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

G. Bender, H.N. Dinh

Presenter: Guido Bender, NREL

Date: 5/20/2020

Venue: 2020 DOE Annual Merit Review

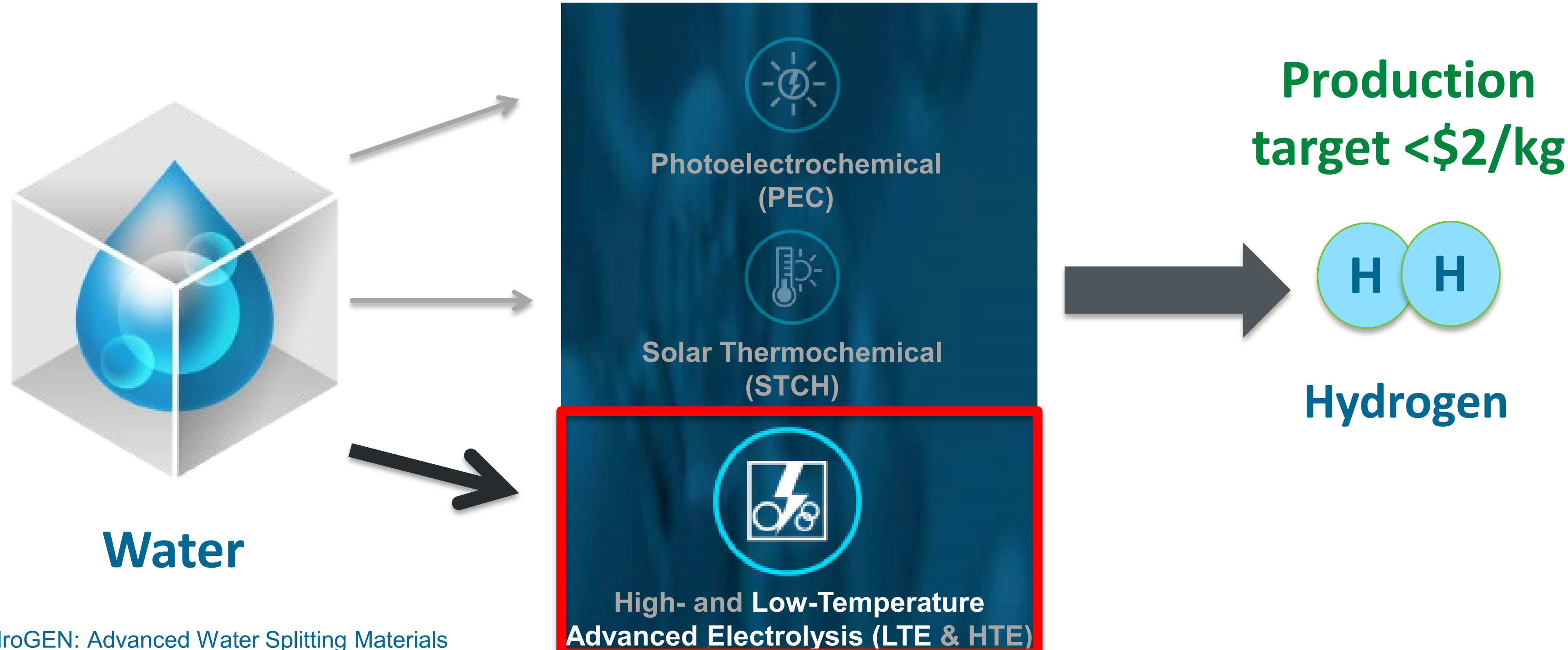
Project ID # P148A

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:



Relevance – Impact on LTE Technology

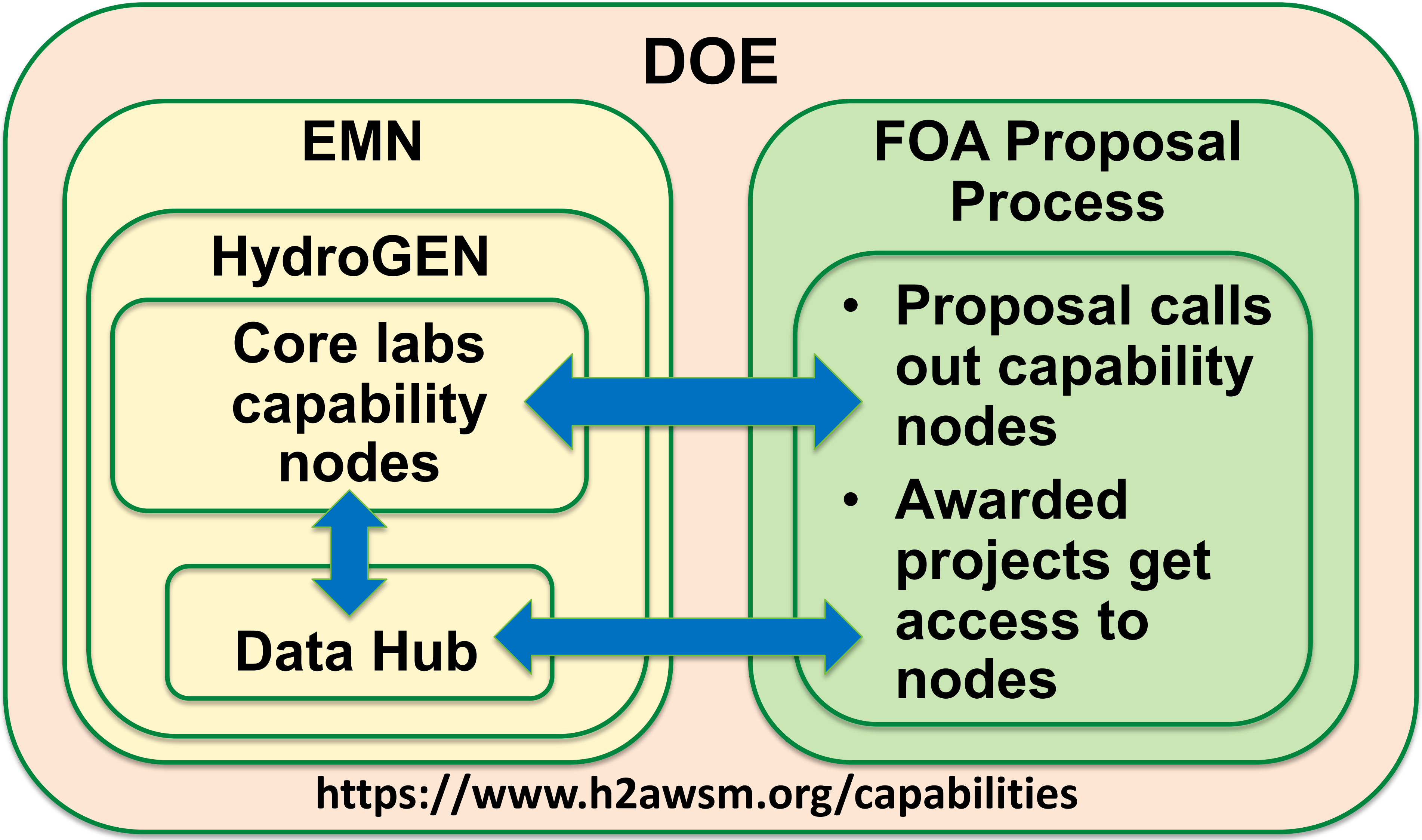
PEM
- Gas Crossover
- Membranes
- Catalyst Materials
- Catalyst Loading
- PTL Materials

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



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

University of Oregon

Chemours

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
	H. Dinh B. Pivovar	Linking LTE/Hybrid Materials to Electrode Properties to Performance

Discussed in PEC, P148A

Discussed here in LTE, P148C

PEM: Understanding and improving materials

AEM: Developing and understanding materials

Accomplishments and Progress:
Node Utilization for Project Support

NREL NATIONAL RENEWABLE ENERGY LABORATORY | Lawrence Livermore National Laboratory | Sandia National Laboratories | BERKELEY LAB

49 nodes requests for LTE

↓

19 nodes used by LTE projects

→

Node Classification

27x Characterization

10x Computation

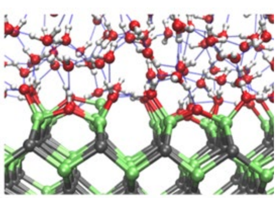
7x Material Synthesis

5x Process and Manufacturing Scale-Up

Collaboration and Coordination - Node Utilization

FY'20 Projects (Chem, UO, GT)

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 (red: NREL Data Hub, LLNL, LBNL, SNL LAMMPS)

Processing & Scale Up (black: NREL Multi-Comp. Ink Development)

Material Synthesis (blue: NREL Novel Membrane Fabrication, SNL Separators)

Collaboration and Coordination - Node Utilization

FY’20 Projects (Chem, UO, GT)

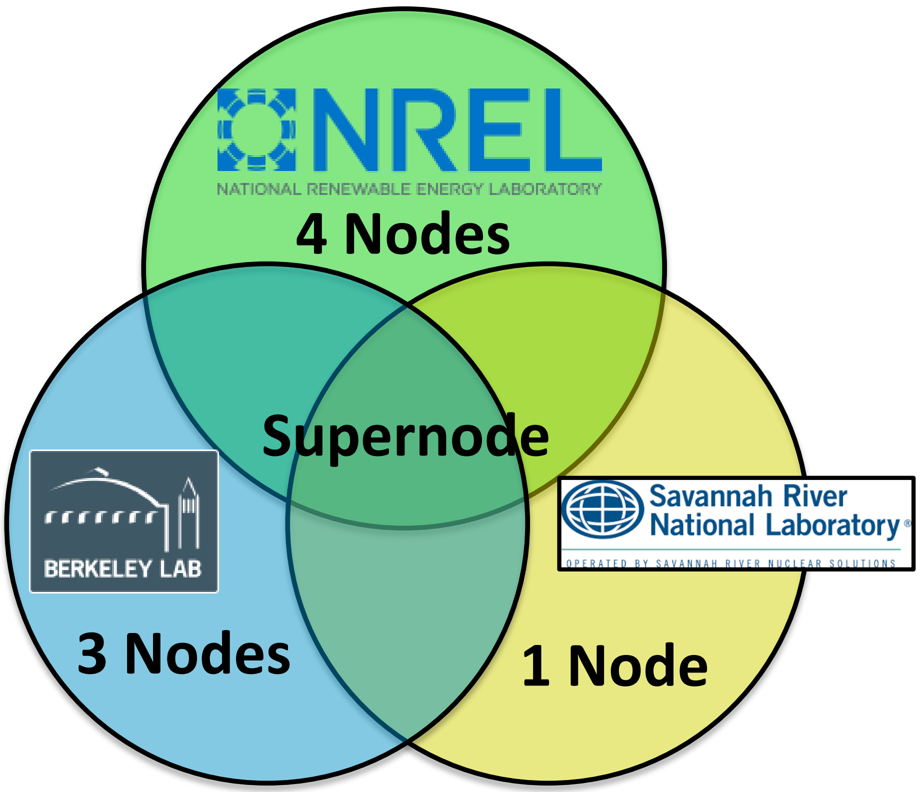
Lab	Node	LTE Super	Chem	UO	GT	Nel	ANL	NEU	LANL 1	LANL 2
SNL	Advanced Electron Microscopy						✓			
NREL	Catalyst Synthesis, Ex situ Characterization & Standardization	✓				✓	✓			
LBNL	Ionomer Characterization and Understanding	✓	✓	✓		✓		✓		✓
NREL	In Situ Testing Capabilities	✓	✓	✓	✓	✓		✓	✓	✓
LBNL	Understanding 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



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

Supernode Goals

NREL | Savannah River National Laboratory | BERKELEY LAB | 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.

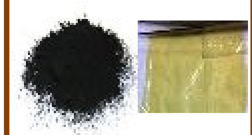
Project Elements:

- 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)**

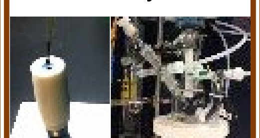
Expertise:

Materials → Devices

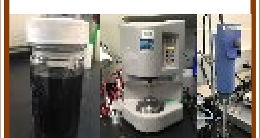
Materials: Catalyst, Ionomer & Membranes



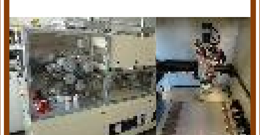
Electrochemical Characterization Catalyst



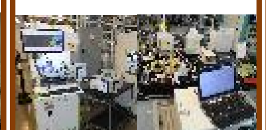
Ink Composition Catalyst, Ionomer & Solvents



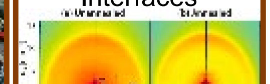
MEA Integration Fabrication in Parameter Space



Performance Evaluation In-Situ PEM, AEM

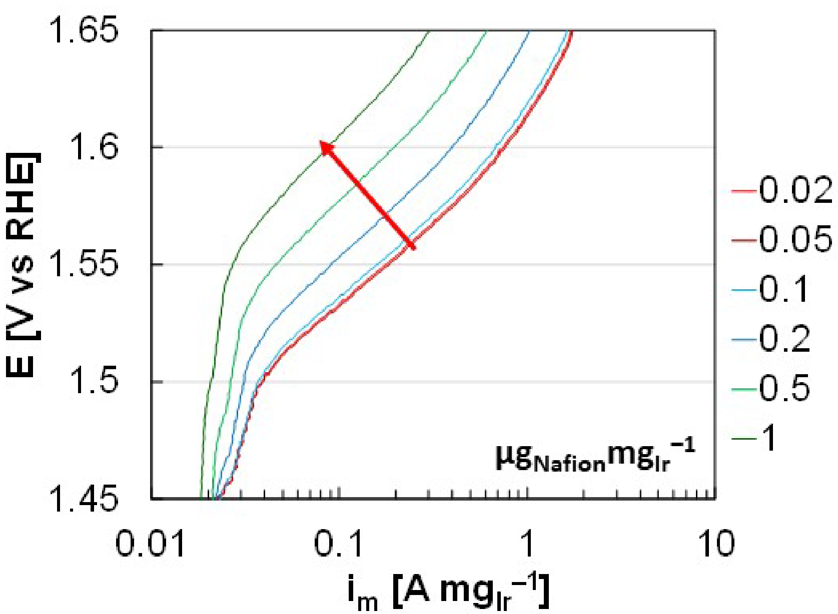


Advanced Characterization Morphology, Material Properties, Interfaces

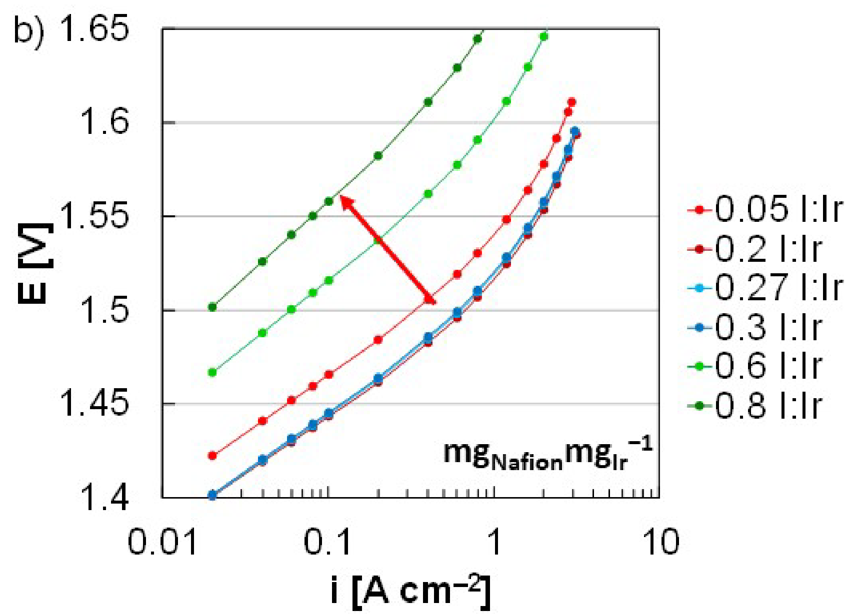


Supernode Accomplishments: RDE/MEA Correlation

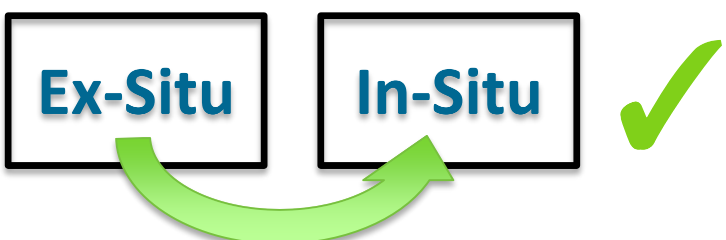
RDE Ex-Situ



MEA In-Situ



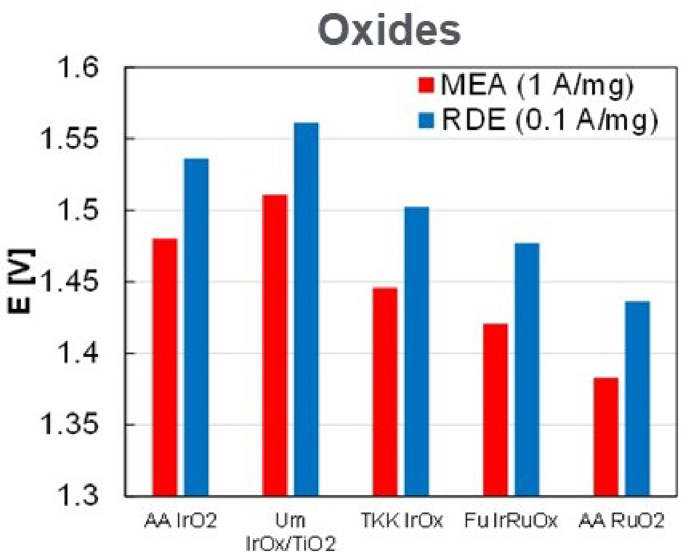
- **Correlation between RDE and MEA systems confirmed**
- **Same trend in RDE observed in MEA for effects of:**
  - **Catalyst loading**
  - **Ionomer content**
  - **Catalyst used**

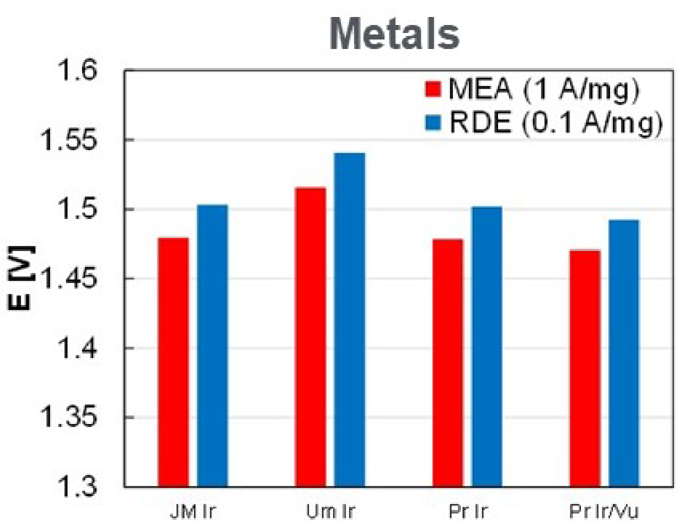


S.M. Alia, G.C. Anderson, *J. Electrochem. Soc.*, **2019**, *166*(4), F282-F294. DOI:10.1149/2.0731904jes

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

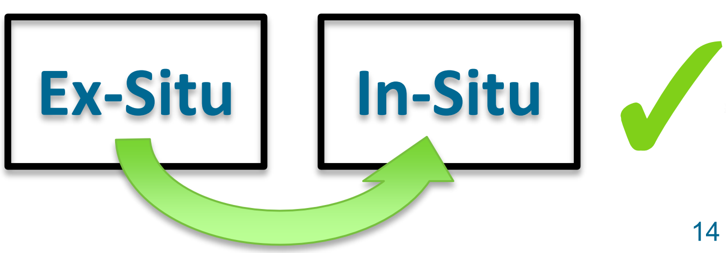
Supernode Accomplishments: Met Go/No Go Milestone by Correlating Ex- with In-situ Performance





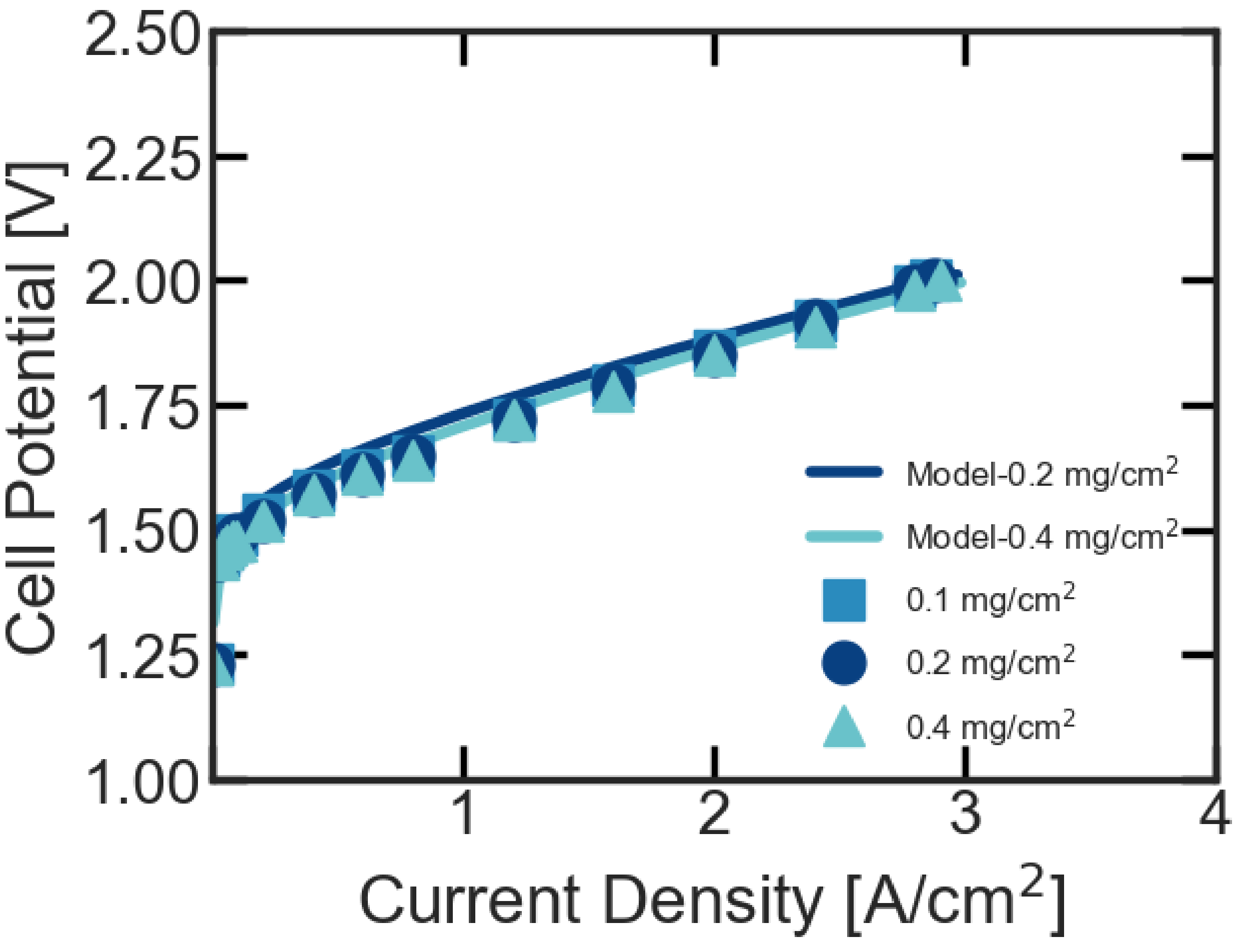
		E^{MEA} [V]	ΔE [mV]	E^{RDE} [V]	ΔE [mV]	ΔE [%]
Alfa Aesar	IrO_2	1.480	–	1.536	–	–
Umicore	IrO_x/TiO_2	1.511	30.5	1.561	25.4	-16.7
TKK	IrO_x	1.446	-34.5	1.502	-33.6	-2.6
Furuya	$IrRuO_x$	1.421	-59.6	1.477	-58.8	-1.3
Alfa Aesar	RuO_2	1.383	-97.4	1.436	-99.5	2.2
Johnson Matthey	Ir	1.480	–	1.503	–	–
Umicore	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.

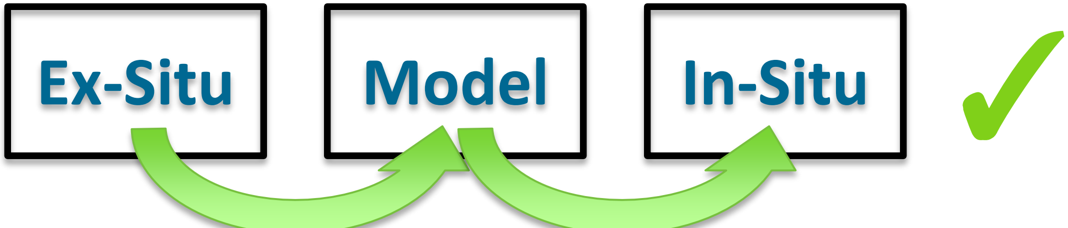


S.M. Alia, M.-A. Ha, G.C. Anderson, C. Ngo, S. Pylypenko, R.E. Larsen, *J. Electrochem. Soc.*, **2019**, *166*(15), F1243-F1252. DOI:10.1149/2.0771915jes

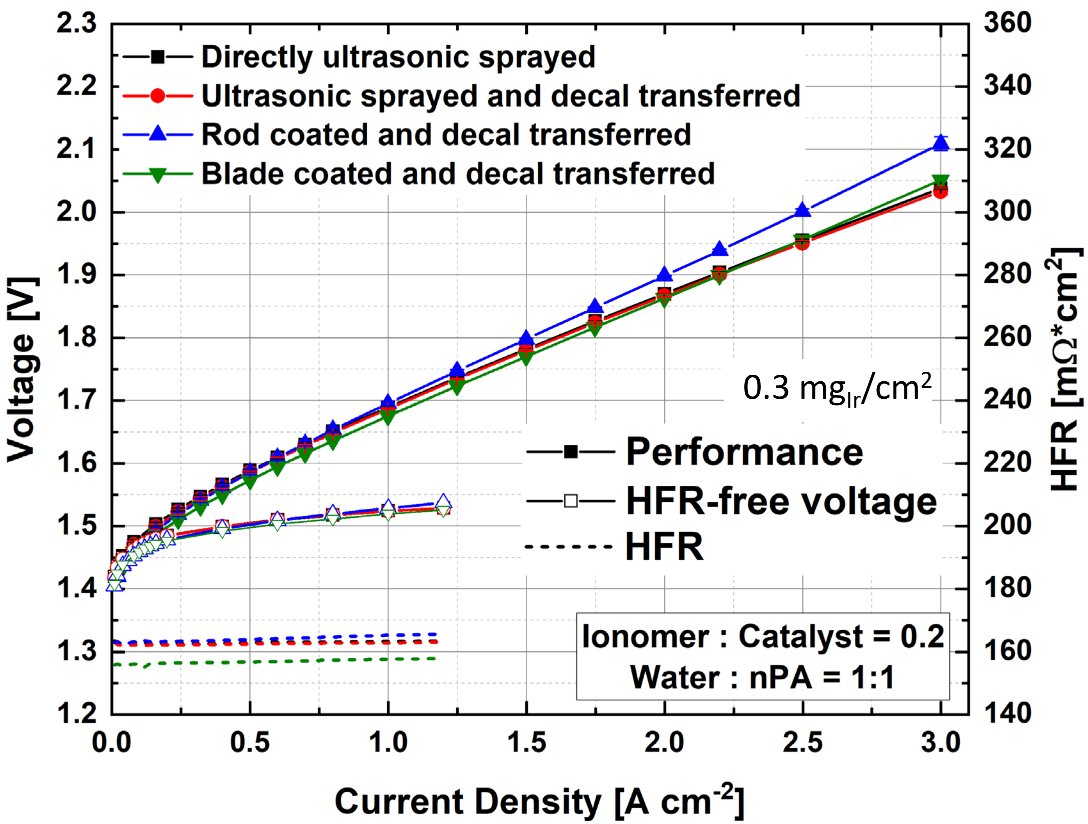
Supernode Accomplishments: Multiscale Modeling Agrees with Experimental Data



- 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

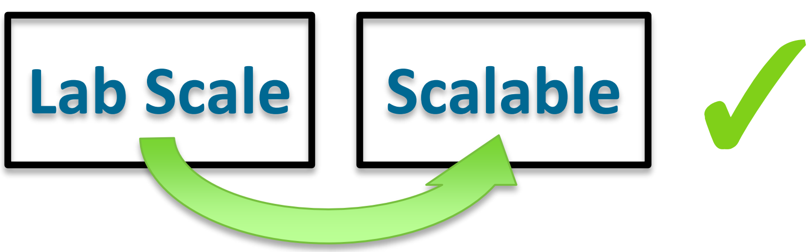


Supernode Accomplishments: Doctor Blade and Mayer Rod Comparisons



- 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)**

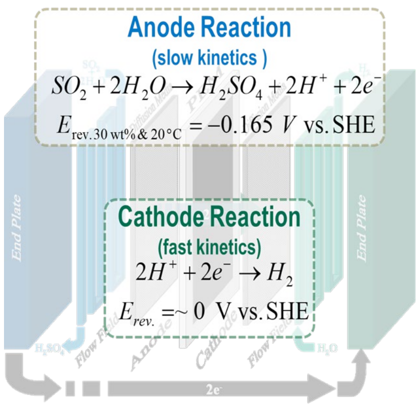
- Ionomer : catalyst ratio from 0.1 to 0.3 did not significantly impact performance for blade- or rod-coated methods.
- Ink composition found to be less impactful at higher loading. Thicker catalyst layers may mask nonuniformities.



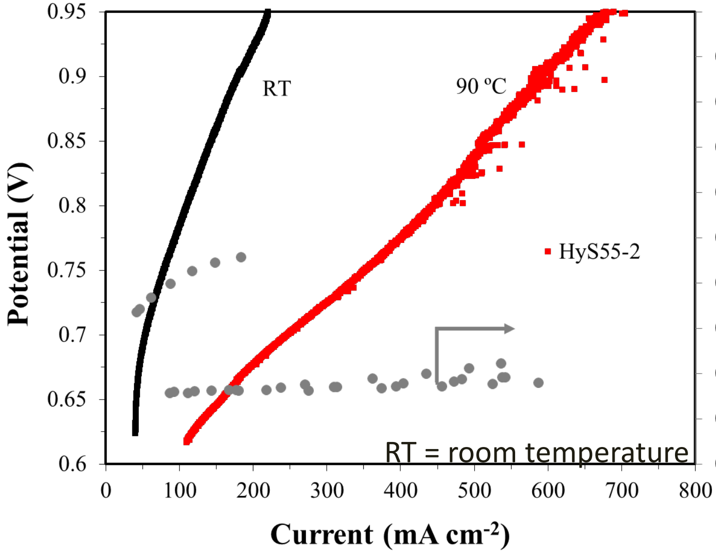
Supernode Accomplishments: Hybrid Cycle Correlating Ex- & In-situ Testing

NREL NATIONAL RENEWABLE ENERGY LABORATORY | Savannah River National Laboratory OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS | BERKELEY LAB

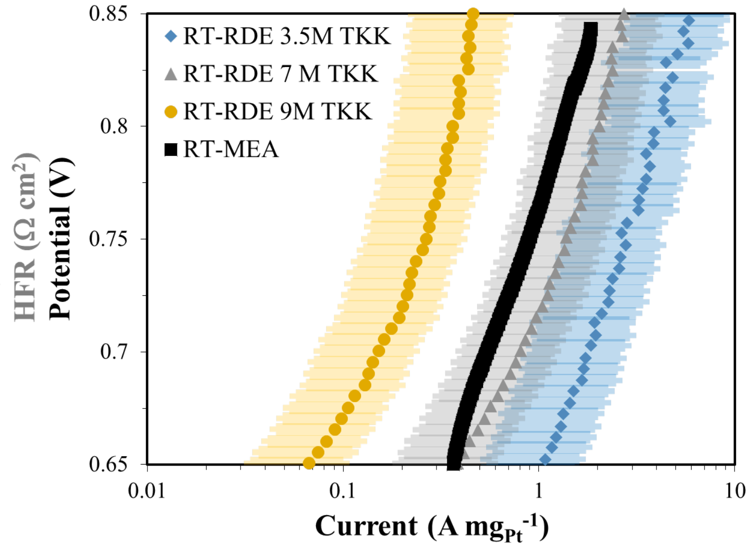
Hybrid Cycle



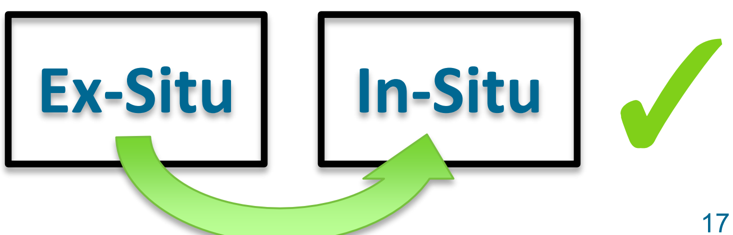
MEA testing



RDE vs MEA testing

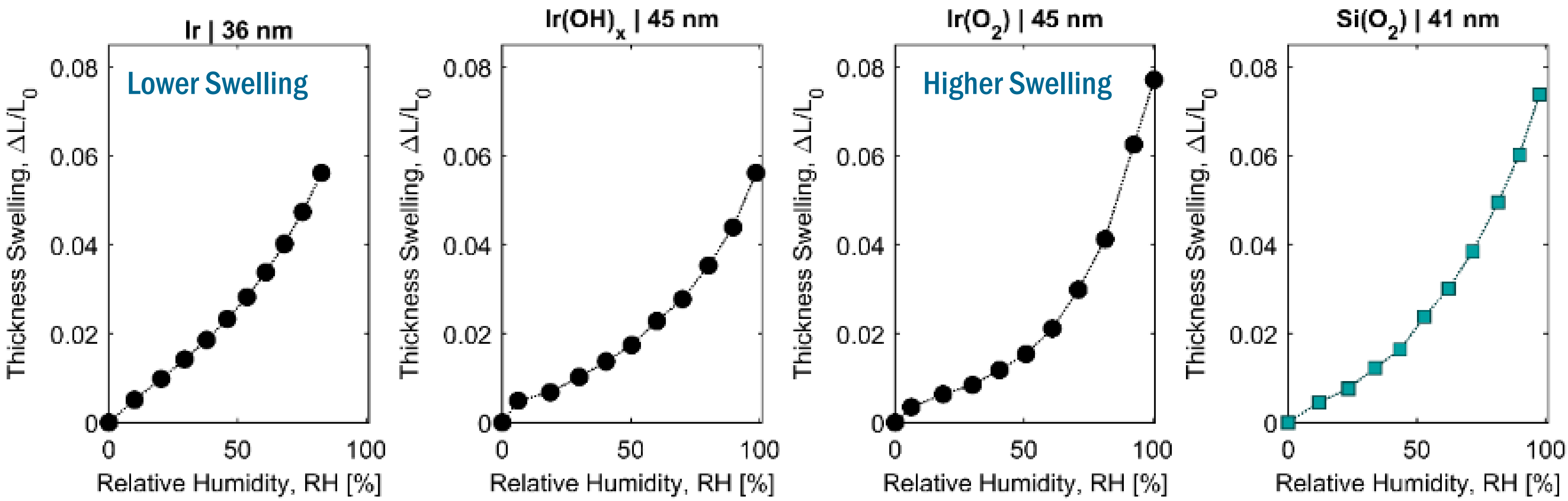


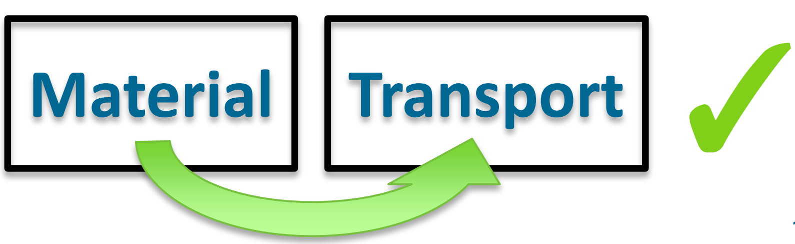
- Met Go/No Go milestone criteria of 50 % agreement between RDE and MEA testing (indicated by shaded area)
- Good agreement between in-situ and ex-situ measurements at acid concentration of 7 M
- Performance sensitivity to coating method and catalyst ink formulation is similar to that observed in fuel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



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

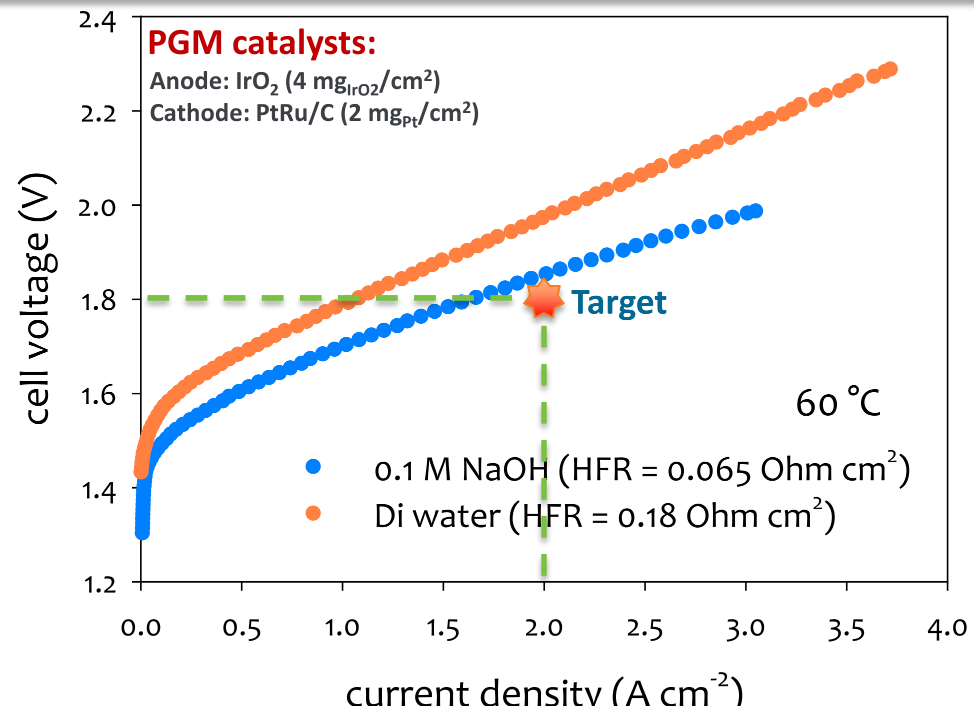
Example Project Accomplishment Slide

Los Alamos National Laboratory · Rensselaer · nel · Sandia National Laboratories · NREL National Renewable Energy Laboratory · Berkeley Lab · **5 Nodes**

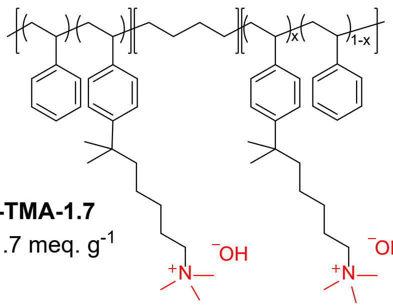
Scalable Elastomeric Membranes for Alkaline Water Electrolysis

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/cm^2 at 1.8 V) for AEM electrolyzer

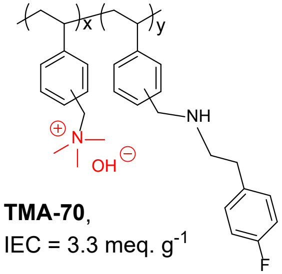


AEM (RPI)



SES25-TMA-1.7
IEC = 1.7 meq. g^{-1}

Ionomer (LANL)



TMA-70,
IEC = 3.3 meq. g^{-1}

Y. Kim, PD159

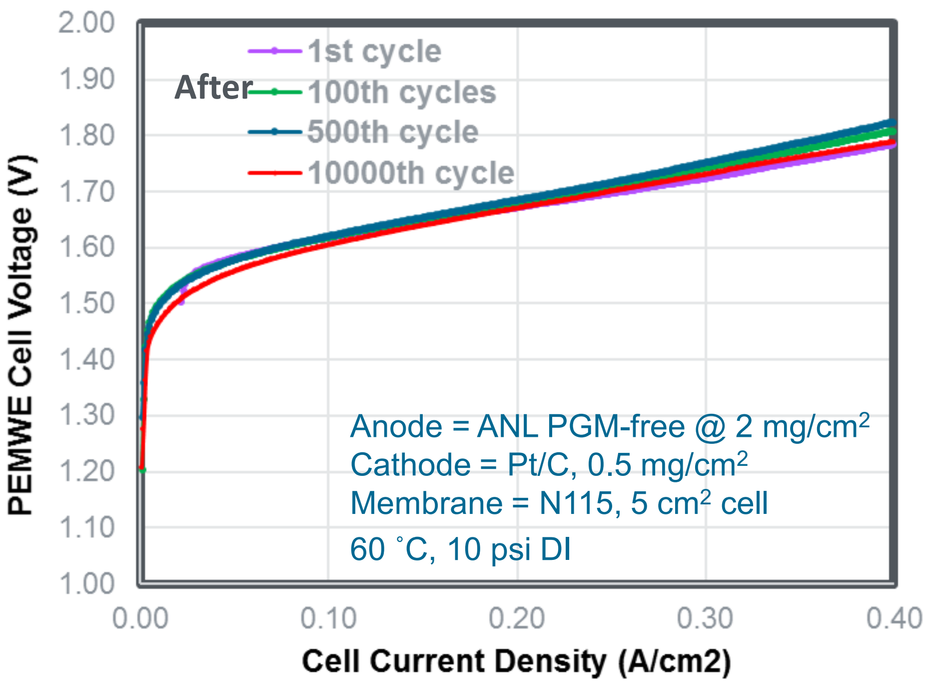
PGM-free OER Catalysts for PEM Electrolyzer

Argonne National Laboratory · University at Buffalo, The State University of New York · Giner Labs · Berkeley Lab · Lawrence Livermore National Laboratory · Sandia National Laboratories · NREL

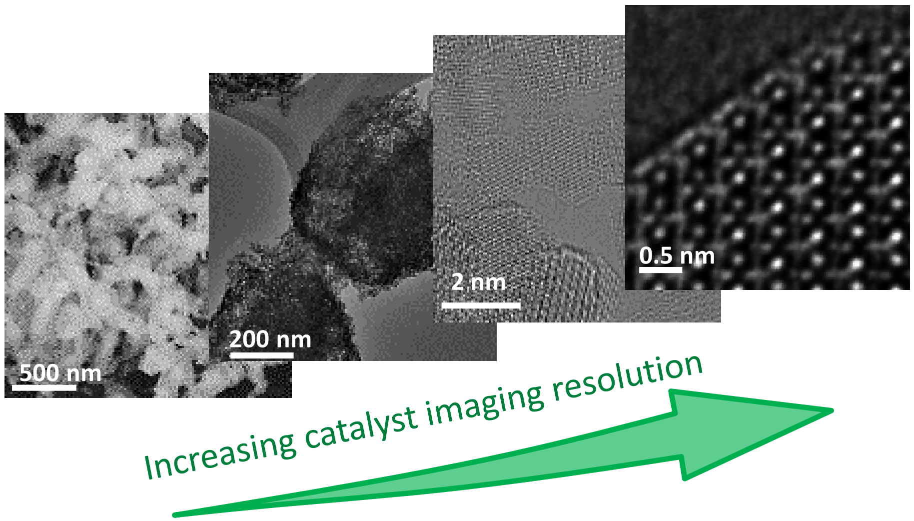
5 Nodes

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/cm² @ 1.8 V and stability over 10,000 voltage cycles in PEMWE.



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



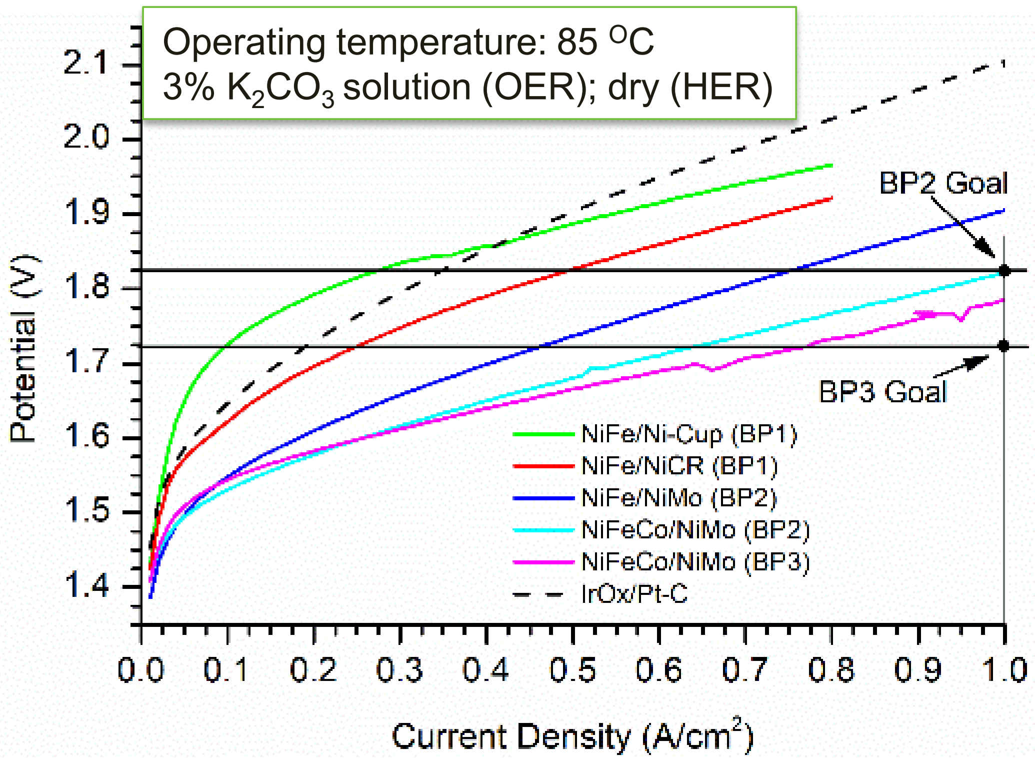
D-J Liu, PD157

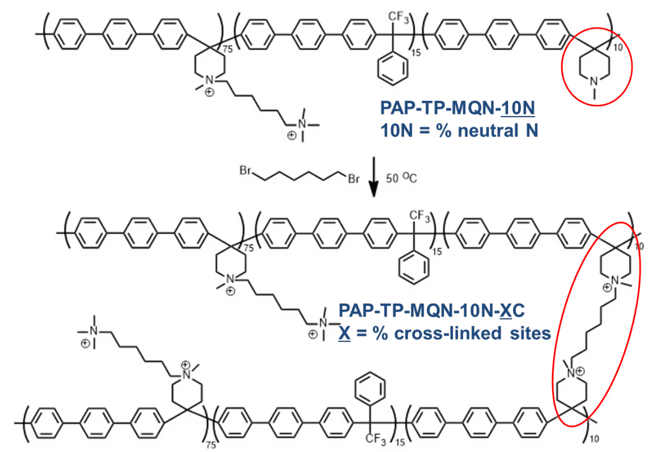
Novel PGM-free Catalysts for Alkaline HER and OER

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°C: 12%

ASR @ 80 °C: 0.65 Ω·cm^2
IEC loss after 1000hr in 90°C KOH: ~8.13%

S. Mukerjee, P156

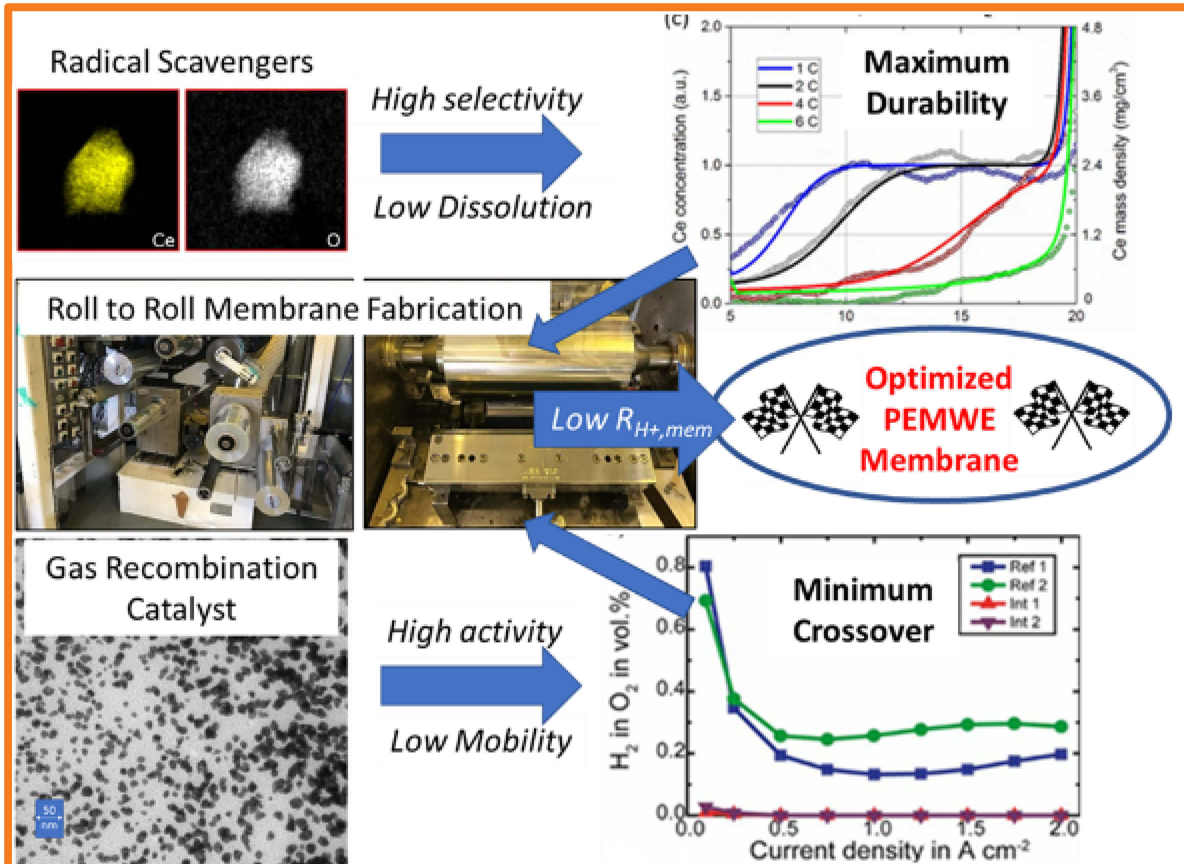
Performance and Durability Investigation of Thin, Low Crossover Proton Exchange Membranes for Water Electrolyzers

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:

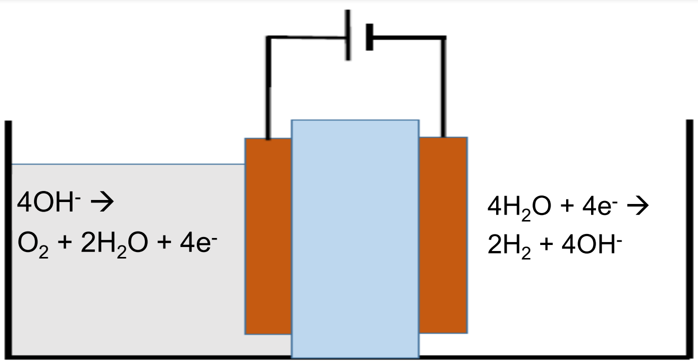
1. **Engineering a thin, reinforced membrane on roll to roll scale**
2. **Remediating gas crossover with recombination catalyst**
3. **Preventing membrane degradation with immobile radical scavengers**

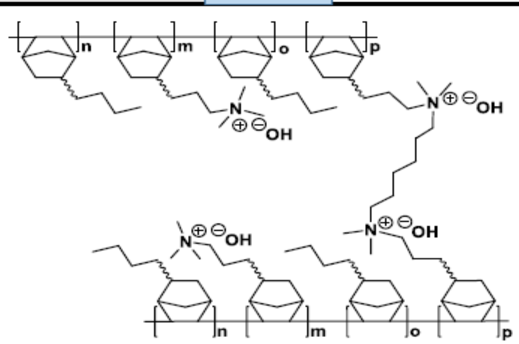
A. Park, P186

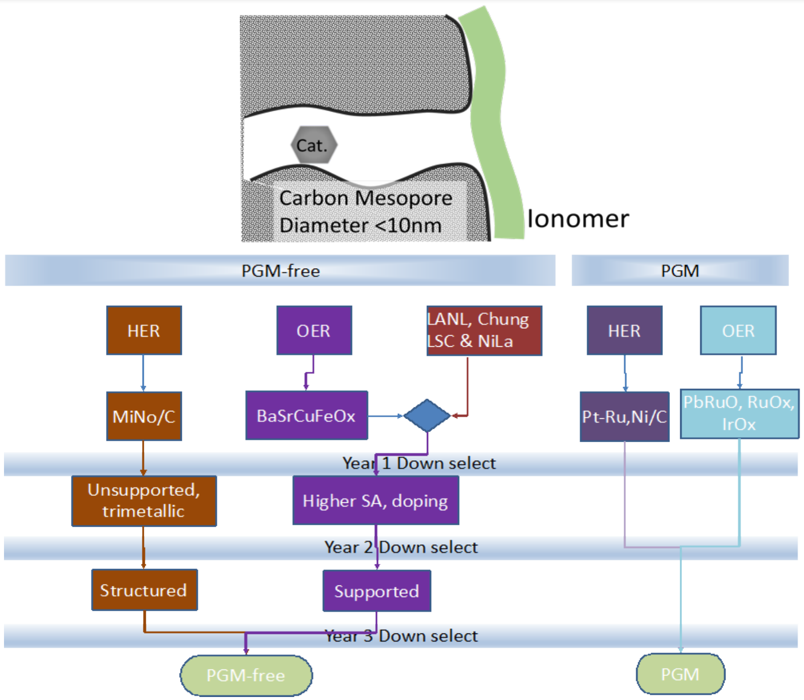
Georgia Institute of Technology | South Carolina | nel | PAJARITO POWDER FUEL CELL CATALYSTS | NREL NATIONAL RENEWABLE ENERGY LABORATORY | BERKELEY LAB | 3

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







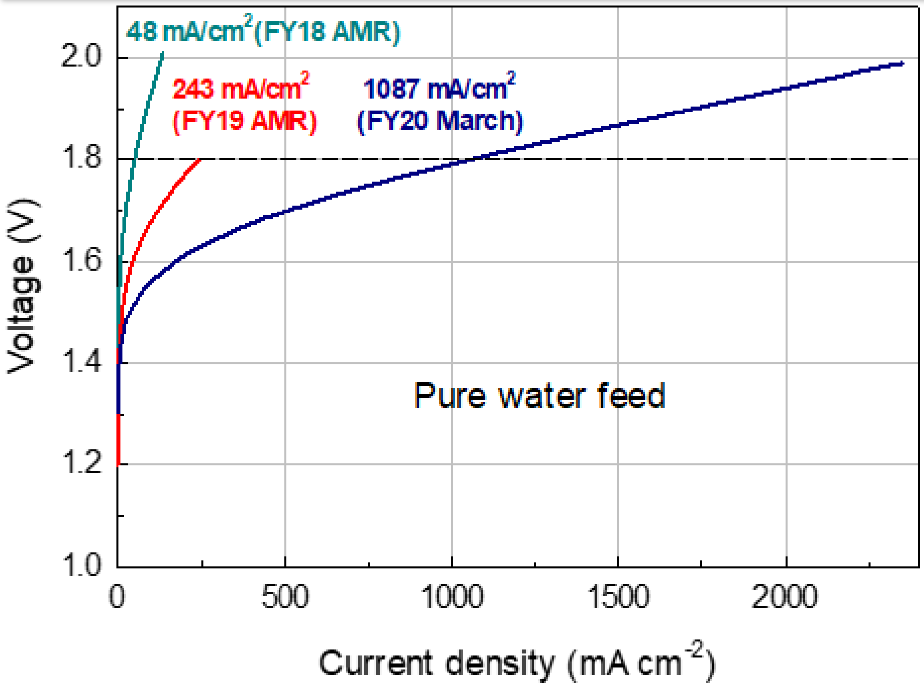
P. Kohl, PD185

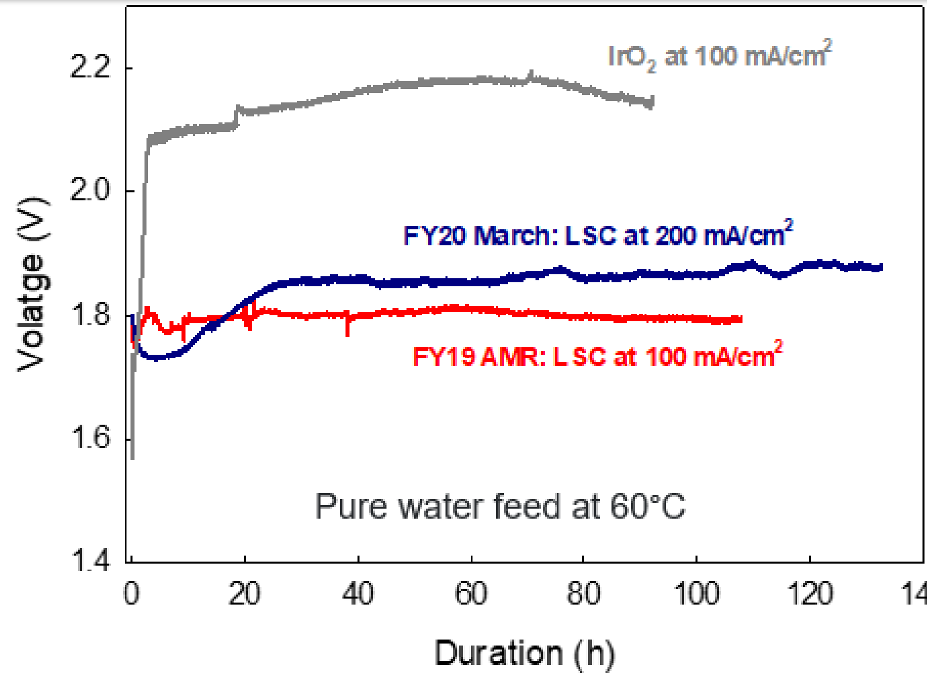
PGM-free OER Catalysts for Alkaline Water Electrolysis

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/cm² at 1.8 V at 85˚C and slowed degradation rates to 0.2 mV/hr at 200 mA/cm² at 60˚C.





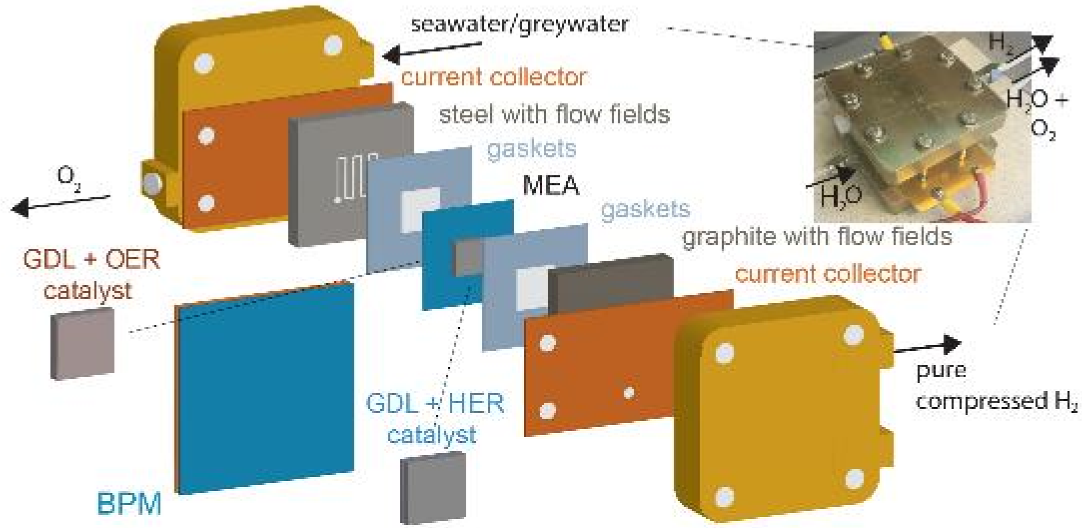
H. Chung, PD158

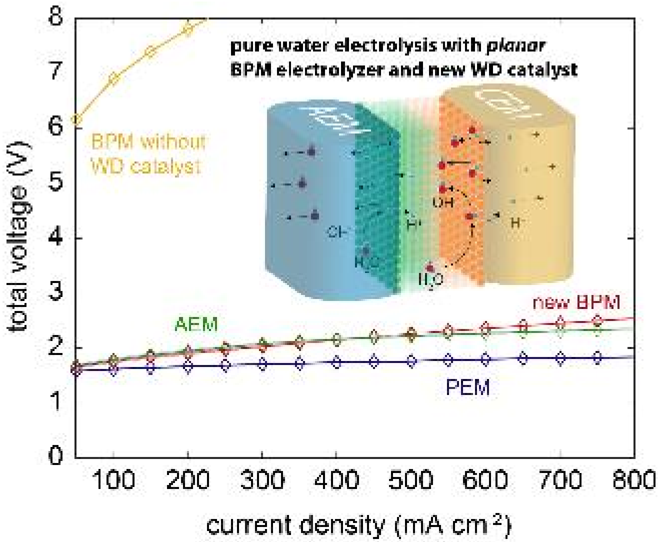
Hydrogen From Membrane Electrolysis of Dirty Water

UNIVERSITY OF OREGON | Sandia National Laboratories | NREL | 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

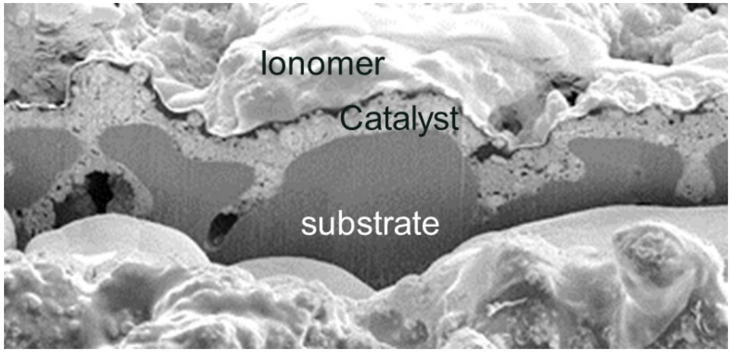
nel · UC Irvine · Oak Ridge National Laboratory · NREL National Renewable Energy Laboratory · Berkeley Lab

7 Nodes

Goals: Develop ultra-efficient PEM electrode per targets below

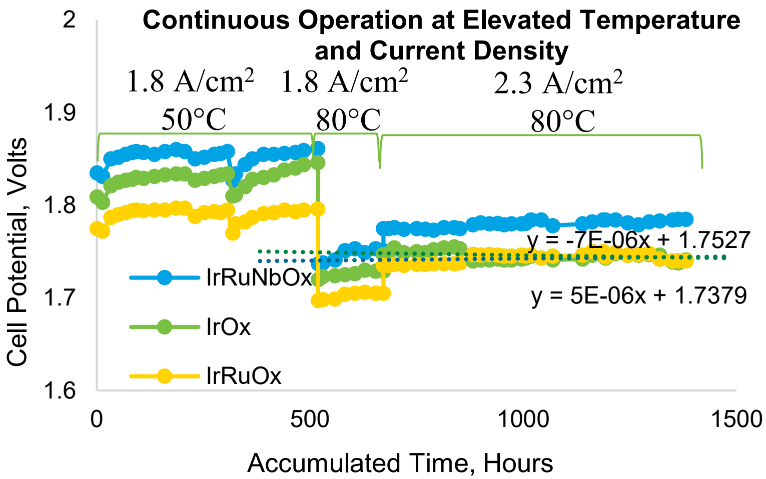
Metric	State of the Art	Proposed
Membrane thickness	*175 microns*	*50 microns*
Operating temperature	*58°C*	*80-90°C*
Cell Efficiency	*53 kWh/kg*	*43 kWh/kg*

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

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

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HydroGEN
Advanced Water Splitting Materials

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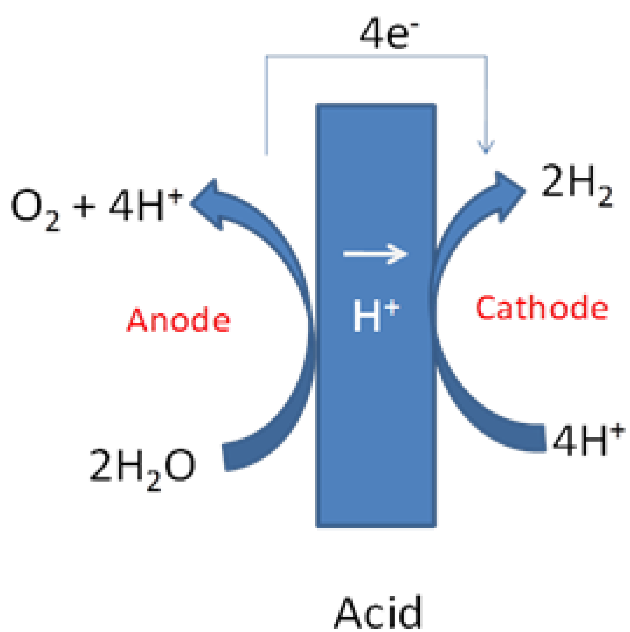
Technical Backup Slides

Overview - LTE Technology

Schematic PEM*

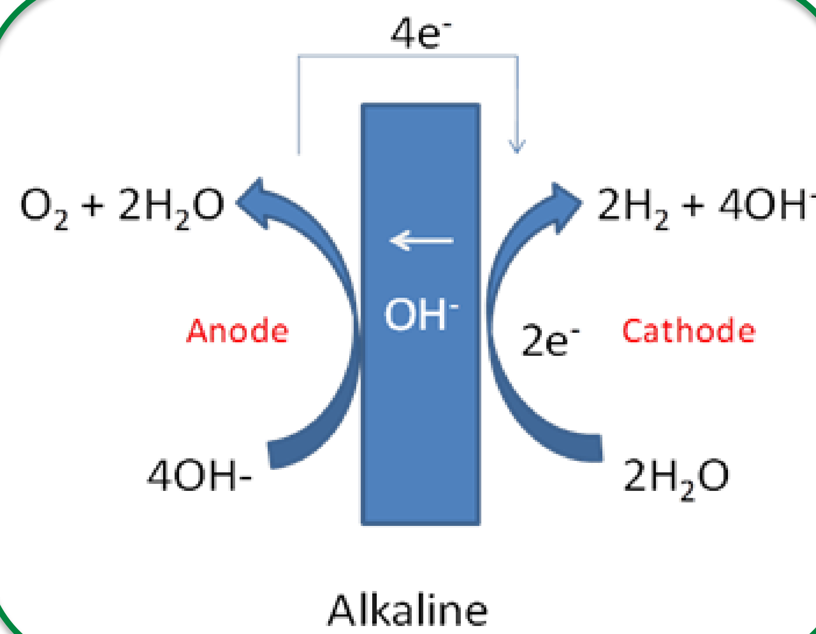


Anode: $2H_2O \Rightarrow O_2 + 2H^+ + 2e^-$

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*

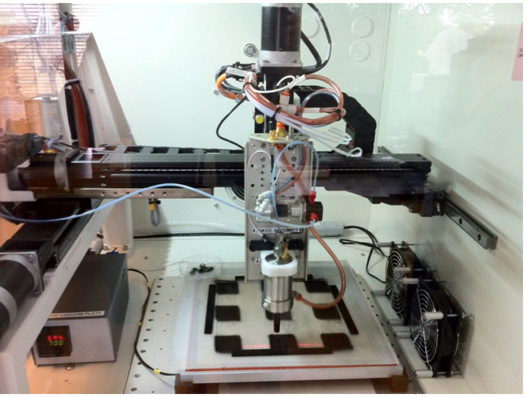
State-of-Art AEM

- 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

*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

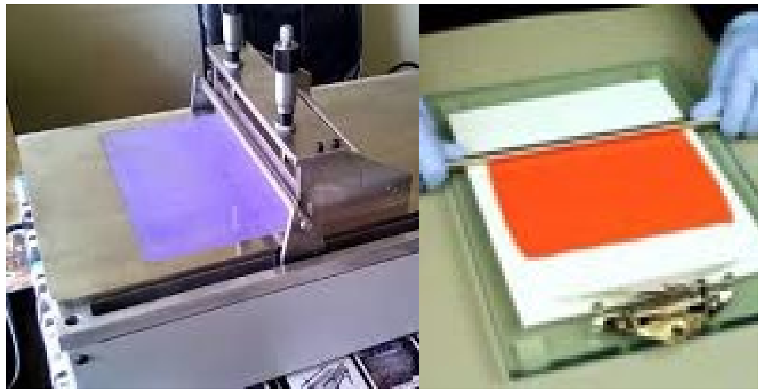
Ultrasonic Spray



Used to demonstrate new materials and for fundamental studies

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

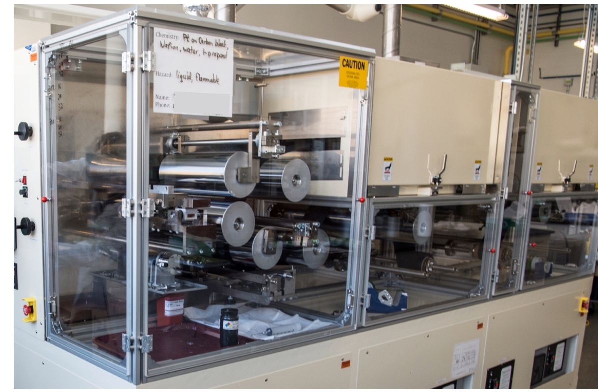
Doctor Blade/Mayer Rod



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

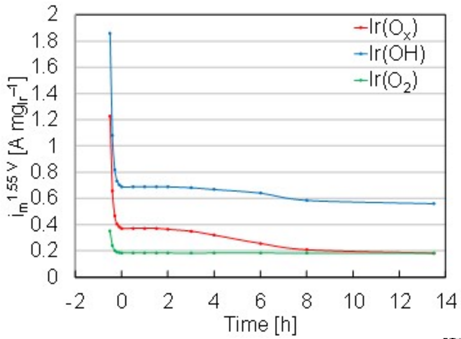
Roll-to-Roll



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)