Advanced Electro-Catalysts through Crystallographic Enhancement

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Los Alamos National Laboratory

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Project ID FC161

Overview

Timeline:

- Project Start Date: October 1, 2016*
- Project End Date: September 30, 2019

**Subcontracts in place February 2017*

Budget:

- Total Project Budget: $3.335M
  - Total Recipient Share: $335K
  - Total Federal Share: $3M
  - Total DOE Funds Spent: $1.1M*

  **As of 3/31/2018*

Barriers

A. Durability

B. Cost

C. Performance

Partners

- LANL (J. Spendelow)
- Brown University (S. Sun, A. Peterson)
- University of Pennsylvania (C. Murray)
- SUNY University at Buffalo (G. Wu)
- EWII Fuel Cells (M. Odgaard)

Relevance

Objectives

- **Design active and durable nanoparticle ORR catalysts based on fully-ordered intermetallic alloys on highly graphitized nitrogen-doped carbon supports**
  - Binary and ternary alloys of Pt with Co, Ni, other base metals
  - Project will avoid Fenton-active metals
  - Commercial supports used initially; N-doped C supports later
- **Demonstrate catalysts in high-performance, durable MEAs and scale up to 50 cm^2**

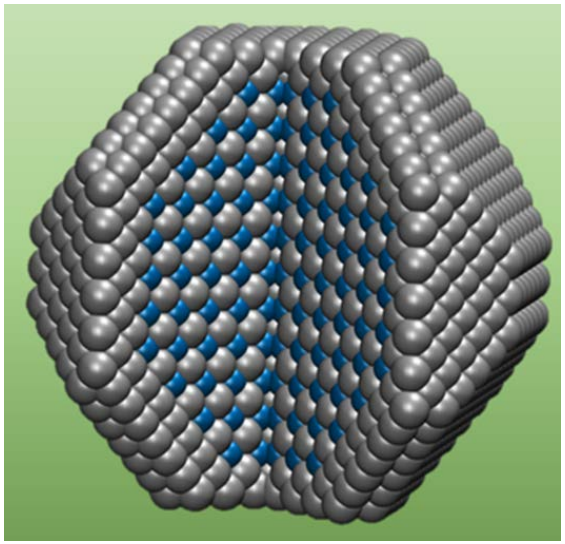
Project Targets:

- Mass activity > 0.44 A/mg_{PGM} @ 0.9 $V_{iR\text{-}free}$
- <40% mass activity loss after catalyst AST
- <30 mV loss at 0.8 A/cm^2 after catalyst AST
- PGM total loading < 0.125 mg/cm^2
- Power density > 1 W/cm^2
- <40% mass activity loss after support AST
- <30 mV loss at 1.5 A/cm^2 after support AST

Approach: Synthesis

Use atomic-level ordering to increase performance and durability of Pt-based catalysts

- Synthesize intermetallic nanoparticles (CoPt, NiPt, ternaries)
  - Prepare fully-ordered cores to stabilize base metal
  - Further protect core with Pt skin
  - Use theory and computation (DFT, machine-learning techniques) to guide nanoparticle design
- Support nanoparticles on Fe-free, N-doped graphitic carbon



Approach: Characterization and Testing

Use atomic-level ordering to increase performance and durability of Pt-based catalysts

- Integrate supported nanoparticles into MEAs, test initial performance and durability
- Perform MEA diagnostics (impedance, limiting current methods) to characterize loss mechanisms and guide electrode design
- Perform initial and post-mortem characterization (XRD, XAS, XRF, SEM-EDS, TEM, STEM-HAADF, STEM-EDS) to guide synthetic work and determine effect of structure and composition on performance and durability
- Scale-up and validate MEA performance (5 cm^2 → 50 cm^2)
- Scale-up catalyst synthesis (multi-gram batches)

Approach: Catalyst Structures

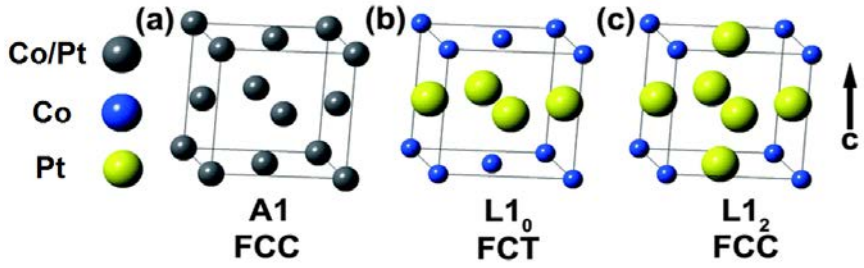
Ordered intermetallic catalysts

Primary material set:

1. fct-MPt (face-centered tetragonal, $L1_0$ structure, M = Co, Ni, other transition metals)
2. fct-M_1M_2Pt (ternaries)

Alternative materials (risk mitigation):

1. $L1_2$ structures (Pt_3M)
2. Doping with other elements
3. Other intermetallics

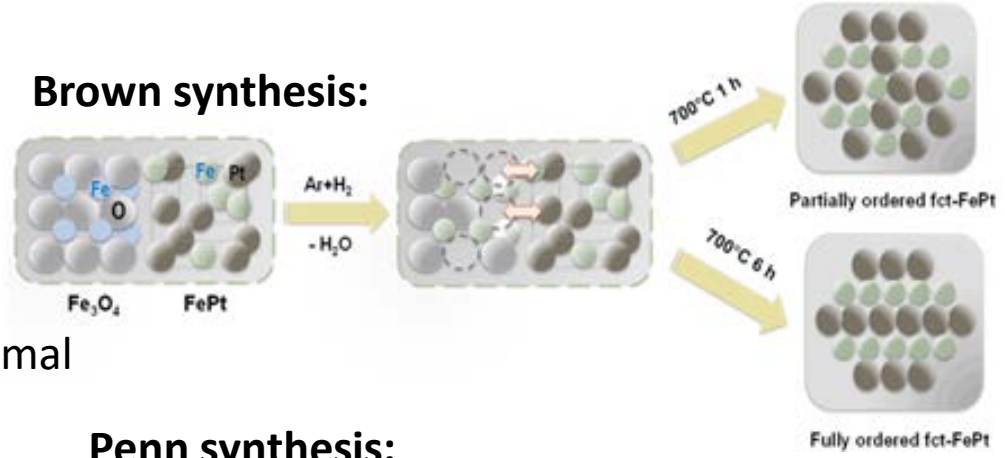


Adapted from Johnston-Peck et al., *Nanoscale*, 2011, **3**, 4142

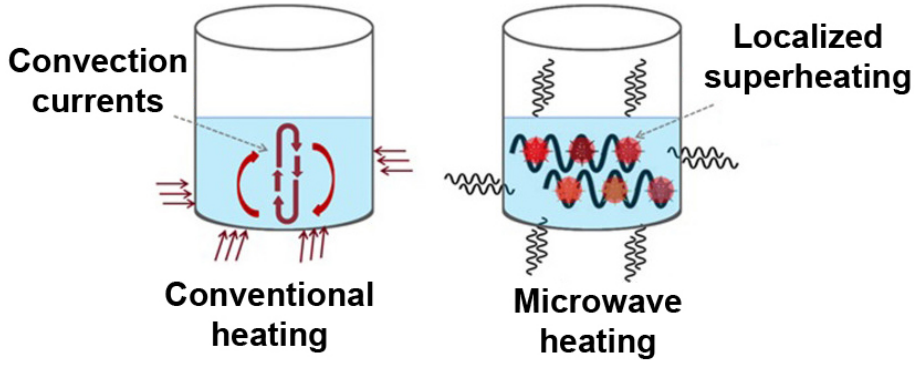
Approach: fct-MPt Synthesis

1. Brown: wet chemical synthesis of alloy nanoparticles in high-boiling solvents, followed by thermal annealing to create ordered structures (highest control, lowest scalability)
2. Penn: microwave synthesis and rapid thermal annealing (high risk, but may provide enhanced ordering, improved scalability)
3. LANL: seed-mediated synthesis by metal salt impregnation in Pt/C, followed by annealing to produce ordered structures (lowest control but highest scalability)

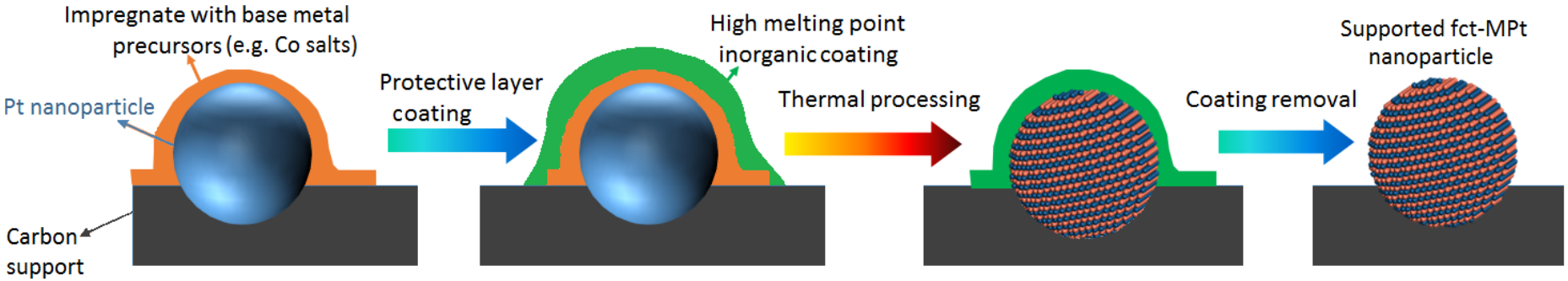
Brown synthesis:



Penn synthesis:



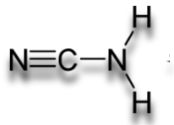
LANL synthesis:



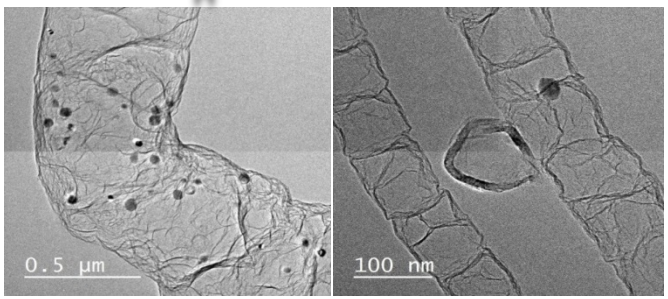
Approach: N-doped Carbon Supports

We are developing several classes of advanced carbon supports:

N-doped graphene tube supports

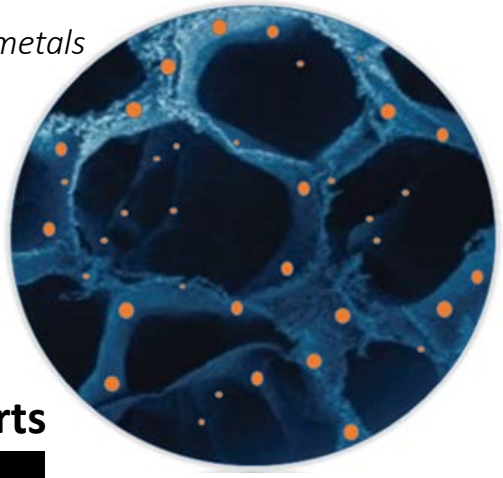


Dicyanamide + metals



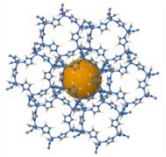
Hydrogel-based nanocarbon supports

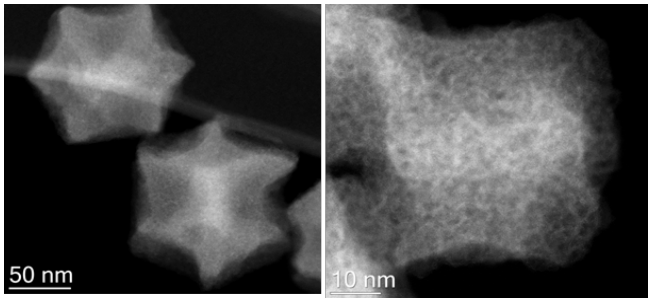
Polyaniline + metals



MOF-derived nanocarbon supports

ZIF-8 + metals





Key attributes:

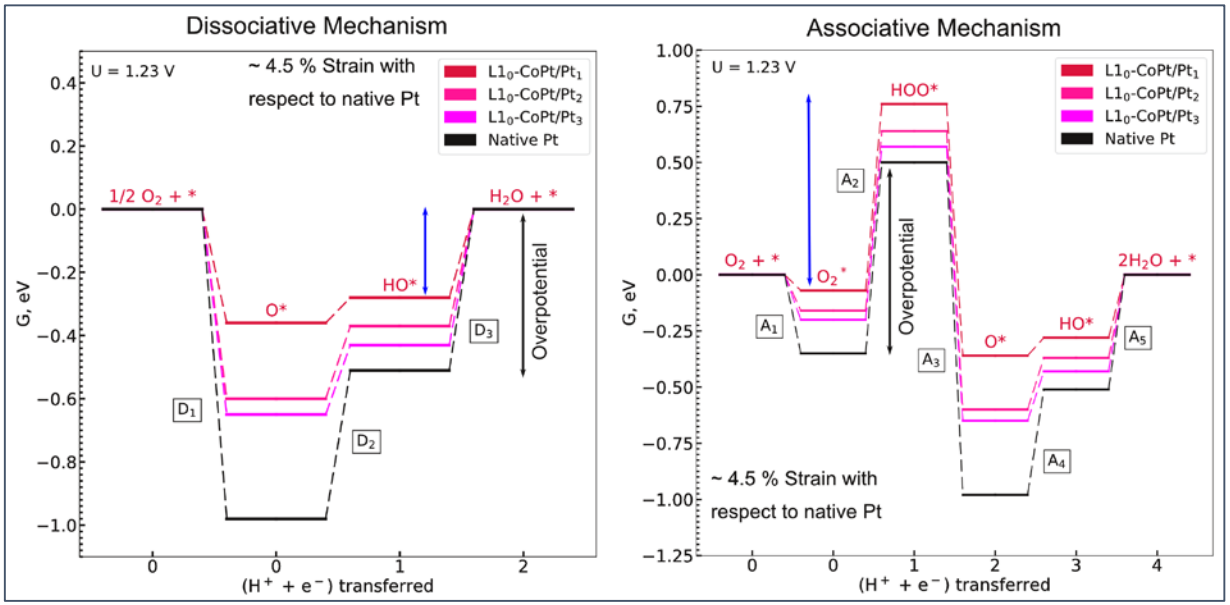
- **Highly graphitized** - improved durability
- **N-doped** - improved dispersion and stabilization of nanoparticle catalysts
- **Fe-free** - avoids Fenton degradation

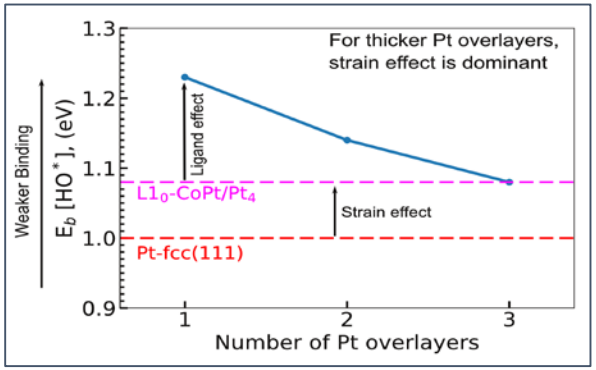
Milestones

12/16	Synthesize fully ordered fct-FePt nanoparticles from FePt-Fe_3O_4 precursors and perform initial electrochemical characterization
3/17	Incorporate at least two distinct ordered intermetallic catalysts into MEAs and perform fuel cell testing including mass activity and high-current performance
6/17	Perform initial durability testing (square-wave AST) on ordered intermetallic catalysts in MEA
9/17	Demonstrate 5-7 nm fct-MPt with durability in electrocatalyst AST superior to baseline Pt/C
12/17	Synthesize at least two distinct N-doped supports and compare their properties as catalyst supports
3/18	**GO/NO-GO:** Demonstrate 0.44 A/mg_{PGM} mass activity on an Fe-free system under operating conditions specified for DOE mass activity target
6/18	Develop alternative fct-CoPt synthetic pathway using deposition on Pt nanoparticle seeds
9/18	Develop atomistic models that attribute reactivity changes to strain, ligand, and crystal structure for fct-CoPt system
12/18	Demonstrate ordered intermetallic nanoparticle catalyst meeting mass activity and 30,000 cycle AST durability targets in 5 cm^2 MEA
3/19	Demonstrate supported catalyst meeting 5,000 cycle support AST durability targets in 5 cm^2 MEA
6/19	Demonstrate ordered intermetallic nanoparticle catalyst meeting mass activity and 30,000 cycle AST durability targets in 50 cm^2 MEA
9/19	Validate MEA performance of 1 W/cm^2 or greater and achievement of mass activity and durability targets in 50 cm^2 MEA

Accomplishments and Progress: DFT Computation

Effect of strain and ligand on the reaction intermediates of ORR: DFT study on (111) surfaces

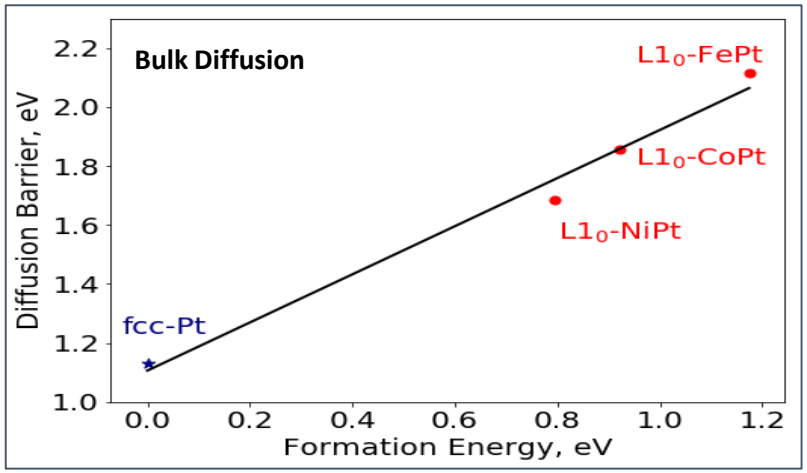


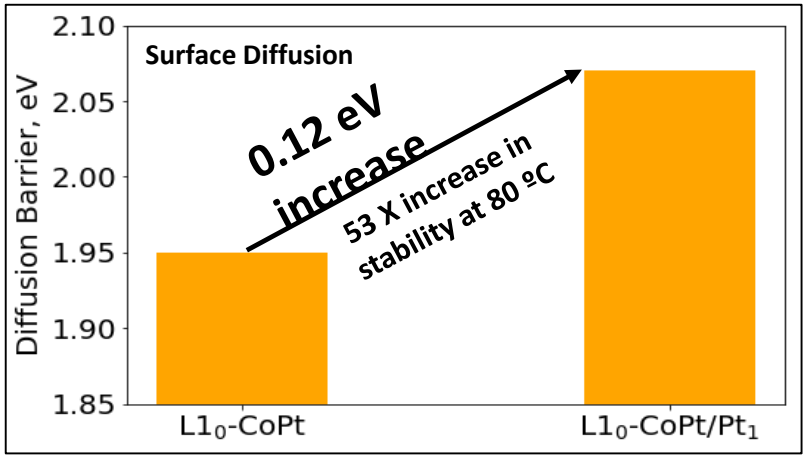


- Compressive strain raises the free energies of the reaction intermediates
- Ligand effect can further tune the overpotential at BOL (1-2 layer shell), but ligand effect is attenuated by thick shell (3-4 layers) after AST

Accomplishments and Progress: DFT Computation

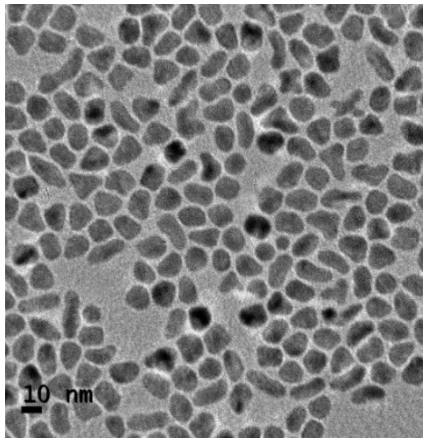
Kinetic Stability: Bulk and Surface diffusion





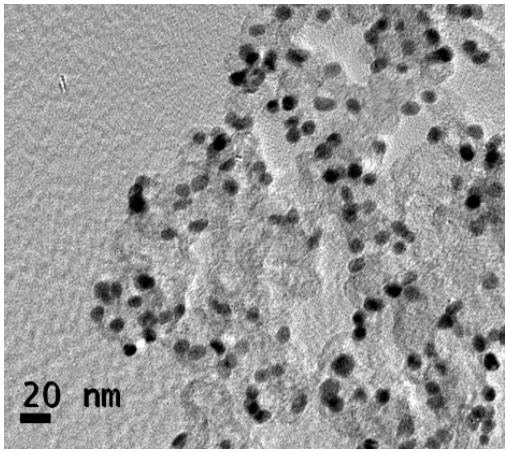
- We use vacancy mediated diffusion as a descriptor of kinetic stability
- Helps understand and predict long-term stability, which is difficult to measure experimentally
- Helps predict the number of Pt layers to expect, which greatly influences whether strain or ligand effect dominates
- We are developing principles and correlations to guide searches for new materials e.g., diffusion barrier vs. formation energy

Accomplishments and Progress: Brown fct-CoPt/Pt

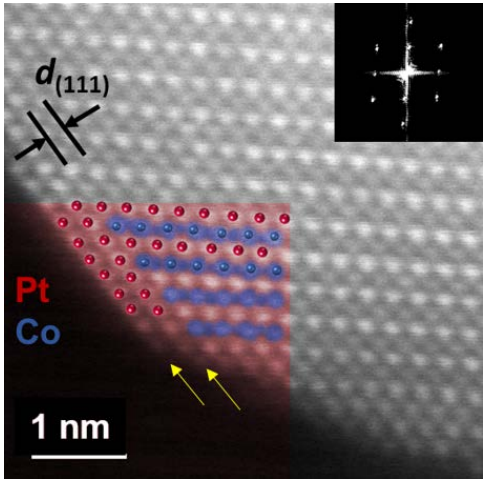


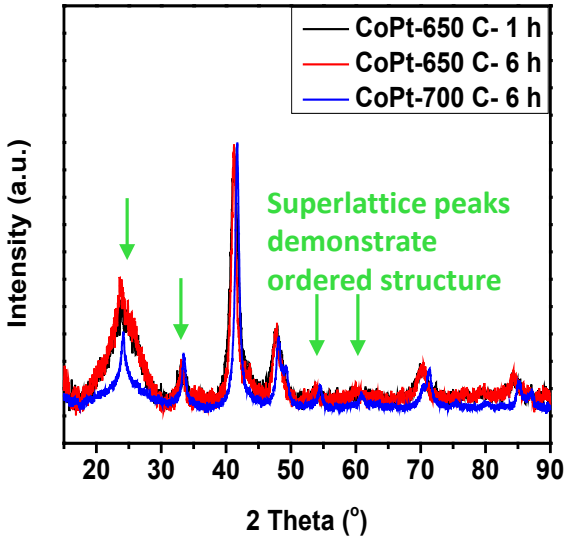
9 nm $Co_{49}Pt_{51}$

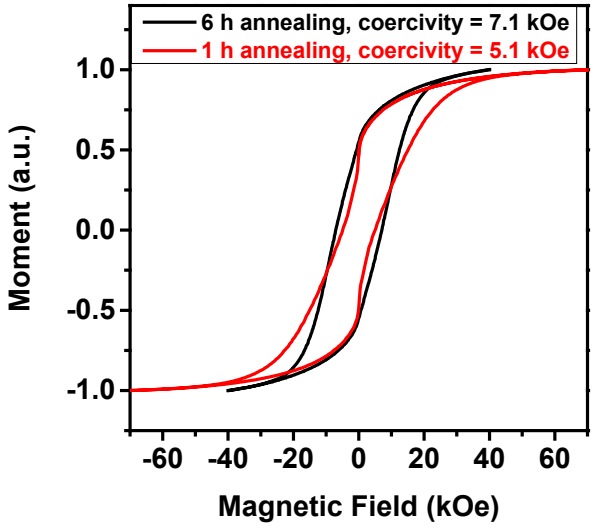
Loaded on carbon, annealed at 650°C for 6 h in 5% H_2/Ar



After acid leach:



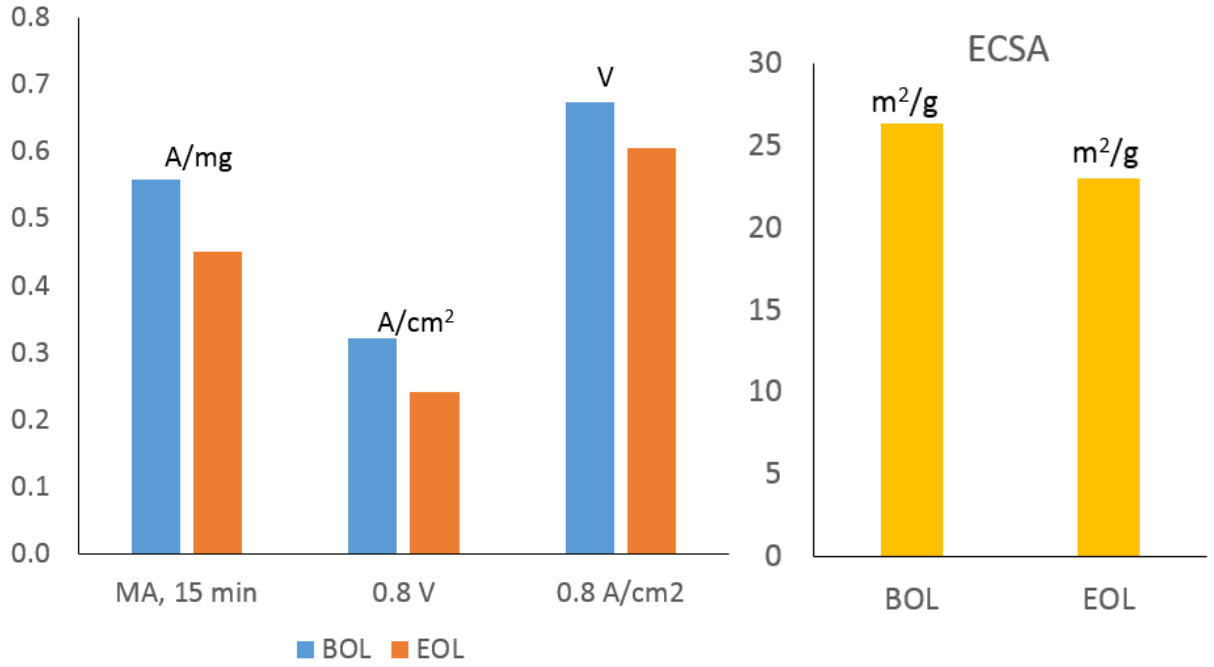


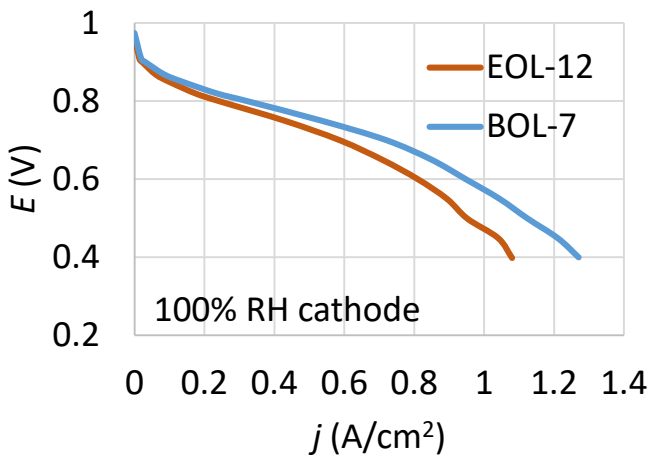


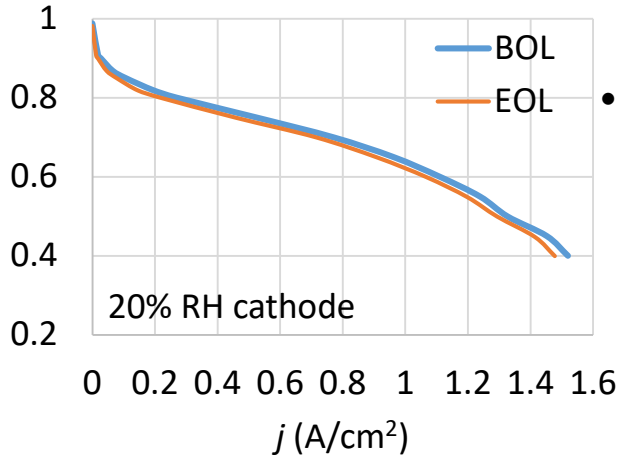
- XRD, coercivity measurements, and TEM all demonstrate high degree of ordering
- Pt shell (~2 atoms thick) after acid leach

BROWN

Accomplishments and Progress: Brown fct-CoPt/Pt



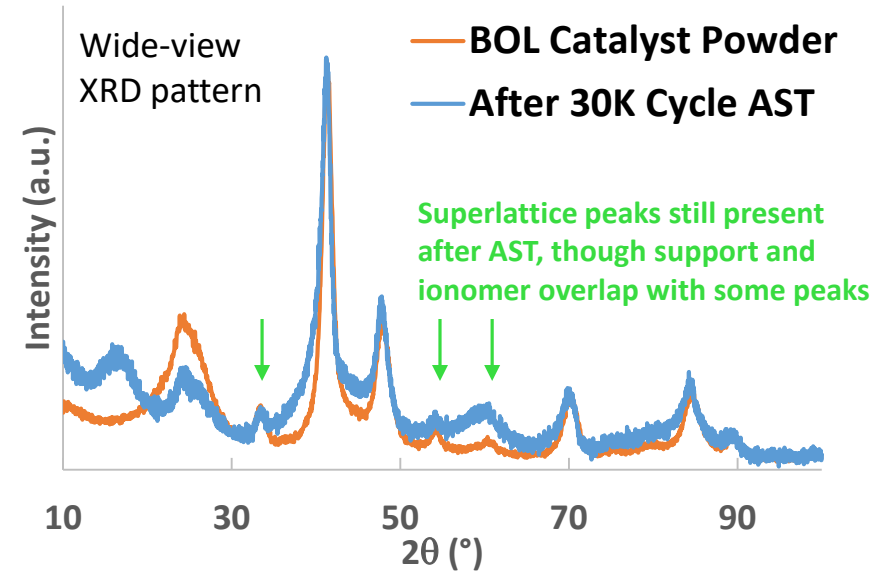


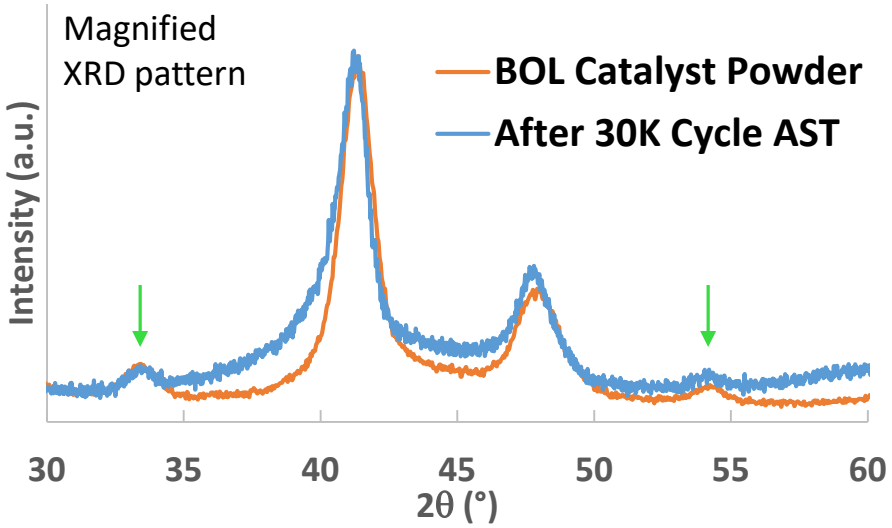


- BOL mass activity: **0.56 A/mgPGM** (Target: >0.44 A/mgPGM)
- Post-30K cycle mass activity (measured at 15 min): **0.45 A/mgPGM**
- Loss after 30K cycles: **20%** (Target: <40%)
- Loss at 0.8 A/cm² after 30K cycles: **69 mV** (Target: <30 mV), but mostly due to flooding
- Power density: **0.58 and 0.73 W/cm²** at 150 and 250 kPa (Target: 1 W/cm²)

150 kPaabs, 500/1000 sccm H_2/air, 5 cm², 0.105 mgPt/cm² cathode, Nafion 211

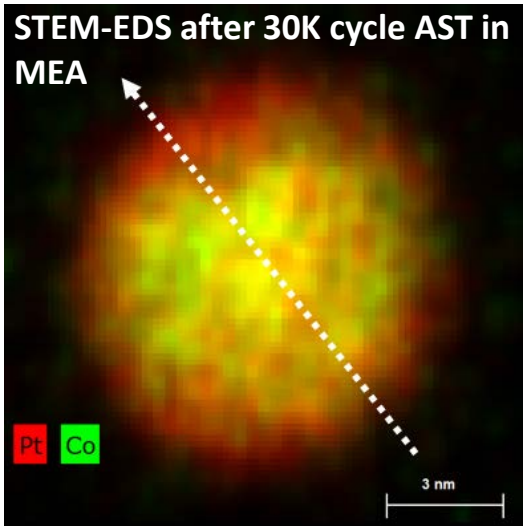
Accomplishments and Progress: Brown fct-CoPt/Pt

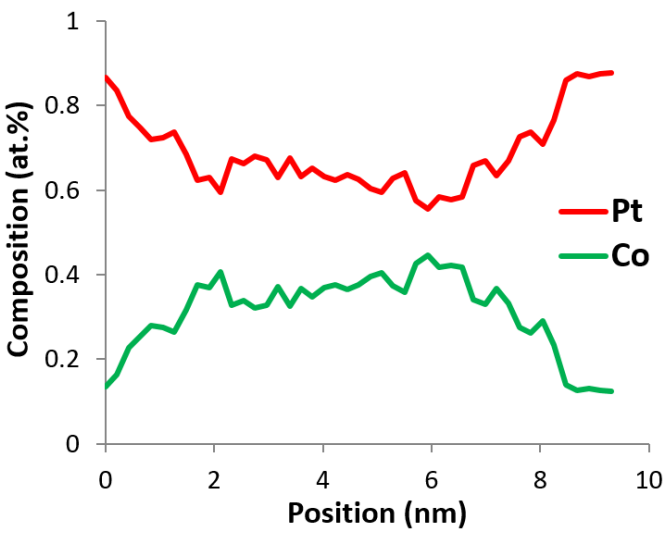




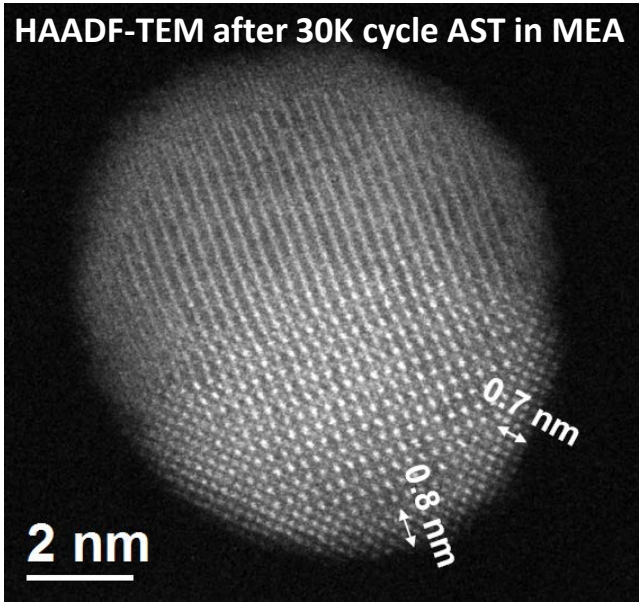
- XRD superlattice peaks are slightly smaller after 30K cycle AST, suggesting surface leaching
- Insignificant shift in peak position, indicating **lattice established by ordered core remains unchanged**
- XRF indicates composition change ($Pt_{0.61}Co_{0.39}$ → $Pt_{0.71}Co_{0.29}$) corresponding to **1-2 atom shell at BOL, 3-4 atom shell at EOL**

Accomplishments and Progress: Brown fct-CoPt/Pt





- STEM-EDS shows ~1 nm Pt shell surrounding $Pt_{50}Co_{50}$ core after AST (total particle composition $Pt_{70}Co_{30}$)
- HAADF-TEM shows highly ordered core remains after AST, coated with a ~0.7-1.0 nm Pt shell (3-4 atoms thick)



Key conclusions:

- Ordered core remains intact even after AST
- Co leaching occurs only from surface, forming Pt shell that protects particle interior from further leaching
- Pt shell is too thick for significant ligand enhancement after AST, but **kinetic enhancement due to strain remains even after 30K cycles**

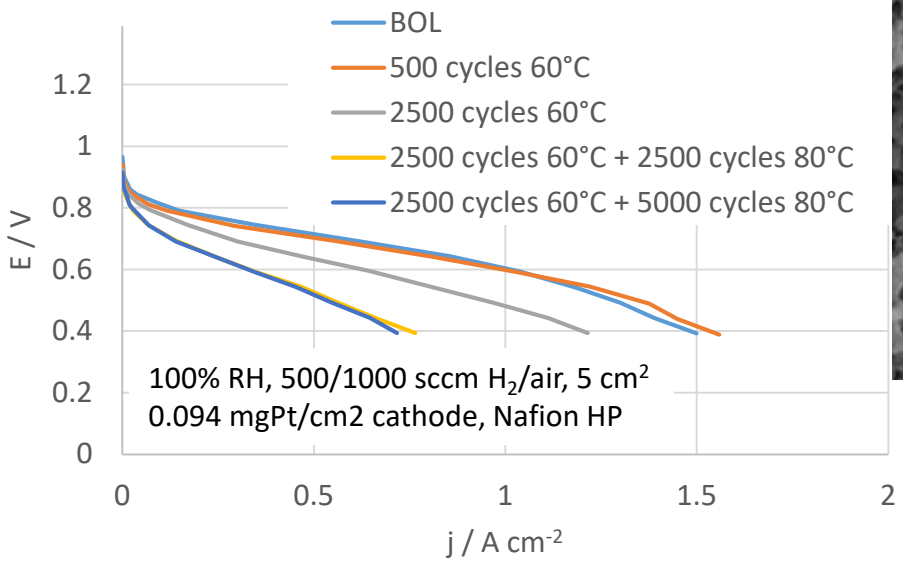
OAK RIDGE National Laboratory

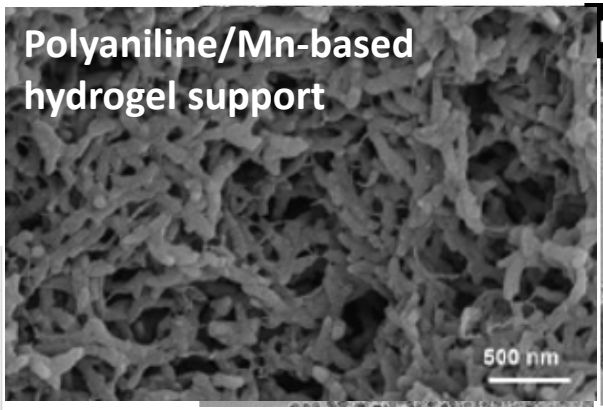
Accomplishments and Progress: Brown fct-CoPt/Pt

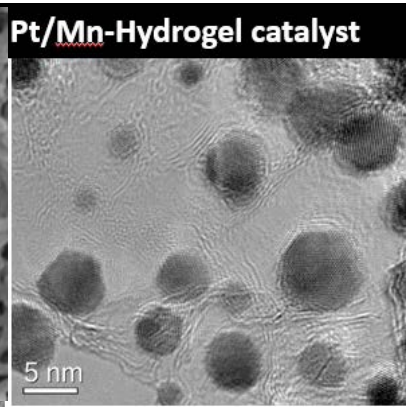
	Units	Measured	Target
Mass Activity	A/mgPGM	**0.56**	**0.44**
Mass Activity after Catalyst AST	A/mgPGM	**0.45**	**0.264**
Degradation at 0.8 A/cm^2 (Catalyst AST)	mV	**69**	**30**
Current Density at 0.8 V	A/cm^2	**0.32**	**0.3**
Power at 0.67 V, 150 kPa$_{abs}$	W/cm^2	**0.58**	**1**
Power at 0.67 V, 250 kPa$_{abs}$	W/cm^2	**0.73**	**1**
Cathode PGM Loading	mg/cm^2	**0.105**	**0.125**
Robustness, Cold		**0.64**	**0.7**
Robustness, Cold Transient		**0.68**	**0.7**
Robustness, Hot		**0.19**	**0.7**
ECSA	m^2/gPt	**26**	
ECSA after Catalyst AST	m^2/gPt	**23**	
Crystallite Size (XRD)	nm	**7.8**	
Crystallite Size after Catalyst AST	nm	**9.6**	
Particle Size (TEM)	nm	**8.9**	
Particle Size after Catalyst AST	nm	**8.7**	
Composition	%	**Pt61Co39**	
Composition after Catalyst AST	%	**Pt71Co29**	
Degree of Ordering (XRD)	%	**85**	
Ordering after Catalyst AST (XRD)	%	**77**	

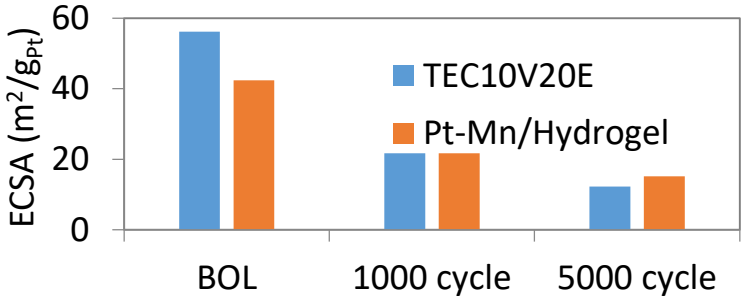
- High mass activity at BOL; only 20% loss after AST
- Excellent ECSA retention (but low ECSA from the start)
- Degradation at 0.8 A/cm^2 due to increased flooding after AST
- High power performance is too low – probably due to thick electrode (~25 µm)

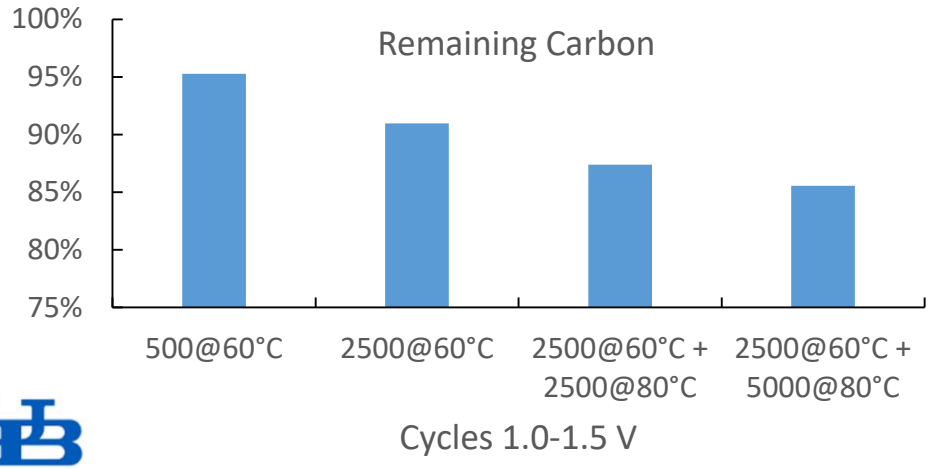
Accomplishments and Progress: Pt/C Hydrogel Support





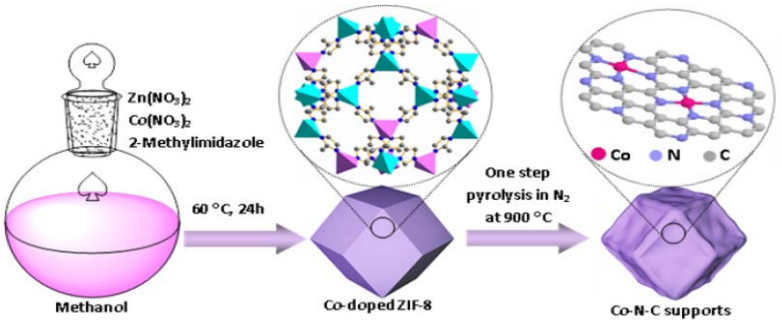




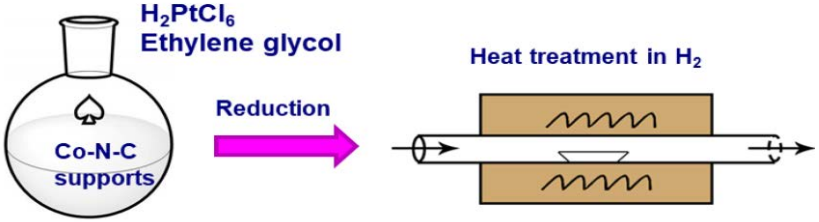


- Pt on carbon support from polyaniline hydrogel precursor (Mn-based) provides good BOL polarization performance
- High stability for first 500 cycles, and better ECSA retention than Pt/Vulcan, but substantial improvement needed to meet 5000 cycle durability target at 80°C

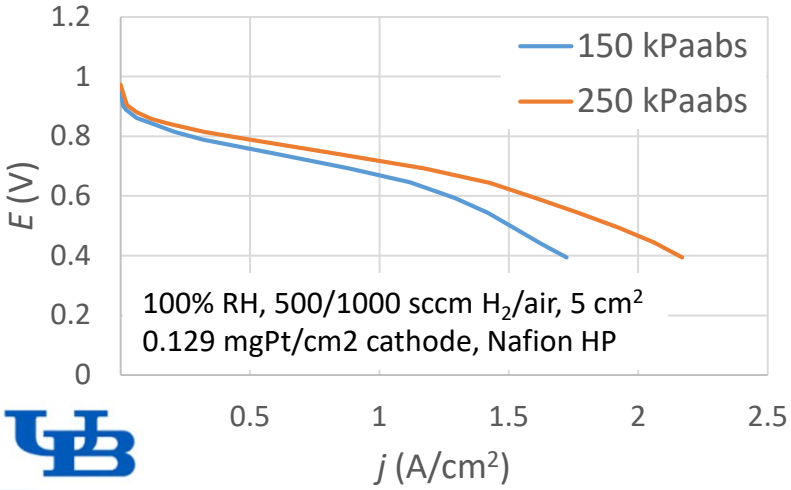
Accomplishments and Progress: PtCo-ZIF

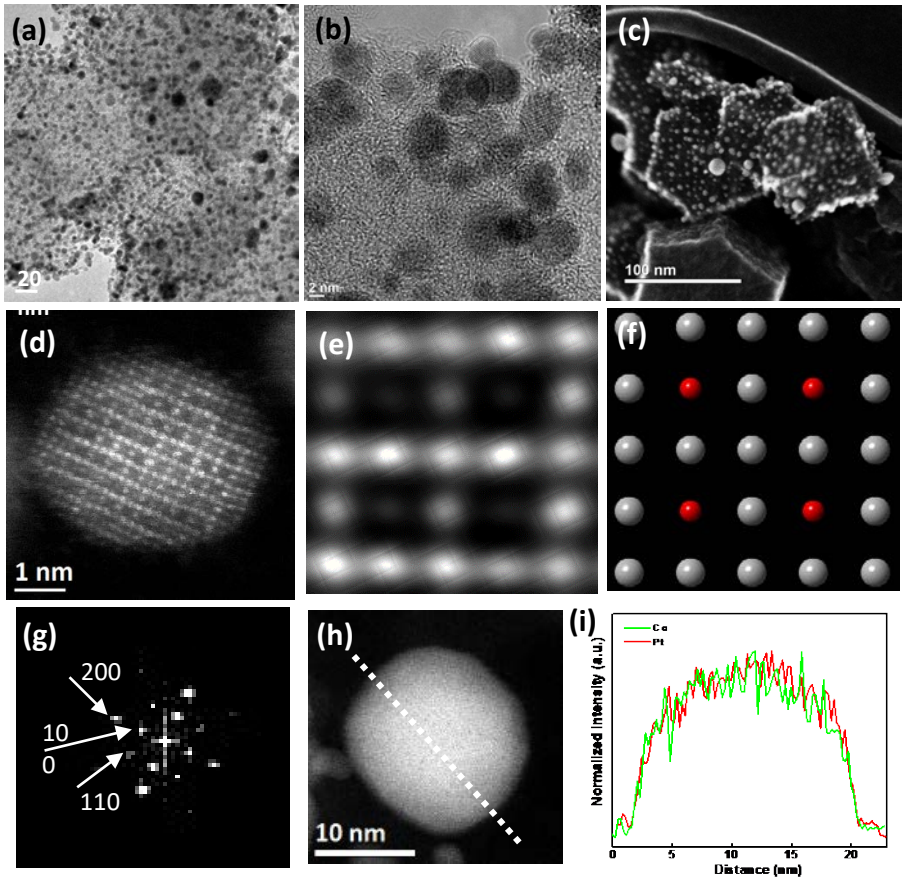


Preparation of ZIF derived carbon supports



Preparation of Pt-Co intermetallic catalysts





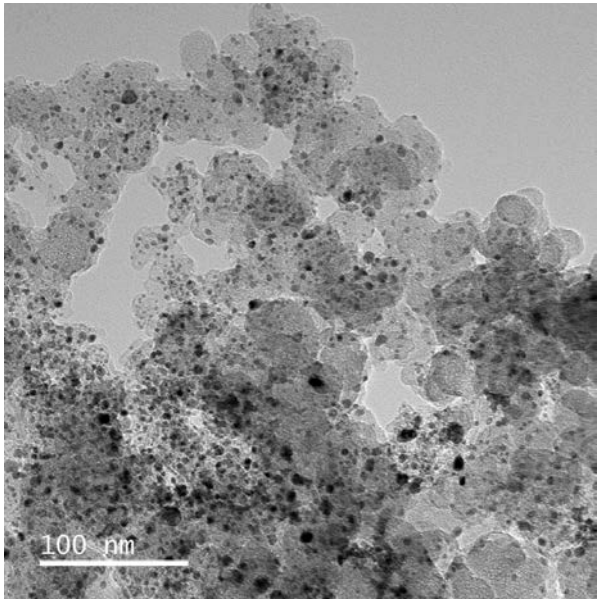
- Heat treatment of Co ZIF, followed by Pt impregnation and additional heat treatment, produces ordered PtCo on N-doped carbon
- High fuel cell performance in unoptimized MEA

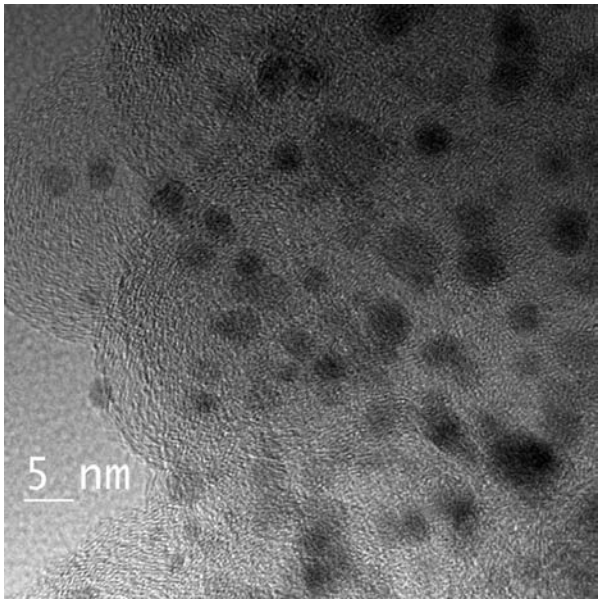
Accomplishments and Progress: PtCo-ZIF

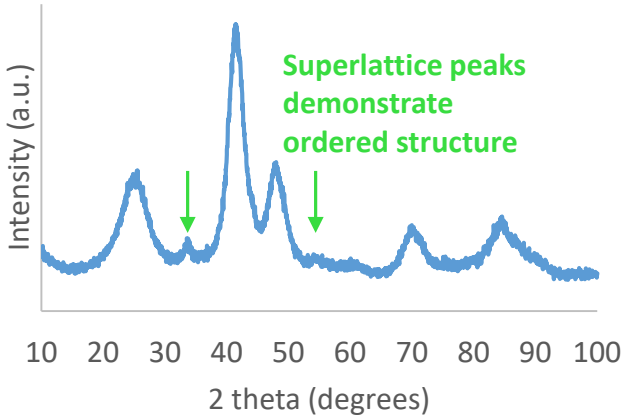
	Units	Measured	Target
Mass Activity	A/mgPGM	**0.47**	**0.44**
Mass Activity after Catalyst AST	A/mgPGM		**0.264**
Degradation at 0.8 A/cm^2 (Catalyst AST)	mV		**30**
Current Density at 0.8 V	A/cm^2	**0.26**	**0.3**
Power at 0.67 V, 150 kPa$_{abs}$	W/cm^2	**0.67**	**1**
Power at 0.67 V, 250 kPa$_{abs}$	W/cm^2	**0.86**	**1**
Cathode PGM Loading	mg/cm^2	**0.129**	**0.125**
Robustness, Cold			**0.7**
Robustness, Cold Transient			**0.7**
Robustness, Hot			**0.7**
ECSA	m^2/gPt	**28**	
ECSA after Catalyst AST	m^2/gPt		
Crystallite Size (XRD)	nm		
Crystallite Size after Catalyst AST	nm		
Particle Size (TEM)	nm	**6.0**	
Particle Size after Catalyst AST	nm		
Composition	%	**Pt55Co45**	
Composition after Catalyst AST	%		
Degree of Ordering (XRD)	%		
Ordering after Catalyst AST (XRD)	%		

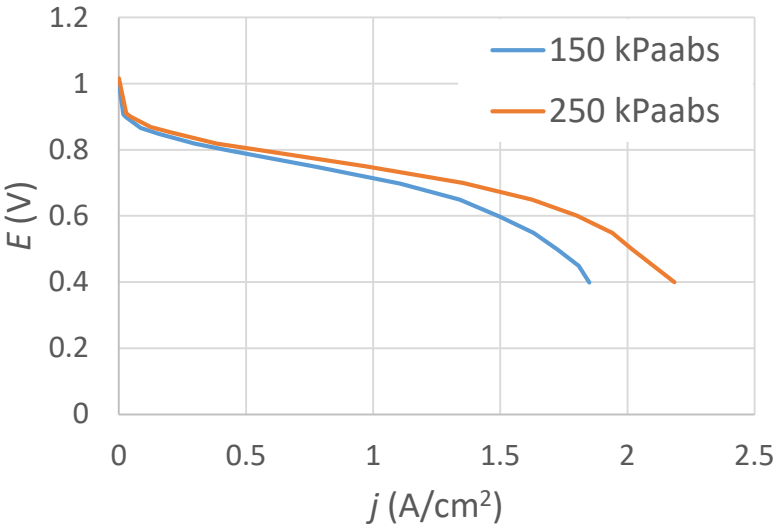
- Promising initial results with un-optimized MEA
- Testing still underway at time of slide submission

Accomplishments and Progress: LANL fct-CoPt









- New LANL fct-CoPt/Vulcan catalyst has small particles (mostly 3-5 nm) and high degree of ordering (80%), enabling excellent BOL performance in un-optimized MEA
  - **Mass activity = 0.71 A/mgPGM**
  - **Power density = 1.06 W/cm²@250 kPaabs)**
- First initial results shown here; durability testing and characterization still underway

Accomplishments and Progress: LANL fct-CoPt

	Units	Measured	Target
Mass Activity	A/mgPGM	**0.71**	**0.44**
Mass Activity after Catalyst AST	A/mgPGM		**0.264**
Degradation at 0.8 A/cm^2 (Catalyst AST)	mV		**30**
Current Density at 0.8 V	A/cm^2	**0.42**	**0.3**
Power at 0.67 V, 150 kPa$_{abs}$	W/cm^2	**0.83**	**1**
Power at 0.67 V, 250 kPa$_{abs}$	W/cm^2	**1.06**	**1**
Cathode PGM Loading	mg/cm^2	**0.103**	**0.125**
Robustness, Cold			**0.7**
Robustness, Cold Transient			**0.7**
Robustness, Hot			**0.7**
ECSA	m^2/gPt	**44**	
ECSA after Catalyst AST	m^2/gPt		
Crystallite Size (XRD)	nm	**4.4**	
Crystallite Size after Catalyst AST	nm		
Particle Size (TEM)	nm	**3.7**	
Particle Size after Catalyst AST	nm		
Composition	%	**Pt60Co40**	
Composition after Catalyst AST	%		
Degree of Ordering (XRD)	%	**80**	
Ordering after Catalyst AST (XRD)	%		

- Promising initial results with un-optimized MEA
- Mass activity = **0.71 A/mgPGM** using 15 min hold protocol (DOE/FCTT) used in this project; **0.88 A/mgPGM** using H_2/O_2 pol curve protocol
- Testing still underway at time of slide submission

Accomplishments and Progress: Status vs. Targets

	Catalyst Label	Co-Imp Pt/C #15c	Co-Imp Pt/C #6g	Co-Imp Pt/C #8e	Brown 110317_3	Penn CoPt_2017	Buffalo_043018#2	
	MEA Label	YP050418b	YP040418c	YP040418b/ YP041818	YP112017	YP041618b	YP050418a	
		LANL fct-CoPt	LANL fct-CoPt	LANL fct-CoPt	Brown fct-CoPt	Penn fct-CoPt	Buffalo PtCo-ZIF	Target
Mass Activity	A/mgPGM	**0.71**	**0.46**	**0.57**	**0.56**	**0.28**	**0.47**	**0.44**
Mass Activity after Catalyst AST	A/mgPGM		**0.21**	**0.3**	**0.45**	**0.16**		**0.264**
Degradation at 0.8 A/cm^2 (Catalyst AST)	mV		**27**	**41**	**69**	**62**		**30**
Current Density at 0.8 V	A/cm^2	**0.42**	**0.35**	**0.29**	**0.32**	**0.16**	**0.26**	**0.3**
Power at 0.67 V, 150 kPa$_{abs}$	W/cm^2	**0.83**	**0.75**	**0.69**	**0.58**	**0.41**	**0.67**	**1**
Power at 0.67 V, 250 kPa$_{abs}$	W/cm^2	**1.06**			**0.73**		**0.86**	**1**
Cathode PGM Loading	mg/cm^2	**0.103**	**0.113**	**0.096**	**0.105**	**0.089**	**0.129**	**0.125**
Robustness, Cold				**1.1**	**0.64**			**0.7**
Robustness, Cold Transient				**1.05**	**0.68**			**0.7**
Robustness, Hot				**0.25**	**0.19**			**0.7**
ECSA	m^2/gPt	**44**	**35**	**41**	**26**	**28**	**28**	
ECSA after Catalyst AST	m^2/gPt		**21**	**23**	**23**	**12**		
Crystallite Size (XRD)	nm	**4.4**	**11.3**	**5.6**	**7.8**			
Crystallite Size after Catalyst AST	nm				**9.6**			
Particle Size (TEM)	nm	**3.7**	**9.0**	**4.0**	**8.9**		**6.0**	
Particle Size after Catalyst AST	nm				**8.7**			
Composition	%	**Pt60Co40**	**Pt53Co47**	**Pt55Co45**	**Pt61Co39**	**Pt39Co61**	**Pt55Co45**	
Composition after Catalyst AST	%		**Pt67Co33**	**Pt74Co26**	**Pt71Co29**			
Degree of Ordering (XRD)	%	**80**	**91**	**86**	**85**			
Degree of Ordering after Catalyst AST	%			**71**	**77**			

Response to 2017 Reviewer Comments (1)

Comment: Reporting on mass activities of electrocatalysts in MEAs is likely to be a problem, and no clear benchmarks exist in this regime (other than the 2005 Gasteiger paper). Key issues are catalyst and ionomer dispersion in the ink method of making the catalyst coated membrane, the gas diffusion media, and the cell compression, plus the usual parameters of relative humidity and backpressure.

Response: FC-PAD has established benchmarks using commercial catalysts. All MEA testing reported here uses MEAs made using standard techniques:

- **Water/n-propanol inks, with catalyst and ionomer dispersed by sonication, and deposited by ultrasonic spray**
- **I/C = 0.9 for high surface area carbon or 0.5 for Vulcan carbon**
- **GDLs are 29BC (SGL), compressed by 20-25%**
- **Testing used 5 cm^2 differential cells at 500/1000 sccm anode/cathode**
- **Target electrode loading 0.1 $mgPt/cm^2$ (some sample-to-sample variation as reported in the test results)**
- **All testing was performed at 150 kPa_{abs} and 100% RH unless noted otherwise**

Response to 2017 Reviewer Comments (2)

Comment: It should be made clear what all of the team members are doing, especially the various team members doing catalysts synthesis (i.e., what each of them does that is unique).

Response:

- **Brown is adapting solvothermal synthetic technique to improve control of size, composition, and ordering of fct-MPt (M = Co, Ni, Fe)**
- **Penn is developing alternative synthetic pathways based on novel metal precursors, microwave irradiation, and rapid thermal annealing**
- **LANL is developing alternative seed-mediated synthetic pathways, as well as testing catalysts provided by Brown and Penn**
- **Buffalo is producing novel supports**
- **EWII is validating materials and scaling up MEAs**

Comment: It is not clear why ordered nanocrystals should be more stable and active, as the materials will reach equilibrium while under use and presumably go to a disordered state.

Response: Ordered face-centered tetragonal alloys of Pt with transition metals such as Fe, Co, and Ni are thermodynamically more stable than disordered alloys. Testing results demonstrate that ordered structure remains intact even after 30K cycle AST.

Collaboration and Coordination

LANL

- Coordinate project
- Synthesize, characterize, and test catalysts
- Produce and test MEAs

Brown

- Solvothermal catalyst synthesis
- Characterize catalysts and supply to partners
- Provide theory-based design principles

Buffalo

- Synthesize and characterize supports; supply to partners

Penn

- Alternative catalyst synthesis based on microwave and rapid thermal annealing
- Characterize catalysts and supply to partners

EWII

- Scale up MEA production
- Catalyst/MEA validation

Other collaborators in FY18:

- **ANL** (Synchrotron X-Ray studies)
- **ORNL** (TEM, STEM)
- **BNL** (TEM, STEM)

Remaining Challenges and Barriers

- Scale up synthesis to multi-gram batches using cost-effective chemistry
- Improve durability of N-doped supports
- Incorporate N-doped graphitized supports into high-performance electrodes
- Develop optimized electrode structures with effective transport properties to enable consistent achievement of >1 W/cm^2 operation

Proposed Future Work

FY18:

- Prepare fct-MPt catalysts on N-doped graphitized supports and compare with baseline Pt/C and fct-MPt/C
- Perform computational studies to guide synthetic work and interpret experimental findings
- Scale up successful synthetic chemistries to gram scale
- Downselect promising catalysts for MEA optimization

FY19:

- Scale up MEA testing from 5 cm^2 to 50 cm^2
- Optimize electrode structures for high-current operation (> 1 W/cm^2)
- Continue catalyst and support development using feedback from MEA testing and characterization

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

Tech Transfer Activities

- Engaged a US catalyst supplier (Pajarito Powder LLC) to discuss possible licensing and scale-up activities

Summary

Objective: Design active and durable ORR catalysts based on fully-ordered intermetallic alloys on highly graphitized nitrogen-doped carbon supports, and demonstrate in high-performance, durable MEAs.

Relevance: Project directly addresses cost, durability, and performance through key DOE targets:

- MEA mass activity > 0.44 A/mg_{PGM} @ 0.9 ViR-free
- <40% MEA mass activity loss after catalyst and support ASTs
- <30 mV loss at 0.8 A/cm^2 and 1.5 A/cm^2 after catalyst and support ASTs
- PGM total loading < 0.125 mg/cm^2
- Power density > 1 W/cm^2

Approach: Fully-ordered intermetallic Pt alloy catalysts supported on highly-graphitized N-doped carbon supports are being developed and tested in MEAs. Synthetic work is guided by computational ORR kinetic studies. Feedback from MEA testing and from characterization studies guides each round of synthetic development.

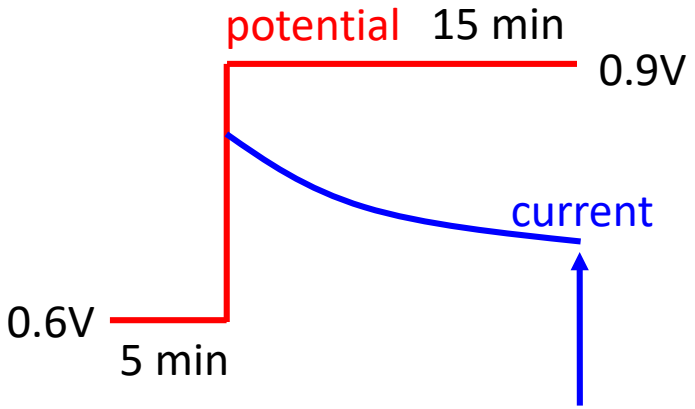
Accomplishments: Intermetallic fct-CoPt catalyst meets mass activity and durability targets. New N-doped supports also show promising initial durability, but not yet stable for 5000 cycles.

Collaborations: Strong team consists of a national lab with extensive catalyst synthesis, MEA testing, and characterization capabilities, three universities with excellent synthetic and computational capabilities, and an industrial partner with experience in MEA validation and scale-up. External collaborators provide additional characterization capabilities.

Technical Backup Slides

MEA Testing Protocols

Mass Activity: 15 min hold at 0.9 V

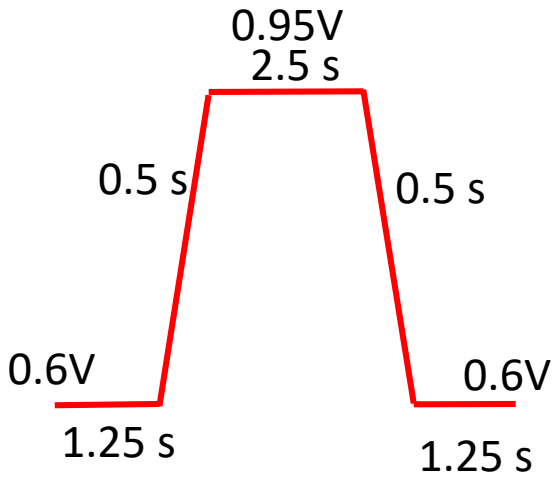


Mass activity measured during last 1 min of 15 min hold

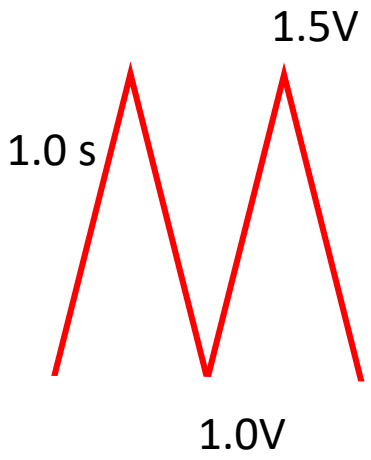
H_2/O_2, 500/1000 sccm; 80°C; 100% RH; 150 kPa_{abs}; cathode: 0.1mg_{Pt}/cm^2 ; anode: 0.1mg_{Pt}/cm^2

This protocol provides a conservative estimate of mass activity, approximately 20% lower than the common H_2/O_2 pol curve method used in other labs.

Catalyst AST: square wave between 0.6 and 0.95 V with 0.5 s rise time



Support AST: triangle wave between 1.0 and 1.5 V at 500 mV/s

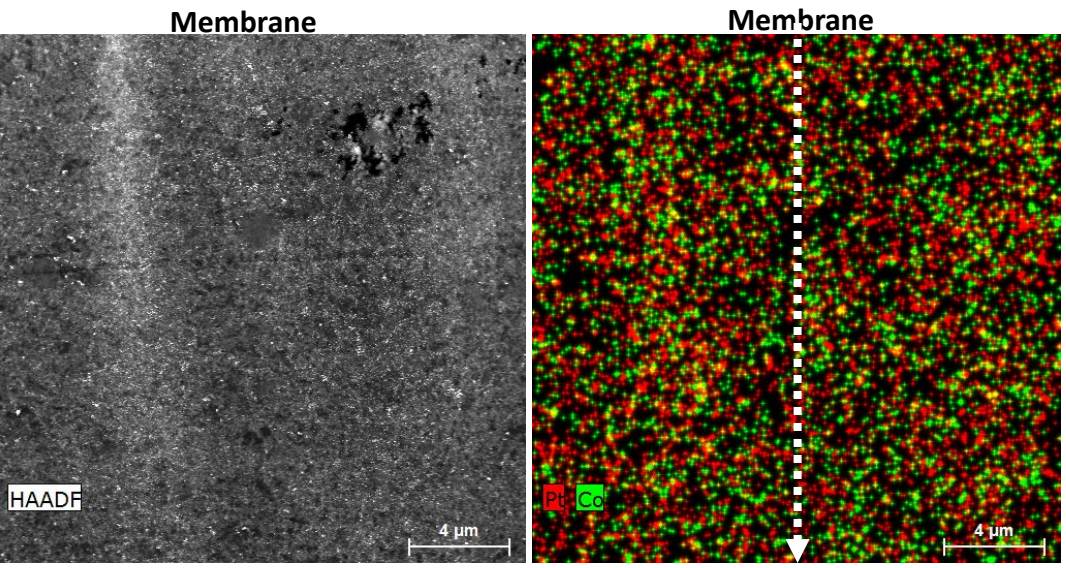


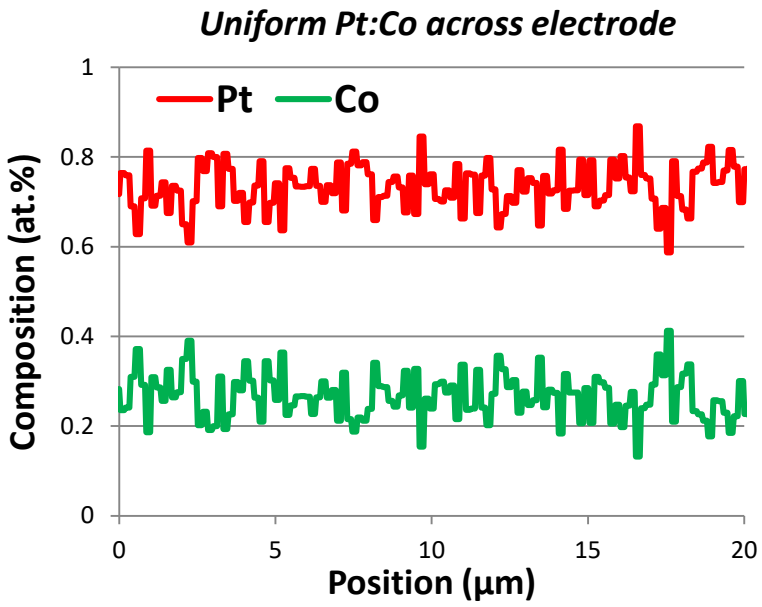
H_2/N_2, 200/200 sccm; 80°C; 100% RH; 150 kPa_{abs}; cathode: 0.1mg_{Pt}/cm^2 ; anode: 0.1mg_{Pt}/cm^2

Brown fct-CoPt/Pt MEA EDX Characterization

Overall Composition: Fresh Powder: $Pt_{62}Co_{38}$ After AST: $Pt_{70}Co_{30}$

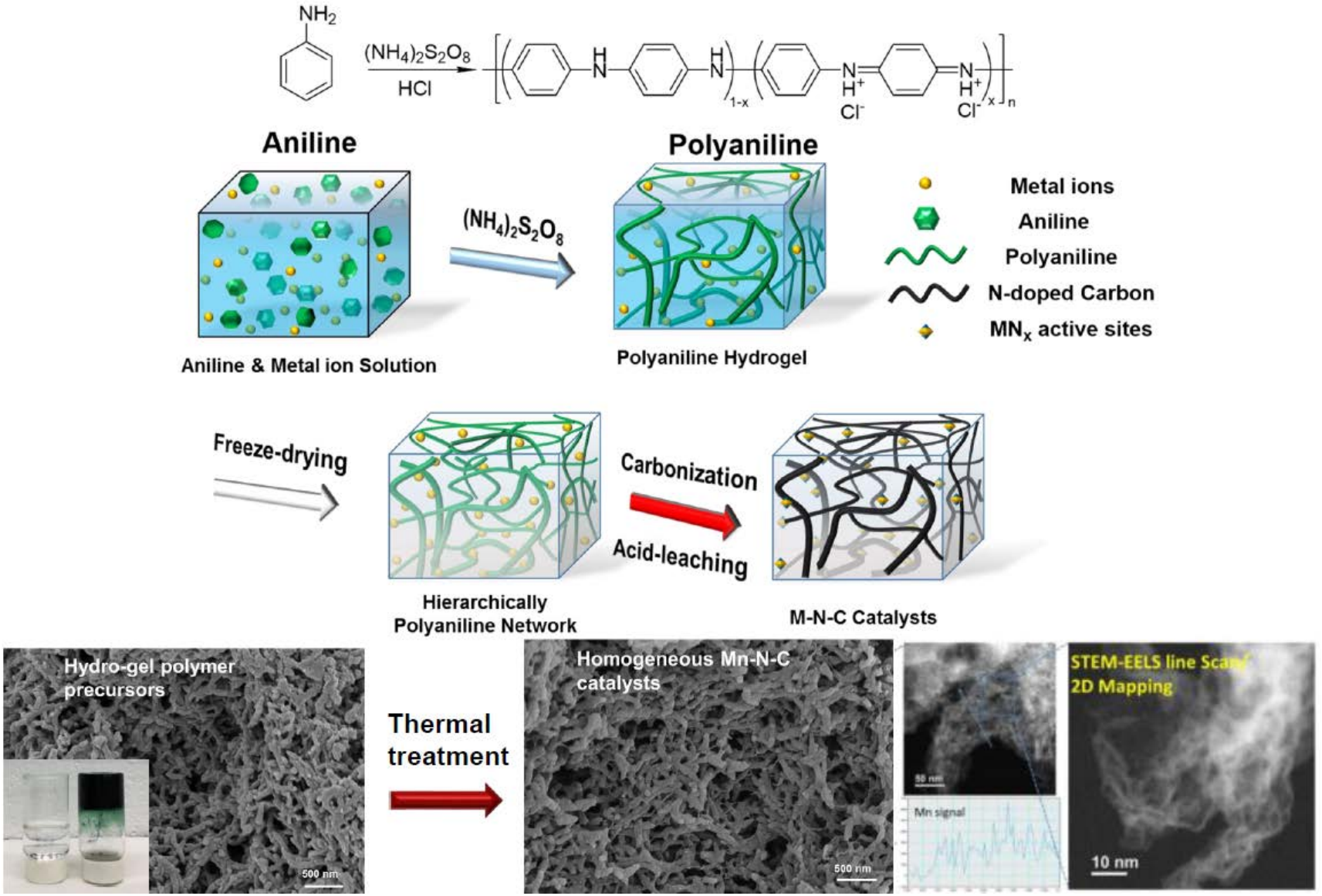
Tested MEA: YP112017





Tested MEA: YP112017

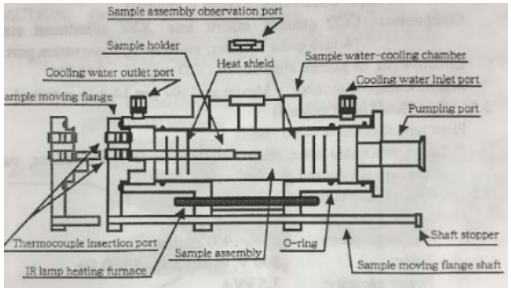
Hydrogel Supports (Mn precursor)

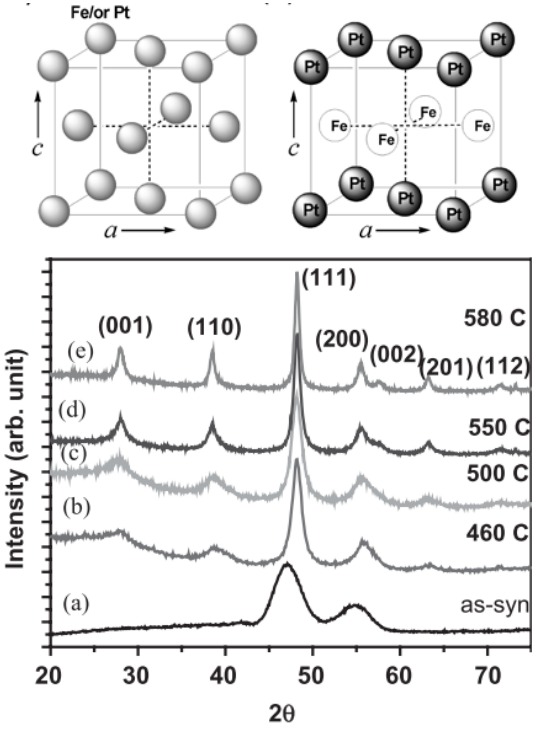


Methods for FCC to FCT: RTA

Desktop rapid thermal annealing (RTA) furnace

- **Temp range:** RT to 1200°C
- **Max. heating rate:** 50°C/sec in vacuum
- **Heating environment:** vacuum, air, Ar and H_2/He

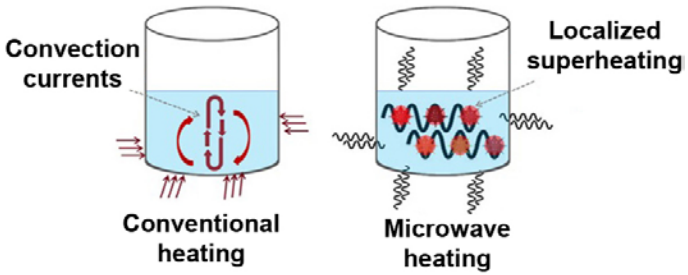


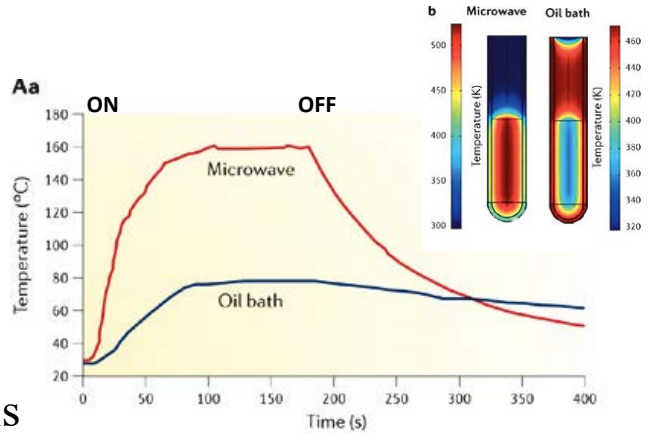


S. Sun et al., *J. Am. Chem. Soc.* **2010**, *132*, 4996–4997
S. Sun and C.B. Murray *et al.*, *J. Magn. Magn. Mater.* **2003**, *266*, 227-232

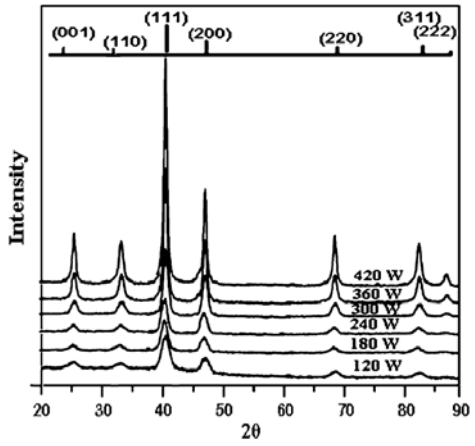
Methods for FCC to FCT: microwave

- Microwave heating: localized





- Microwave-assisted direct synthesis
  - Different power levels



Microwave exposure time 25 min		
Microwave power (W)	Fe–Pt particle size (nm) from	
	XRD	TEM
120	7	
180	8	
240	10	7
300	13	10
360	15	15
420	17	20

Angew. Chem. Int. Ed. **2004**, *43*, 6250
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