

Development of Durable Active Supports for Low Platinum Group Metal Catalysts

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Overview



Timeline

 Project Start Date: 02/21/17 •Project End Date: 11/20/17 •Total Project Budget: \$149,973

Budget

- Total Project Budget: \$149,973
- Total recipient share: \$0
- •Total Federal share: \$149,973
- •Total DOE funds spent: \$3,615

Barriers

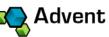
 Durability, Cost, and Performance low-PGM content PEMFC catalyst supports is insufficient (FCTO MYRDD)

Partners

Pajarito Powder

-Durable Active Carbon Supports & catalysts

•Advent Technologies • Advent



-Electrodes and MEAs

June 5, 2017

Relevance: Objectives

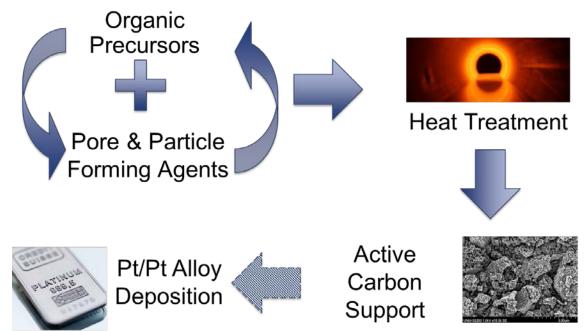


- Develop Pajarito's Durable Active Carbon Supports (DACSs)
 - Optimize materials characteristics
 - Optimize Pt/support interactions
 - Increase support carbon corrosion resistance
 - Increase active support/Pt activity and durability synergism
- Deploy Pt/DACS in MEAs for validation
 - Integrate Pt/DACS into the MEA structure
 - Test and provide MEAs for validations
- Impact
 - Increase the durability of fuel cell electrocatalysts
 - Improve economics of PEMFC

Approach

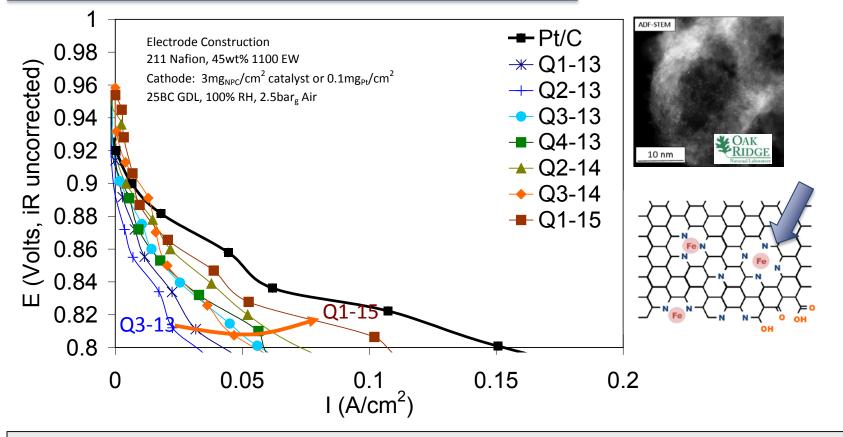


- Improve stability of M-N-C type PGM-free ORR catalysts developed with assistance of FCTO projects (DACS). Design Pt/DACS catalysts with increased durability
 - Fe, Co, and Mn based



Approach



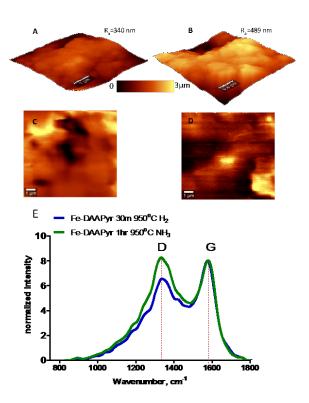


Precious Metal Free Supports performance now exceed 50mA/cm² @0.8V

Approach



- Iteratively modify catalysts
 - Modify the precursors used in making PGM-free catalysts to
 - Control DACS morphology (surface area, porosity etc) through the design of synthetic approach
 - Increase Pt dispersion via increased Pt-support interaction
 - Increase graphitic content through selection of graphene-forming organic molecules (N-C precursors)
 - Resist membrane and ionomer degradation trough decrease of H₂O₂ generation



Approach: Timeline and Milestones



ID	Task Name	Start	Finish	Duration	Q1 17 Q2 17			Q3 17		Q4 17		
					Mar		May	Jun	Jul	Aug	Sep	Oct
1	Synthesis of active supports for initial testing.	3/1/2017	6/1/2017	67d								
2	Testing of AS in RRDE and RDE MEA	3/1/2017	4/28/2017	43d								
3	Downselect to 3 supports	4/28/2017	4/28/2017	0d								
4	Decoration of AS with Pt	4/3/2017	6/1/2017	44d								
5	Testing of Pt/AS in MEA	6/1/2017	6/30/2017	22d								
6	Re-optimization of new catalysts for MEA	7/3/2017	9/1/2017	45d								
7	Achieve 3nm Pt particles on 2-3 DACS	7/3/2017	7/3/2017	0d								
8	Testing re-optimized catalysts in MEA	9/1/2017	9/29/2017	21d								
9	Downselect to 2 supports	10/2/2017	10/2/2017	0d								
10	Scale up of re-optimized catalysts to 25gr level	9/1/2017	11/1/2017	44d								
11	Collection of results and writing report, Phase 2 plan	11/2/2017	9/20/2018	231d								
12	Deliver MEAs for validation	11/20/2017	11/20/2017	Od								

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

Pajarito participated at SBIR Kick-off meeting. Project started according to the schedule.

Accomplishments: Commercial Baseline



Kinetic Region (Log Scale)

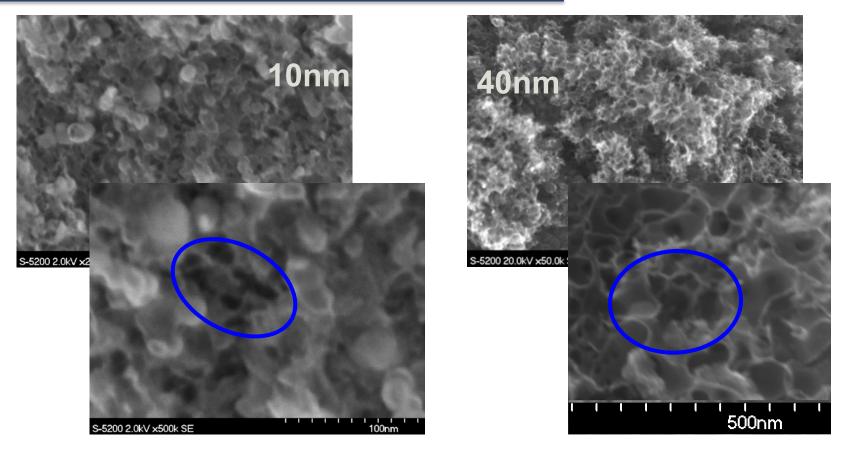
0.94 1 0.92 0.9 -HiSpec 9100 BoE 0.88 -HiSpec 9100 BoE --- HiSpec 9100 EoE 0.86 --- HiSpec 9100 EoE 0.84 0.82 0.3 0.5 1.0 1.5 2.0 0.8 0.0 $I(A/cm^2)$ 0.01 0.1 1

Commercial catalyst degrades drastically under DoE Start-Stop AST: 89% activity loss

Polarization Curve

Approach and progress: Pore Size Control

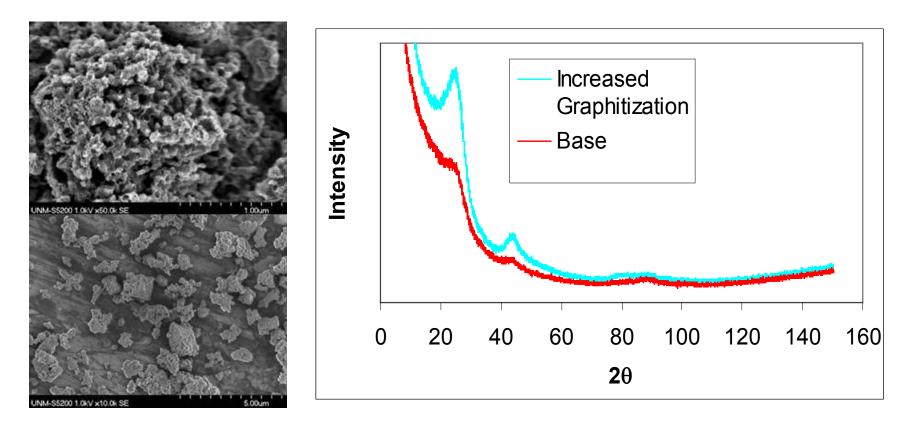




Design of DACS by VariPore[®] approach allows control support morphology

Approach and progress: Graphitization

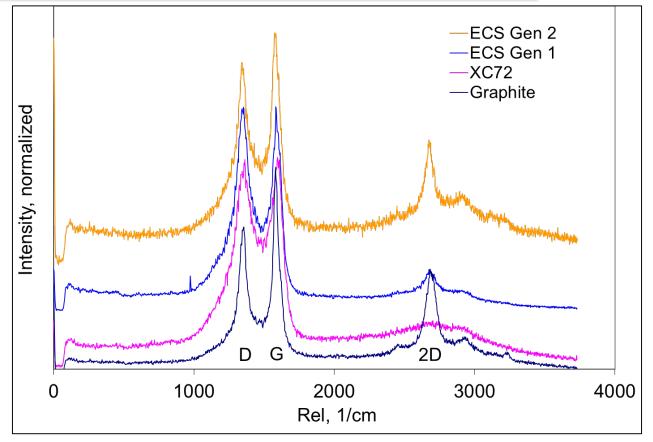




Higher graphitization leads to more corrosion resistance under Start-Stop AST protocol

Approach and progress: Graphitization

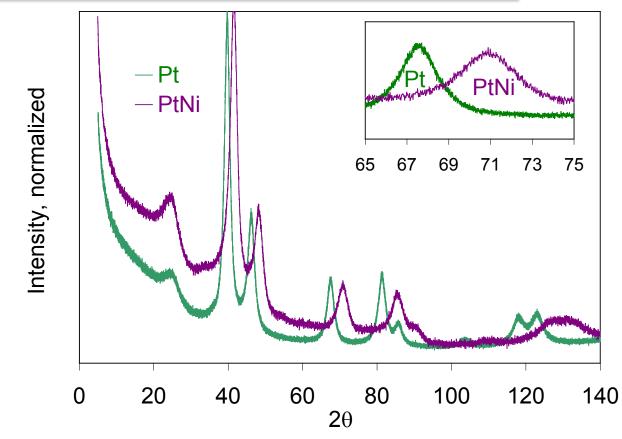




Graphitic content is controlled by heat treatment conditions and nature of organic precursor

Approach and progress: Platinization





Proprietary Pt deposition method with control of: particle size and chemical composition (alloys)

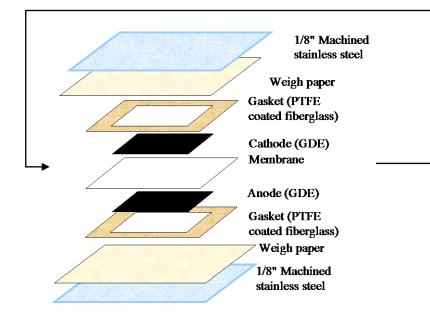
Approach and progress: MEA Manufacturing at Pajarito



Silverson high shear mixer 2-5wt% solids Ink

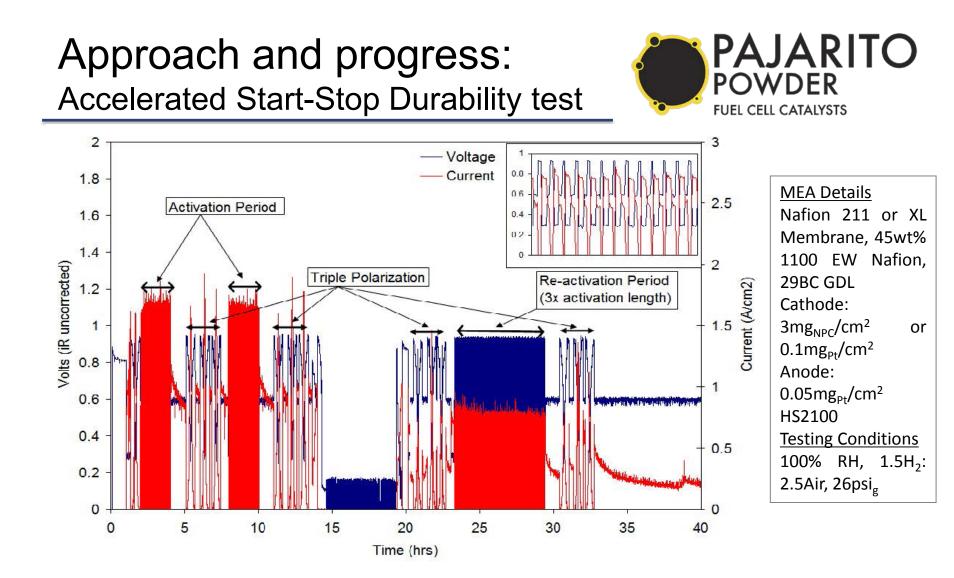
Sonotek ExactCoat
→ 25kHz sonic nozzle
2-200µgr/cm²/pass





Carver 3860 heated press ~6+ minutes 131°C+ 75psi for 25cm² 60psi for 50cm²



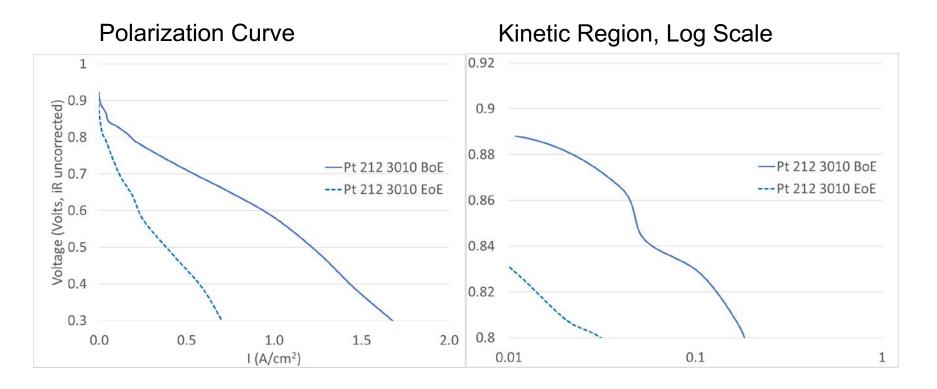


1-1.5V, 5000 cycle Start-Up/Shut-Down test procedure

June 5, 2017

Approach and progress: Start-Stop Results for Gen 1





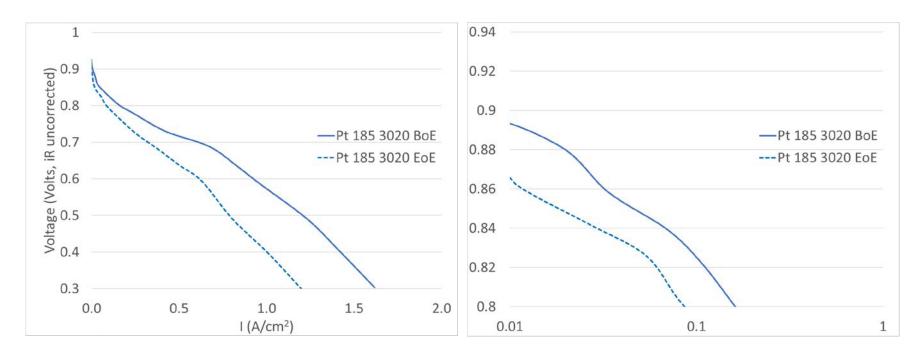
Gen 1 of Pajarito Pt/DACS 3010 degrades on the same level as a commercial Pt/C (89% activity loss)

Approach and progress: Start-Stop Results for Gen 2



Polarization Curve

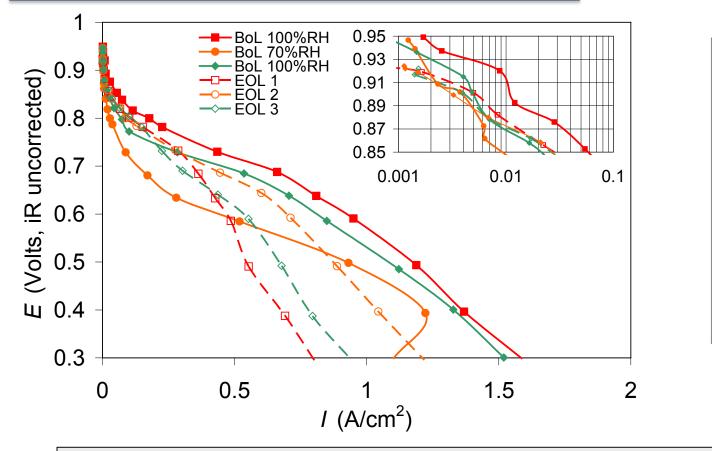
Kinetic Region, Log Scale



Gen 2 Pt/DACS 3020 catalyst, degrades 63%

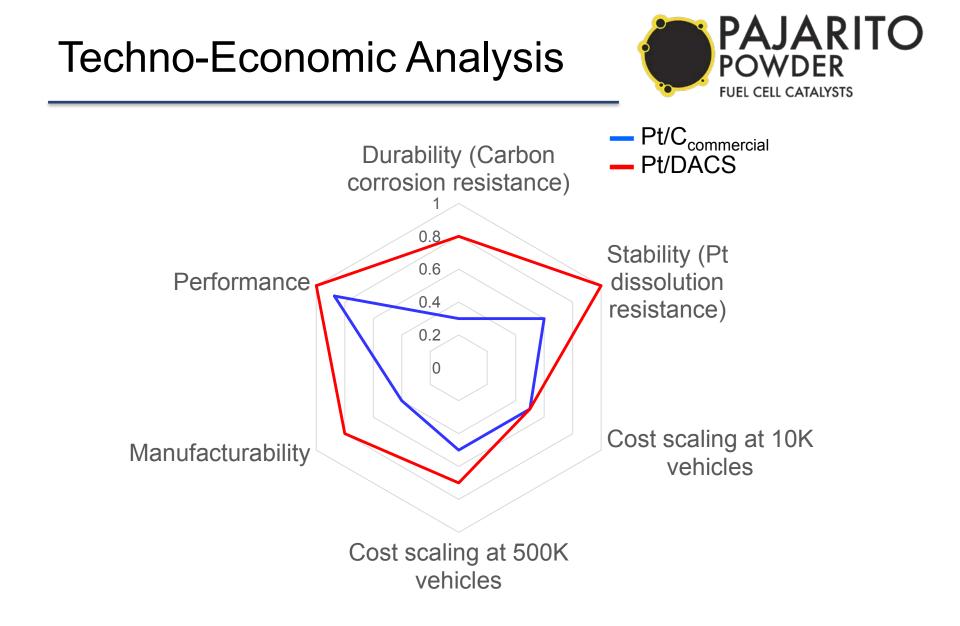
Approach and progress: Start-Stop Results Summary





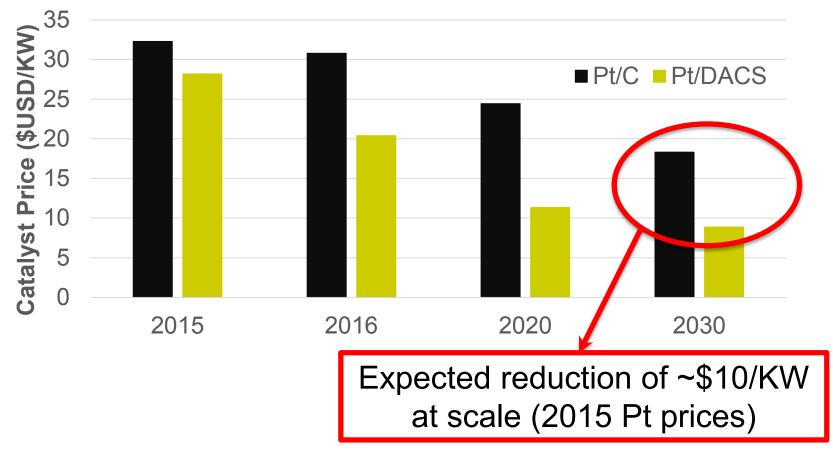
MEA Details 211 or XL Membrane, 45wt% 1100 EW Nafion, 29BC GDL Cathode: 0.1mg_{Pt}/cm² Anode: 0.05mg_{Pt}/cm² HS2100 <u>Testing Conditions</u> 100% RH, 1.5H₂: 2.5Air, 26psi_g

Integration of Pt/DACS into the catalytic layer needs improvement. Excellent durability confirmed.





Comparison of Pt/C to Pt/DACS Cost



Pt/C Cost from DOE "Fuel Cell System Cost – 2015"; Pt price fixed at 2015 levels

Collaborations



- Pajarito Powder (Prime)
 - Management
 - Synthesis of Durable Active Supports (DACS)
 - Platinization of DACS
 - Manufacturing and testing of MEAs
- Advent Technologies (sub)
 - MEA catalyst layer optimization
 - Feedback on electrode/catalyst interaction
 - Improved MEA construction
- Pajarito and Advent have been collaborating closely under an MOU as well as partnership in a DoE Incubator program (FC132)





 Pajarito Powder performed analysis of preliminary data on DACS synthesis and characterization

Early results show ~40% increase in durability

- Commercial Pt/C materials were selected for benchmarking activity and durability
- Initial parameters for synthesis of DACS and Pt/DACS were identified and materials are being synthesized by Pajarito
- Advent began making electrodes

Future Activities



- Phase 1
 - Demonstration of concept
 - Identification of challenges, barriers, and advantages
 - Proposing mitigation scenarios
- Phase 2
 - Continued refinement of approach
 - Include Pt-alloys in the catalysts
 - Increasing of support carbon corrosion resistance
 - Inclusion of additional PGM-free manufacturing platforms
 - LANL-based approach recently licensed by Pajarito
 - Promising technologies arising from EMN/ElectroCat
 - Perform electrode and MEA modification and improvement
 - Testing and diagnostics of MEA performance
 - Feedback of testing results to modify catalyst supports
 - Automotive and lab validation, diagnostics and feedback
- "Any proposed future work is subject to change based on funding levels."