H2NEW Hydrogen (H2) from Next-generation Electrolyzers of Water:
H2NEW LTE: Task 2 Benchmarking and Performance

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Project ID # 196b

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DOE Hydrogen Program
2023 Annual Merit Review and Peer Evaluation Meeting

Project Goals

Goal: H2NEW will address components, materials integration, and manufacturing R&D to enable manufacturable electrolyzers that meet required cost, durability, and performance targets, simultaneously, in order to enable $2/kg hydrogen (by 2025 on way to H_2 Shot target, $1/kg by 2031).

H2NEW has a clear target of establishing and utilizing experimental, analytical, and modeling tools needed to provide the scientific understanding of electrolysis cell performance, cost, and durability tradeoffs of electrolysis systems under predicted future operating modes

H2NEW Task 2: Performance and Benchmarking Overview

Timeline and Budget

- Start date (launch): **October 1, 2020**
- Awarded through September 30, 2025
- FY22 DOE funding for Task 2: **$1.75M**
- Planned FY23 DOE funding for Task 2: **$2.9M**
- Task 2 funds expected by end of FY'23: **$6.1M**

Barriers

- **Capital Cost**
- **Performance**

Consortium Task Team

Deputy Director:
Debbie Myers (ANL)

Task Liaisons:
Guido Bender (NREL)
Adam Weber (LBNL)

Subtask Leads:
Rajesh Ahluwalia (ANL)
Siddharth Komini Babu (LANL)
James Young (NREL)
Dave Cullen (ORNL)
Ethan Crumlin (LBNL)
Daniel Hussey (NIST)

Discretionary Leads:
Svitlana Pylypenko (Mines)
Shawn Litster (CMU)
Iryna Zenyuk (UCI)

Relevance and Impact

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Technical Targets for PEM Electrolyzer Stacks and Systems [a,b]

CHARACTERISTIC	UNITS	2022 STATUS[c]	2026 TARGETS	ULTIMATE TARGETS
Stack				
Total Platinum Group Metal Content (both electrodes combined) [d]	mg/cm^2	3.0	0.5	0.125
	g/kW	0.8	0.1	0.03
Performance		2.0 A/cm2 @ 1.9 V/cell	3.0 A/cm2 @ 1.8 V/cell	3.0 A/cm2 @ 1.6 V/cell
Electrical Efficiency [e]	kWh/kg H_2 (% LHV)	51 (65%)	48 (69%)	43 (77%)
Average Degradation Rate [f]	mV/kh (%/1,000 h)	4.8 (0.25)	2.3 (0.13)	2.0 (0.13)
Lifetime [g]	Operation h	40,000	80,000	80,000
Capital Cost [h]	$/kW	450	100	50
System				
Energy Efficiency	kWh/kg H_2 (% LHV)	55 (61%)	51 (65%)	46 (72%)
Uninstalled Capital Cost [h]	$/kW	1,000	250	150
H_2 Production Cost [i]	$/kg H_2	>3	2.00	1.00

Source: https://www.energy.gov/eere/fuelcells/technical-targets-proton-exchange-membrane-electrolysis
For details on footnotes a-i see above-referenced website.

- **Task 2 “performance” activities specifically focus on the capital cost and efficiency targets:**
  - ➢ **Membrane selection and crossover characterization to improve efficiency and reduce capital cost**
  - ➢ **Catalyst selection and screening to reduce capital cost and help improve lifetime**
- **Modeling and characterization work within Task 2 cross cuts with Tasks 1 and 3 and help address the durability/lifetime target.**

Approach: Task 2 Breakdown

Task 2a: Performance benchmarking, baselining, validation

i. Coordination with domestic (HydroGEN Topic 2b) and international partners (IEA)
    - Development of protocols, reference cells, round robin
    - Development of reference materials and standardized tests for cell material evaluation

Task 2b: Cell performance testing in support of electrode development

i. In-situ cell testing independent of durability testing
    - Advanced diagnostics, operating condition studies, segmented cell

ii. In-operando (beam-line) characterization of cells for performance enhancement & degradation studies

Task 2c: Ex-situ studies focused on performance factors

i. Advanced characterization of structure, morphology and properties of inks, catalysts, cell components and electrodes

Task 2d: Cell level model development

Approach: Task 2 Quarterly Progress Measures

Milestone Name/Description	Criteria	End Date	Status
(Tasks 1 and 2) Membrane swelling impact on performance and durability	Quantify the impact of membrane swelling processes at the membrane/anode and PTL/anode interfaces on cell performance, (via voltage breakdown and impedance analysis), and assess potential impact on durability, by systematically varying at least one material or processing variable (such as membrane equivalent weight, membrane pretreatment, membrane thickness, membrane reinforcement, or PTL modification). (NREL, LBNL)	9/30/2022	Complete, see slides #17, #21 and Task 1 poster
(Task 2bi) Baselining conditioning procedure.	Establish the correlation of the in-situ performance (polarization curve, EIS, CV) to the changes in morphology on the different break-in procedures. (LANL, ANL, ORNL, NREL)	03/31/2023	Complete, see slide #12
(Task 2bii) PTL coating and PTL oxidation state with anode potential	Report on impact of anode potential on the Pt PTL coating and Ti PTL using ex-situ and operando X-ray absorption spectroscopy and other techniques. (ANL, NREL, LANL, ORNL)	03/31/2023	Complete, see slide #14
(Task 2c) Ionomer impact on OER kinetics	Report on impact of PFSA on OER kinetics for Alfa Aesar IrOx and Umicore Ir/TiOx core-shell catalyst by comparing micro-cavity and RDE kinetics. Visualize differences in ionomer distribution by cryo-EM. (ANL, LBNL, ORNL)	09/30/2023	In preparation

Approach for LTE Performance in Detail

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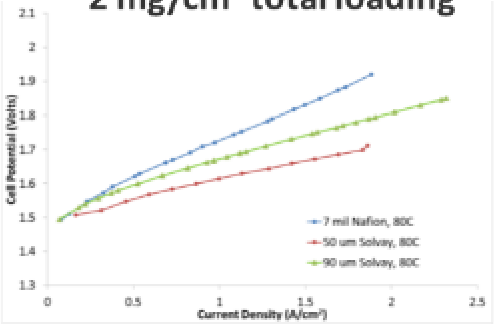
NEL 2016 Status
2 mg/cm² total loading



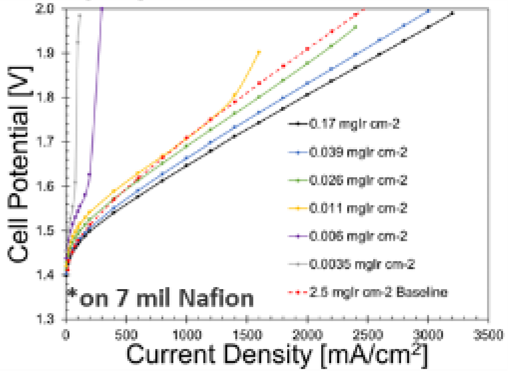
N. Danilovic, ECS Transactions, 75 (2016) 395

- Commercial MEAs are "overdesigned" with high catalyst loadings and thick membranes
- Poor understanding of structure, interfaces and performance impacts

Circumvent Edisonian Approach

Commercial materials:
0.4 mg_{Ir}/cm^2
2 mil membrane
Ti PTL and Pt coating

Spray coated CCM Status



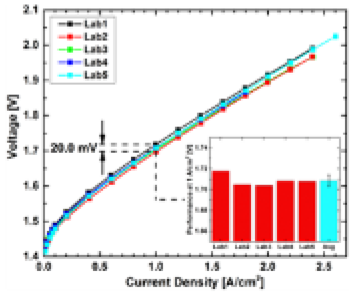
Z. Taie, ACS AM&I, 12 (2020) 52701

- Academic MEAs have demonstrated loadings as low as 0.05 mg/cm²
- Translation to high-throughput MEAs is not feasible without an understanding of underlying properties and function

Understanding — Structure Property Relationships — Rational Solutions

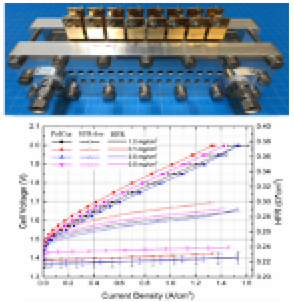
2026
69% LHV @ 3 A/cm²
0.5 mg_{PGM}/cm^2

Performance & In-Situ Characterization



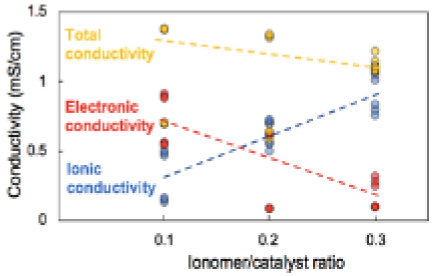
Utilize in situ characterization techniques to evaluate materials and components. Establish protocols and benchmark across institutions.

Advanced In-Situ Characterization



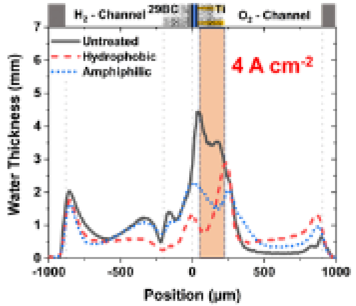
Develop and utilize advanced electrochemical diagnostics.

Ex-Situ Diagnostics



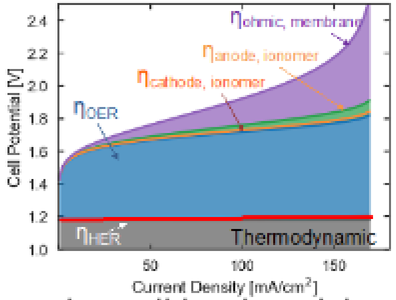
Develop and utilize ex-situ diagnostic techniques to extract component level parameters and to determine performance impacting characteristics.

In-Operando Characterization



Develop and utilize operando techniques to identify performance and durability limiting phenomena in operating cells.

Cell Level Modeling



Utilize cell level modeling to understand underlying mechanisms and to guide in design and optimization of cells and components

Accomplishments: Expanded Single Cell Test Capabilities

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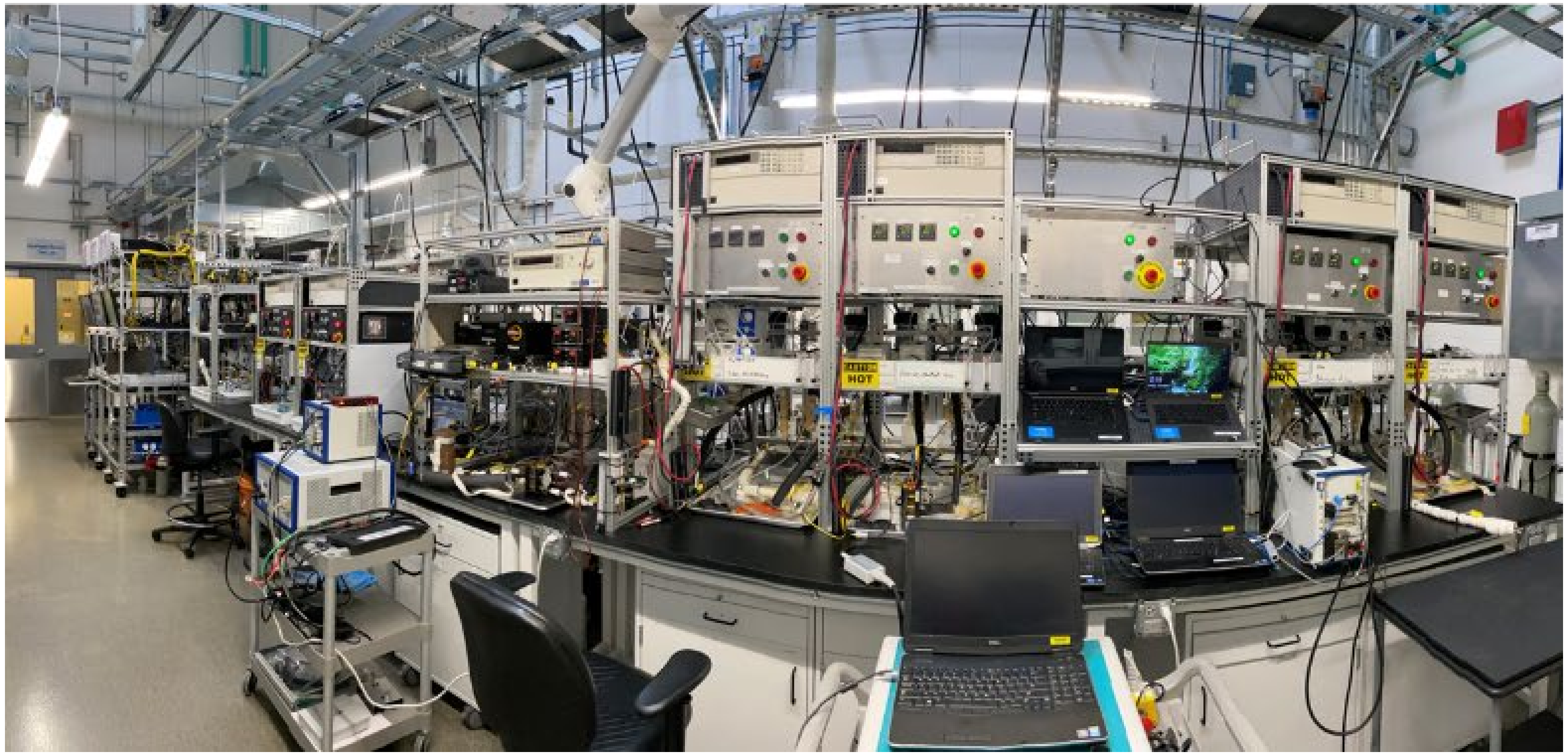
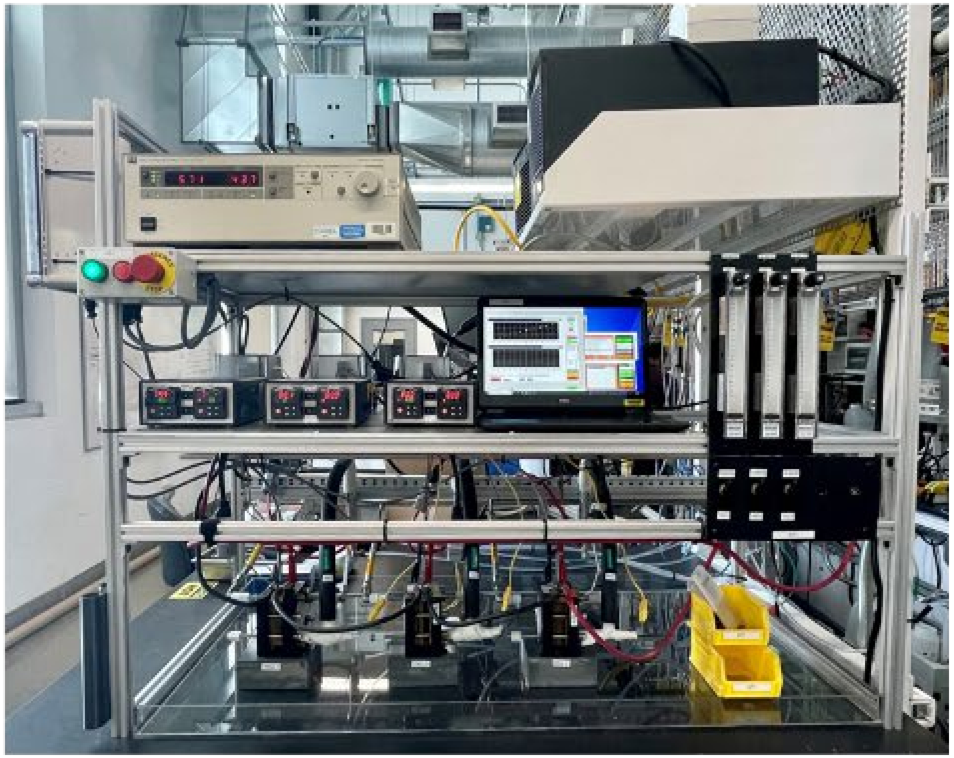
Single Cell Test Stations



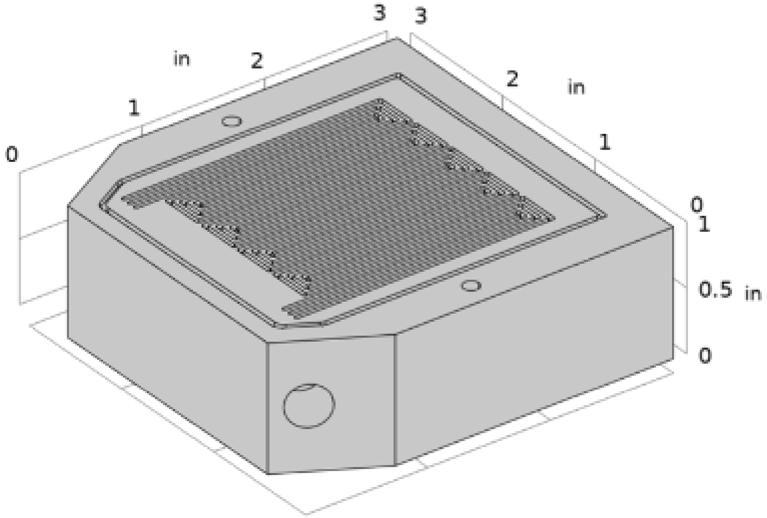
High Throughput Test Stations



- **Performance testing for Task 1, Task 2, & Task 3**
- **Majority of test stations can do single cell ambient/ambient testing**
- **7 / 14 NREL test station have H2Xover capabilities**
- **2x can do high pressure**

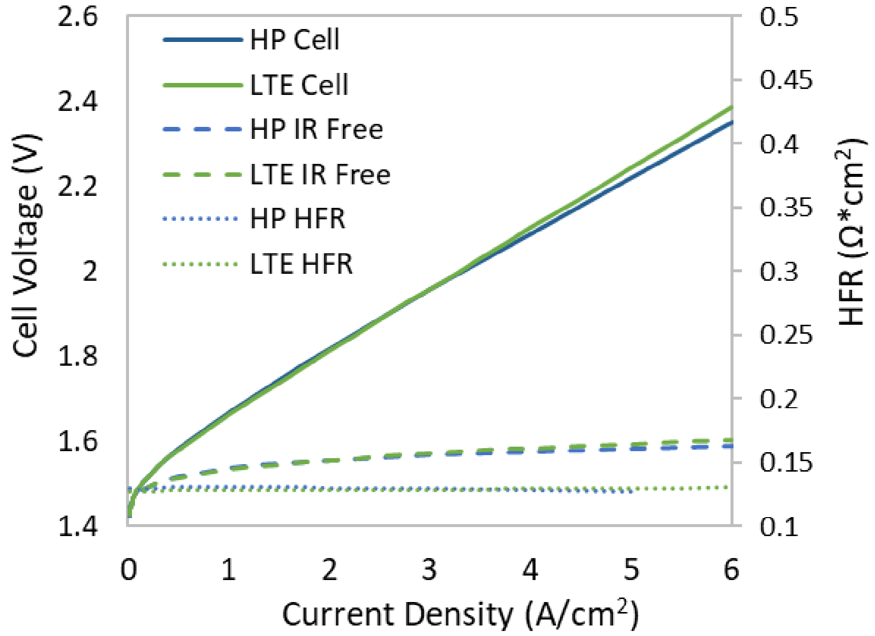
Task 2a: Accomplishments – High Pressure LTE Hardware

- Developed 30 bar_g rated LTE hardware with 25 cm^2
- Mask down cell area for sizes < 25 cm^2
- Research hardware: PTLs & GDLs of various thicknesses can be used (unlike existing commercial options)
- Drawings will be released as open source on H2NEW website
- 50 cm^2 cell version under development



- High pressure sealing enabled by:
  - Stringent tolerances for flatness and parallelism
  - O-seal fittings (>2000 psi) for inlets/outlets (see picture at right)
  - Increased flow field thickness for reduced deflection

O-seal fitting



Task 2a: Accomplishments – International Benchmarking

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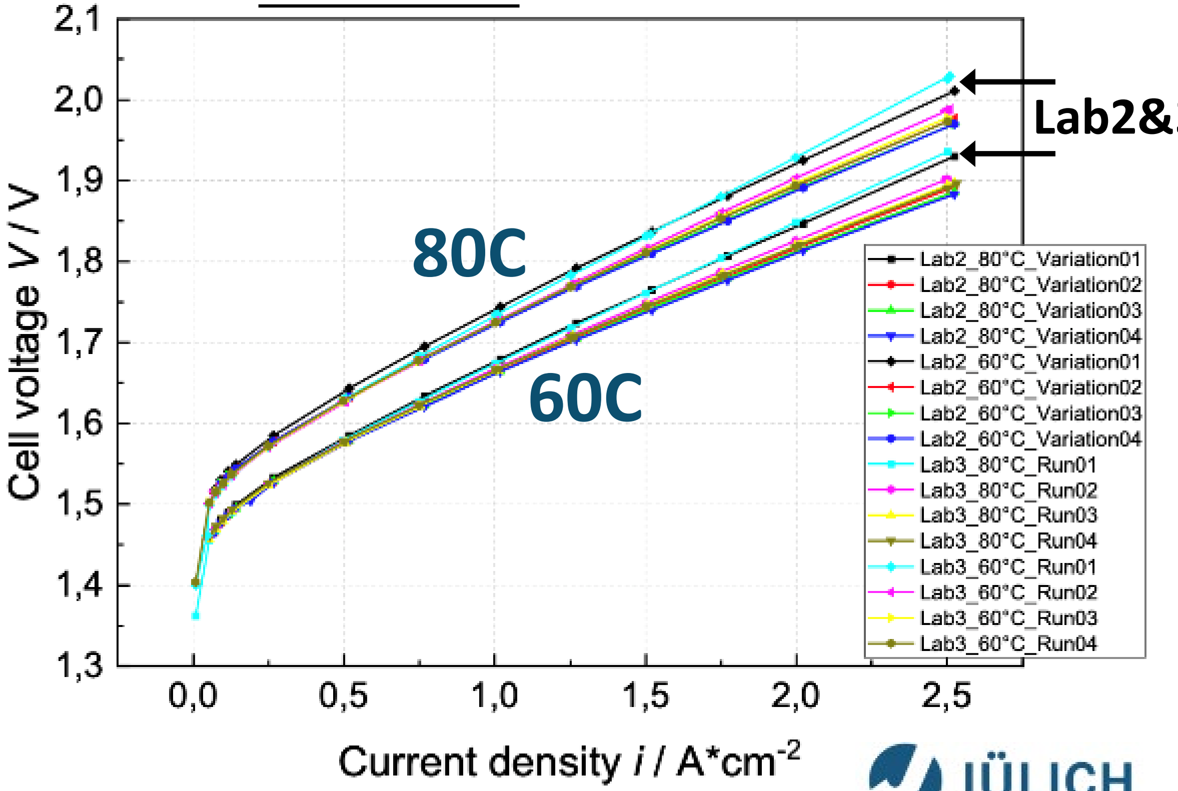
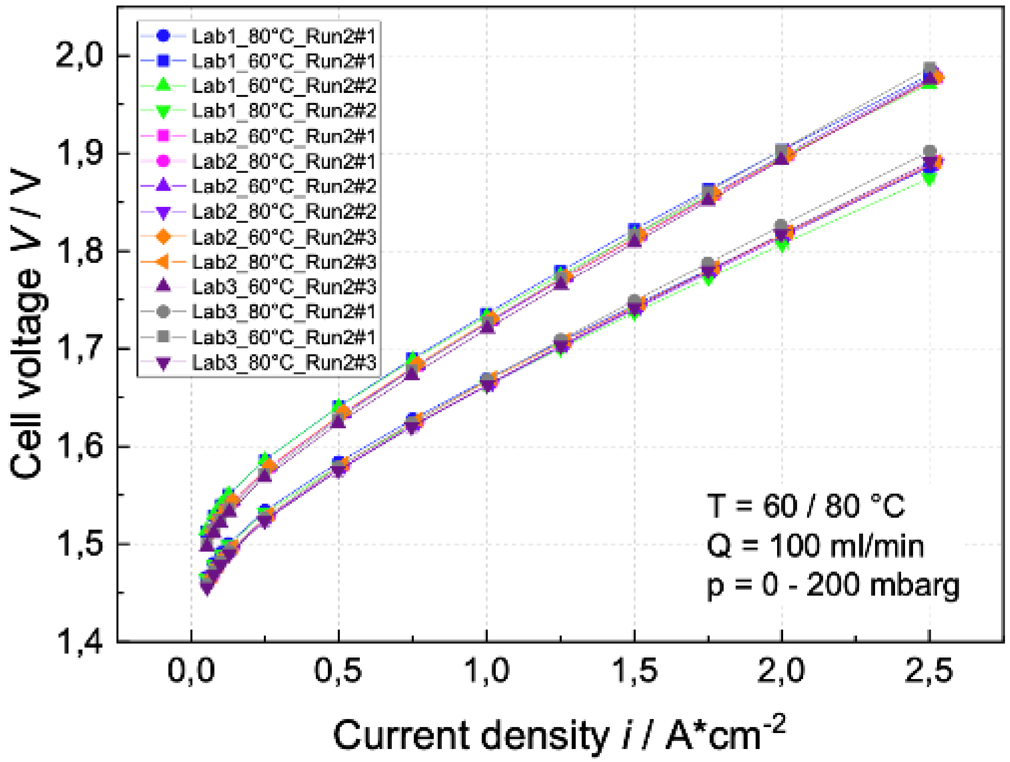
Runs #1-4 from All Labs



Runs #2-4 from All Labs



JÜLICH FORSCHUNGSZENTRUM

Fraunhofer ISE

Lab1 stable for **Runs #1-4**

Lab2 and **Lab3** stable for **Runs #2-4**

-> Conditioning not complete until after at least one polarization curve is taken

Current density / A*cm²	ΔUmax / mV (T = 60 °C)	ΔUmax / mV (T = 80 °C)
0.1	18	12
1	15 (27)	8 (20)
2	10	19

< 20 mV X-Lab variation is possible

In-house reproducibility of < 10 mV (2 A/cm²) typical

Task 2a: Accomplishments – H2NEW Internal Benchmarking

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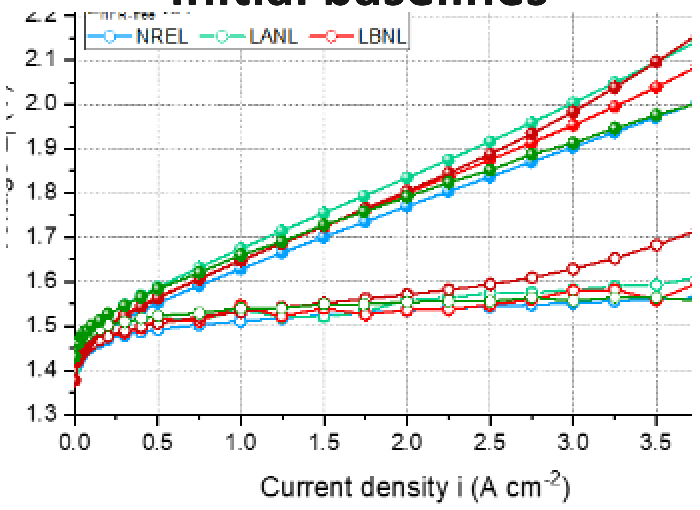
- **Several test iterations dramatically improved comparability across labs using traveling cell hardware**
- **Improvement from 200 mV variation down to 4 mV**
  - ✓ Removal of water quality issues
  - ✓ Protocol expansion to include reconditioning between separate experiments
- **In-situ experimental results are fully reproducible across labs**
- **Cell assembly & fabrication are next**

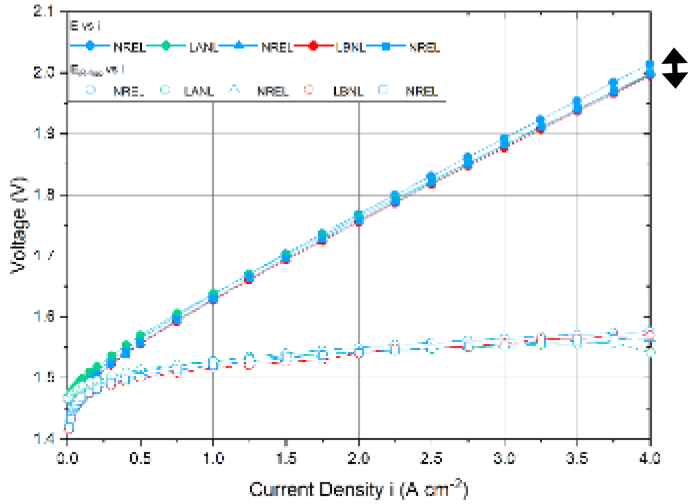
Initial baselines



200 mV @ 4 A/cm^2

All: 19 mV

Phase 1 traveling cell



All after re-conditioning: **4 mV**

Task2bi: Accomplishments – Determined Effects of Different Cell Conditioning Protocols

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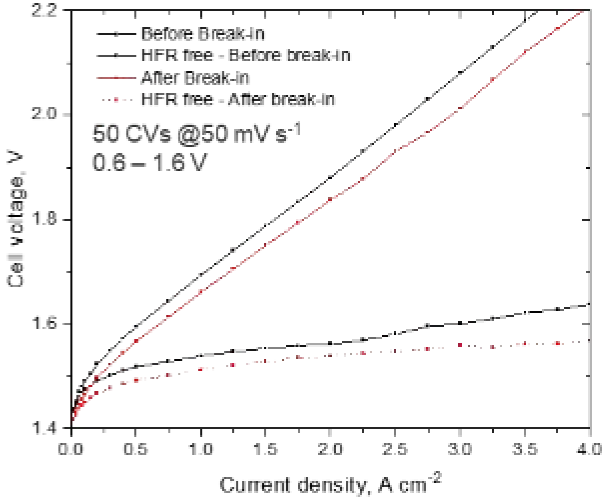
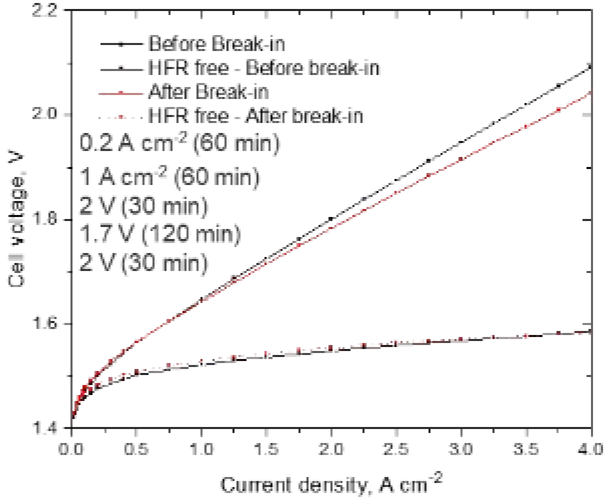
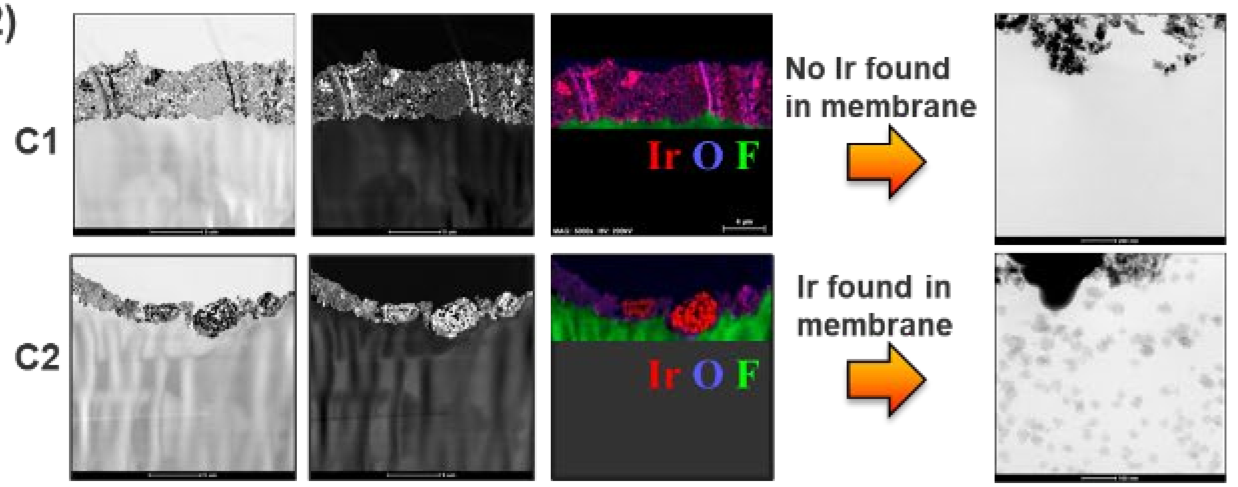
Conditioning Procedure 1 (C1)

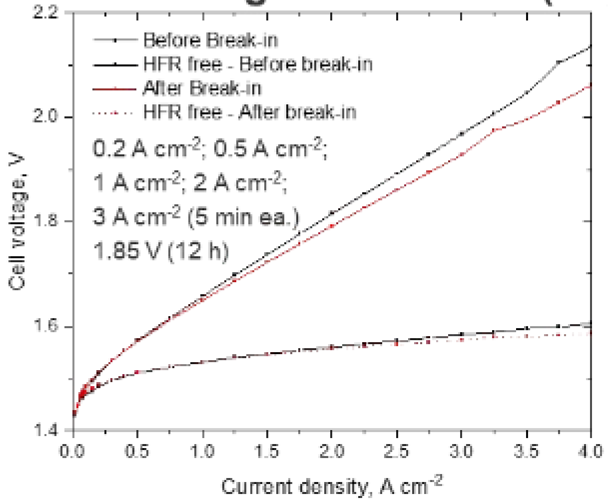


Conditioning Procedure 2 (C2)

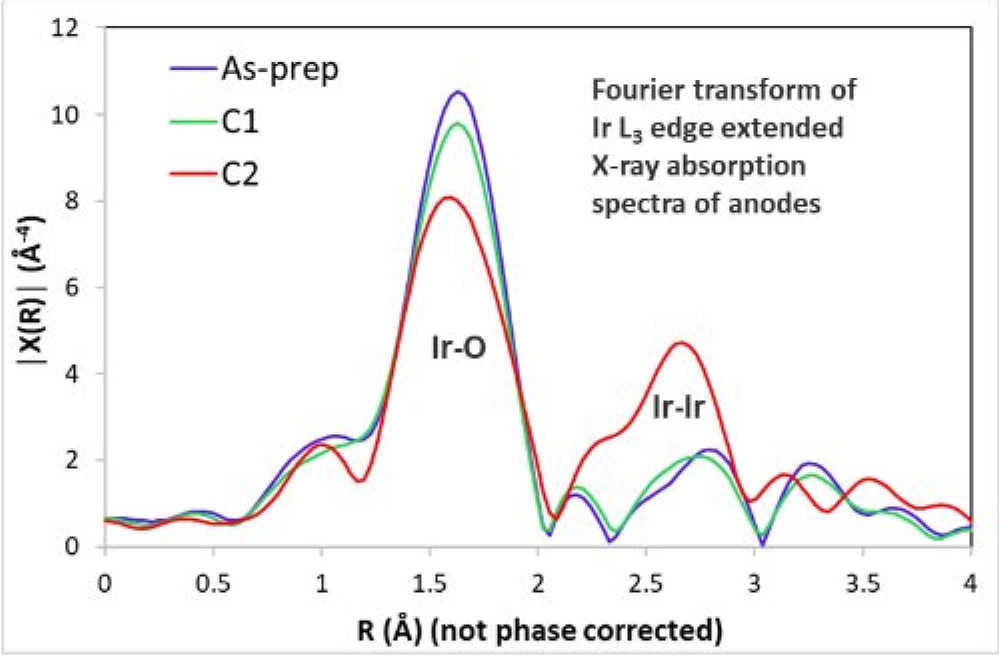




Conditioning Procedure 3 (C3)



- **Dissolution of Ir and its transport into the membrane**
  - **Not observed with conditioning C1 with short CVs up to 1.6 V**
  - **Observed with conditioning C2 with long voltage holds up to 2V**
- **Conditioning C2 results in the presence of metallic Ir**

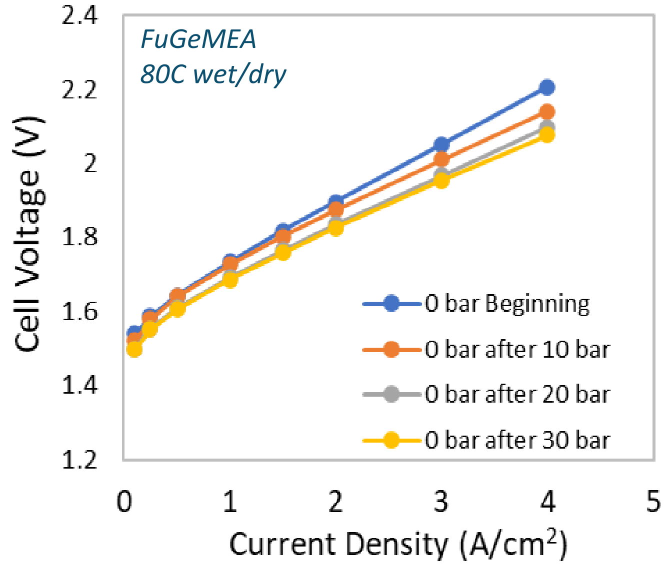


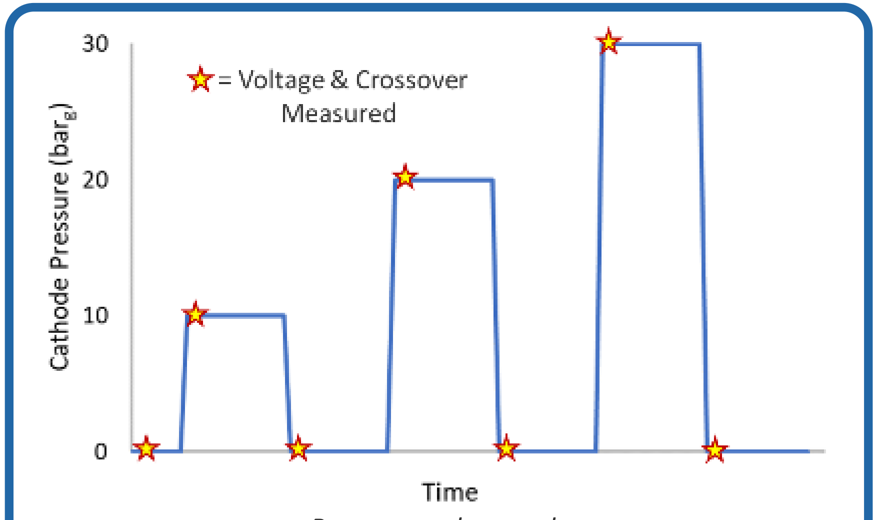
Conditioning details given in backup slides.

80 °C, 5 cm^{-2}, 10 ml min^{-1}; **Membrane**: N115; **Anode:** IrO_2 0.1 mg cm^{-2}; **Cathode:** Pt/C, 0.1 mg cm^{-2}

Task 2bi: Accomplishments – Elucidated Effects of Pressure Cycling

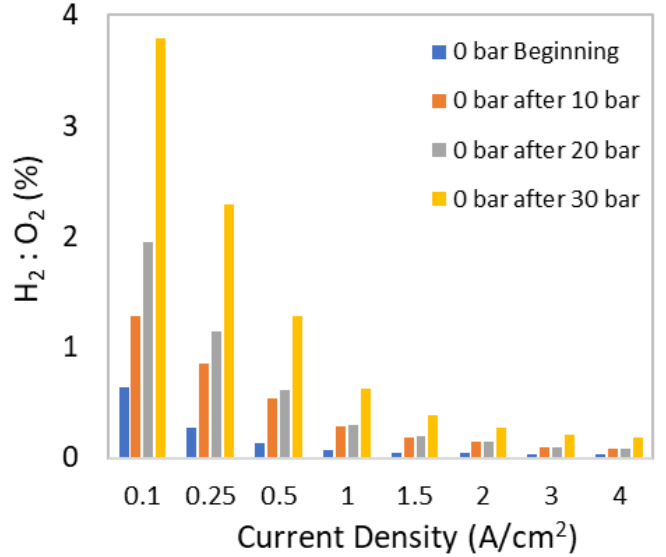
- **Studied impact of differential pressure cycling on ambient pressure VI performance and H_2 crossover**
- **Cell voltage decreased after cell exposure to increasing Δp**
  - **In contrast to expectations from Nernst equation**
  - **A second process is occurring**
  - **We are suspecting a decrease in membrane thickness as cathode pressures force CCM intrusion into the PTL**





Pressure cycle test plan

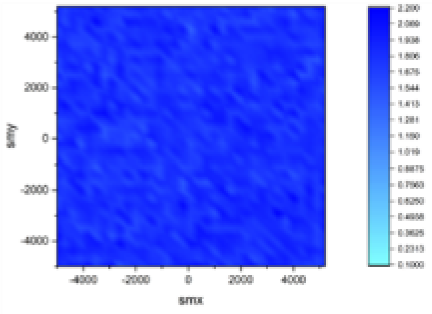
- **H_2 crossover increases dramatically after exposure to elevated pressures**
  - **This supports the membrane thinning theory**
- **Future work will include EIS to enable a full voltage breakdown analysis**



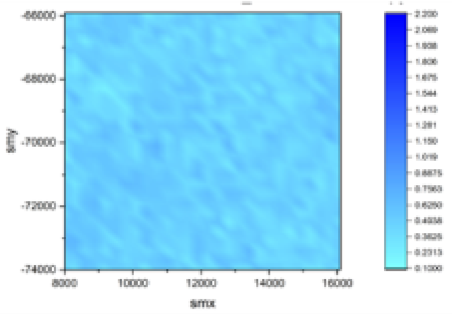
Task 2bii: Accomplishments
– Elucidated Effects of Anode Potential on Pt-coated PTL

Ex situ Synchrotron X-ray Mapping of Pt Loading

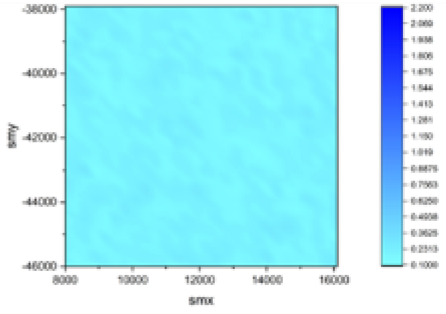
As-prepared Pt-coated Ti PTL



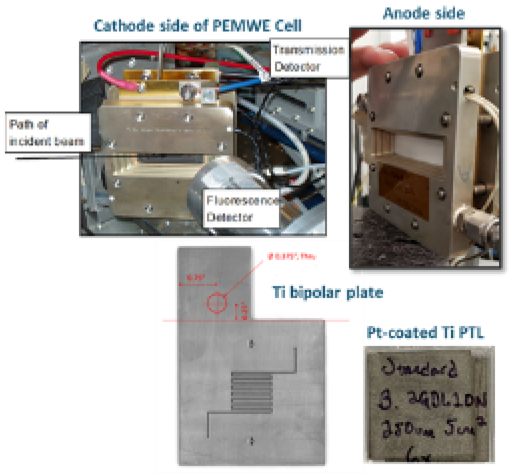
Pt-coated PTL after 2V to OCV; cathode purge

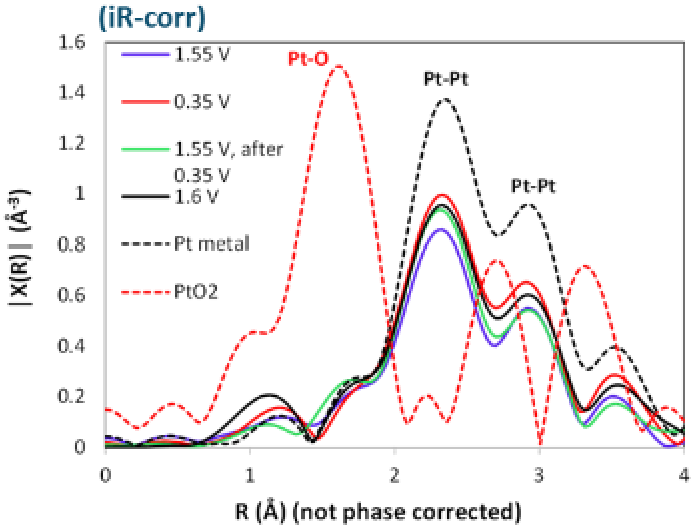


Pt-coated PTL after 2V to OCV; no cathode purge



Operando Pt X-ray Absorption Spectra of PTL Coating as a function of Anode Potential

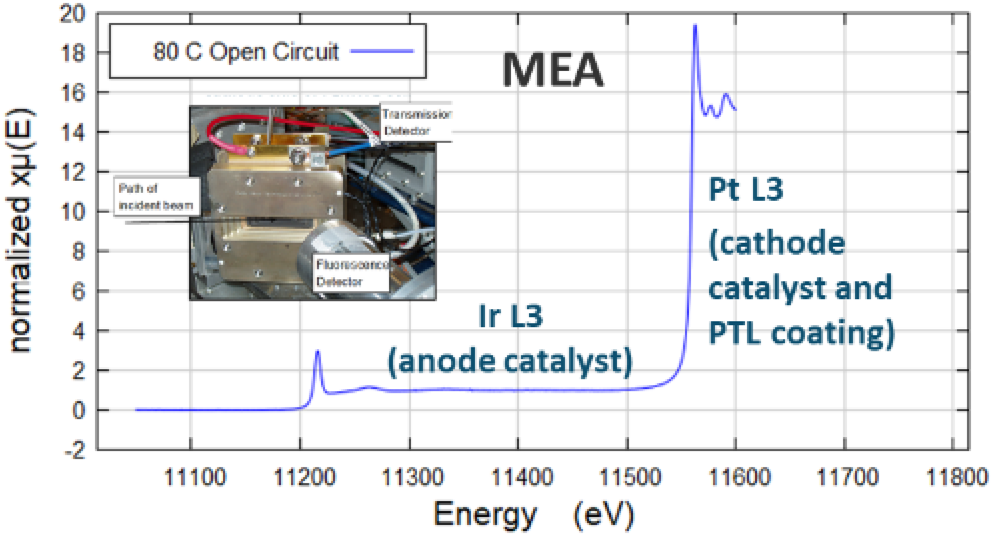


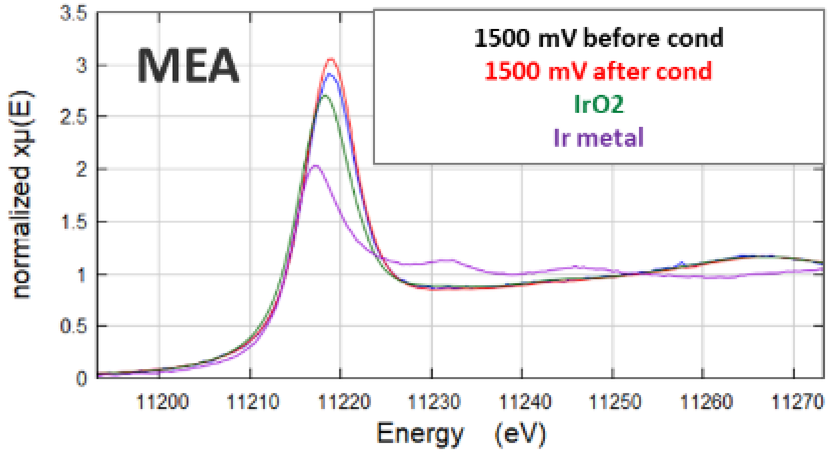


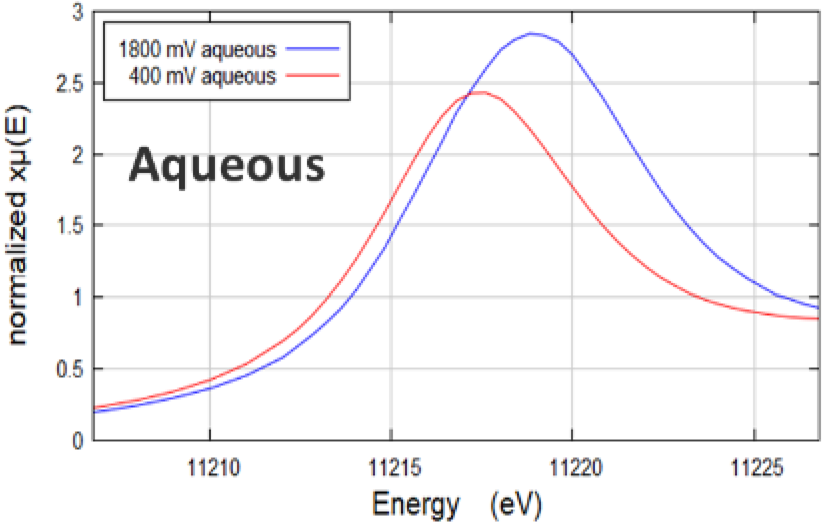
- Higher loss of Pt from PTL coating without cathode purge
- No significant oxidation of Pt coating up to anode potentials of 1.6 V (iR-corrected); evidence of Pt coarsening is noted

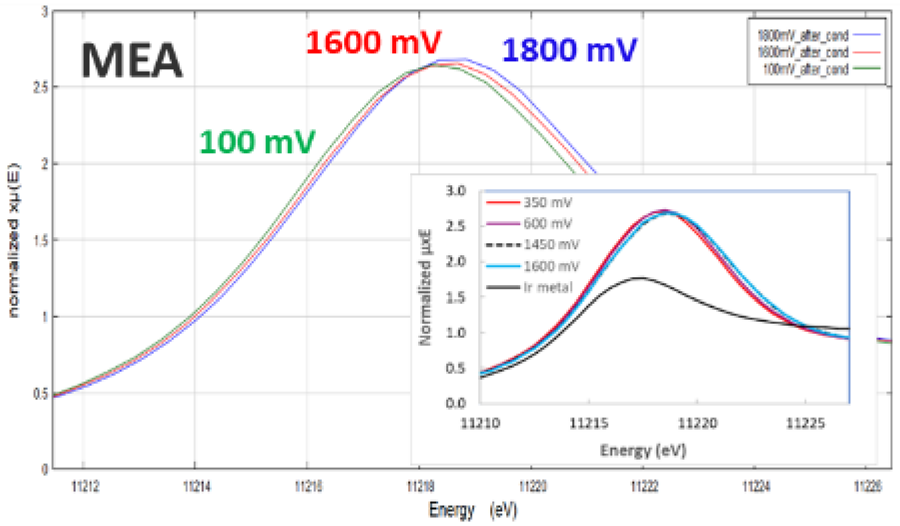
Task 2bii: Accomplishment – Determined extent of Ir oxidation using Operando X-ray Absorption Spectroscopy

- **Conditioning:**
  - 0.6 to 1.6 V, 2.5 s hold at each voltage, 2 V/s, 500 cycles
  - Alfa Aesar IrO_x average oxidation state > 4+ at 1550 mV
  - Ir oxidation state increase after conditioning
- **Potential:**
  - Increase in IrO_x oxidation state with potential observed in MEA, but to a much lesser extent than in aqueous electrolyte







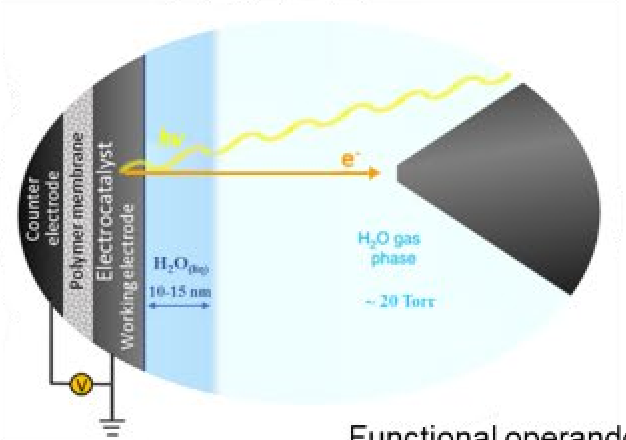


•FuGeMEA CCMs: 0.4 mgIr cm-2 Alfa Aesar IrOx; 0.1 mgPt cm-2 TKK +46wt% Pt/HSC

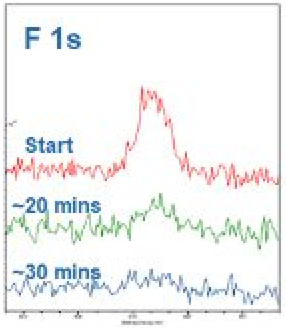
Task 2bii: Accomplishments – Further Developed & Demonstrated Operando Characterization of Ir with Ambient Pressure XPS

- **Flow cell 2-chamber APXPS electrolyzer facilitates operando characterization of working electrode exposed to 20 Torr water vapor (100% RH at room temperature).**
- **Tender X-rays allow probing 10-30 nm's into the composite electrode interface while operating**

Understanding of beam influence & kinetics

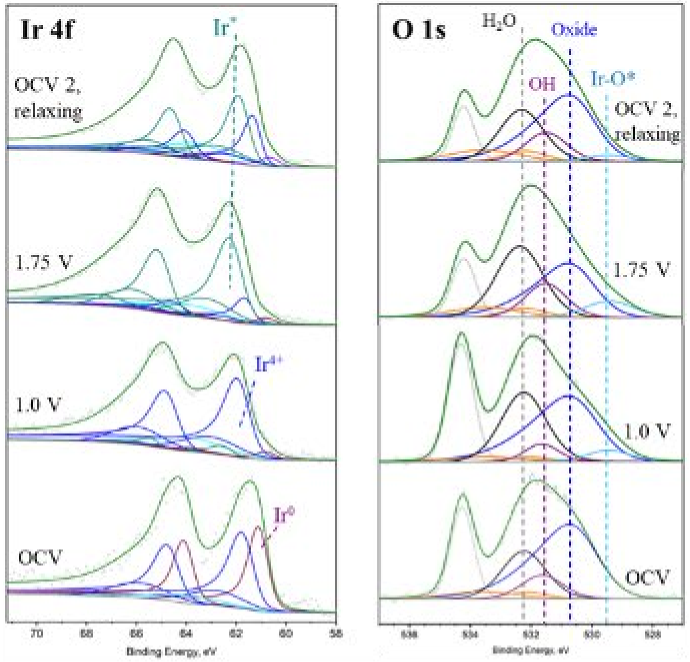


Functional operando cell with access to catalyst layer



⇦ Faster beam damage to polymer components: Beam induced C-F bond cleavage

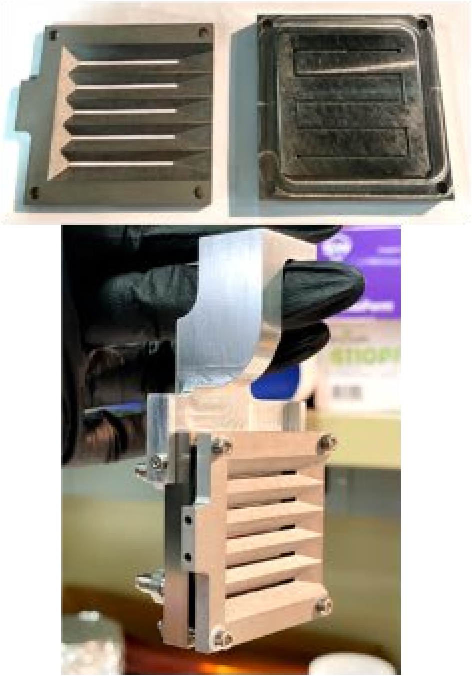
Data for TKK Ir Black Anodes



TKK Ir Black: Catalyst oxidation with application of potential:

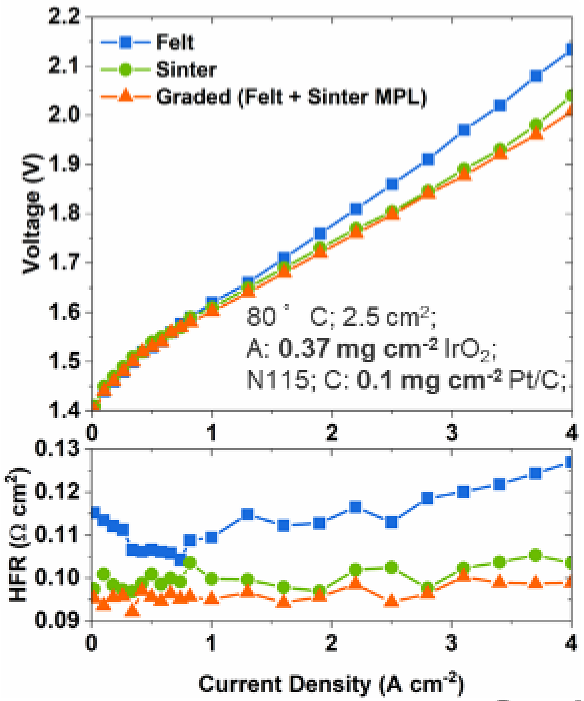
- Loss of Metallic Ir and formation of Ir* (3+ or 5+)
- Development of O* moiety at ~530 eV (deprotonated O/ -OOH)

Heterogeneity in response to potential: Upgrade of cell design for better performance



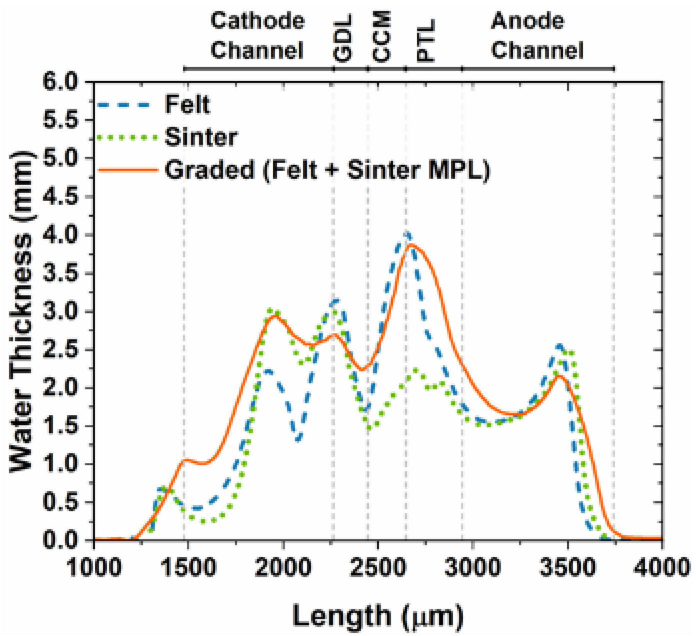
- Narrow channels prevent MEA bulging with pressure and more even application of potential.
- Provides more area to investigate

Task 2bii: Accomplishments – Operando Characterization of Effect of PTL Morphology

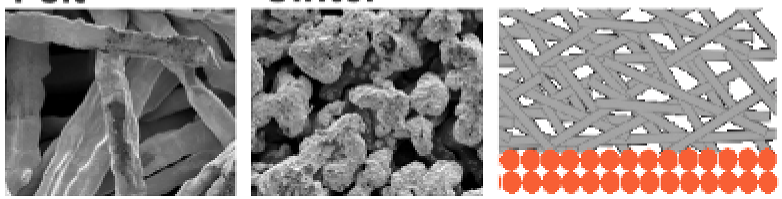


- Performance of cell with microporous layer (MPL) on PTL is higher due to better contact with anode catalyst layer
- Hierarchical structure of PTL with MPL provides better water management
- Able to compare same PTL with neutrons and X-ray imaging

Water thickness from neutron Imaging at 2 A cm^{-2}

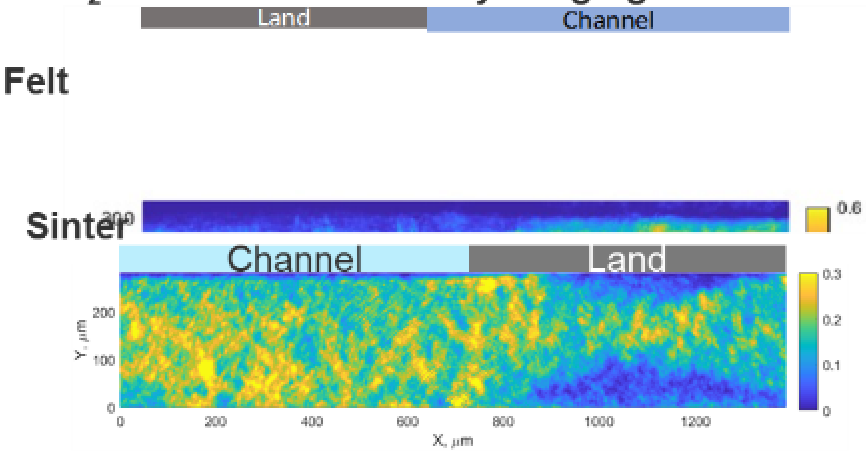


Felt Sinter Graded



PTL	Thickness (μm)	Porosity	Average Pore Size (μm)
Felt	~240	0.67	15.40
Sinter	280	0.37	4.90
Graded	240+60	0.67+0.40	15.40 + 3.97

O_2 saturation from X-Ray Imaging at 2 A cm^{-2}



- Combination of operando Neutron and X-Ray imaging to study O_2 bubble in PTL and water distribution across the MEA.
- Future Work: Ex-situ and operando characterization of new PTLs (laser modified, PTLs with MPL, PTLs graded porosity) being studied in H2NEW

Task 2c: Accomplishments
– Studied Effects of PTL Coatings using New Techniques

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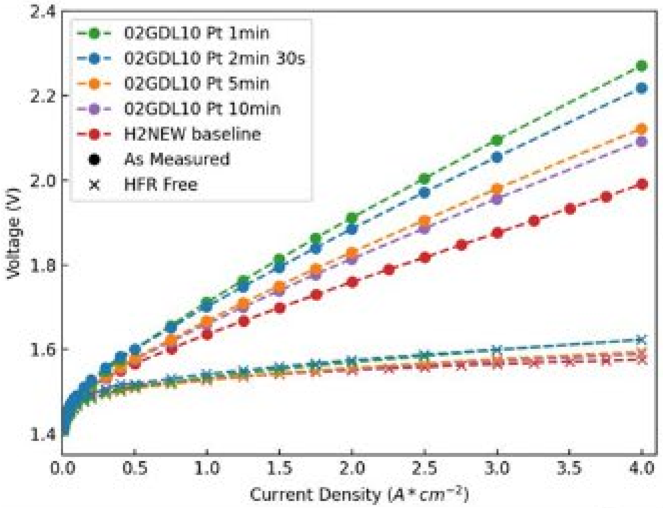
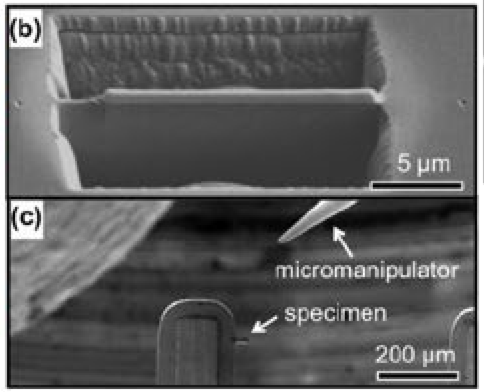
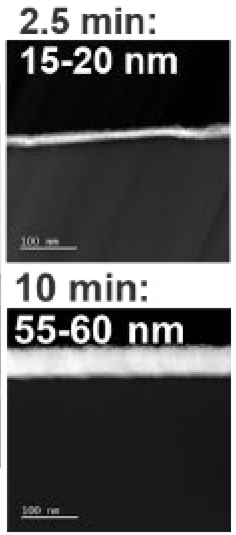
In-Situ Results

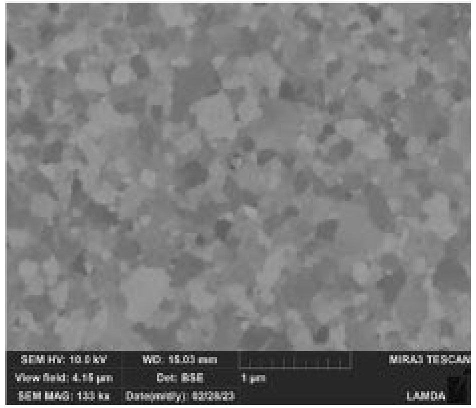


FIB lift-out of surface of Pt-coated PTLs



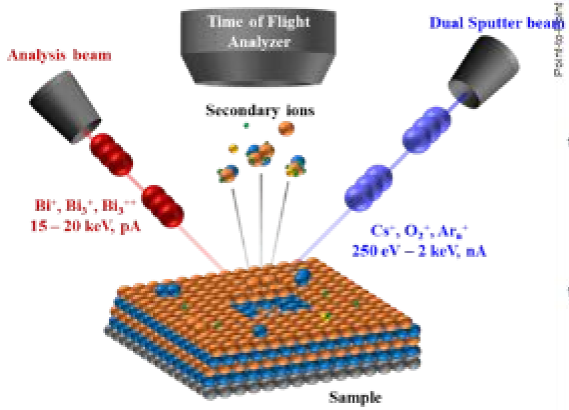


Emergence of Porosity and Grain Growth after Annealing

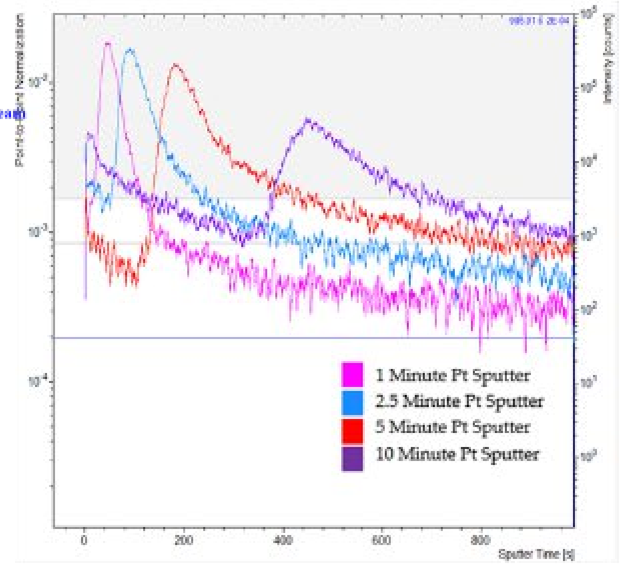


- **Ohmic & kinetic performance effects due to thrifting PGM protective coatings**
- **Characterizing thickness and composition of Pt protected porous transport layer (PTL)**

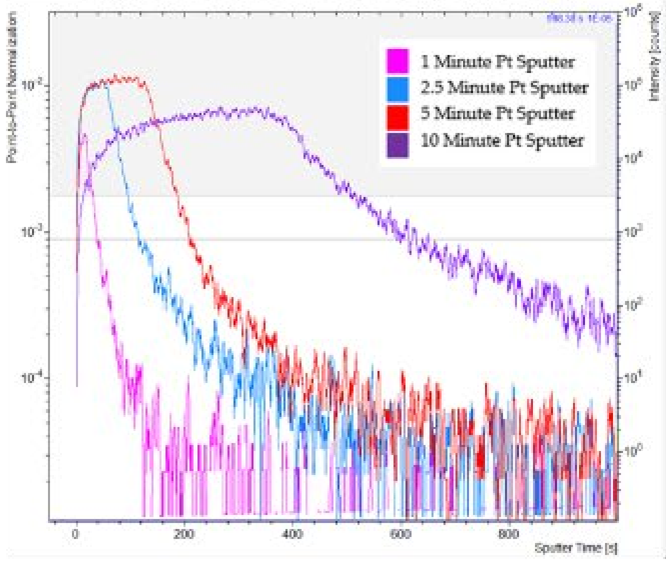
TOF-SIMS of model Pt-coated Ti plates



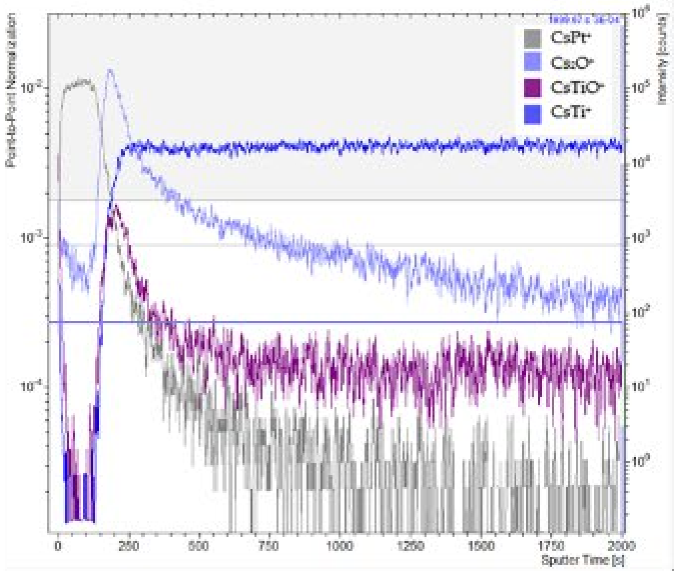
Depth Profile of Oxide vs sputter time



Depth Profile of Pt vs sputter time



Layer Composition 2.5 Minute Pt Sputter



Task 2c: Accomplishments – Characterized PTL surfaces; added capabilities

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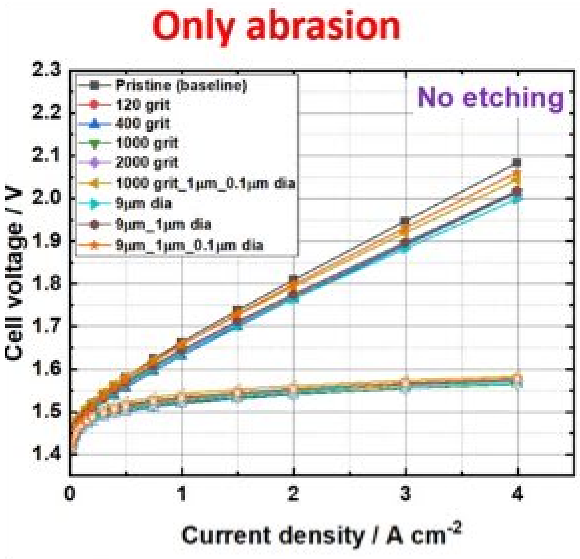
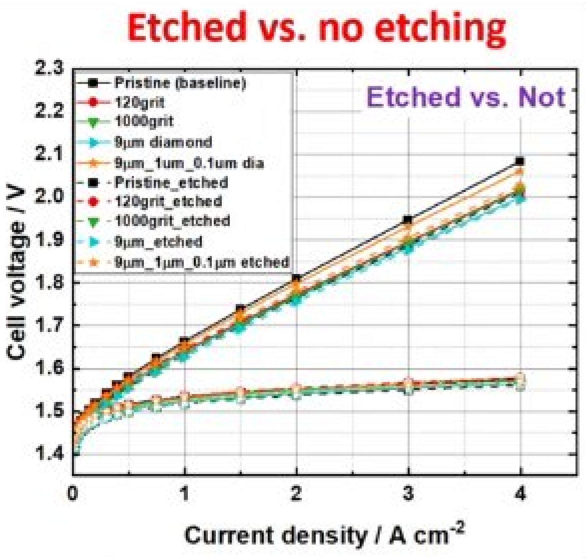
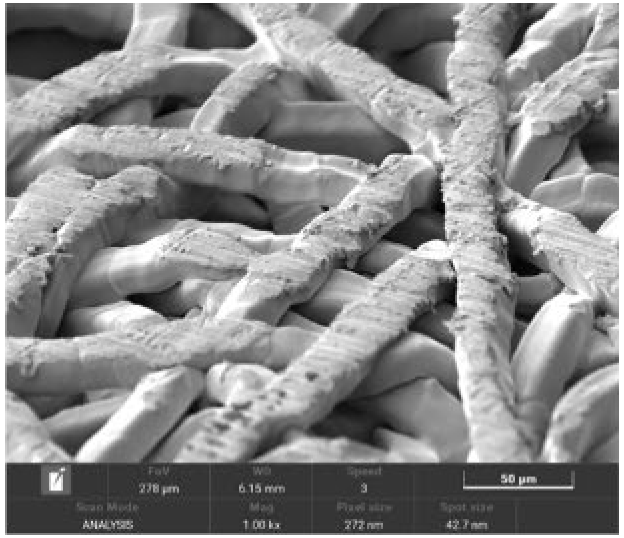
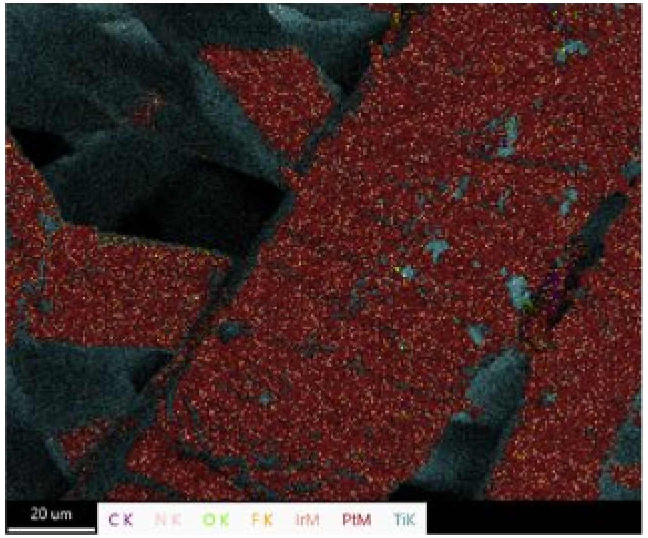
Example: Studying impact of PTL surface roughness





1000 Grit Abrasion + Pt Coating with platinum)





In-situ VI-performance observations

- Abrasions of the PTL may result in performance improvement which may be assigned to
  - ➢ change in roughness increasing the extent of PTL-anode contact
  - ➢ removal of TiOx
- Microscopy characterization tools give insight into the applied morphology and elemental changes

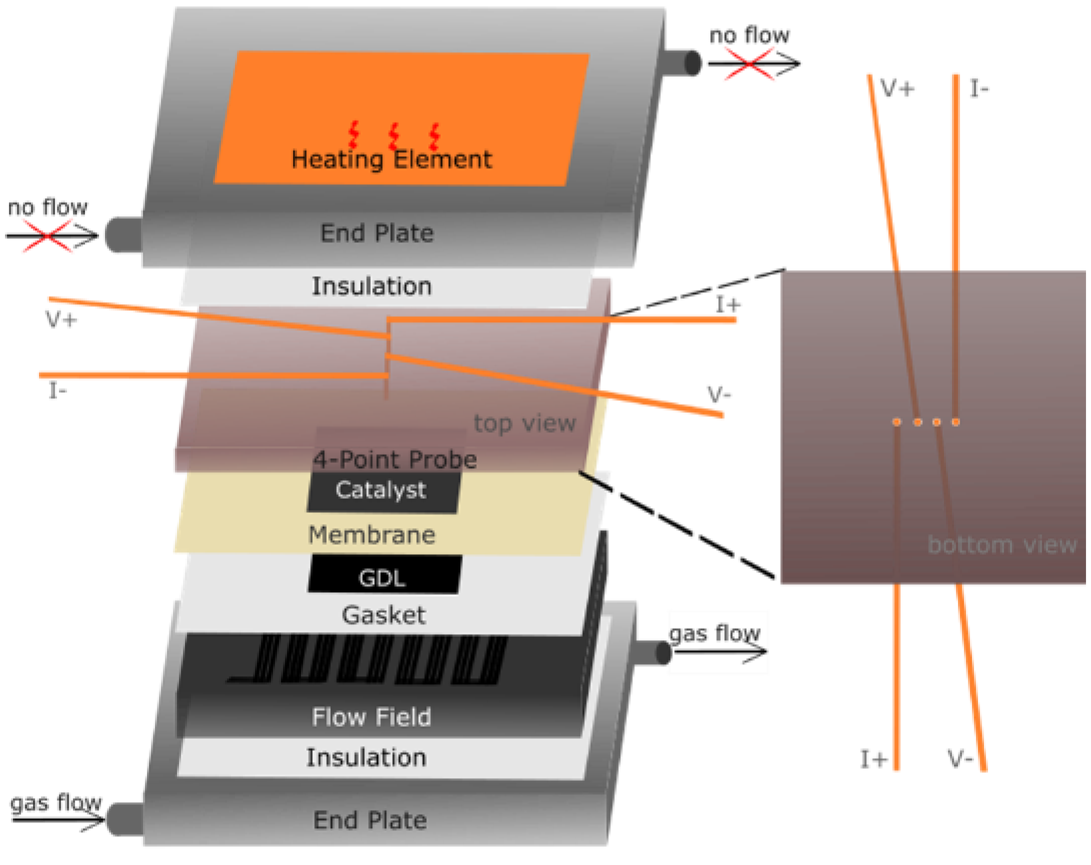
More characterization capabilities have been added to portfolio

- Conductive atomic force microscopy (C-AFM) scanning microwave impedance microscopy (sMIM) to probe permittivity and conductivity of PTL-catalyst interfaces
- *In situ* neutron reflectometry (NR) and cryo-electron microscopy to study native structure of ionomer-catalyst interfaces and their degradation

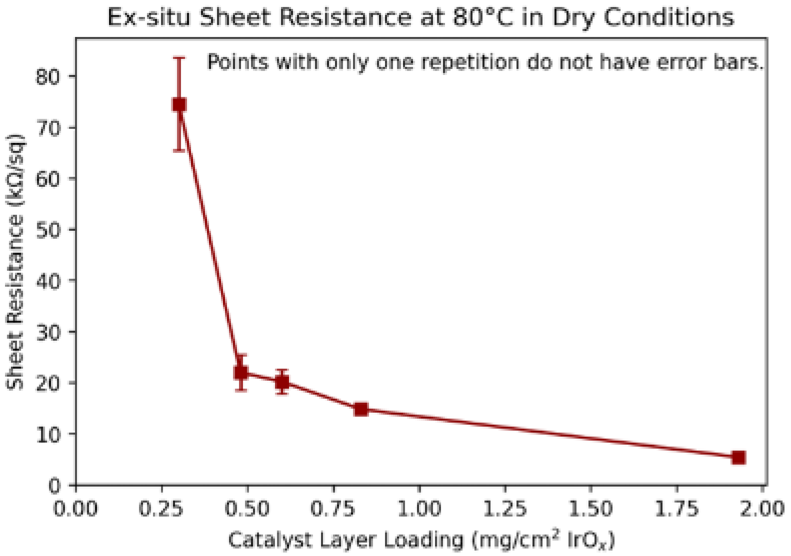
Coming in FY23: ThermoFisher Helios Hydra Cryo-PFIB

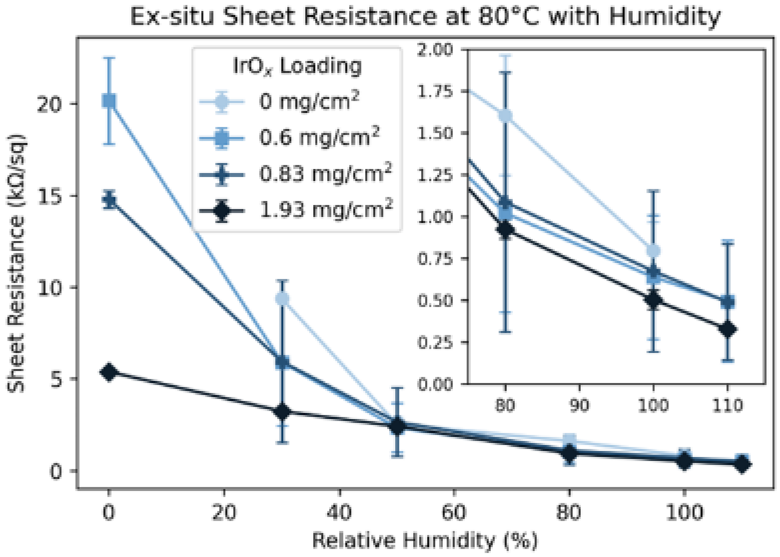
Task 2c: Accomplishments – Measured Conductivity of Humidified Catalyst Layer

- **Temperature- and humidity- dependent measurement capability developed**



Ex-situ Conductivity Cell Assembly **4-Point Probe Insert**

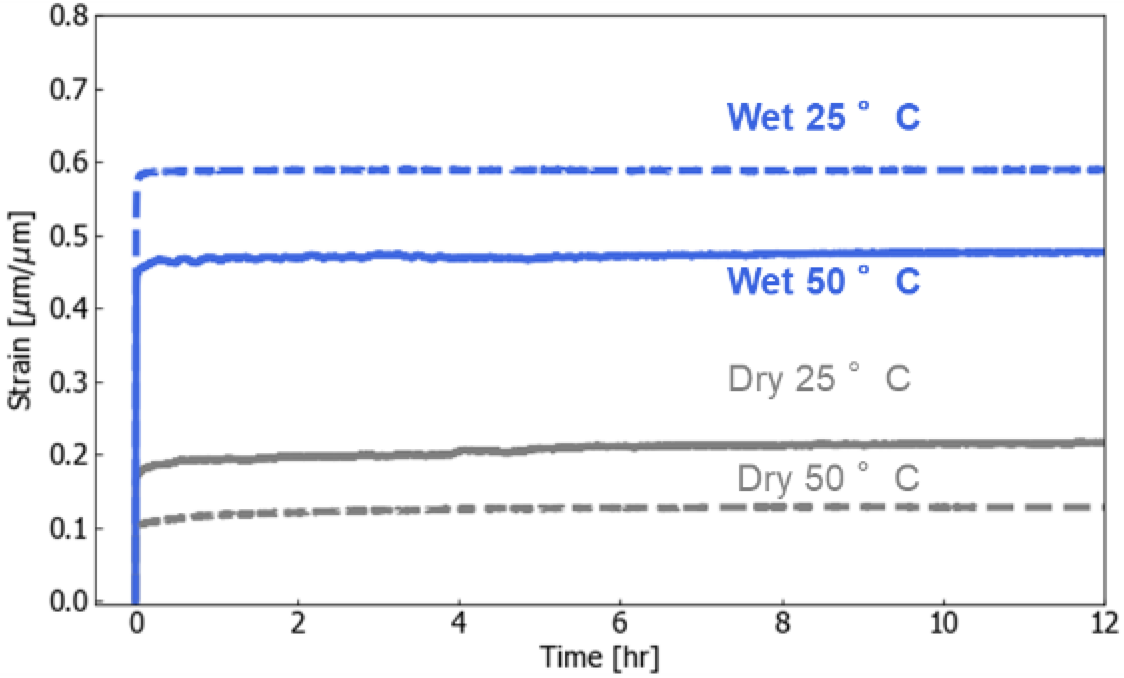




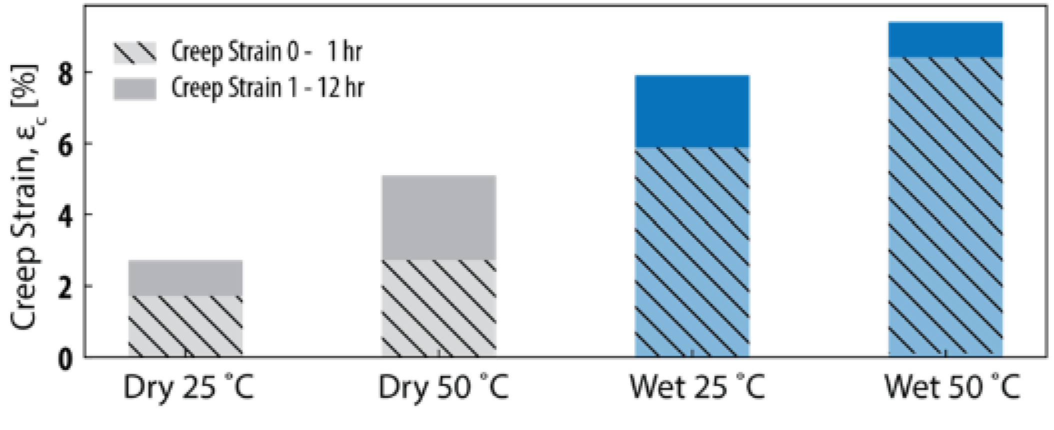
- **Sheet resistance of spray-coated IrO_x increases dramatically with decreasing catalyst loading**
- **Sheet resistance at 80 °C decreases with increasing relative humidity**
- **Catalyst layer dry out may cause high resistance especially for low IrO_x loading (Factor of 10x observed)**

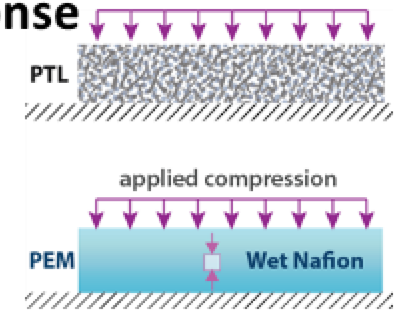
Task 2c: Accomplishments – Measured Nafion Creep with Hydration and Temperature

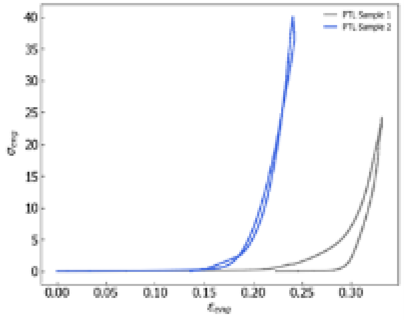
- **Modified compression-creep procedure to include temperature control for wet PEMs**
  - Compression of hydrated Nafion 117 at 35 MPa (5000 psi) in water
  - Increased creep (thickness reduction) observed at higher temperature (50 vs 25 °C)
  - Hydration accelerates creep behavior
  - Creep response is more dominant within the first 1-2 hours followed by a continuous thickness change
- **Membrane will be tested with PTLs**
  - Preliminary mechanical testing of PTLs completed
  - PTLs exhibit rate-dependent, nonlinear mechanical response



Compression Creep Temperature Hydration Effect (35 MPa, 12 hrs)

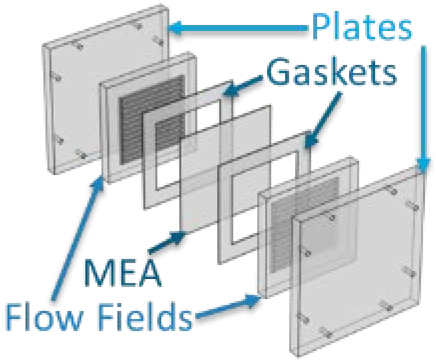




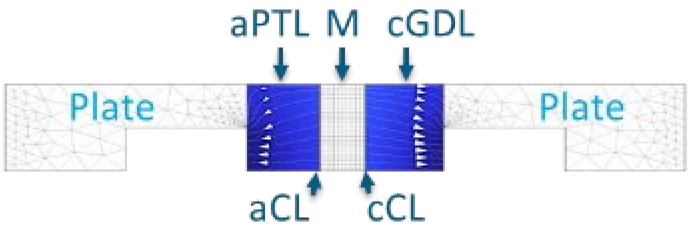


Task 2d: Accomplishments – Developed and Utilized Model for Cation Contamination and Recombination Layer

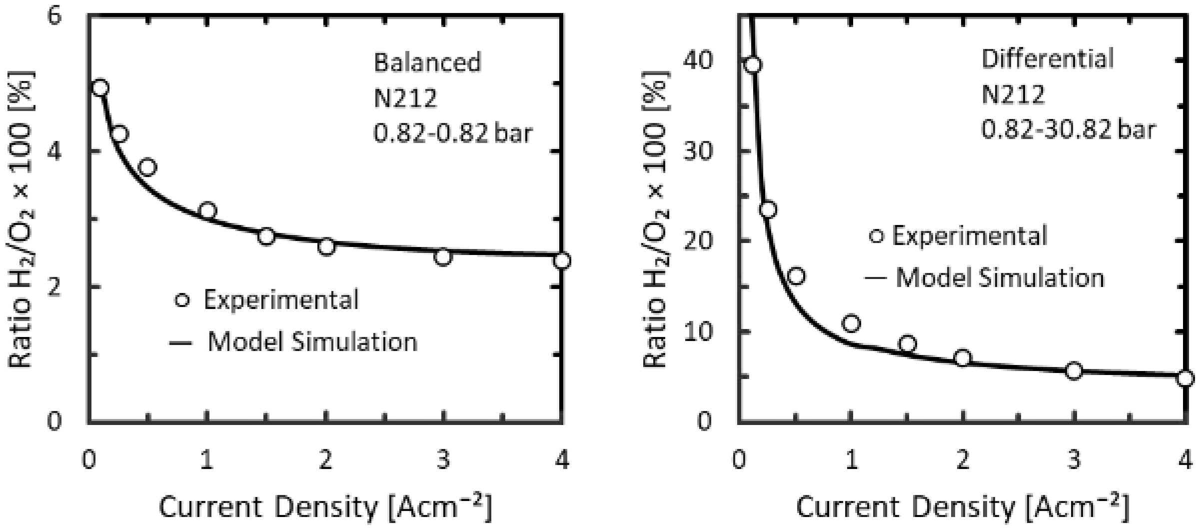
- **Mathematical Modeling**
  - ➢ **Finite-element models in COMSOL Multiphysics®**
  - ➢ **Development of mechanistic models describing PEMWE cell performance**
    - **First-principle modeling of heat, electronic, fluid, porous media and charged-species transport.**
- **Achievements**
  - ➢ **Differential-Pressure Gas-Crossover Model**
    - **Model describes crossover mechanism of two solubilized-gas pathways**
    - **Accounts for specific gas-membrane diffusion and solubility in Nafion™**
      - – **Oxygen - higher solubility and slower diffusion**
      - – **Hydrogen - lower solubility and faster diffusion**



Exploded schematic representation of PEMWE Cell



Cross-section modeling domain



Experimental and simulated gas crossover of a PEMWE cell operated under balanced-pressure and differential-pressure modes. Experimental measurements performed by NREL.

Task 2d: Accomplishments – Developed and Utilized Model for Cation Contamination and Recombination Layer

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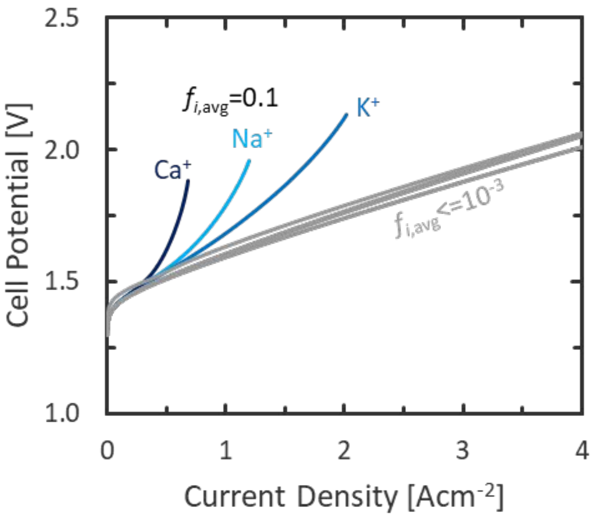
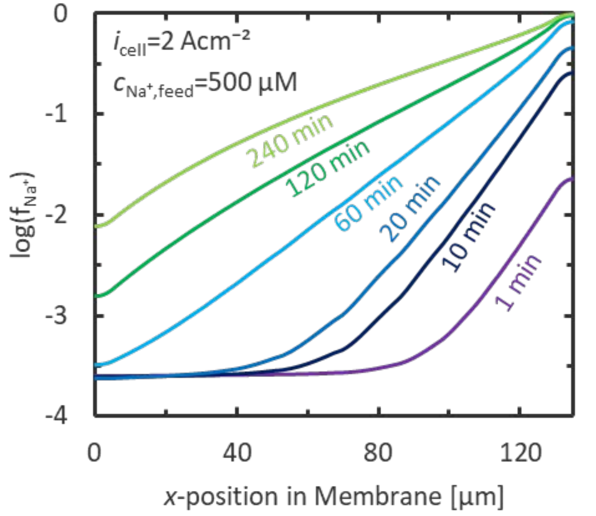
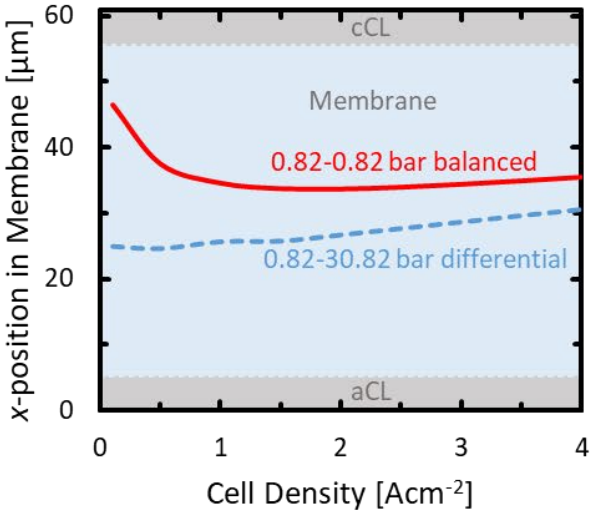
- **Achievements (cont.)**
  - ➢ **Cation-contaminated PEMWE cell**
  - ➢ **Low-concentration (µM) cation exposure leads to cell failure within hours due to cations replacing protons in cathode catalyst layer**
- **Future Work**
  - ➢ **Recombination layer optimization for placement and loading**
    - **Initial theoretical optimization show optimal recombination layer placement is dependent of operation conditions**
  - ➢ **Strategies for recovering PEMWE cell performance after contamination events**
    - **Extent of recoverable performance**
    - **Time to recovery**



Steady-state polarization curves. f_i is the fraction of cation-exchanged sulfonic acid sites. Three cations were simulated Na^+, K^+, and Ca^{2+}.



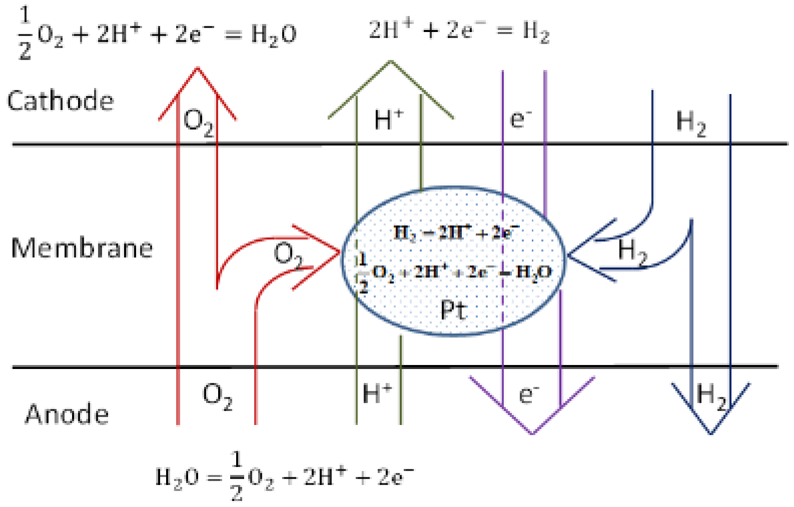
Cation content as a function of position in the membrane with elapsed exposure time to 500 µM Na^+ feed as a parameter at 2 Acm^{-2}.

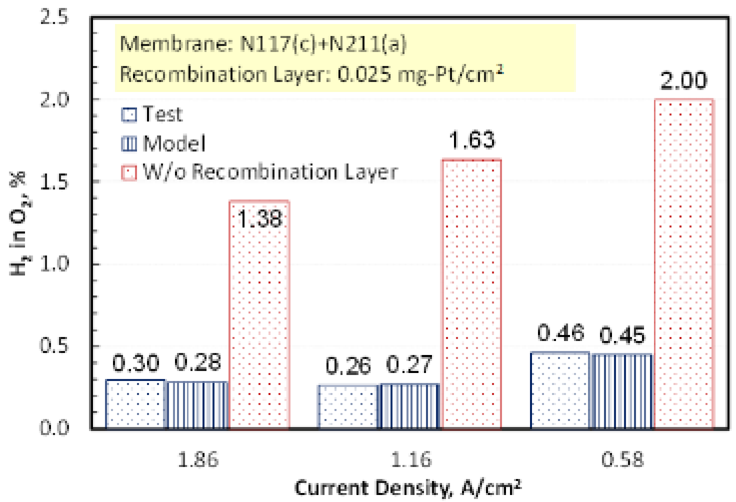


Theoretical optimal placement of recombination layer in N212. Optimum was defined as the location in the membrane where the ratio of dissolved gas concentrations of H_2 and O_2 were stoichiometrically balanced at 2:1.

Optimal position is a function cathode channel pressure and operating current density.

Task 2d: Accomplishments – Developed Model for Hydrogen and Oxygen Crossover and Effects of Mitigated Membranes

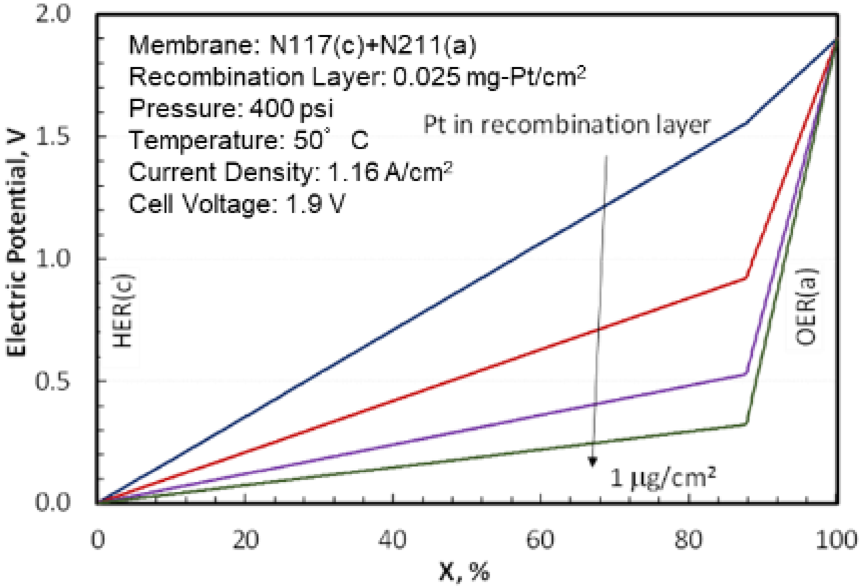


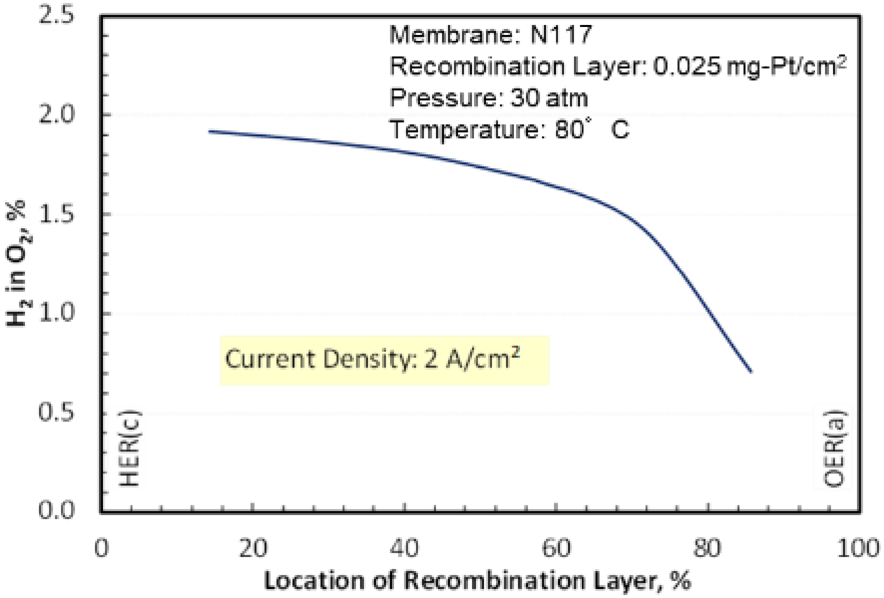


Model Validation

- **Pt interlayer causes 4 to 5-fold reduction in H_2 crossover to anode.**
  Data from R. J. Ouimet, et al., 2020 ECS Meeting Abstract, Polymer Electrolyte Fuel Cells & Electrolyzers, MA2020-02 2469

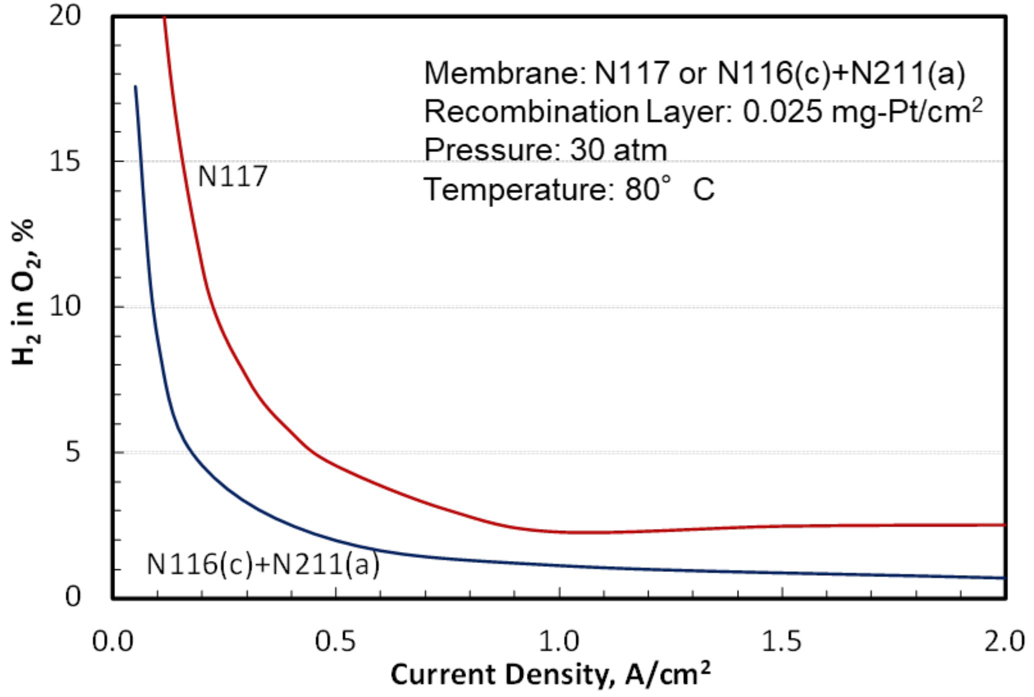
- **Pt interlayer lowers the electric potential and promotes electrochemical reaction between crossover H_2 from cathode and O_2 from anode.**
- **Locating the interlayer closer to the anode mitigates H_2 crossover to anode with only a small increase in H_2 crossover from cathode. However, interlayer location may affect Pt stability.**

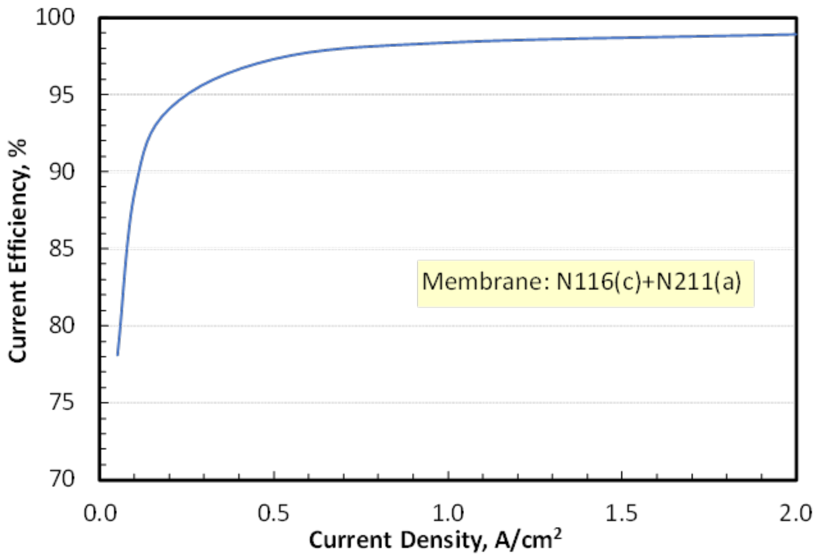


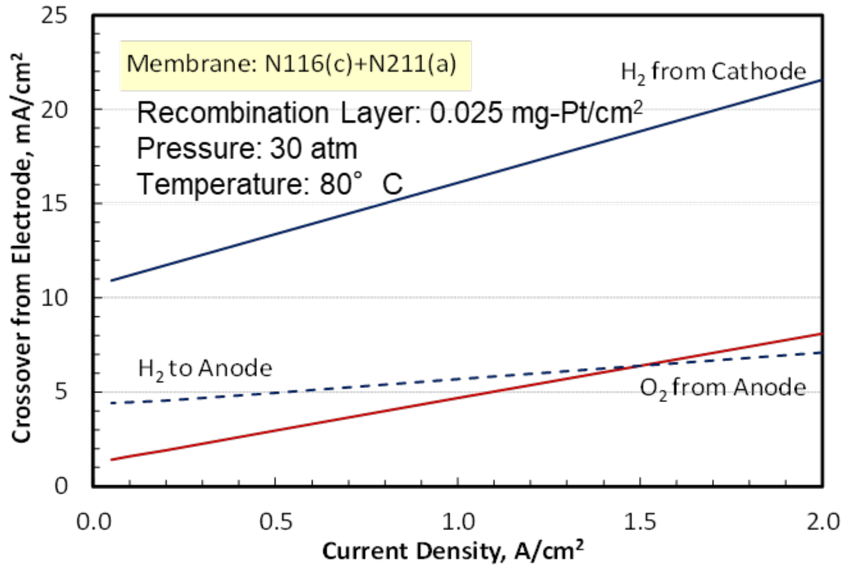


Task 2d: Accomplishments – Developed Model for Hydrogen and Oxygen Crossover and Effects of Mitigated Membranes

- Even with the Pt interlayer, H_2 concentration in O_2 exceeds the lower flammability limit below 0.25 A/cm^2.
- At lower current density, H_2 crossover from cathode decreases more than H_2 crossover to anode.
- Current efficiency defined on the basis of net H_2 collected at the cathode (produced-crossover) and cell current density: ~99% at 2 A/cm^2, ~94% at 0.25 A/cm^2.







Collaboration and Coordination

H2NEW Consortium Partners:

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NIST Affiliate Team: Daniel Hussey, David Jacobson, Jacob LaManna

Discretionary Partners:

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Carnegie Mellon University Team: Shawn Litster (PI)

University of California, Irvine, NFCRC Team: Iryna Zenyuk (PI), Devashish Kulkarni, Cliff Wang, Jack Todd Lang, John Stansberry

Other Partnerships and Collaborations:

MIT, B. Khaykovich; **FZ Jülich**, Germany; **Fraunhofer ISE**, Freiburg, Germany; **Paul Scherrer Institute**, Aargau, Switzerland, **IEA Annex 30 Working Group**

Proposed Future Work

Task 2a: Performance benchmarking, baselining, validation

- **Complete development of 50 cm^2 high pressure research hardware and publish as open source**
- **Benchmark FuGeMEA performance with various state-of-the-art single and multi-layer PTL materials and close structure property relationships**

Task 2b: Cell performance testing in support of electrode development

- **Continue to support Tasks 1 and 3 through capability development, performance & operando experiments**
- **Close molar flow balance of H_2 with crossover diagnostics to enable in-depth recombination catalyst studies**
- **Include EIS in studies of effects of pressure cycling to enable full voltage breakdown analysis**

Task 2c: Ex-situ studies focused on performance factors

- **Continue to support all Tasks with characterization capabilities through**
- **Determine impact of ionomer content on OER kinetics using various diagnostics**
- **Measure Nafion™ creep with PTLs present**
- **Probe permittivity and conductivity of PTL-catalyst interfaces using advanced spectroscopy capabilities**
- **Study native structure of ionomer-catalyst interfaces and their degradation using neutron reflectometry**

Task 2d: Cell level model development

- Utilize cell model to determine optimum placement and loading for of recombination layer

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

Summary—Task 2

- The Task 2 effort focuses on cell performance and characterization with the goal of achieving higher cell efficiency, as well as supporting Task1 and Task 3 with its capabilities
- Benchmarking efforts improved the capability to compare results within the domestic as well as the international community
- Expansion of test capabilities with regards to throughput and portfolio
- Effects of operating conditions on conditioning and H_2 crossover determined
- Detailed studies on the role of PTL properties and PTL interfaces conducted
- Electrode resistance and membrane creep investigated in ex-situ studies
- Cell modeling expanded to include H_2 crossover processes