant to impurities, if appropriately designed, which would increase system longevity, allow for less-stringent input water purity, and lower costs.





S. Boettcher, PD187

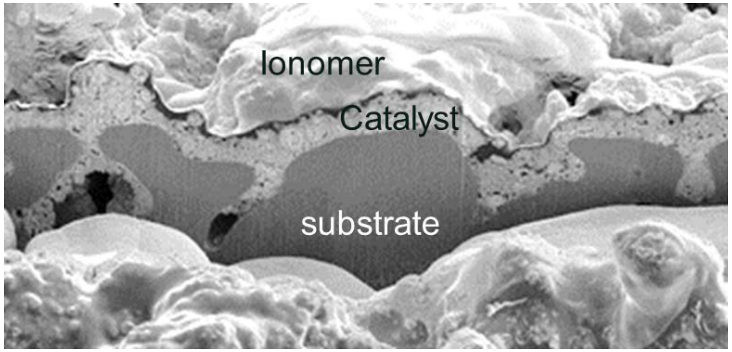
High Efficiency PEM Water Electrolysis

7 Nodes

Goals: Develop ultra-efficient PEM electrode per targets below

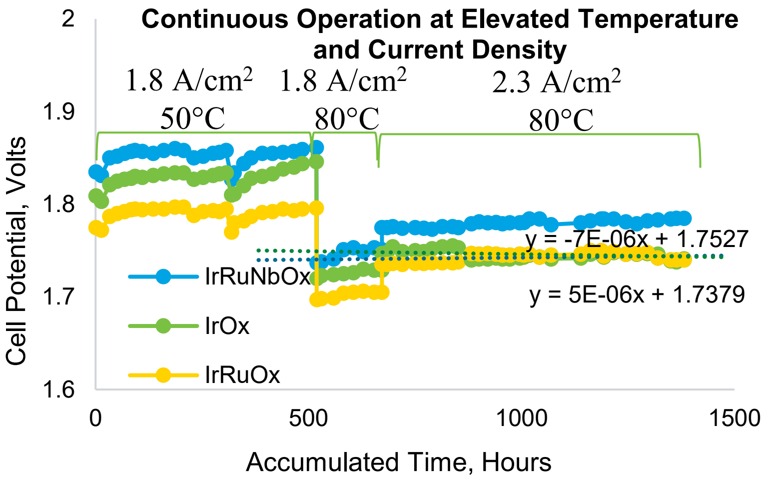
Metric	State of the Art	Proposed
Membrane thickness	*175 microns*	*50 microns*
Operating temperature	*58°C*	*80-90°C*
Cell Efficiency	*53 kWh/kg*	*43 kWh/kg*

Approach: Look at materials and manufacturing holistically to optimize



Accomplishments in Phase 2

Met voltage and durability targets with advanced catalyst and thin membrane



Focus of Phase 2

1. Development of hydrogen cross-over mitigation strategy
2. Integrate catalysts and hydrogen mitigation into integrated assembly
3. Scale-up and conduct durability tests in multi-cell stack
4. Conduct final cost analysis

K. Ayers, PD155

Accomplishments and Progress – LTE Benchmarking

- **Collaborated with HydroGEN Benchmarking Project**
  - Workshop participation
  - Session chairing
  - Progress on Protocols and Standard vocabulary & definitions
- **Interfacing HydroGEN & IEA Annex 30 Benchmarking activities**
  - Communicating RR phase II progress
  - Discussing common hardware platform
- **Contributing to Meta Data development of HydroGEN Data Center**

Summary - HydroGEN LTE Projects

- HydroGEN LTE is actively supporting
  - 8 FOA projects with 41 node call outs
  - 2 Supernodes with 14 node call outs
- FOA Projects demonstrate improvements in PEM & AEM technologies
- LTE Supernode interlinks Ex-Situ, In-Situ and Modeling Results and supports upscaling
- Working closely with the project participants and benchmarking activities to advance knowledge and utilize capabilities

Future Work

- **Fully integrate recently started FOA awarded seedling projects (~March/April 2020)**
- **Continue to enable and support research of the funded FOA Projects through lab nodes and expertise**
- **Utilize and expand Supernodes to help accelerate LTE research**
- **Work with the 2B team and LTE working group to establish testing protocols and benchmarks**
- **Continue to utilize data hub for increased communication, collaboration, generalized learnings, and making digital data public**

Acknowledgements

Energy Materials Network
U.S. Department of Energy

HydroGEN
Advanced Water Splitting Materials

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Rensselaer
Pajarito Powder
Advent
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Giner Labs
University of Oregon
University at Buffalo The State University of New York
Chemours
Georgia Institute of Technology
Los Alamos National Laboratory EST. 1943
National Fuel Cell Research Center University of California · Irvine
Oak Ridge National Laboratory
UofSC South Carolina

NREL National Renewable Energy Laboratory
Berkeley Lab
Sandia National Laboratories
INL Idaho National Laboratory
Lawrence Livermore National Laboratory
SRNL

Acknowledgements

Energy Materials Network
U.S. Department of Energy

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NREL
NATIONAL RENEWABLE ENERGY LABORATORY

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Guido Bender
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Allen Kang
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Janghoon Park
Jason Pfeilsticker
Bryan Pivovar
Michael Ulsh
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Savannah River National Laboratory®
OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

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Héctor Colón-Mercado

BERKELEY LAB
Lawrence Berkeley National Laboratory

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Ahmet Kusoglu
Jessica Luo
Adam Weber
Guosong Zeng
Jeremy Zhou

NREL · BERKELEY LAB · Sandia National Laboratories · INL Idaho National Laboratory · Lawrence Livermore National Laboratory · SRNL

Acknowledgements

Energy Materials Network
U.S. Department of Energy

HydroGEN
Advanced Water Splitting Materials

Publication Number: NREL/PR-5900-76549

This work was authored in part by the National Renewable Energy Laboratory, operated by Alliance for Sustainable Energy, LLC, for the U.S. Department of Energy (DOE) under Contract No. DE-AC36-08GO28308. Funding provided by the U.S. Department of Energy Office of Energy Efficiency and Renewable Energy Fuel Cell Technologies Office. The views expressed in the article do not necessarily represent the views of the DOE or the U.S. Government. The U.S. Government retains and the publisher, by accepting the article for publication, acknowledges that the U.S. Government retains a nonexclusive, paid-up, irrevocable, worldwide license to publish or reproduce the published form of this work, or allow others to do so, for U.S. Government purposes.

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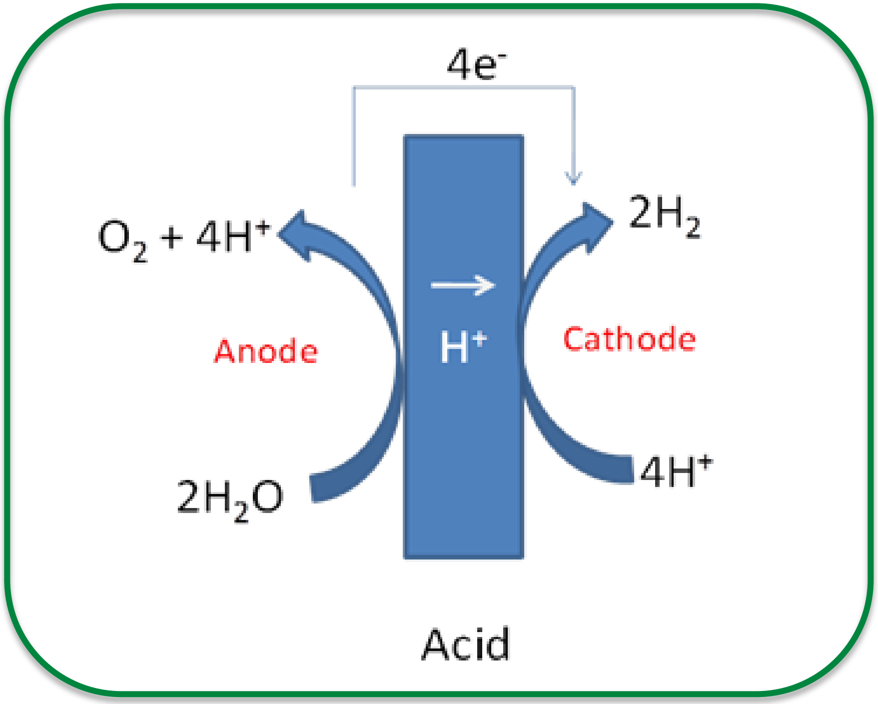
Technical Backup Slides

Overview - LTE Technology

Schematic PEM*

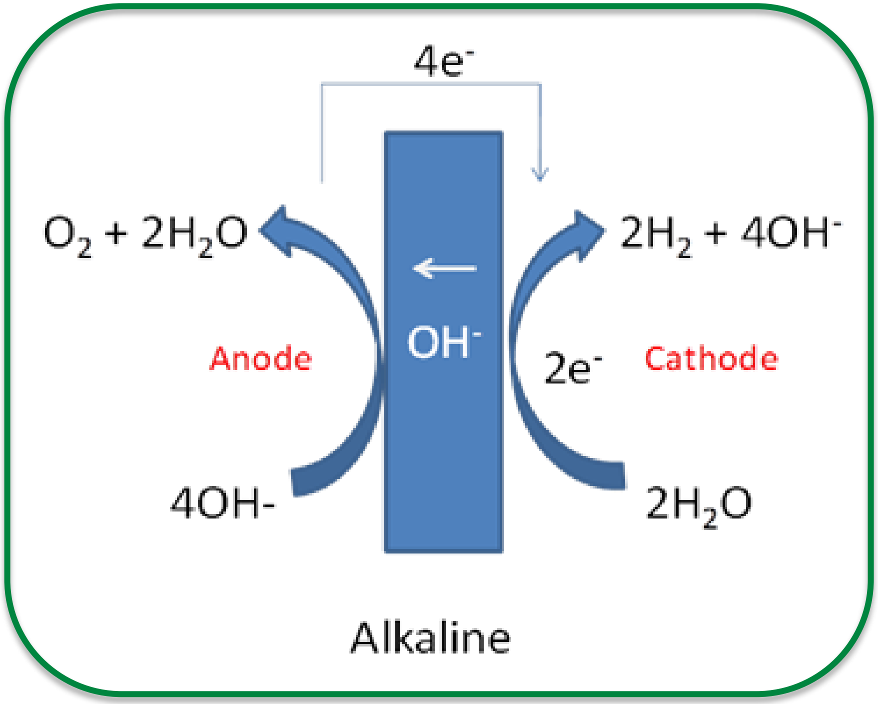


Anode: $2H_2O \Rightarrow O_2 + 2H^+ + 2e^-$

Cathode: $2H^+ + 2e^- \Rightarrow H_2$

- **Niche Application Deployment**

Schematic AEM*



Anode: $4OH^- \Rightarrow O_2 + 2H_2O + 4e^-$

Cathode: $4H_2O + 4e^- \Rightarrow 2H_2 + 4OH^-$

- **Low TRL Technology**
- **Research Stage**

* K.Ayers, AMR Presentation PD094, 06/2014

Overview - LTE Technology Relevance / Impact

State-of-Art PEM

- 2V @ 2A/cm^2
- 2-3 mg/cm^2 PGM catalyst loading on anode & cathode
- 60k – 80k hours in commercial units
- Niche applications
  - Life support
  - Industrial H_2
  - Power plants for cooling
- \$3.7/kg H_2 production*

State-of-Art AEM

- 2V @ 0.2A/cm^2 in H_2O
- Improved performance in basic solution
- 2-3 mg/cm^2 PGM-free catalyst loading on anode & cathode
- ~2k hour at 27˚C demonstrated **
- No commercial units
- \$/kg production not available

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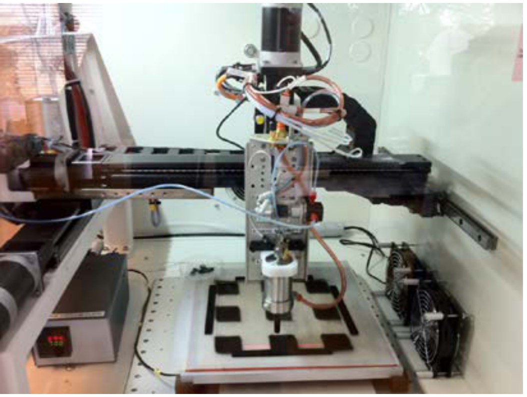
*High volume projection of hydrogen production for electrolysis: https://www.energy.gov/sites/prod/files/2017/10/f37/fcto-progress-fact-sheet-august-2017.pdf

** K.Ayers, AMR Presentation PD094, 06/2014

Supernode Accomplishments: Electrode Fabrication Platforms

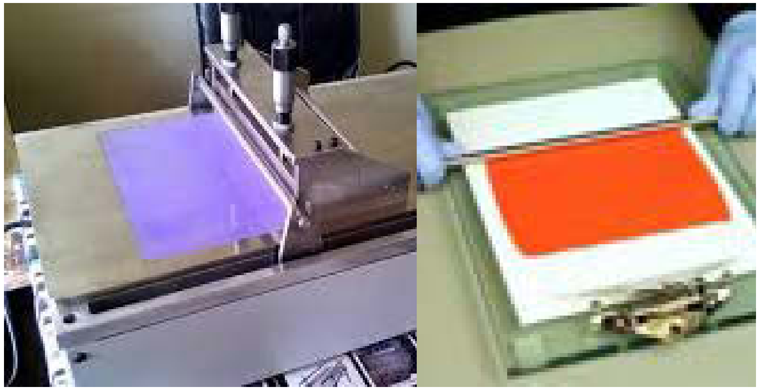
Ultrasonic Spray



Used to demonstrate new materials and for fundamental studies

Conditions
- Dilute ink
- Sequential build up of layers
- Heated substrate
- Vacuum substrate
- Batch Process

Doctor Blade/Mayer Rod



Used to demonstrate new materials and for fundamental studies. Prove out ink formulations or processes prior to R2R

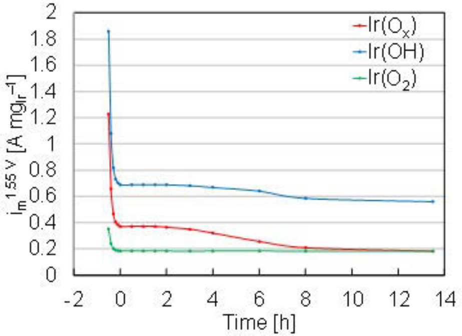
Conditions
- Concentrated inks
- Single layer coating
- Heated substrate
- Vacuum substrate
- Batch Process

Roll-to-Roll

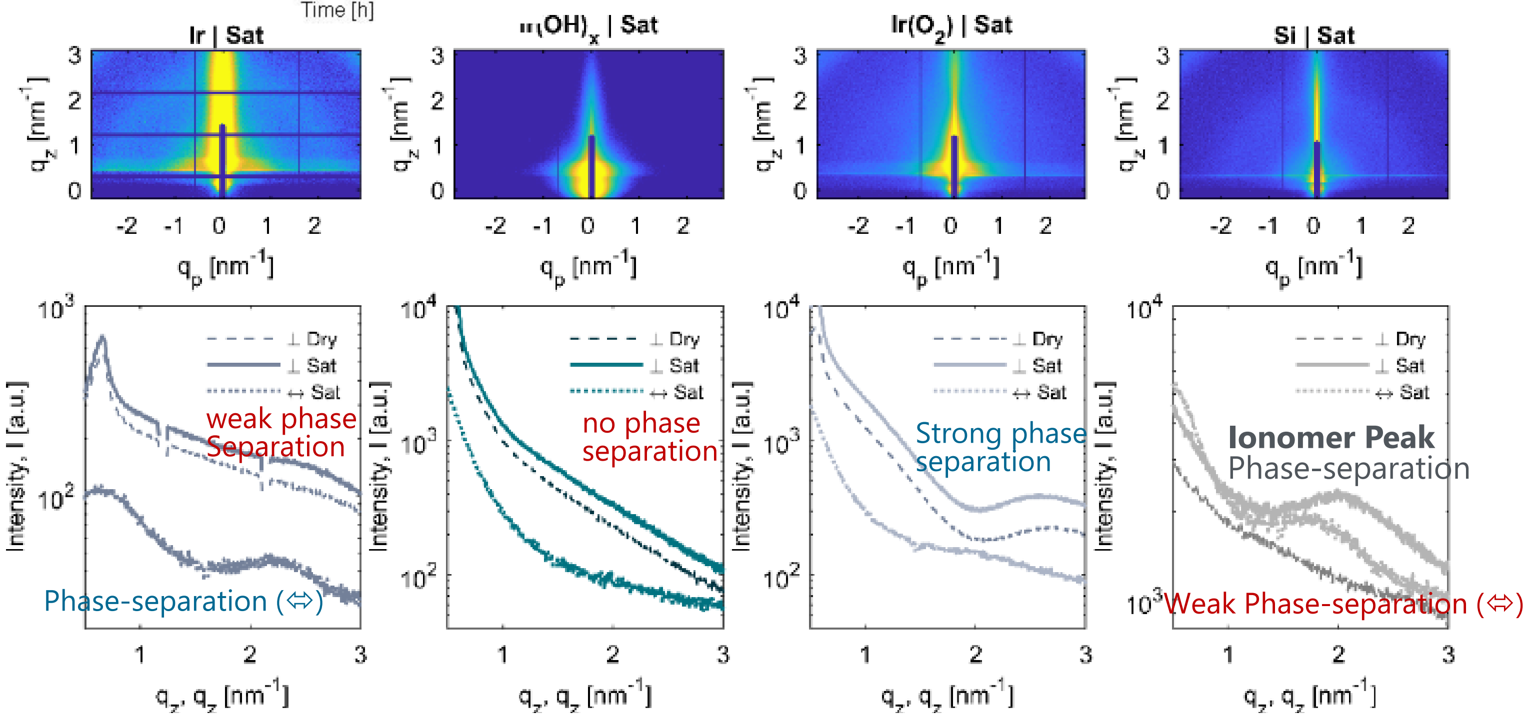
Demonstrate scalability of materials and MEA/cell designs. Studies of process variables

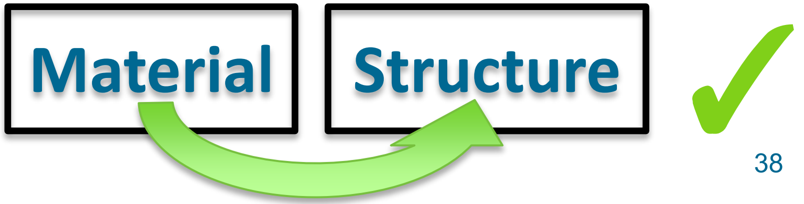
Conditions
- Concentrated inks
- Single layer coating
- Room temperature substrate
- Convection drying
- Continuous Process

Supernode Accomplishments: Thin Film Morphology: GISAXS



- Nafion morphology on Iridium substrates
  - Overall phase-separated nanostructure
  - Broader peaks and weaker phase-separation on OH
- Ir(O_x) [Metal]: phase-separation (**in-plane** ordering)
- Ir(OH) [functionalized]: **no** phase-separation (both directions)
- Ir(O_2) [Bulk Oxide]: phase-separation (**thickness** ordering)





Material **Structure** ✓