

Novel ionomers and electrode structures for improved PEMFC electrode performance at low PGM loadings



DoE Annual Merit Review

Washington, DC, April 30, 2019

Project FC155: PI: Andrew Haug, 3M



BUDGET & Status

Timeline

- Project start date: 10/1/16
- Project end date: 9/30/19
 - 29 of 36 months complete @ AMR

Budget

- Total Project Budget: \$3,245,349
 - Total Recipient Share: \$649,071
 - Total Federal Share: \$2,596,278
 - Total Project Costs:* \$2,148,352
 - Current Recipient Share: \$428,089
 - Current DOE Share: \$1,712,356
 - * As of 1/31/19
 - ** Sub expenses as of 1/1/19
- Running roughly 3 months underspent

Barriers addressed

- Cost, durability, performance
- Operational robustness

Partners

- SUBCONTRACTORS
 - Michigan Technological University
 - Tufts University
 - FCPAD:
 - LBNL, ORNL, NREL, LANL, ANL
- PROJECT LEAD:
 - 3M

Key Barrier: Cathode Transport limitations

Dispersed Cathodes at SEF's below $100 \text{ cm}^2_{\text{PGM}}/\text{cm}^2_{\text{planar}}$

- Transport losses become significant

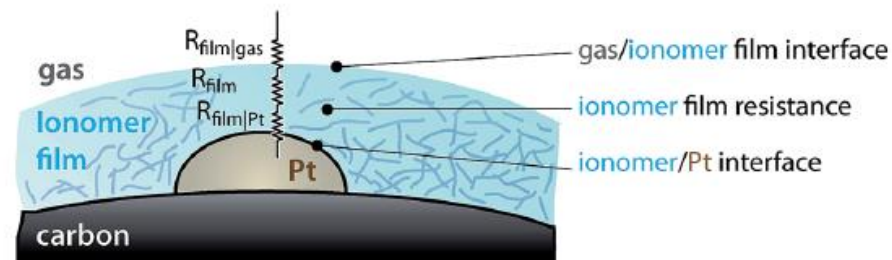
Traditional NSTF cathodes break this trend

- SEF's as low as 10.

Likely that oxygen transport through ionomer near the reaction site is a key limitation

FC155 goal is to

- Understand and improve Ionomer, bulk & local electrode transport
- Integrate NSTF into a dispersed electrode
- Maintain NSTF activity and durability
- Achieve high performance and robustness



A. Weber, J. Mater. Chem. A, 2014, 2, 17207–17211

IMPROVED IONOMER

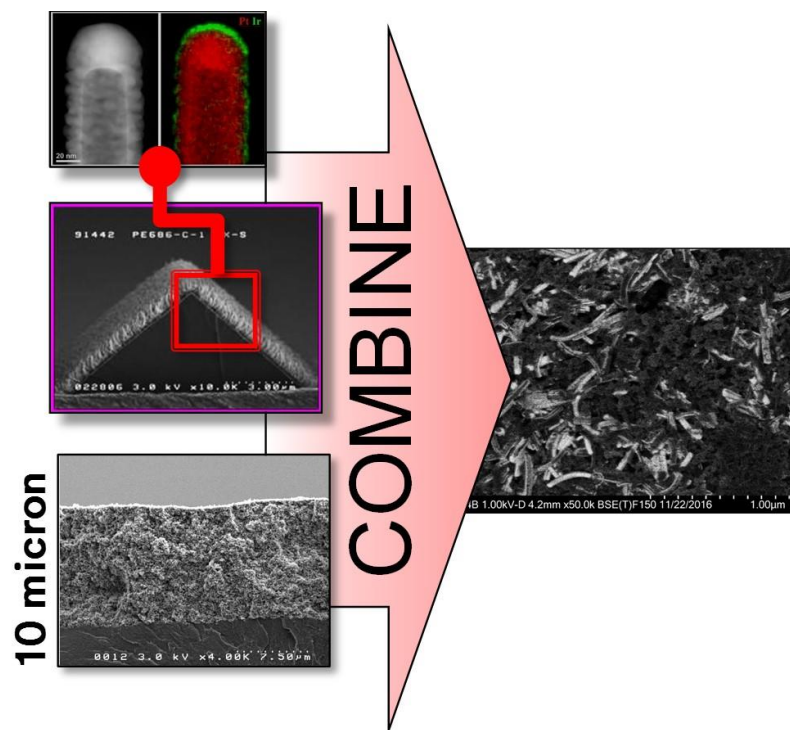
Dispersed NSTF

2 methods to improve transport



Incorporate NSTF into powdered electrode

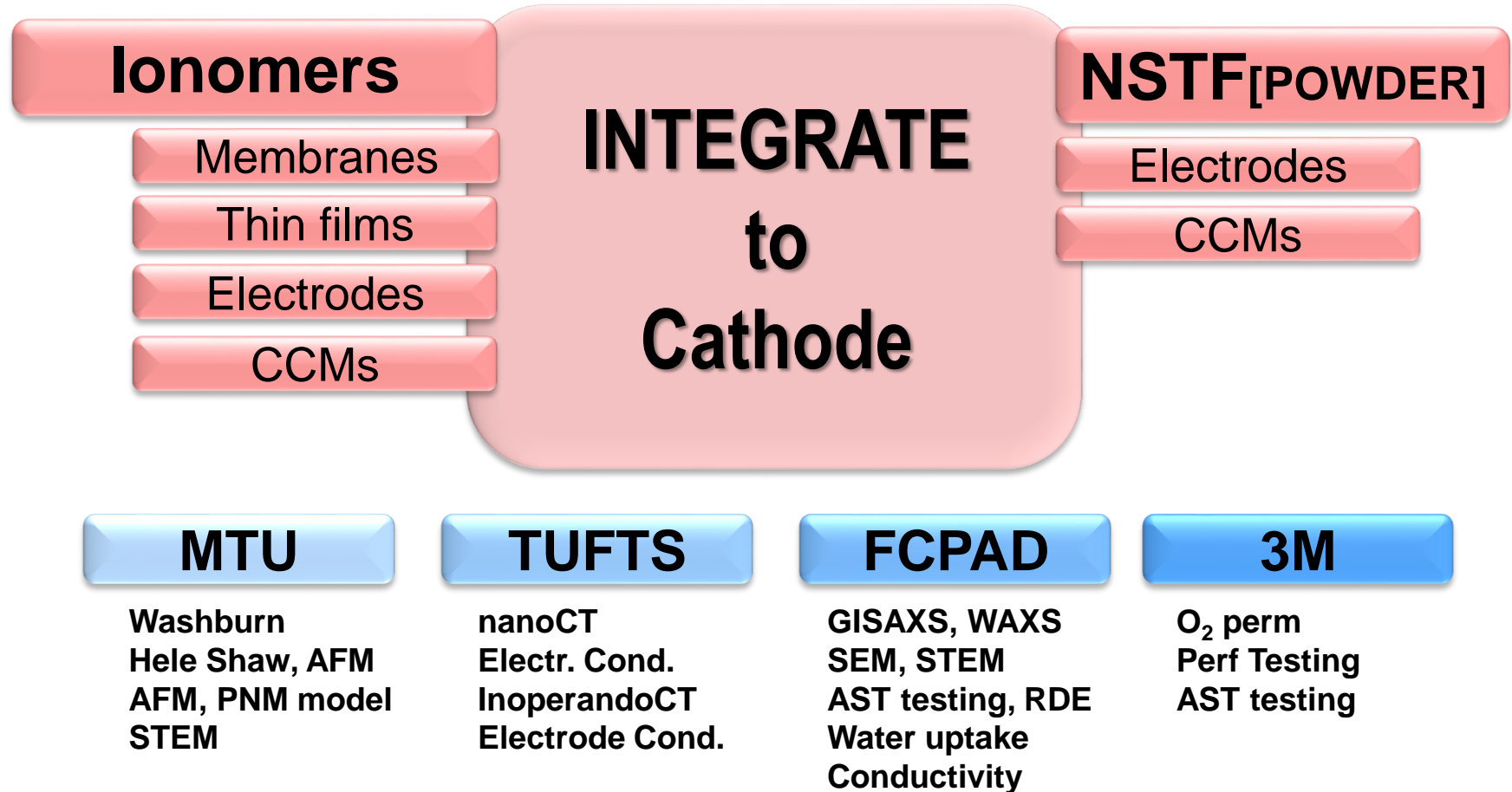
- 10-100X thicker than NSTF
- Contains ionomer
- Improved operational robustness
- Not constrained to planar NSTF loadings



Relevance, Objectives & Status

METRIC		2020 ¹ Target	FC155 Target	3/2017	3/2018	2/2019
PGM total loading, mg/cm ²		0.125	0.125	0.102 ²	0.102 ²	0.095 ²
PGM total loading, g / kW [150 kPa abs]	NSTF Ionomer	0.125	0.125	0.172 ²	0.172 ² 0.125 ^{2,4}	0.172 ² 0.125 ^{2,4}
Mass activity @ 900 mV iR-free, A/mg	NSTF Ionomer	0.44	0.44+	0.28+ 0.15	0.28+ 0.21	0.31 0.36
Support AST , % mass activity loss, 5k cycles	NSTF Ionomer	< 30	< 30	28% (Pt)	<10% (Pt)	<10% (Pt) 27% (PtCo)
Electrocatalyst AST, mV loss @ 0.8 A/cm ²	NSTF Ionomer	< 30	< 30	NA	80 ⁵ 134 ⁵	80 ⁵ 69 ⁵
Electrocatalyst AST, % Mass activity loss	NSTF Ionomer	< 40	< 40	45% (Pt) 83% (Pt)	40% (Pt) 54.5%	41% (Pt/Ir) 54.5%
MEA Robustness (cold/ hot / cold transient)	NSTF Ionomer	0.7/0.7/0.7	>0.7/>0.7/>0.7	0.83/0.79/>1.0	0.93/0.84/0.90 0.97/0.90/0.94	0.93/0.84/0.90 0.97/0.90/0.94
Ionomer Conductivity (S/cm, 80C, 50%RH)		---	0.087	0.050	0.070	0.099
Ionomer Bulk O ₂ perm (mol-cm-s ⁻¹ -cm ⁻² -kPa ⁻¹), 80C, 50RH		---	1.8E-13	2.0E-13	2.3E-13	2.1E-13
¹ All metrics and DOE 2020 targets are taken from DE-FOA-0001412		4 At 0.661V for 80/68/68C. 7.5 psig, 0.686V for 90/84/84C, 21/6 psig				
² 0.025 mgPt/cm ² anode		5 At 70/70/70C, 0 psig				
³ 3M transient protocols used for NSTF testing						

Collaboration & Coordination



Progress and Objectives

Milestone Summary Table

Q/M %

BP1	Go/NoGo: NSTF electrode ECSA $\geq 15 \text{ m}^2/\text{g}$, $40 \text{ cm}^2/\text{cm}^2$, 0.7 robustness. Ionomer bulk O ₂ perm + conductivity > 3M825 baseline		100
TASK	1,2 Synthesize IMIDE#1, Make 20+ grams of NSTF 25 ugPt/cm ² powder.	1/3	100
	1,2,4 Validate DoE AST tests, specialty tests, run baseline with 3 ICs, 3 loadings..	2/6	100
	1, 2 Characterize ionomer, Pt/C, and powder NSTF (SEM, TEM, NanoCT, etc)	3/9	100
	1,2,4 NSTF powder electrode $\geq 0.30 \text{ A/mg Pt}$, NanoCT disp NSTF,	4/12	100
BP2	Go/NoGo: Ionomer exceeds 3M825 O ₂ perm by 33% with similar or improved conductivity. 0.35 A/mg Pt, 0.175 g/kW power output		75
TASK	4 Reaction-kinetics model added to PNM framework. PNM predicts pol curves at T = 40 °C and 80°C.	5/15	100
	2 NSTF Cathode ECSA $\geq 25 \text{ m}^2/\text{g}$.	6/18	100
	4 MTU/Tufts: Baseline structures, electrochem input to PNM, delivering initial predictions.	7/21	100
	2 NSTF activity $\geq 0.35 \text{ A/mg Pt}$ in an electrode. 0.2 g/kW with NSTF containing electrode. *0.31 A/gm _{PGM} achieved with NSTF, 0.36 A/mg _{PGM} with durable dispersed alloy	8/24	95*
BP3	END: See Targets slide		60
TASK	4 MTU/Tufts: PNM - continuum predicts pol curves for T = 40 and T = 80C within 10%	9/27	80
	1-3 Support AST targets achieved. Metal cycle AST <40% activity loss.	10/30	100
	1 Ionomer with 50% greater O ₂ permeability and 50% greater H ⁺ conductivity than 3M825	11/33	100
	1-3 $\geq 0.44 \text{ A/mg PGM}$ in electrode. Metal AST $\leq 30\%$ activity loss. 0.125 g/kW.	12/36	40

TASK1: Ionomer Development

Bulk O₂ Perm, Conductivity

Bulk O₂ permeability

- GM (Zhang ECS 2013) method

Imide #4 (vs 825): **+92%**

Imide #6 (vs 825): **+105%**

Imide #8 (vs 825): **+64%**

Bulk conductivity

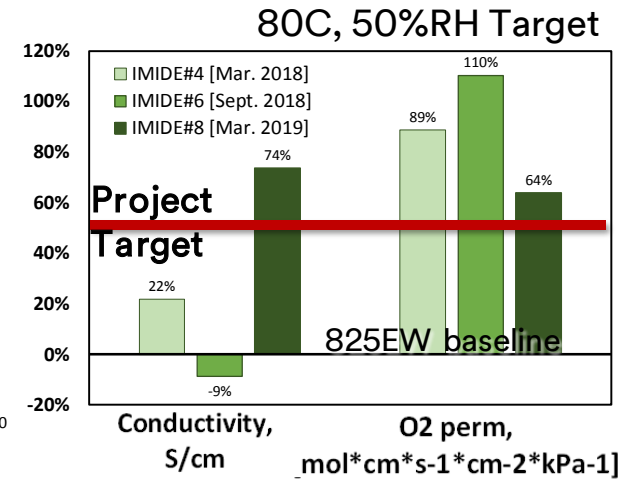
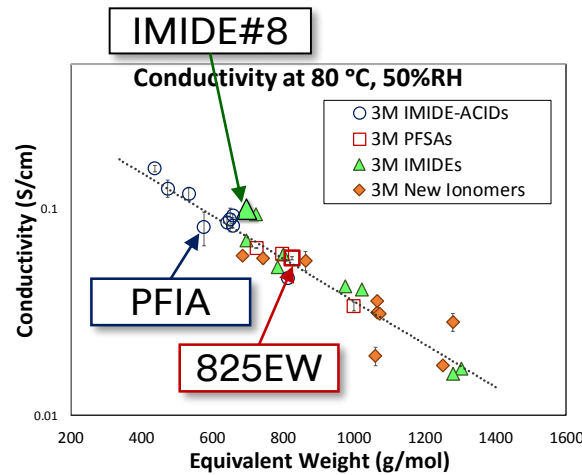
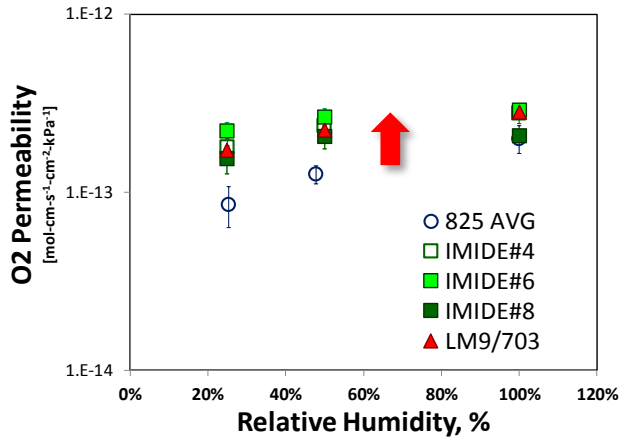
- 4 point probe

IMIDE#4 (vs 825): **+22%**

IMIDE#8 (vs 825): **+74%**

2nd Validation of Imide#6

- Oxtran O₂ transmission
 - (vs 825): **+64%** [23C, 0%RH]



TASK1: Ionomer Structure

Thin film, *LBNL*

Ionomer thin films have been evaluated

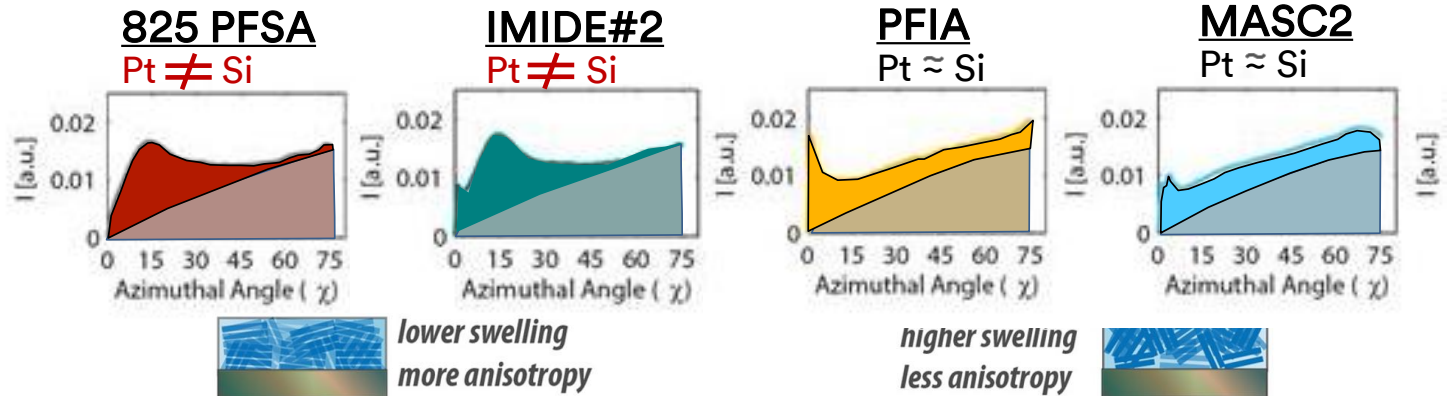
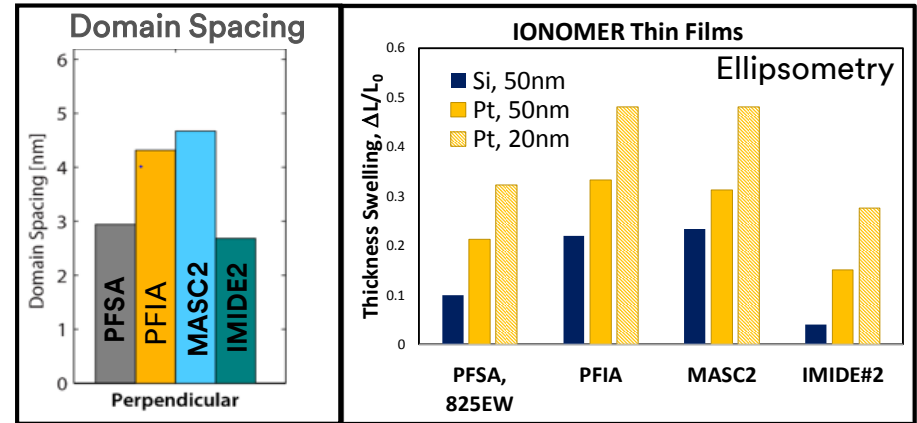
- GISAXS, Ellipsometry
- On Pt and Si substrates

PFIA and MASC thin films have

- Larger ionomer domain spacing
- Stronger nano-phase separation
- Reduced preferential orientation parallel to Pt,Si
- More swell with Pt vs. Si

PFSA & IMIDE#2 more oriented on Pt

- More likely to lay flat on catalyst



TASK1: Ionomer Conductivity, Uptake

Electrode, Tufts/MTU

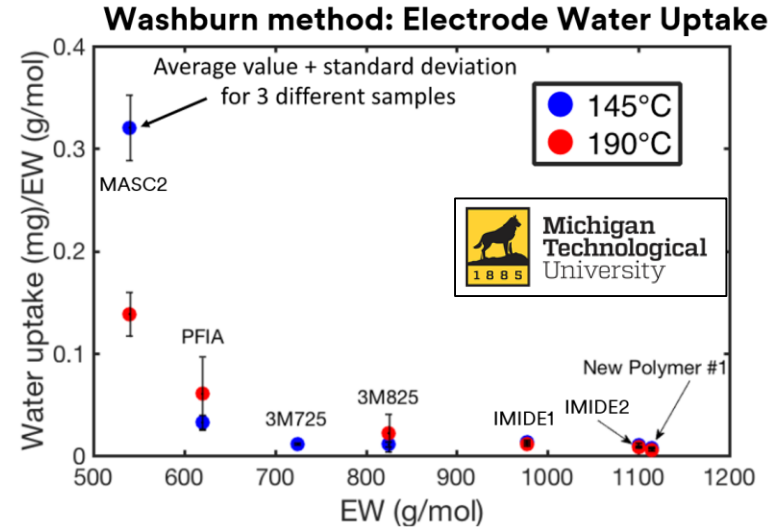
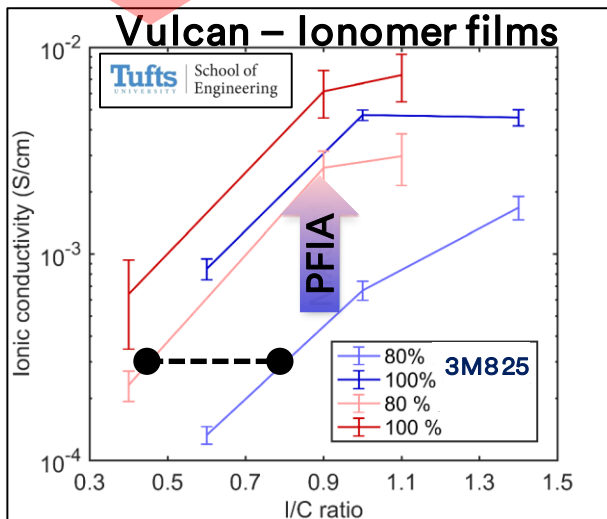
MTU: Water uptake vs. IONOMER

- All using I/C=0.9, 10V50E
- Water uptake increases for PFIA, MASC

Tufts evaluating IONOMER conductivity

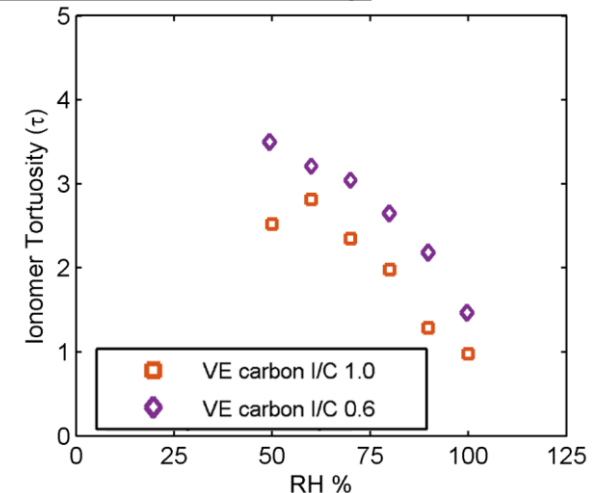
- DC Technique using H₂ pump
- PFIA conductivity @ 80%RH: 8,12X [vs 825, 1000EW]
- $I_{PFIA}/C=0.4$ equivalent to $I_{825}/C=0.8$

KEY



Tufts evaluating IONOMER tortuosity

- Compare DC Technique and AC(EIS) techniques
- Ratio results to estimate H⁺ tortuosity vs. RH



TASK1: Ionomer Local Gas Transport

Electrode, *NREL/LBNL/3M*

Less ionomer reduces resistance

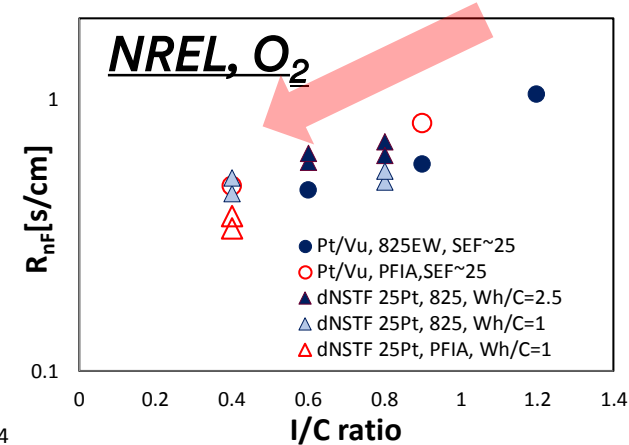
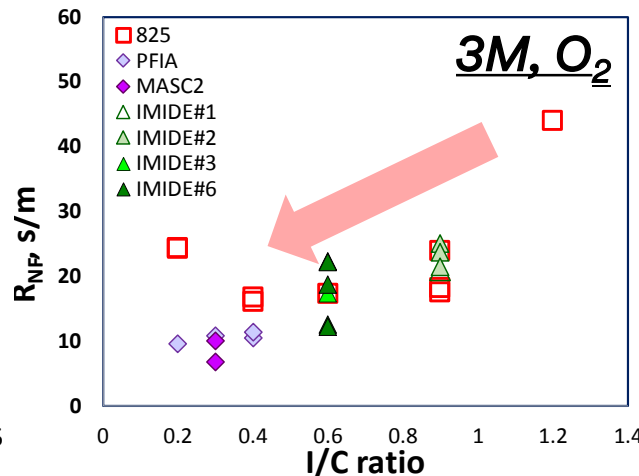
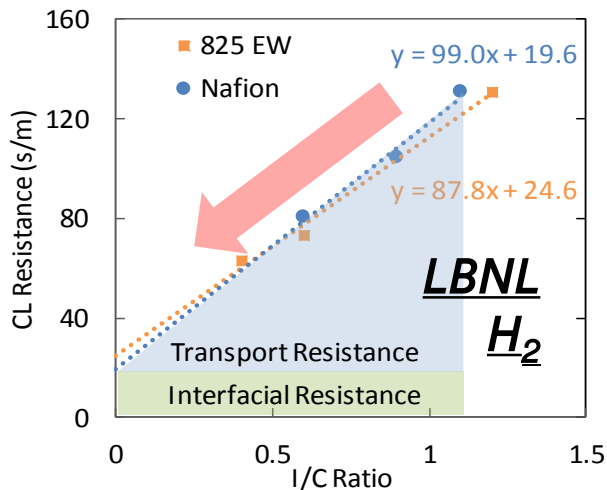
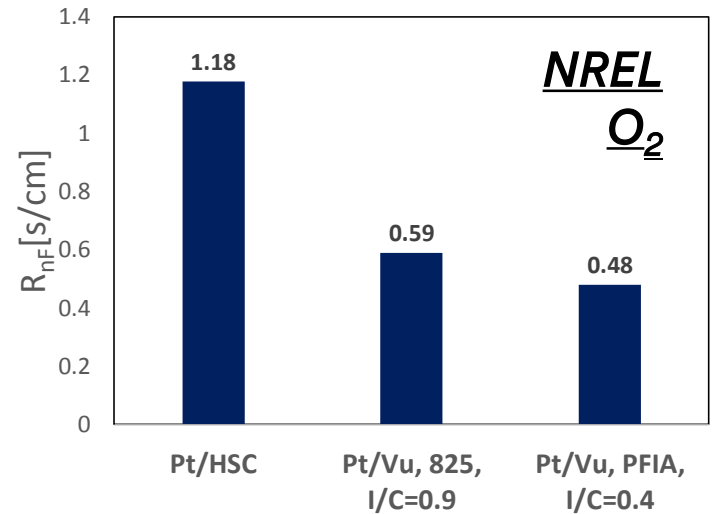
- I/C=0.9 to 0.4 reduced resistance 19-33%
- Seen for H₂ & O₂ transport
- dNSTF and Pt/C systems

Not yet clear differentiation of ionomer type

- Results similar to PFSA baselines at 3M, NREL
- Testing more now at NREL

UNUSUAL Behavior with I/C<0.4

- 825 PFSA shows increase vs. I/C=0.4
- PFIA & MASC2 do not
- Possible agglomeration, catalyst de-activation at <100%RH



TASK1+3: Ionomer Integration

Lower I/C = Higher catalyst activity

- Low-Mid SA carbons
- Gr2 carbon activity increased 61%
- Consistent with Shinozaki et al (on RDE)

Near 0.3 A/mgPt with Pt/Vu

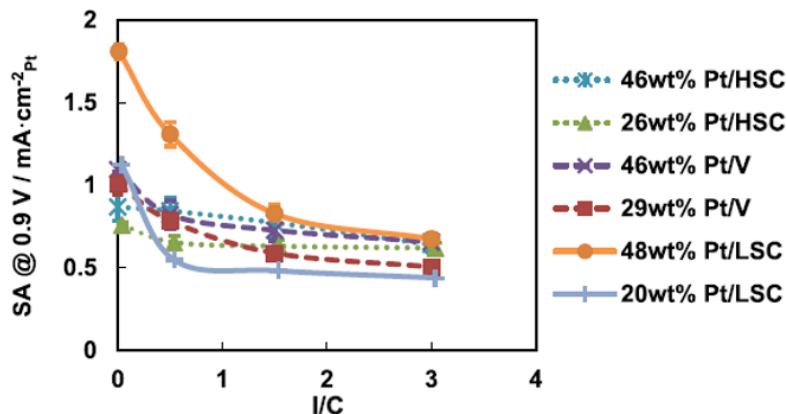
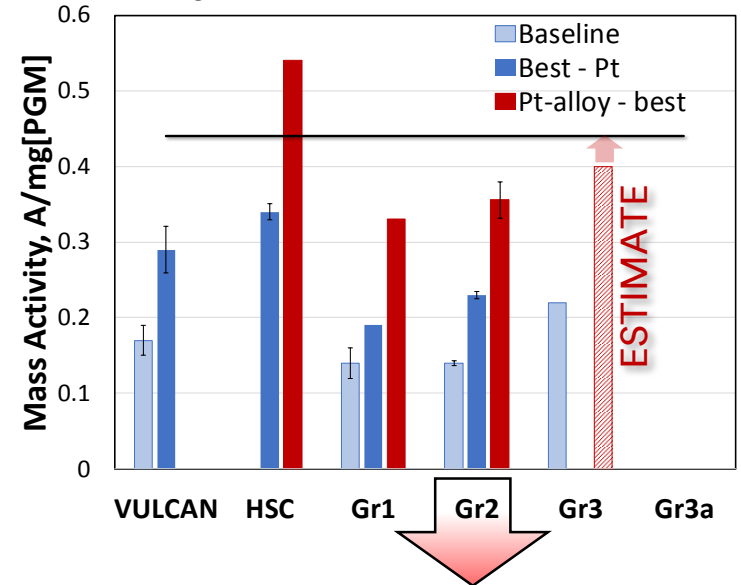
Pt-alloy/Gr2 = 0.36 A/mgPt

- BP2 GNG

Gr3, Gr3a promise higher activity

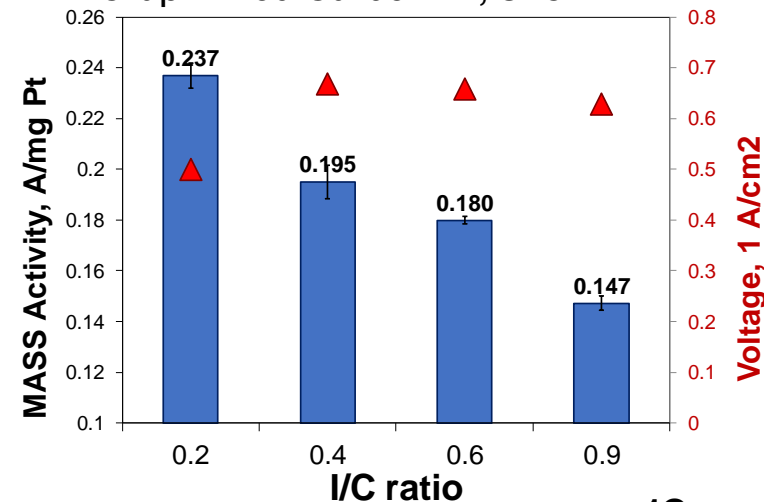
- 3000+ support cycles

I/C vs. Activity



K. Shinozaki et al. / Journal of Power Sources 325 (2016) 745

Graphitized Carbon#2, 825EW



TASK1+2+3: Ionomer Integration

Processing, ANL

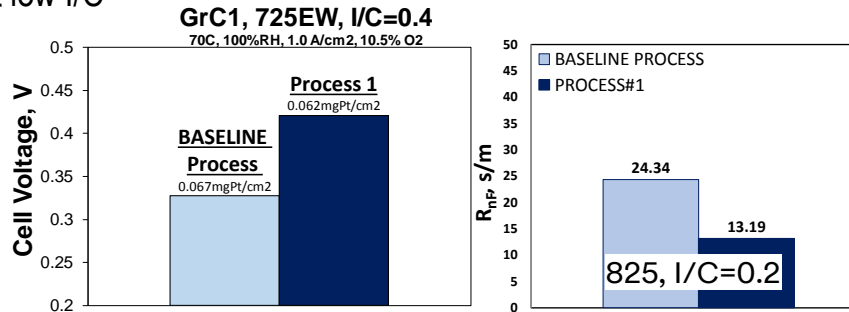
Aggregates & Agglomerates influenced by

- C-type, %M, Ionomer type, I/C ratio
- ANL using USAXS to quantify
- Low I/C & dNSTF electrodes more agglomerated

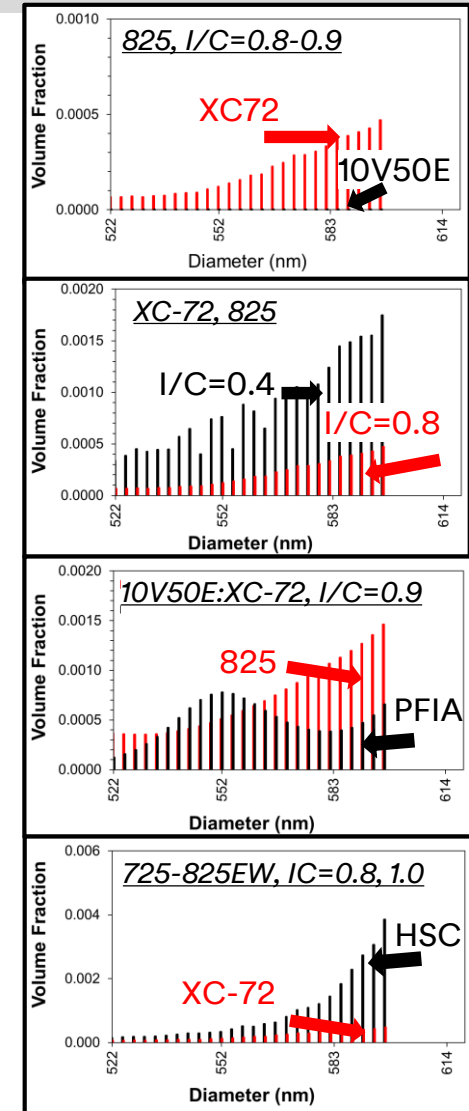
PFIA reduces agglomeration

Processing can reduce agglomeration

- & Increase performance at low I/C



	Type 1	vs	Type 2	>400nm Aggl.
C-type	HSC		XC72	7X
%M/C	XC72		10V50E	50X
I/C	0.4		0.8	3X
Ionomer	825		PFIA	2X
Electrode	dNSTF, XC72, I/C=0.4		10V50E Baseline	~75X (500X for HSC)



TASK1,3: BEST in CLASS

CCM Package SPECS:

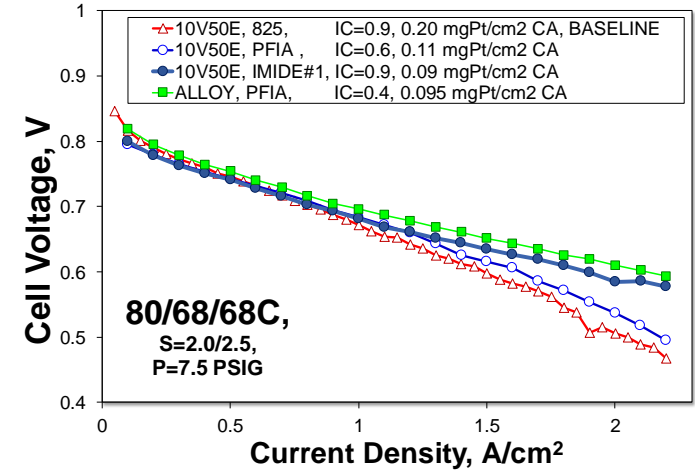
- 0.025 mg Pt/cm² anode
- Better membrane, GDL

Alloy M/Carbon, PFIA, I/C=0.4

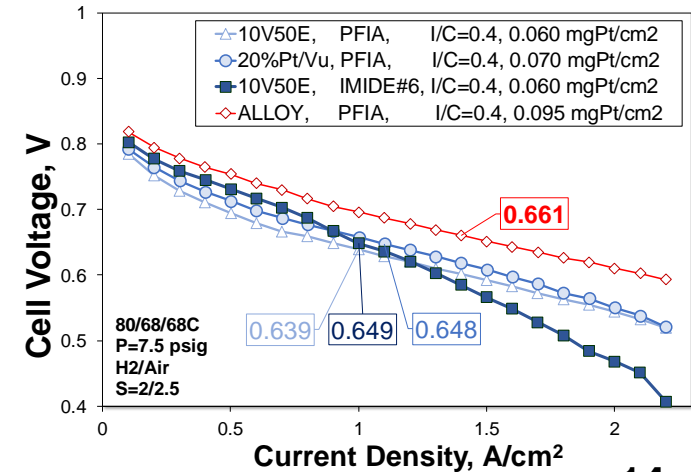
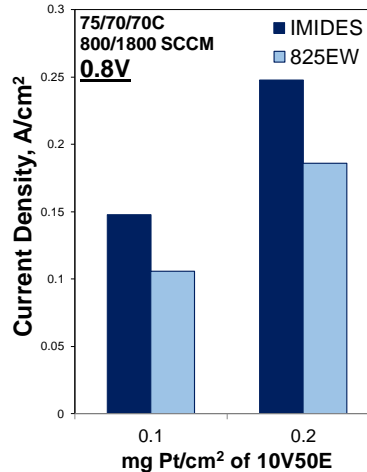
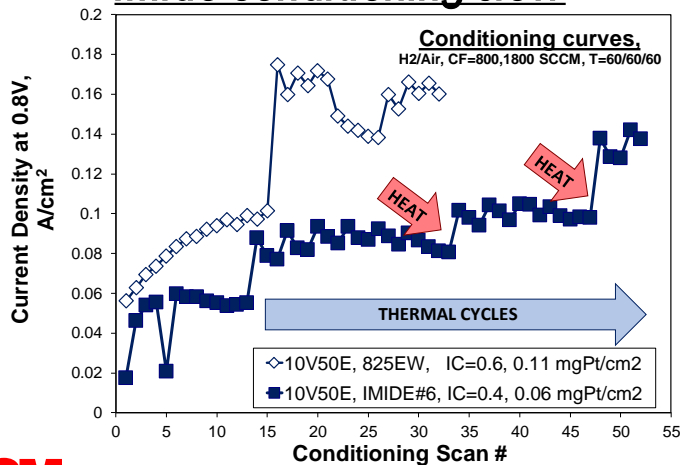
- 0.125 g/kW @ 0.661V [80C, 7.5 psig]
- 0.125 g/kW @ 0.686V [90C, 21.6psig]

Good Pt/C performance at <0.07 mg Pt/cm²

- Imides shows H₂/Air gains



Imide conditioning slow



TASK1,3 : BEST in CLASS

with Durability

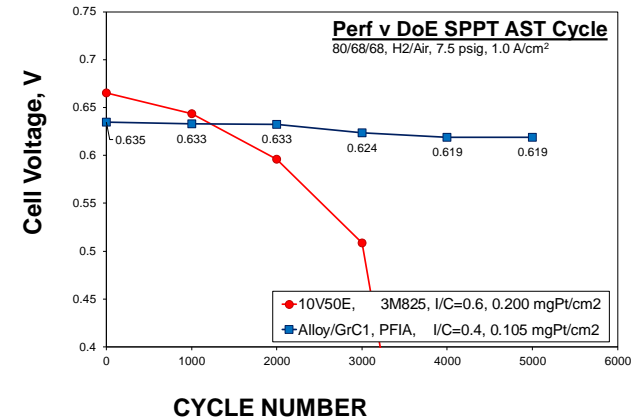
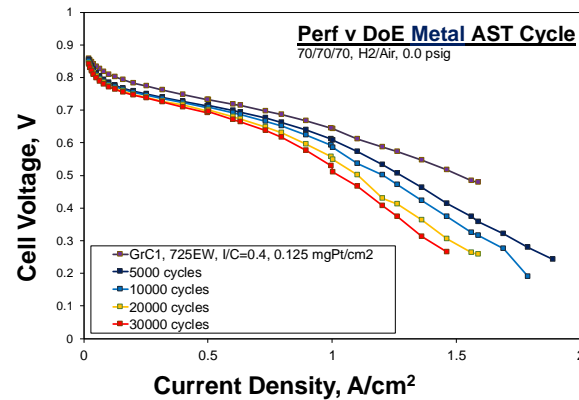
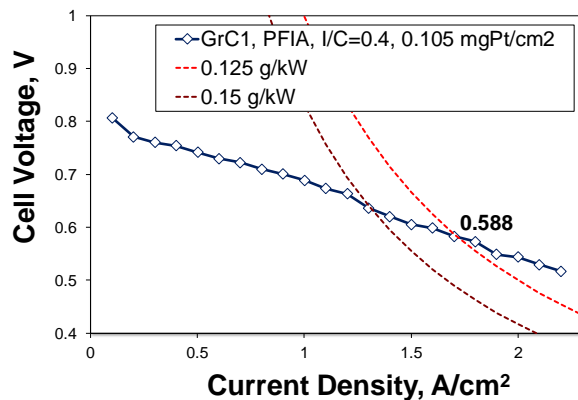
Initial attempts have combined

- Activity & conductivity gains with
- Support stability
- Metal stability

Areas of focus/improvement

- Low initial surface area – increase this
- Metal stability
- Optimizing balance of parts

Property	Performance
Initial activity, A/mgPt	0.31
Local O ₂ resistance, S/cm	-25.9% [vs baseline]
Electrode ionomer thin film conductivity	8X vs. 825 12X vs. 1000
Process Improvement	18% power [@0.067 mgPt/cm ²]
Support stability	5000+ cycles
Metal Stability (will improve with package optimization)	-39.6% ECSA -69mV (0.8 A/cm ²) -39mV (0.5 A/cm ²)



TASK2: Powdered NSTF

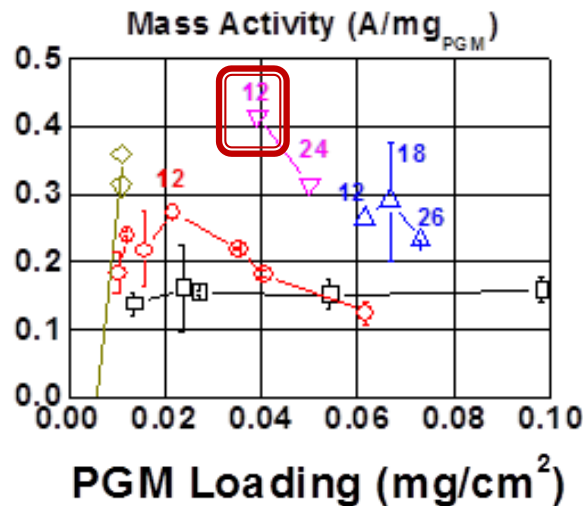
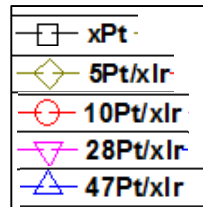
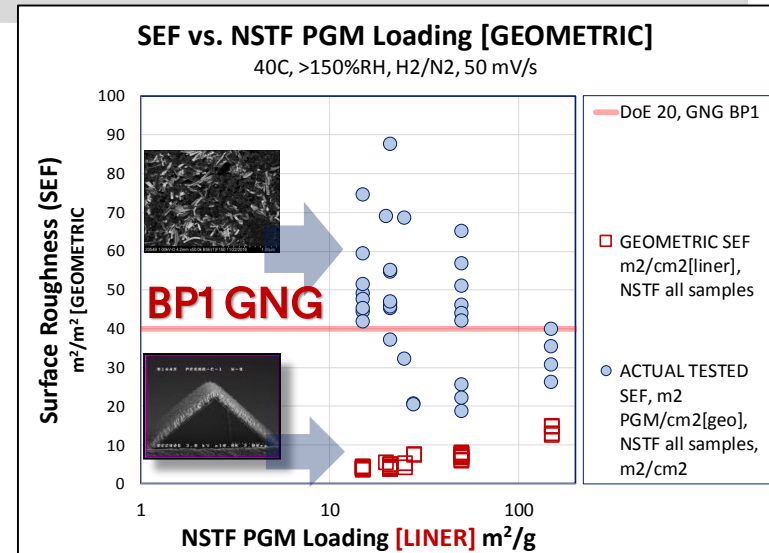
Powdered NSTF

- Eliminates geometric constraint
- Requires new variables (Wh/C, I/C)

Task 2 Targets met: ECSA, SEF

New materials coming

- ECSA= 28-30 m²/g at 40C
- 0.4 A/mgPt with no transition metals



TARGET	Status	Key issue
ECSA = 25 m ² /g	Complete	
Surface Roughness	Complete	
Operating range	Complete	Ensure with downselects
Metal AST – ECSA	Complete	
Support AST	Complete	
Metal AST – 0.8 A/cm ²	80 mV	H+ transport
Activity	0.31	Electrode Structure (Pt/Ir) Transition metal loss (Pt-alloy)

TASK2: dNSTF, Performance Root Cause

ANL, LANL, LBNL, NREL, ORNL, MTU, Tufts

Mid-High current performance loss

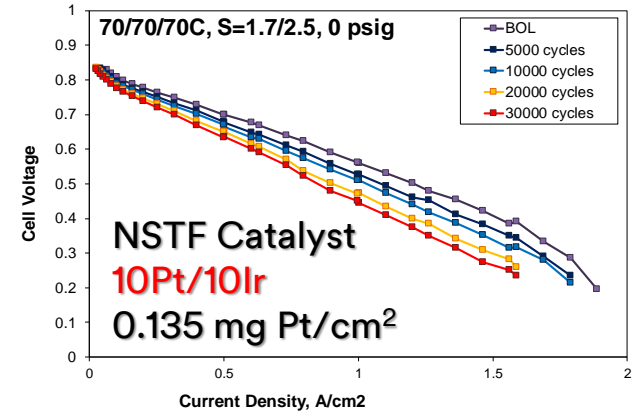
- Lower I/C Improves high currents
- Lower Wh/C Improves high currents
- PFIA Improves @ high currents
- Local O₂ transport EXCELLENT
- Agglomeration is SEVERE
- Proton Transport Poor and RH sensitive

Next slides show the above in detail

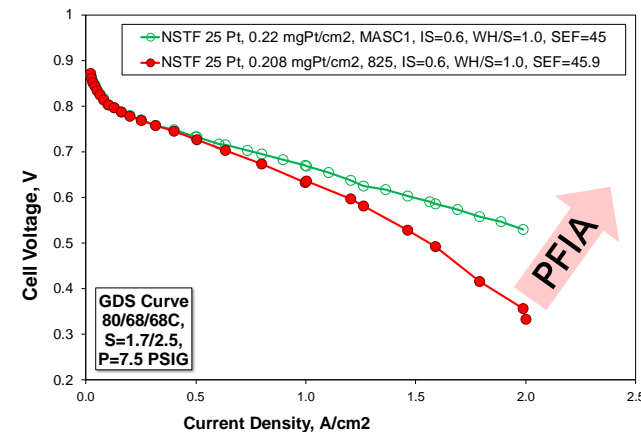
ANL work

	Type 1	vs	Type 2	>400nm Agglomeration
C-type	HSC		XC72	7X
%M/C	XC72		10V50E	50X
I/C	0.8		0.4	3X
Ionomer	825		PFIA	~2X
Electrode	dNSTF, XC72, I/C=0.4		10V50E Baseline	~75X (500X for HSC)

dNSTF electrode HIGHLY agglomerated



So much performance loss
Only 15% ECSA loss!!!



TASK2,3: dNSTF, Performance Root cause

NREL/LANL/3M

Reduced O₂ transport resistance

- More carbon, less ionomer
- I/C=0.8 to 0.4 reduced resistance ~31%
- Wh/C=2.5 to 1.0 reduced resistance ~15%

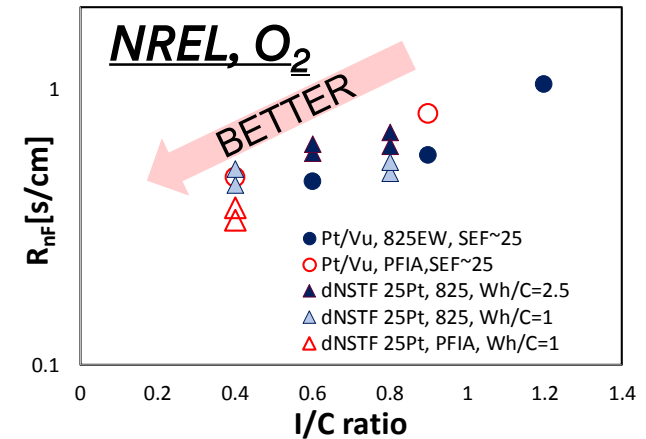
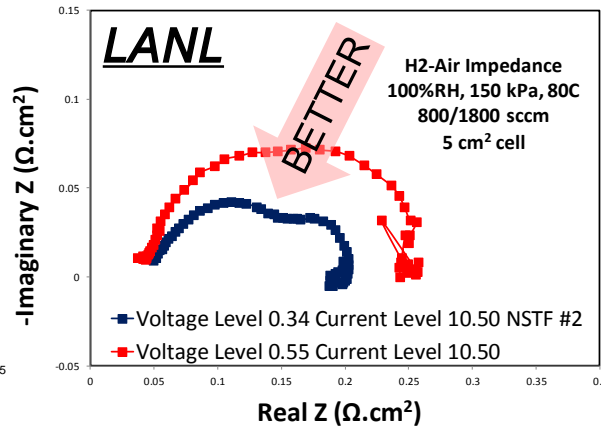
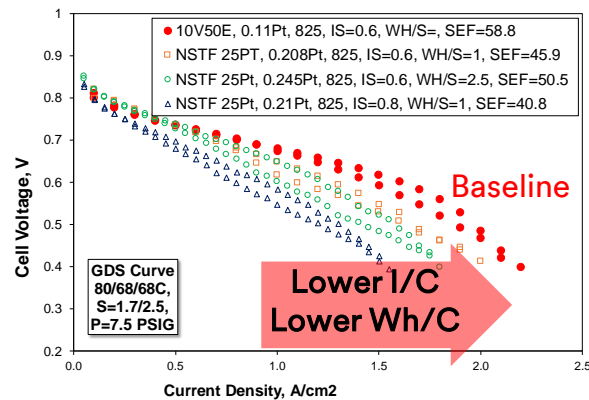
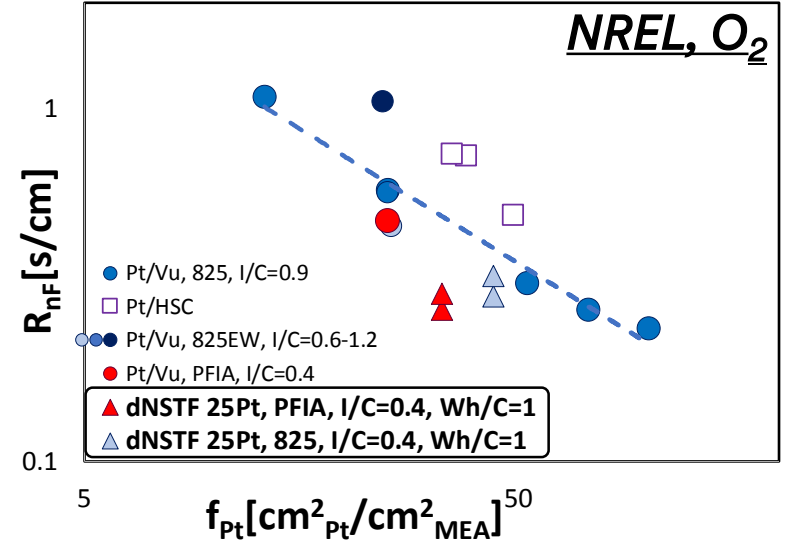
Best local transport achieved (NREL)

- NSTF25Pt, PFIA, I/C=0.4, Wh/C=1.0
- -39.6% vs. Baseline 10V50E
- -61.4% vs. Pt/HSC

Impedance verifies transport gains

Transport Gains = performance gains.

O₂ transport is Great!



TASK2,3: dNSTF, Performance Root cause

NREL / LANL / Tufts

Evaluating key variables

- Gas transport resistance It's Good
- Change with P_{O_2} is small Not kinetic
- H+ resistance (transmission line) Low & RH sens.
- Performance vs. RH (LANL) RH sensitive

Tufts performed CO stripping

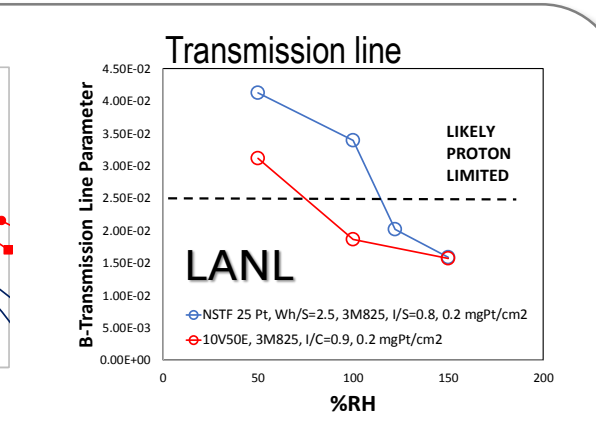
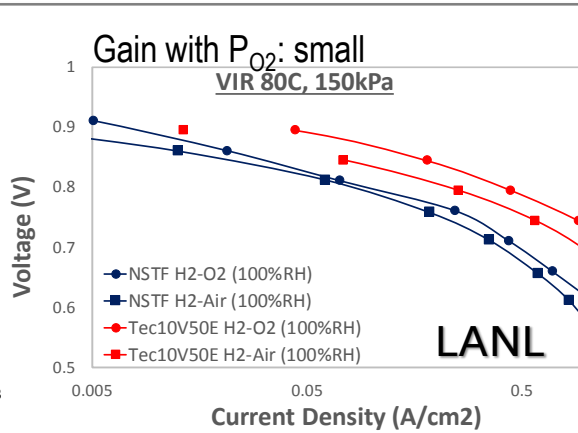
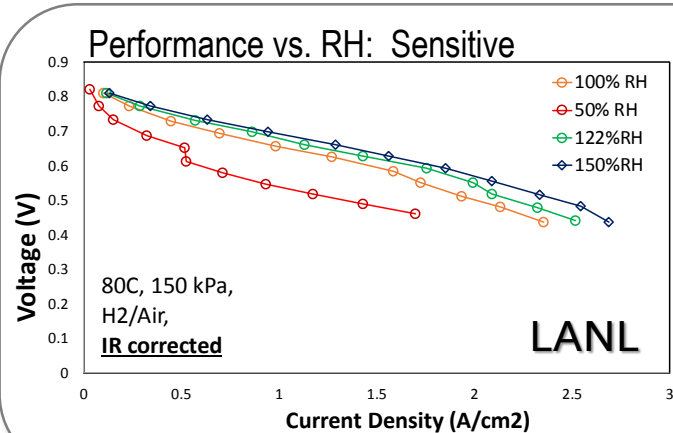
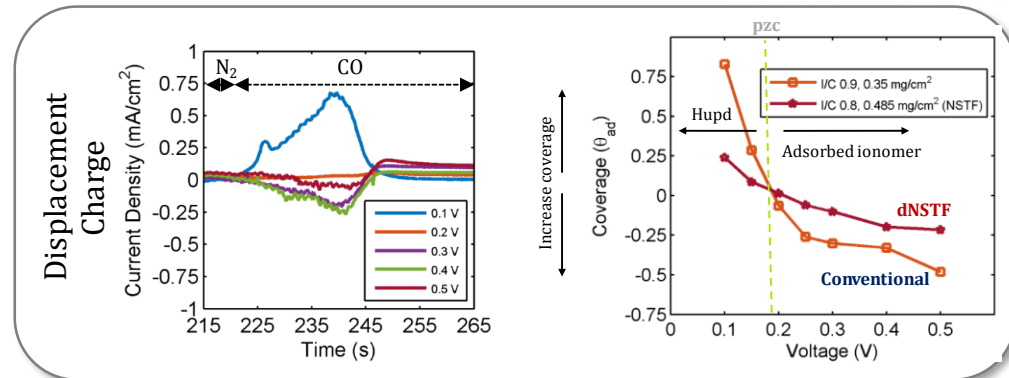
- Disp. NSTF vs. M/C (10V50E)
- Ionomer coverage of whiskers likely low**
 - Due to agglomeration?
 - Contributing to poor conductivity?



Impedance in H₂/Air

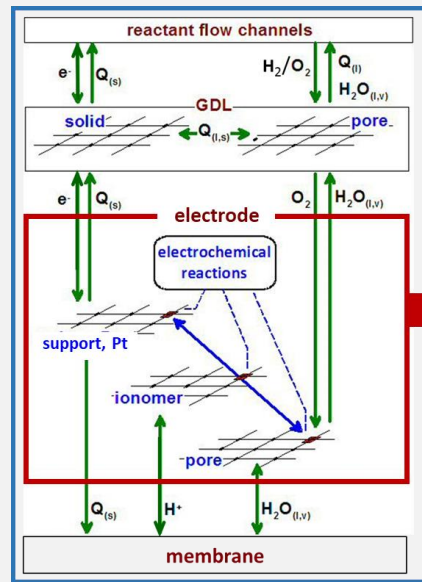
- Low current densities, NSTF much worse
- High current densities, NSTF much better

Dispersed NSTF is likely proton transport limited



TASK4: Tufts-MTU Electrode Transport Model

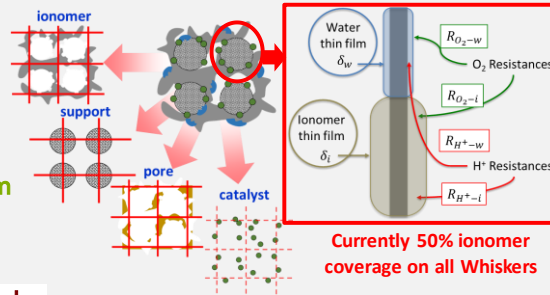
Cathode/Anode transport fluxes Model between the membrane and the gas channel



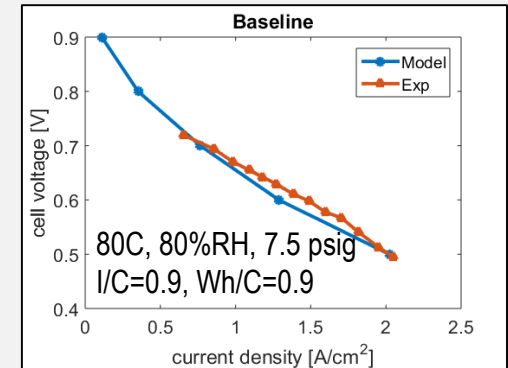
Continuum Model

Pore Network Model

Electrode network approach

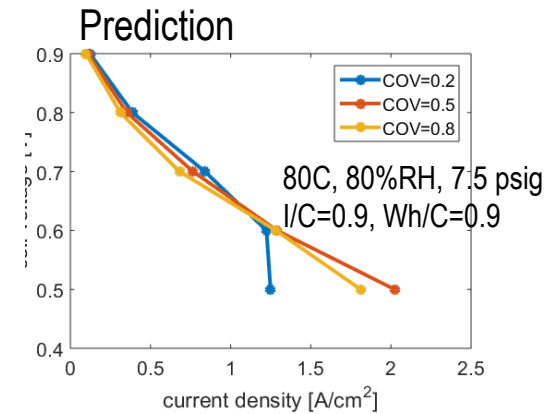
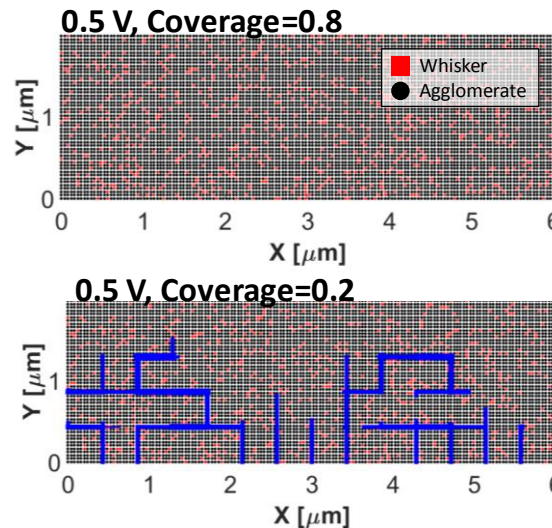


Calibration & Validation of the coupled model



Investigating impacts of whisker coverage by ionomer

- Impacts what the electrode pores see
- Impacts local conductivity
- **Low coverage = more pore flooding**



TASK 2: Best in Class performance

NSTF 25 ug/cm²_[PLANAR] + PFIA

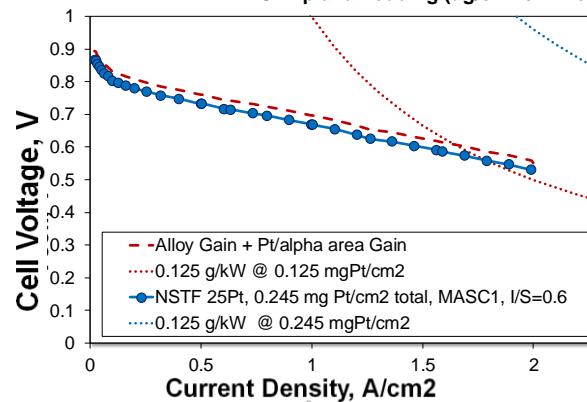
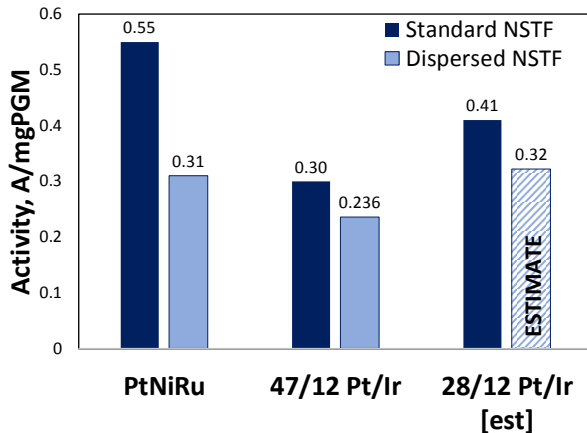
- Best performance
- Mostly overcomes resistance loss issue
- Best local transport of any electrode tested (NREL)

NSTF 28 ug/cm²_[PLANAR] PtNiRu + IMIDE#1

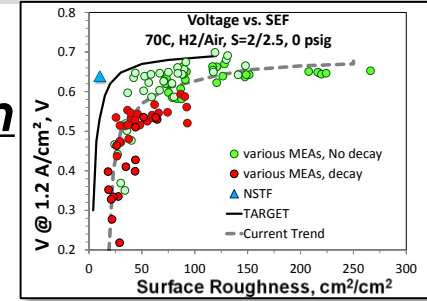
- 0.31 A/mg_{PGM}, highest activity to date

NSTF 47/12 ug/cm²_[PLANAR] Pt/Ir + IMIDE#1

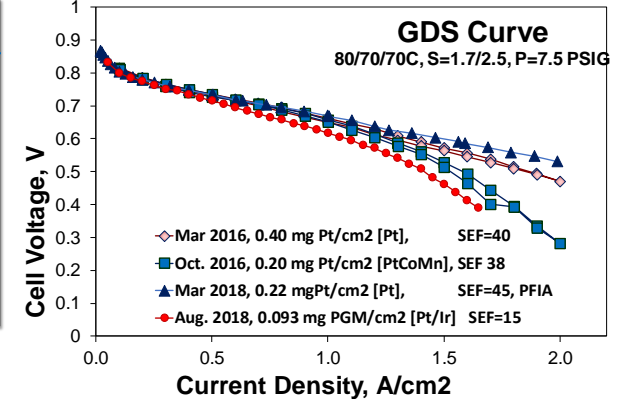
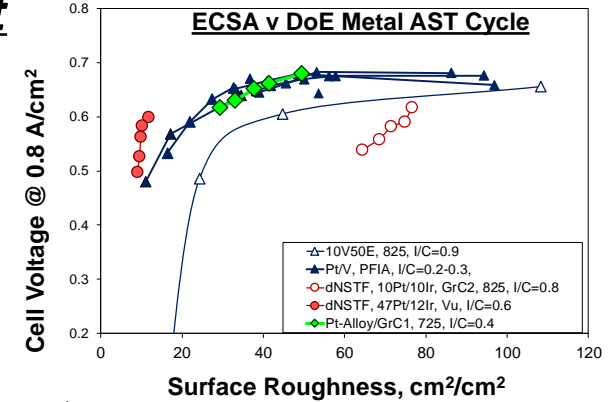
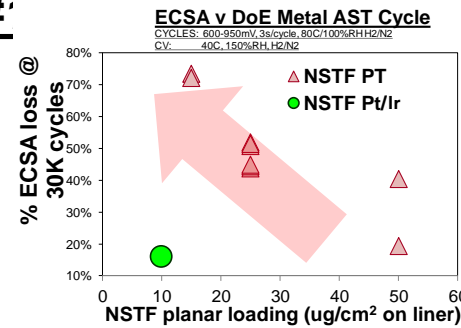
- 0.172 g/kW achieved without best in class package
- 78% activity retained in dispersed format
- Best local transport of any electrode tested [3M]
- 18% ECSA loss
- Can readily pass Support AST



Prediction



Achievement



SELECTED AMR Comments

AMR 2018

Overall, project was good on approach and accomplishments

Presentation was weak on collaboration

- Many results came after 5/2018
- Collaborations shed light on many issues

Multiple comments implying 3M is “layering” NSTF to make a cathode

- This work focuses mainly on dispersing, not layering, NSTF

The future work could be more detailed, and durability should be more thoroughly addressed

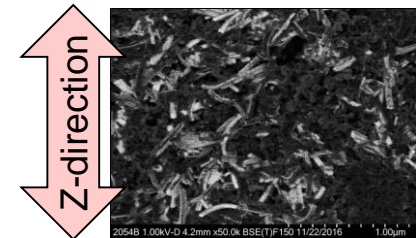
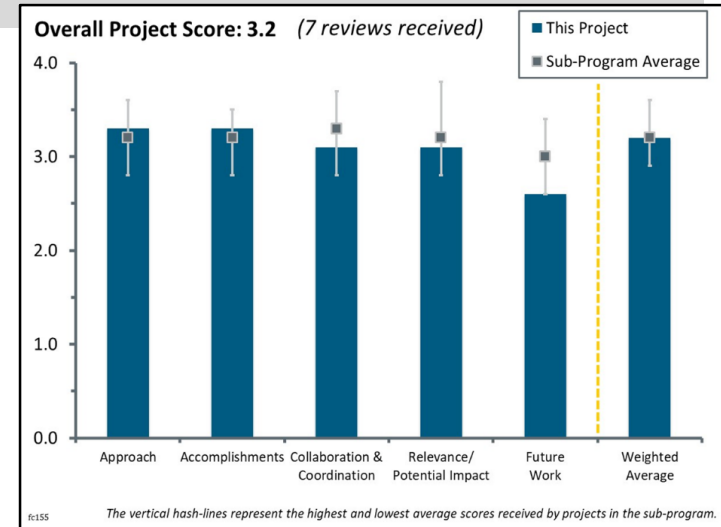
- Hopefully this presentation corrected this

The link between NSTF and novel electrode ionomers is not clear.

- NSTF catalyst was seen as a means to achieving activity and durability targets

Why MASc and imide-based ionomers was chosen is not clear

- Multi-acid side chain ionomers are more conductive
- Imides offer path to higher O₂ permeability



Summary

TASK 1: IONOMER

- **Achieved project targets** (ionomer with >50% oxygen permeability & conductivity vs. 3M825 PFSA)
- Characterized new ionomer thin films, evaluated electrodes, tested CCM for performance, durability
- Showed PFIA 8X more conductive as a thin film vs. 825PFSA, allowing low I/C operation
- Imide ionomers showing mixed gains at low RH H₂/Air operation, minimal at 100% RH or H₂/O₂ operation

TASK 2: DISPERSED NSTF

- Exceptional metal AST shown with Pt/Ir NSTF electrodes but unusual “resistance-like” loss
- Entire team root causing “resistance like” – pointing to protonic conduction issue
- High electrode agglomeration may be contributing to poor whisker coverage by ionomer
- Local gas transport is excellent – lowering I/C and Wh/C raises performance
- Activity of 0.31 A/mgPt achieved

TASK 3: ELECTRODE INTEGRATION

- Low I/C electrodes with PFIA: 18-31% less transport resistance, up to 61% activity gains, improved power
- Achieved 0.36 A/mgPt with Alloy on Graphitized carbon.
- Achieved support stability targets, getting close to metal stability targets
- NSTF transition metals leach out in electrode lowering activity. Pt/Ir active catalyst will be pursued as a result.
- **Achieved support stability targets, Achieved metal AST ECSA targets (NSTF)**

TASK 4: PNM model development in operation

- Looking into impacts of whisker coverage – and impact on water management.
- Will investigate agglomeration and ionomer properties on water management and performance.
- Look at whisker thermal differences vs. dispersed M/C catalysts.

FUTURE WORK

KEY ITEMS

- Resolve dispersed NSTF conductivity issue
- Link ionomer O₂ perm to performance gains
- Optimize new ionomers + durable M/C catalysts
- Optimize processing of low I/C systems
- Complete CCM package optimization for best cathode
- Achieve performance + durability targets

TASK1: IONOMER

- Develop additional ionomer with novel endgroups
- LBNL: Look at super-MASC, IMIDE#6 with GISAXS
- Link bulk membrane oxygen permeability to areas of performance enhancement
- Incorporate more conductive MASC into electrode
- Tufts: Look at imide thin film ionic conduction
- Tufts: CO stripping of low I/C, processed electrodes

TASK2: Dispersed NSTF

- Continue processing to improve conductivity
- ALL: Investigate “un-agglomerated” disp. NSTF electrodes
- Improve ionomer coverage of NSTF whiskers
- Further optimize NSTF 28/12 Pt/Ir to achieve >0.35 A/mgPt
- Incorporate more active materials
- LANL: Define conductivity trends of disp. NSTF electrodes
- Tufts: CO stripping of “processed” NSTF electrodes

TASK3: INTEGRATION

- ALL: Explore processing impacts on low I/C, MASC materials
- ANL,NREL: Further explore performance vs. agglomeration
- LANL: Low I/C conductivity evaluations for M/C materials
- Continue to incorporate NSTF with new ionomers
- Explore new incorporation methods with NSTF
- Optimize activities of new durable catalysts
- If needed: integrate NSTF & dispersed M/C materials
- Tufts: Ionic tortuosity vs. processing for Low I/C

TASK4: MODELING

- MTU: Continue to build fidelity
- MTU/TUFTSRoot cause dispersed NSTF performance issue
- Investigate agglomeration on performance
- Integration low I/C data & identify optimal configuration

BACKUP

Task 3: dNSTF and Transition Metal Issue

Ni and Co leach into electrode pre-test

- PtNi Cathode ionomer is completely neutralized
- Co reduces local O₂ transport and performance

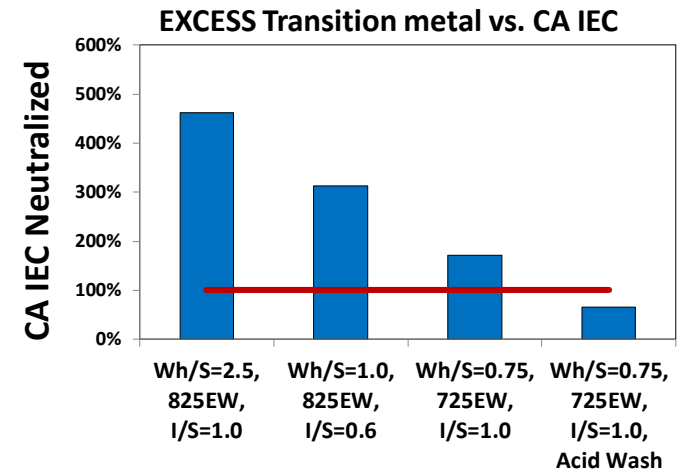
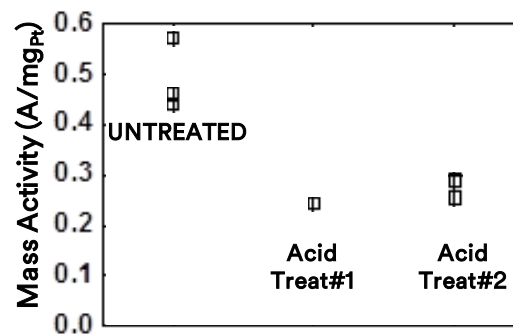
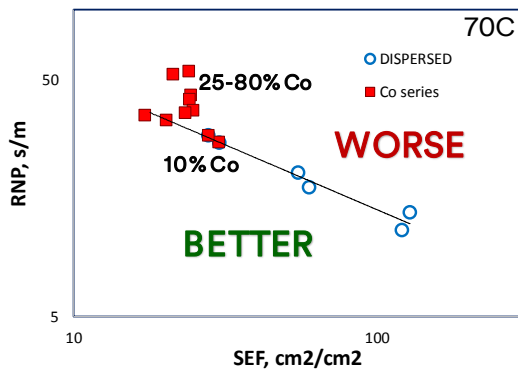
MITIGATION & Understanding Necessary

- Increase electrode ionomer & IEC
- Acid wash catalyst to remove excess Ni
- TMI operating window (NREL local transport)

Status: 1st Acid Treatments caused activity loss

- Similar result happen in ink with ionomer (acid)
- Shift focus more to non-transition metal catalysts (Pt/Ir)
- Work on heat treated NSTF for alloy retention

Catalyst	State	% Transition Metal Retained
PtCoMn	Powder	100 (Co)
	CCM/Untested	33
	Tested	20
PtNi	Powder	100 (Ni)
	CCM/Untested	72.5
	Tested	64



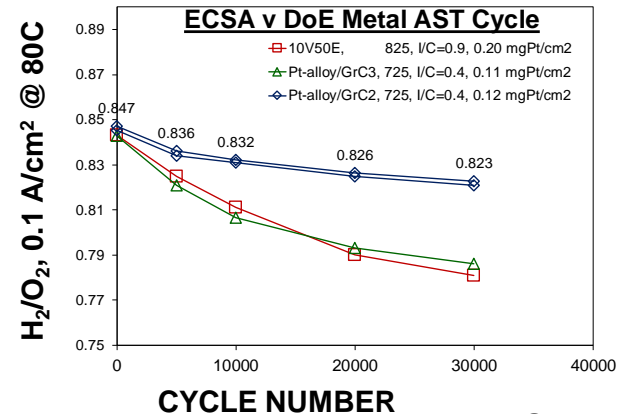
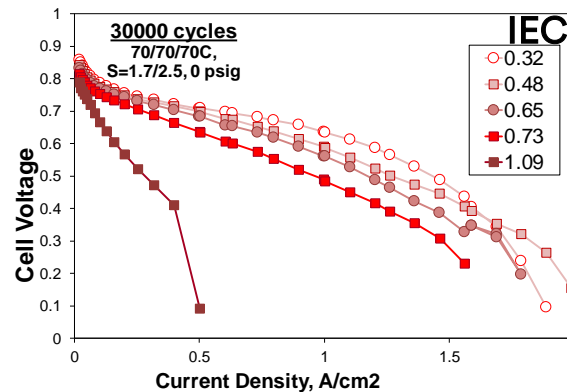
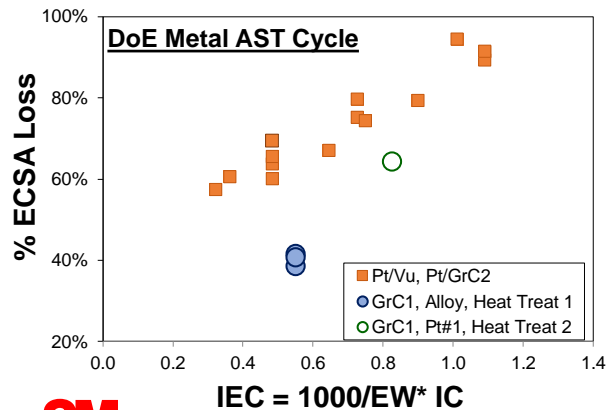
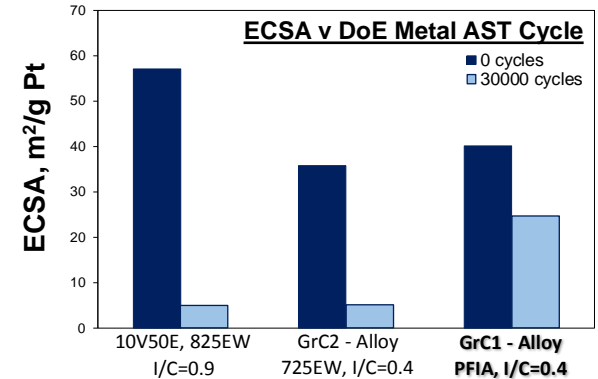
TASK 1,3: Electrode Integration for M/C catalysts

Additional Metal AST Work

OPTIMIZING Durable Carbons

- I/C and IEC vs. durability
- Lowering IEC increases durability
- Tested from 620 to 1200 EW
- End of life performance significantly improved
- Lower I/C limit where high currents suffer

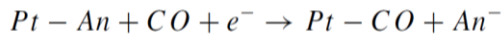
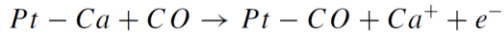
Graphitized carbon choice makes a significant difference in metal stability



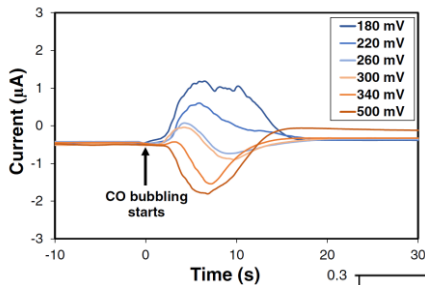
CO displacement Technique

• **Developed by Feliu:**

- Constant potential is applied and zero-charge CO displaces adsorbing species on Pt. Oxidative or reductive current can be measured, depends on what type of species are displaced:

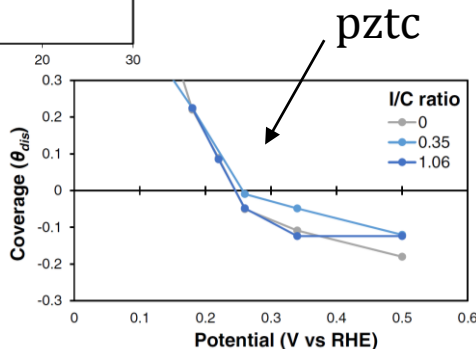


- Measured displacement current densities are integrated. CO-adsorption takes place without change in oxidation state. We can then calculate the coverage using q_{strip} .



$$q_{dis} = q_f - q_i \approx -q_i$$

$$\theta_{dis} = \frac{2 \times q_{dis}}{q_{strip}}$$



AC+DC electrode Technique

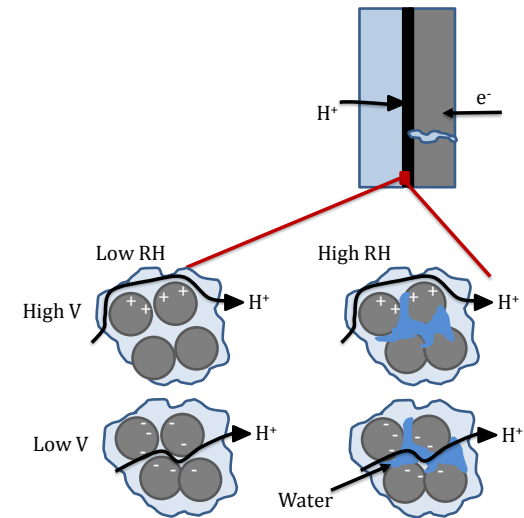
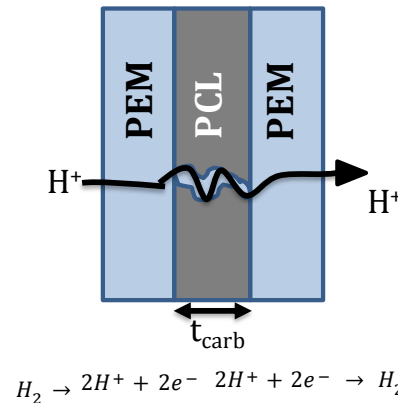


• **DC Technique**

- Easy to interpret data
- Contact resistance and membrane resistance isolated
- Protons pumped through membrane and PCL
- **Method captures ionomer tortuosity**

• **AC technique**

- **Does not capture layer tortuosity**
- Double layer capacitive charging since no Pt present
- Capacitive charging only at PEM/electrode interface



Garrick, T. R.; Moylan, T. E.; Yarlagadda, V.; Kongkanand, A., *J Electrochem Soc* **2017**, *164* (2), F60-F64.



TASK1: Novel Ionomer Development

Ex-Situ vs. In-Situ

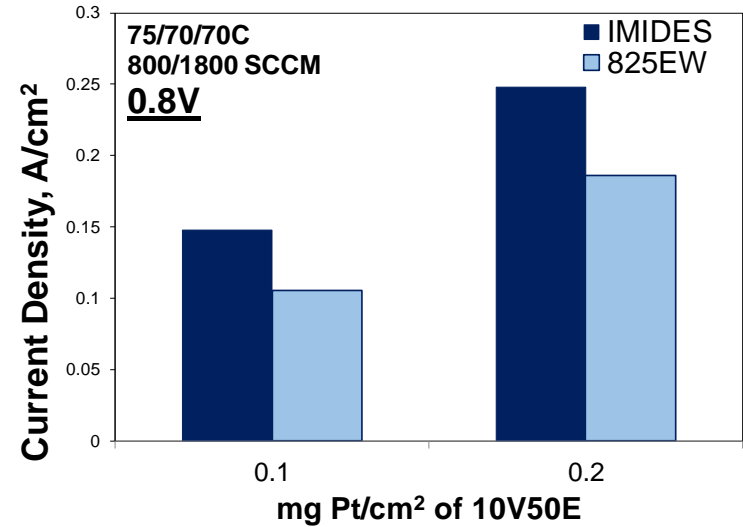
Imide-based materials show gains

- At H₂/Air, sub-saturated, high stoic
- Bulk O₂ perm also better drier
- Imides #1, 2, 3, 6 tested

Imide not showing H₂/O₂ activity gains

Results unlike 1200EW PFSA

- Shows H₂/O₂ activity gains



	<u>ELECTRODE PERFORMANCE GAINS</u>						
	BULK FILM	Local O ₂ Transport	Thin film Conductivity	In-cell Tests	Activity	dNSTF	Metal Stability
PFIA & MASC	No	No	Yes	No	No	Yes	No
IMIDES	YES	No, possibly low RH	Not Tested	H ₂ /Air, <100% RH	Variable	Not Yet	No
Low I/C	---	Yes	No	Yes	Yes	Yes	Yes
PFIA + Low I/C	---	Yes	Yes	Yes	Yes	Yes	Yes

Focus is on Low surface area, durable carbons. Metal is on the surface & better interact with ionomer.

Acknowledgements

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