

High-Throughput/Combinatorial Optimization of Low-Pt PEMFC Cathode Performance

High-Throughput Synthesis, ORR Activity Modeling, and Testing of non-PGM PEMFC Cathode Catalysts

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Nancy Kariuki, and Theodore Krause

Argonne National Laboratory

Annual Merit Review
Arlington, Virginia

Project ID# FC114

June 8-12, 2015

Project Overview

Timeline

- Project Start Date: August, 2014
- Project End Date: September, 2015
- Percentage complete: 64%

Budget

- DOE Funding: \$775K (to ANL)
- Funding spent as of March 31: \$263K (38%)
- Total DOE Project Value: \$1,050K (contingent on go/no-go)
- Cost Share Percentage: 0%

Barriers

- A. Electrode Performance
- B. Cost
- C. Durability

Partners and Project Lead

- **Oak Ridge National Laboratory (Low-Pt portion)**
 - Dave Cullen
- **Los Alamos National Laboratory (non-PGM portion)**
 - Piotr Zelenay, Ted Holby, and Samrat Choudhury
- **Project lead: Argonne National Laboratory**
 - Debbie Myers, Rajesh Ahluwalia, Firat Centibas, Magali Ferrandon, Xiaoping Wang, Sheldon Lee, Voja Stamenkovic, and Dongguo Li



Relevance

■ Project Objectives

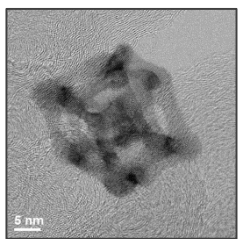
- Overall: develop tools for the rapid synthesis, fabrication, characterization, activity and durability screening, and performance optimization and testing of fuel cell cathode catalysts and catalyst layers in membrane-electrode assemblies (MEAs)
- Low-Pt portion: realize the oxygen reduction reaction activity benefits of the Argonne nano-segregated Pt cathode electrocatalysts (FC-008), demonstrated in aqueous tests, in membrane-electrode assemblies at high current densities and on air
- Non-PGM portion: accelerate the development of high activity and stable non-PGM cathode catalysts for polymer electrolyte fuel cells

Barrier	2020 Target
A. Electrode Performance	PGM: $>250 \text{ mW/cm}^2$ and $>300 \text{ mA/cm}^2$ at 800 mV, $>1000 \text{ mW/cm}^2$ at rated power. Non-PGM: $>200 \text{ mA/cm}^2$ at $0.8 V_{iR\text{-free}}$
B. Cost	$<\$40 \text{ kW}_e$ system, $\$14/\text{kW}_e$ MEA cost target, $<0.125 \text{ mg}_{\text{PGM}}/\text{cm}^2$
C. Durability	$<40\%$ loss of initial catalytic mass activity at 900 mV on O_2 ; $<30 \text{ mV}$ loss at 0.8 A/cm^2



Approach: High-Throughput/Combinatorial Optimization of Low-Pt PEMFC Cathode Performance

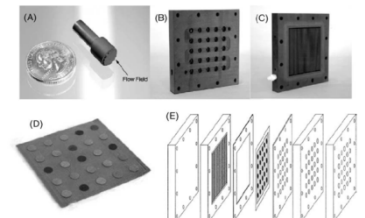
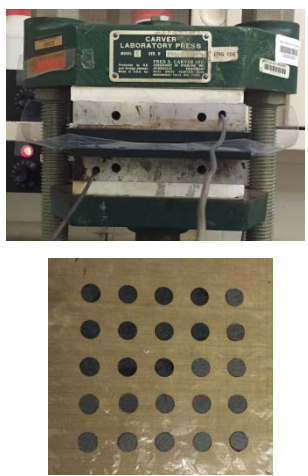
- Overall Approach: High-throughput fabrication, combinatorial testing, and transport modeling of cathode catalyst layers to maximize reactant (O₂ and H⁺) transport to catalyst sites
- Tasks:
 - Synthesis of Pt nano-segregated catalyst powders (Argonne-V. Stamenkovic)
 - High throughput/robotic synthesis of catalyst-ionomer-solvent inks of various ionomer to carbon ratio and solvent type
 - Analysis of microstructure of inks and dried inks via SEM and TEM for microstructure (Oak Ridge NL- D. Cullen)
 - High throughput/robotic deposition of inks on substrates (blanks or gas diffusion layers)
 - Combinatorial performance testing and characterization of twenty-five electrodes in a membrane-electrode assembly
 - High throughput analysis of data for sources of voltage losses and transport modeling (Argonne-R. Ahluwalia)



ANL/LBNL Pt₃Ni Nanoframes



FreeSlate High Throughput Catalyst Ink Synthesis and Deposition



Combinatorial Fuel Cell Test Fixture

NuVant Systems, Inc.
Nuvant.com

Low-Pt-related FY'15 Milestones and Status

Milestone Name/Description	End Date	Type	Status
Description of equipment and methods for the FreeSlate apparatus to allow: synthesis of PGM catalyst-ionomer-solvent inks, measurement of the carbon agglomerate size distribution in the inks, and deposition of inks on a substrate	10/31/2014	Quarterly Progress Measure (Regular)	Met
Fabricate a 25 electrode membrane-electrode assembly from a low-Pt nanosegregated cathode catalyst	1/30/2015	Quarterly Progress Measure (Regular)	Met
Measure the fuel cell performance of 25 PGM catalyst-based cathode layers in parallel	4/30/2015	Quarterly Progress Measure (Regular)	Met – electrode performance was poor
Develop ionomer-solvent-low-Pt nano-segregated catalyst combination which results in a button fuel cell performance of $>0.24 \text{ A/cm}^2$ at 0.8 V with a cathode catalyst PGM loading of 0.1 mg-Pt/cm^2 , an improvement of at least 50% over the FCTO 2011 baseline status of 160 mA/cm^2	7/31/2015	Annual Milestone (Stretch)	

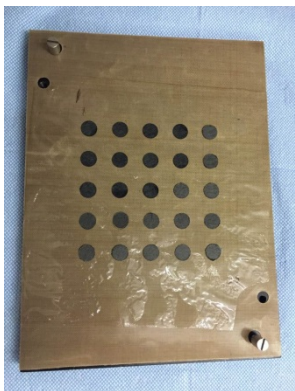


Accomplishments: Low-Pt Portion

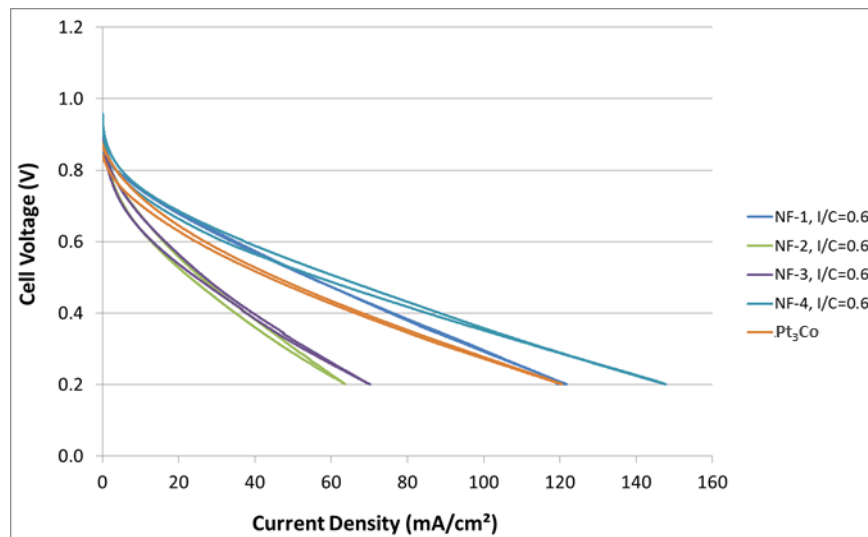
- **Devised a method for deposition of catalyst-ionomer-solvent inks on gas diffusion electrodes using robotic system:**
 - Inks are made by weighing and dispensing the catalyst powder, solvent, and ionomer solutions at varying ratios into vials using the robotic system and mixed using sonication and magnetic stir bars
 - Inks dispensed in circular spots directly onto a heated 1.27 cm² Sigracet® 25 BC gas diffusion layer (GDL) using the micropipette liquid dispensing unit of the robotic system to make a GDE and dried in vacuum oven
 - The GDL pieces were heated at temperatures from 60 to 90°C during the ink deposition.
 - Eleven of the electrodes contained PtNi nanoframe catalyst
 - Four with ionomer to carbon (I/C) weight ratio of 0.6
 - Four had an I/C ratio of 0.9
 - Three had an I/C ratio of 1.0
 - The deposition temperature was varied through a series of electrodes with the same I/C ratio
 - The other fourteen electrodes in the array MEA were PtNi, Pt₃Co, and Pt gas diffusion electrodes (GDEs) with an I/C ratio of 0.8.
 - Twenty-five circular GDE's placed into the MEA template, and hot-pressed, by hand, onto a single Nafion® 211 membrane/anode assembly.



Accomplishment: Combinatorial fuel cell fabrication and testing



$T=60^{\circ}\text{C}$, H_2/Air , 100 kPa(abs.), Cathode PGM loading: $0.1\text{ mg-Pt}/\text{cm}^2$ for Pt_3Ni nanoframe (NF) and $0.53\text{ mg-Pt}/\text{cm}^2$ for Pt_3Co



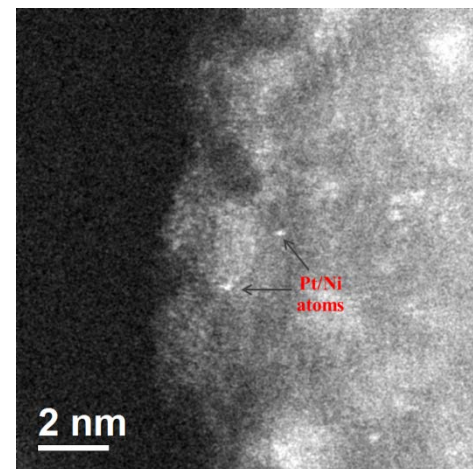
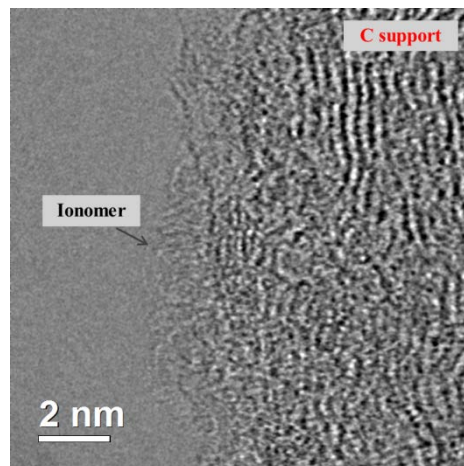
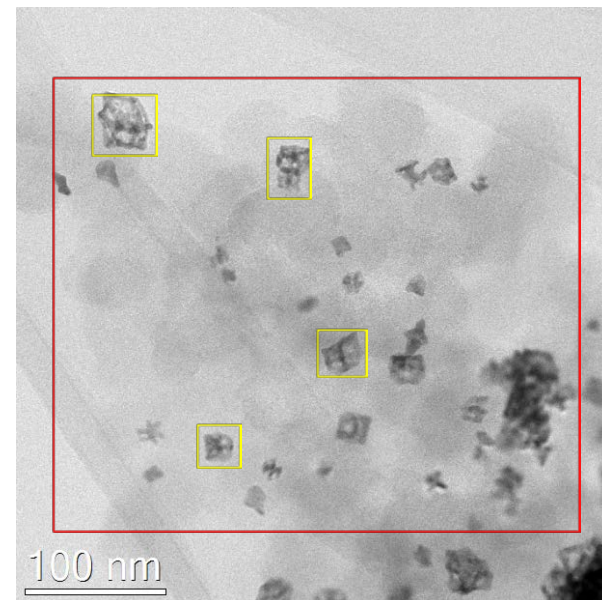
Combinatorial Fuel Cell
Test Fixture and Arraystat
Developed by NuVant Systems, Inc. under DOE SBIR
2005-0017-DE-F602-03ER8374

- **Accomplishment: Fabricated and tested an MEA with twenty-five individually addressable cathodes**
- Performance of this cell, using gas diffusion electrodes, was low, but did show trends in performance with different nanoframe electrocatalyst ink deposition temperatures

Accomplishment: STEM-EDS Characterization of Pt₃Ni-Ionomer-Solvent Inks - I/C Series

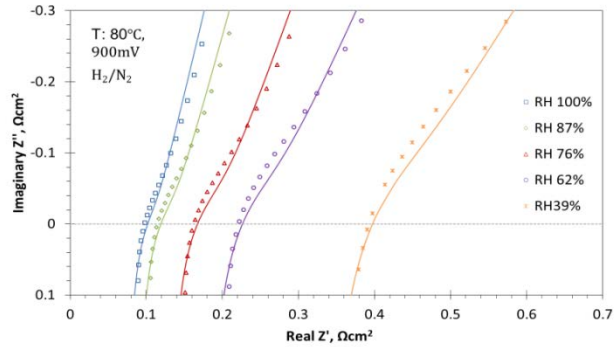
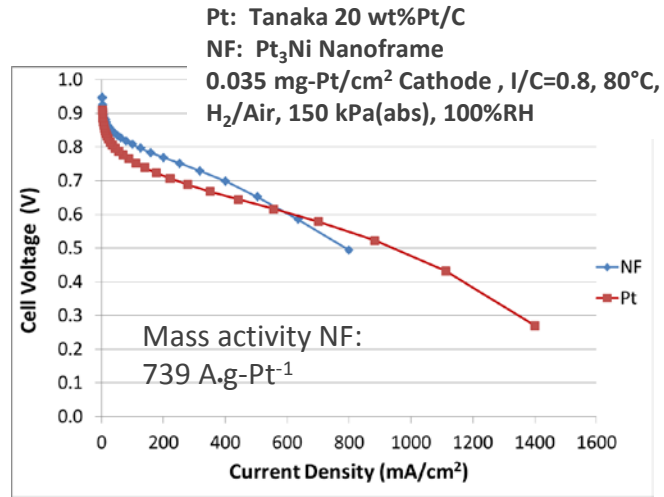
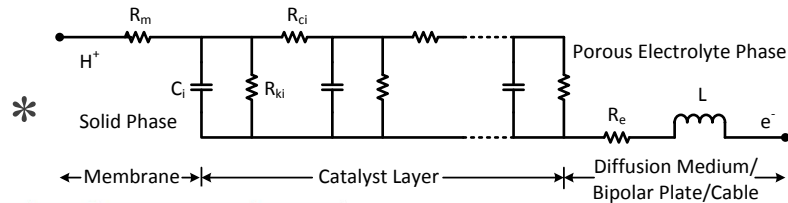
- Pt₃Ni/C-Ionomer-Solvent Inks with I/C of 0.6 and 1.0 were analyzed by STEM-EDS
- EDS quantification of the two samples yielded nearly identical results
 - Frames Only (yellow): Pt₇₄Ni₂₆
 - Frames + support (red): Pt₆₉Ni₃₁
 - Suggest some excess Ni in support, but overall very little Ni leaching, with ionomer loading having no measurable impact on extent of leaching
- Ni or Pt atoms observed in ionomer
- Next step: Ionomer film thickness and carbon agglomerate analysis on MEA for input into electrode structural model

Highlight: As-prepared composition of Pt₃Ni nanoframes stable in ionomer inks (i.e., little or no Ni²⁺ leaching)

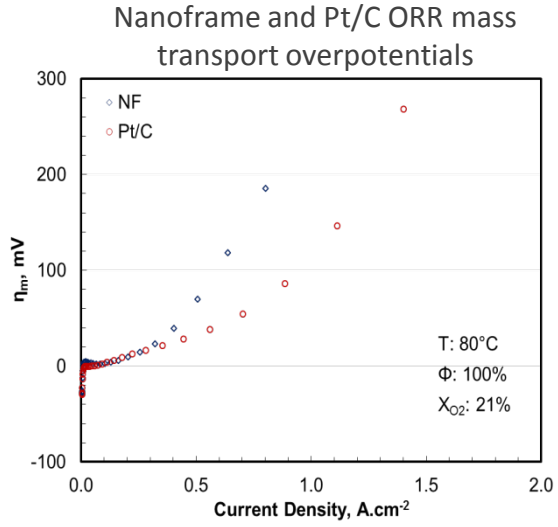
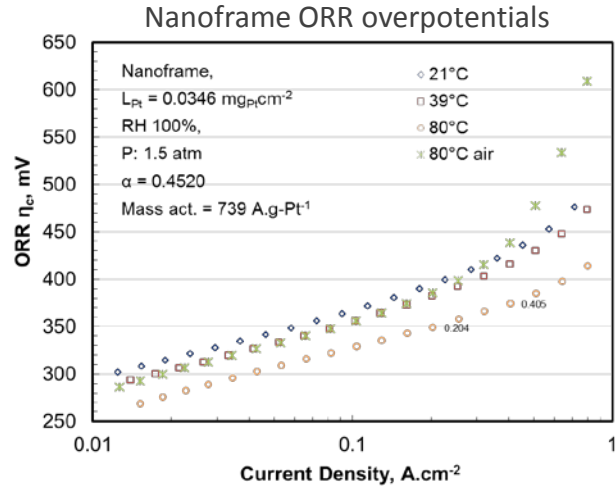
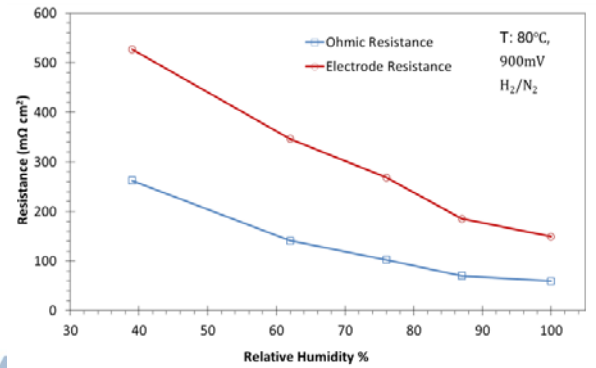


Accomplishments: Analysis of data for sources of voltage losses and transport modeling

- Developed a model which rapidly analyzes impedance and performance data providing mass activity, mass transport and kinetic overpotentials, and electrode resistances to aid design of improved electrodes

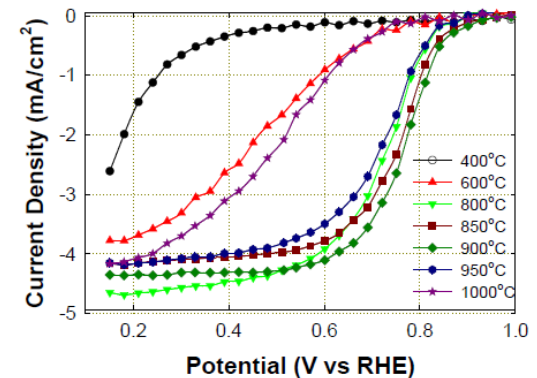
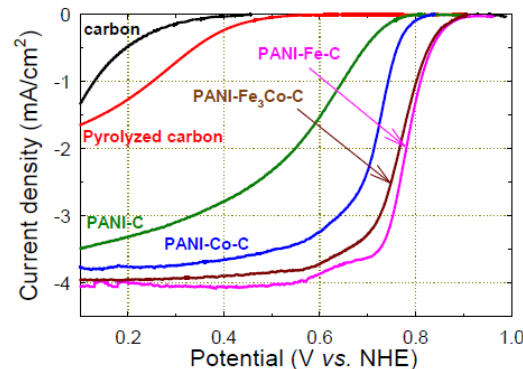
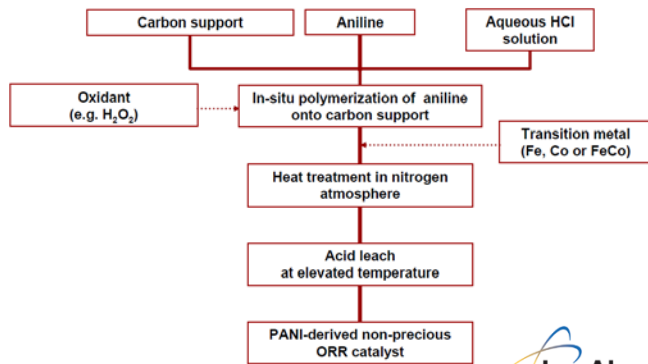


$$E = E_N - iR_{\Omega} - (iR_{\Omega}^c + \eta_c^c + \eta_m^c) - (iR_{\Omega}^a + \eta_c^a + \eta_m^a)$$



Approach: High-Throughput Synthesis, ORR Activity Modeling, and Testing of non-PGM PEMFC Cathode Catalysts

- Overall Approach: Develop and utilize capability for high-throughput synthesis and oxygen reduction reaction (ORR) activity testing of iron, cobalt, polyaniline-based catalysts pioneered by Los Alamos National Laboratory (P. Zelenay, FC-003, “Advanced Cathode Catalysts”, 2007-2011)
- Tasks:
 - Rapid throughput synthesis of catalyst precursor composites (Argonne)
 - Rapid throughput pyrolysis of catalyst precursors (Argonne)
 - Rapid throughput physical and chemical characterization of catalysts (Argonne)
 - High throughput screening of ORR and ORR activity stability of catalysts in a hydrodynamic aqueous cell, similar to rotating-disk electrode (Argonne)
 - Verification of validity of channel flow double electrode cell performance using RDE (P. Zelenay, Los Alamos)
 - Develop capability for high-throughput screening of possible Fe-N-C active site moieties for activity and stability using density functional theory (DFT)-based descriptors (T. Holby, Los Alamos)
 - Scale-up synthesis of the best transition metal-PANI-C catalyst arising from the high-throughput experiments. Fabricate and test membrane-electrode assemblies (P. Zelenay, Los Alamos)



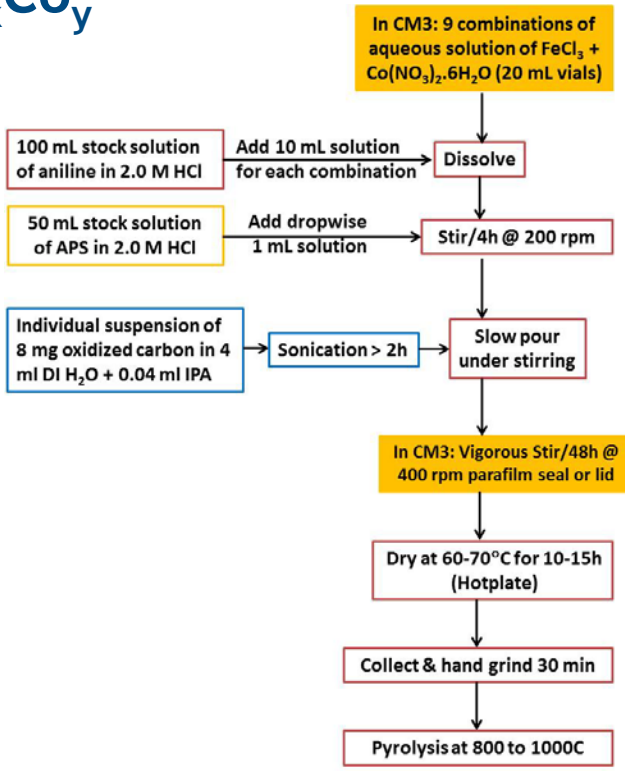
Non-PGM Milestones and Status

Milestone Name/Description	End Date	Status
Fabricate 45 Fe-Co-PANI-C catalysts variants using robotic/rapid throughput system	10/31/2014	Met
Pyrolyze and characterize 45 Fe-Co-PANI-C catalyst variants	1/30/2015	Met
Determine ORR activity of 45 Fe-Co-PANI-C catalyst variants using newly developed high throughput apparatus	4/30/2015	Met with Eiwa Cell
<p>(1) Verify the performance of the multi-channel hydrodynamic cell by comparison with rotating-disk electrode results with a target half-wave potential agreement of 10 mV. (2) Transition metal-PANI-C catalyst composition and synthesis method to achieve a half-wave potential in hydrodynamic measurements exceeding the current status of 0.8 V with a catalyst loading of 0.6 mg/cm². Stretch. 7/31/2015</p>		
<p>Transition metal-PANI-C catalyst from high-throughput synthesis task achieving a half-wave potential in hydrodynamic measurements exceeding the current status of 0.8 V with a catalyst loading of 0.6 mg/cm². Go/No-Go 7/31/2015</p>		
<p>Scale-up synthesis of the best transition metal-PANI-C catalyst arising from the high-throughput experiments and fabricate and test membrane-electrode assemblies to achieve a cell performance of >0.022 A/cm² at 0.9 V(iR-free), a 50% improvement of performance over current status of 0.015 A/cm². Stretch. Annual. 9/30/2015</p>		

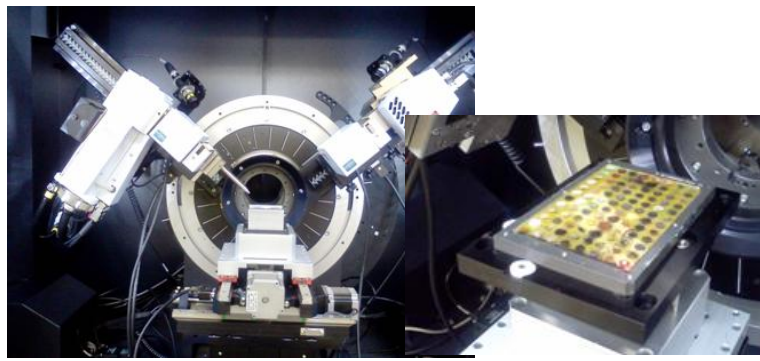
Accomplishment: Developed methods for high-throughput synthesis and characterization of PANI-Fe_xCo_y

- Synthesis of the powder catalyst precursor was executed in three batches on a robotic platform (CM3, FreeSlate Inc.)
- Eleven samples, nine PANI-Fe-Co compositions and two replicates, with the same total amount of metal (Fe+Co=0.0012 moles) were synthesized
- High-throughput X-ray diffraction (D8, Bruker AXS) was performed after pyrolysis from 800°C to 1000°C

Robotic platform



HT X-ray Powder Diffraction



High-throughput (HT) non-PGM catalyst synthesis and phase composition characterization (continued)

Nine Fe to Co ratios

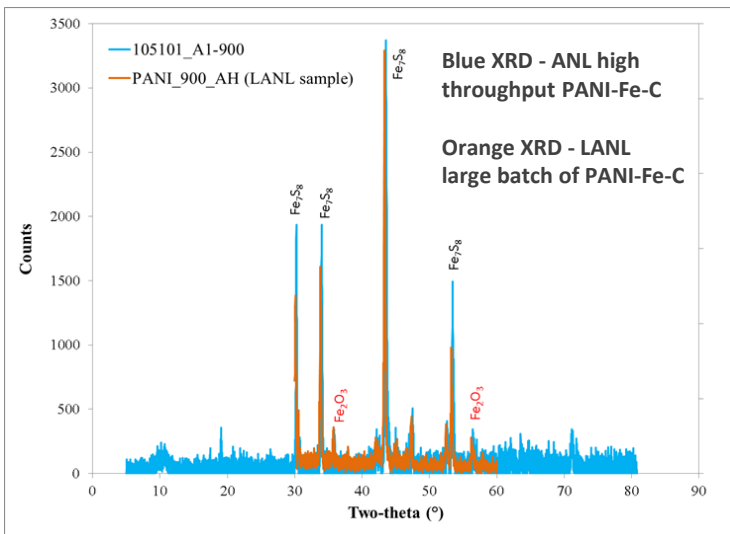
Moles Fe in ratio	Moles Co in ratio
1	0
4	1
3	1
2	1
1	1
1	2
1	3
1	4
0	1

Forty-five variants

**Five pyrolysis temperatures:
800, 850, 900, 950, 1000°C**



(Left) Nine metal salt precursor solutions with iron to cobalt ratios shown in Table (Cobalt only on left and Iron only on right).
(Right) The iron only solution during polymerization of the polyaniline.



Highlight: Argonne's HT process produces PANI-Fe-C powder with same phase composition as large single batch synthesized at LANL

See back-up slides for XRD's of other PANI-Fe,Co-C compositions

Accomplishment: Developed methods for HT acid treatment and washing of non-PGM catalyst precursors

Acid treatment at elevated temperature



Centrifuging (replaces filtering)

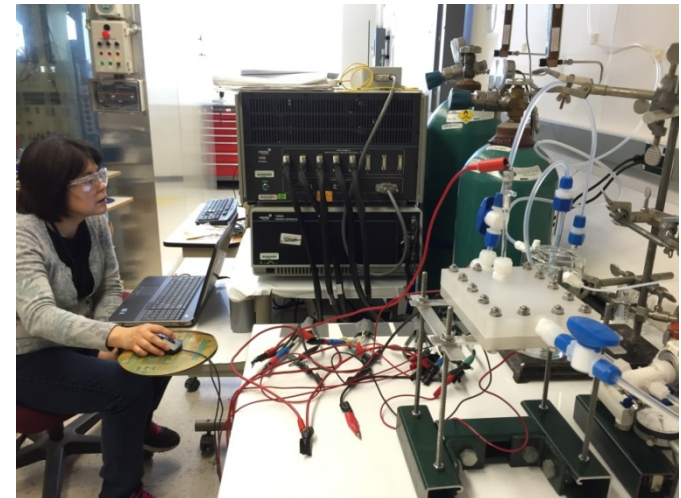
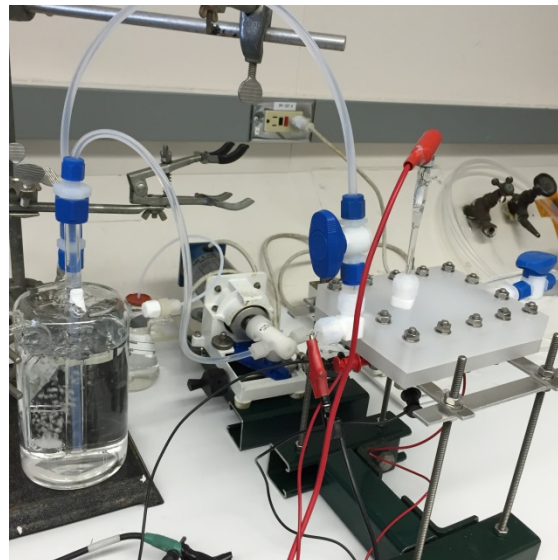
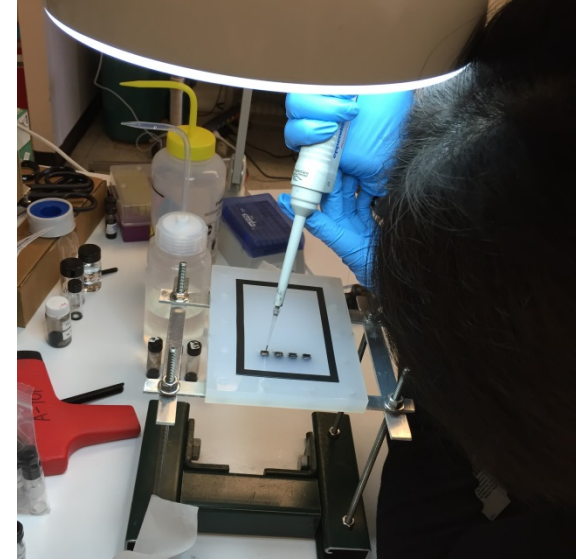
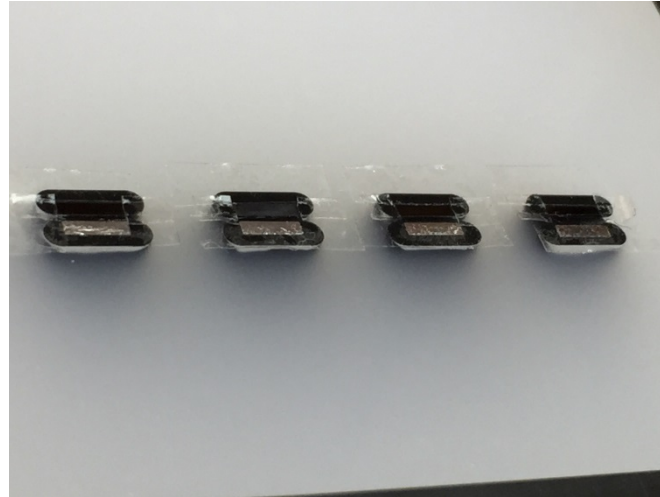
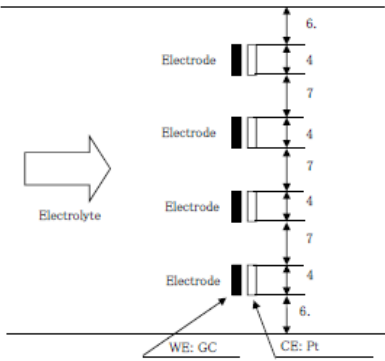


- Acid treatment and washing steps can be high throughput
- Pyrolysis step can be high throughput
- Slow step in catalyst processing is catalyst grinding
 - This step can be mechanized as well

Combinatorial ORR Activity Screening: Eiwa* Four Channel Flow Double Electrode Cell

Electrolyte flow channel: 0.5mmX50mm

Electrode: Working Electrode/Glassy Carbon, Collecting Electrode/ Pt

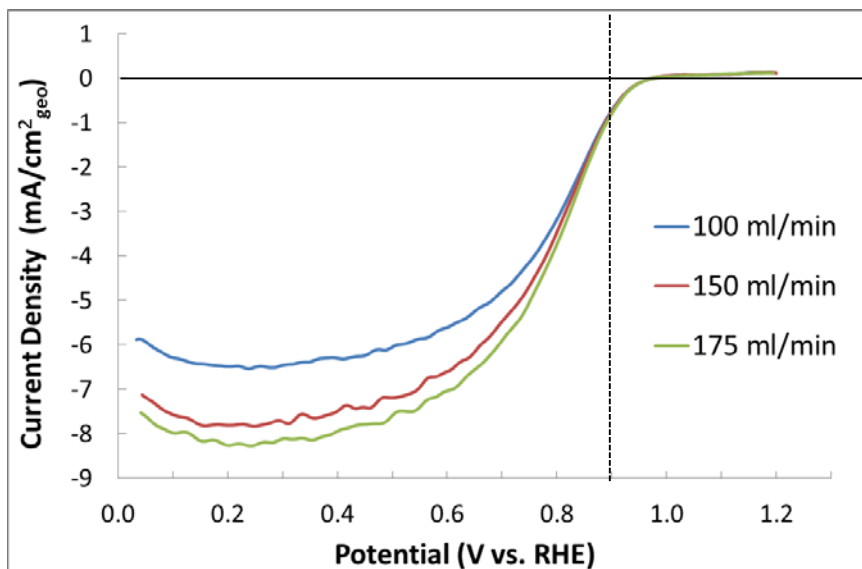


*H. Yano, J.M. Song, H. Uchida, M. Watanabe, J. Phys. Chem. C 112 (2008) 8372.



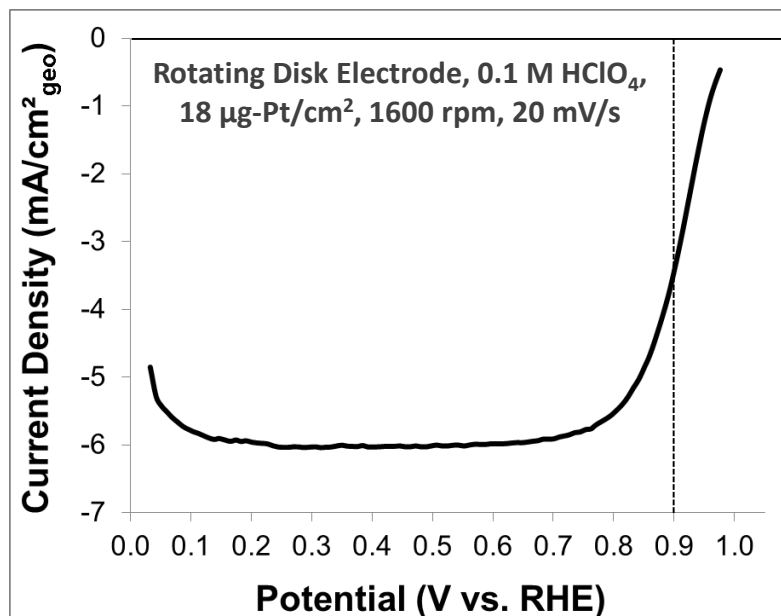
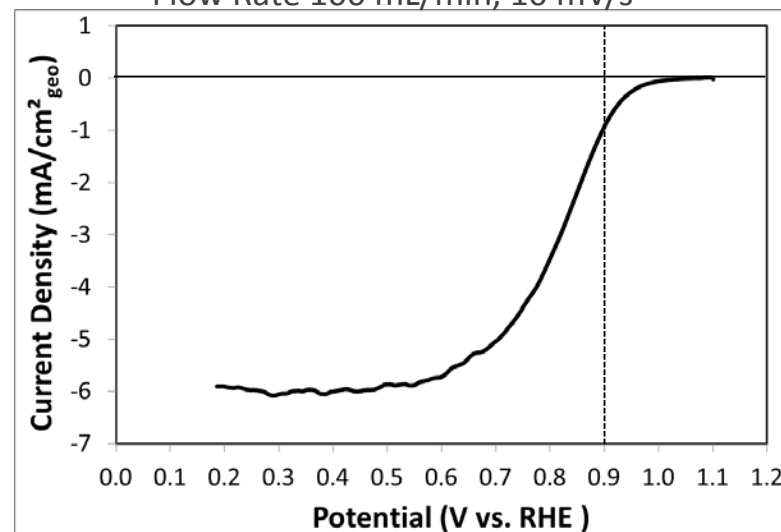
Accomplishment: Verified Eiwa CFDE for ORR using Tanaka 46 wt% Pt/C

Eiwa Channel Flow Double Electrode
0.1 M HClO₄, 18 μg-Pt/cm², no iR comp.,
Flow Rate 100-175 mL/min, 10 mV/s

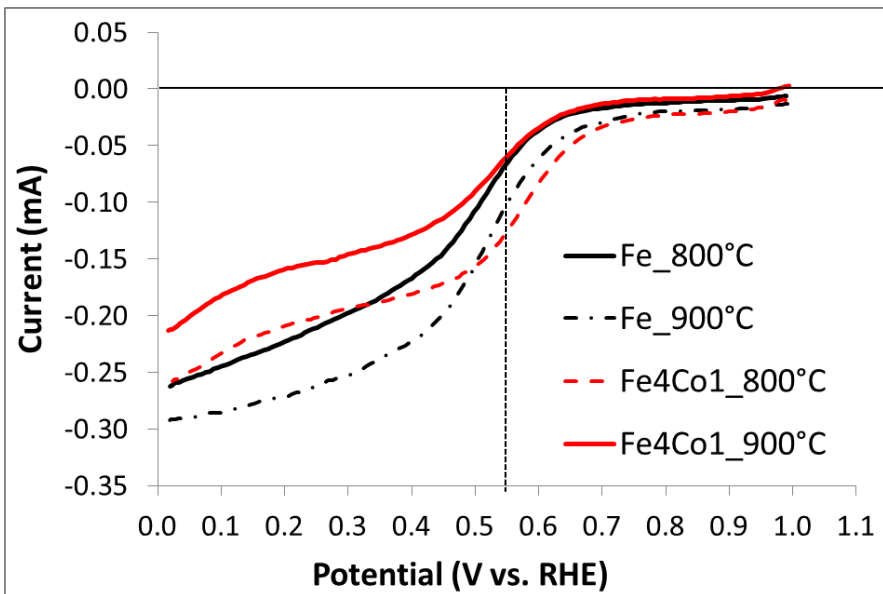
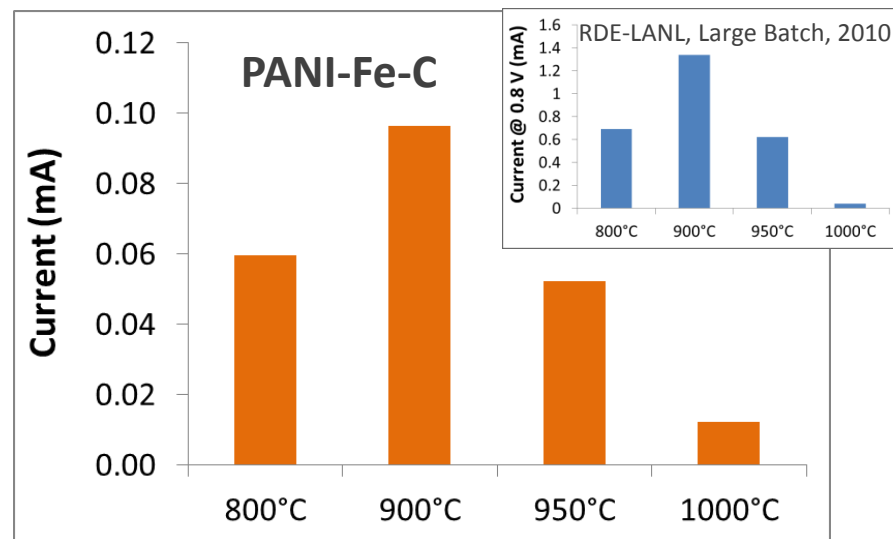
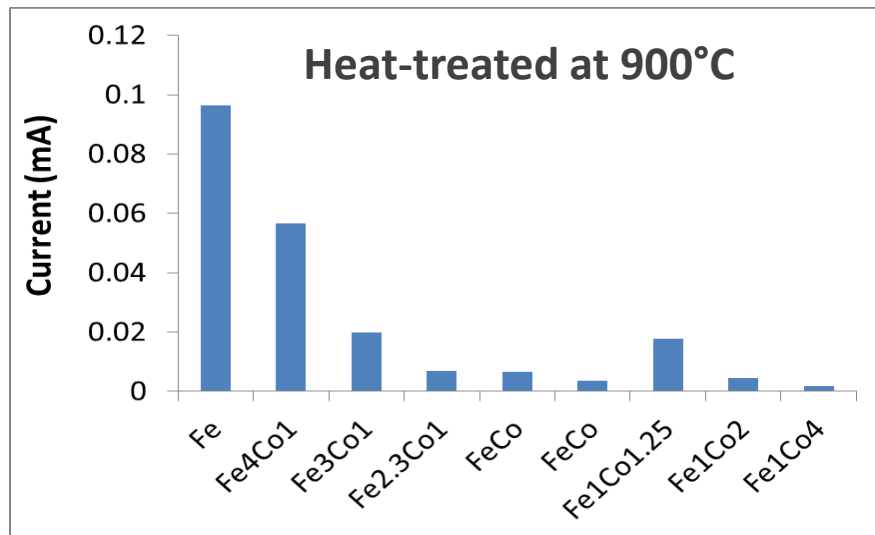


- Achieved diffusion-limited current and reproducible kinetic current for Tanaka Pt/C using Eiwa CFDE
- Identified several issues with Eiwa CFDE cell design, including **high** iR drop between working and reference electrodes
 - Partially addressed with iR compensation
 - Performance lags that of RDE results
 - Issues are being addressed with ANL CFDE cell design

Eiwa Channel Flow Double Electrode
0.5 M H₂SO₄, 24 μg-Pt/cm², iR comp.
Flow Rate 100 mL/min, 10 mV/s



Accomplishment: Achieved agreement of ORR activity trends for HT PANI-Fe,Co-C using CFDE with large batch RDE data



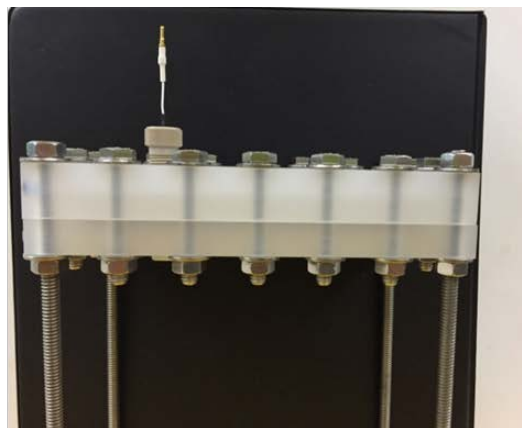
- **Highlight:** Achieved excellent agreement of activity trends, determined using CFDE, for HT small batch samples prepared in this project with trends observed using RDE for large batches prepared by LANL (Zelenay, FC-003)
- **Highlight:** Identified a new PANI-Fe,Co-C composition with higher ORR activity than the previously-identified best in this class: Fe₄Co₁ pyrolyzed at 800°C

Accomplishment: Identified issues with the Eiwa cell design to be addressed with ANL CFDE cell design

- Bubbles easily trapped in the reference electrode port, causing faulty, high working electrode potentials leading to spikes and noisy voltammograms. In severe cases the catalysts are damaged leading to the loss of catalytic activity
- Electrodes are permanently fixed onto the flow plates. If an electrode is damaged and needs to be polished the entire bottom plate of the CFDE must be polished
- Very high iR drop between working and reference electrodes ($>1 \text{ k}\Omega$)
- Due to the lack of aligning mechanism for assembling and disassembling the flow cell, the process is difficult and time consuming.
- These issues are being addressed with Argonne's flow cell design
 - Micro-reference electrode flush with top surface of channel
 - Removable electrodes
 - Aligning pins for cell assembly
 - Filter to remove impurities in electrolyte



Accomplishment: Designed and built a Four Channel Flow Double Electrode (CFDE) Cell at Argonne



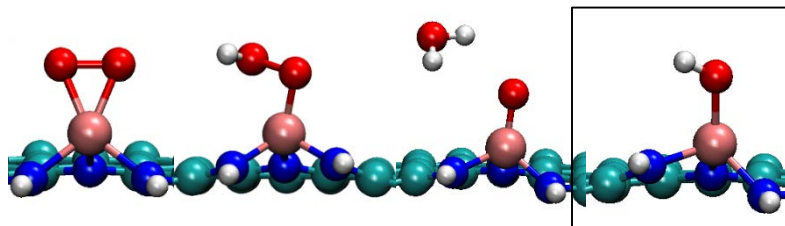
- Argonne CFDE cell has removable electrode holders.
- Electrodes can be removed for polishing and coating with electrocatalyst ink.
- Cell is not ruined if an electrode is destroyed (e.g., by oxidation due to high potentials, etc.)
- Multiple electrode inserts will be made and inks can be deposited with the robotic system for high throughput electrode fabrication.



Non-PGM Catalyst Modeling Task: Goal and Approach

- Correlate atomic scale active site structures to ORR activity and durability
- Develop, test, and utilize high-throughput capability for rapid screening of possible Fe-N-C active site moieties for activity and stability using density functional theory (DFT)-based descriptors
- Apply methodology to generate a library of active site structures with activity and stability descriptors calculated
- Provide guidance for relation between synthesis conditions and realized active site structures
- High-throughput approach dictates minimizing number of simulations
- *OH intermediate state is often potential determining step – activity descriptor
- Utilizing computational hydrogen electrode thermochemical model, simulation of bare and *OH bound site provides rapid feedback for active site screening (ΔE is ideally 1.23 eV)
- Down select based on ΔE calculations for more detailed treatment

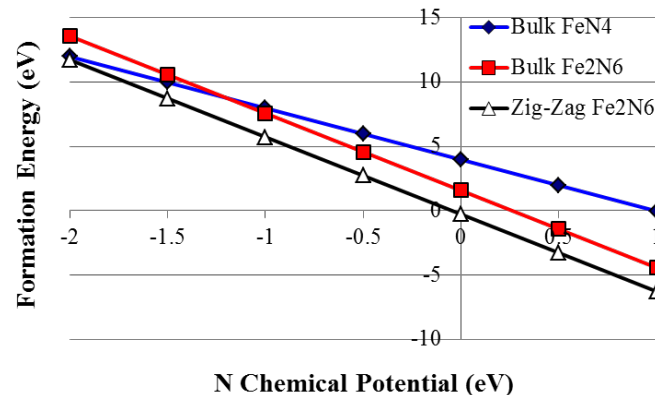
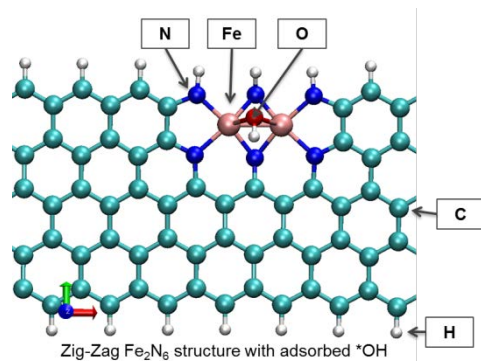
$$\Delta E = E_{*OH} - E_{bare} = (E_{0,*OH} + E_{0,H_2O} + 1/2 E_{0,H_2}) - (E_{0,bare} + 2E_{0,H_2O})$$



Highlight: Activity descriptor determined

Accomplishment: Developed DFT Screening Tool for non-PGM Activity and Stability

- Considered 22 initial structures: down selected to 8 structures based on activity descriptor for full, detailed ORR pathway calculations
 - Bulk FeN₄, Bulk Fe₂N₆, Bulk Fe₂N₅(*OH), Zig-Zag FeN₄, Zig-Zag Fe₂N₆, Zig-Zag Fe₂N₅(*OH), Arm-Chair FeN₄, Arm-Chair Fe₂N₈
- Bulk (2-D periodic graphene) and zig-zag edge host materials seem to act similarly, arm-chair host material seems to behave differently (Fe₂N₆ overbinds *OH)
- Fe₂N₆ structure in addition to previously explored Fe₂N₅(*OH) are attractive target sites
- Calculation of bare M-N-C active site moieties enables estimate of relative active site formation energies $FE = E_{0,bare} - E_{0,host} - \sum \delta n_i \mu_i$
- Chemical potentials are set by synthesis condition so trends between synthesis and active sites formed can be identified



Highlight: Initial library generated and down selection of sites based on activity achieved

Highlight: Stability descriptor and effect of chemical potential demonstrated for down selected active site structures

Collaborations

- Project team (subs) within DOE Hydrogen and Fuel Cells Program
 - Oak Ridge National Laboratory (Low-Pt)
 - Dave Cullen
 - Role: analysis of microstructure of inks, dried inks, and electrode layers via SEM and TEM
 - Los Alamos National Laboratory (non-PGM)
 - Piotr Zelenay, Ted Holby, and Samrat Choudhury
 - Roles:
 - verify validity of channel flow double electrode cell data with RDE
 - develop capability for high-throughput screening of possible Fe-N-C active site moieties for activity and stability using density functional theory (DFT)-based descriptors
 - scale-up synthesis of the best transition metal-PANI-C catalyst arising from the high-throughput experiments. Fabricate and test membrane-electrode assembly
- Collaborator outside of DOE Hydrogen and Fuel Cells Program
 - NuVant Systems, Inc.
 - Eugene Smotkin and Bogdan Gurau
 - Role: In-kind contributor, project advisors on combinatorial MEA cell



Remaining Challenges/Barriers

- Full automation of non-PGM catalyst synthesis including grinding step
- Addressing issues with lack of diffusion-limited current in hydrodynamic measurements of non-PGM catalyst ORR activity
- Addressing high overpotentials observed in non-PGM electrocatalyst ORR voltammograms versus those observed in RDE for large batches
- Attaining high performance in high current density region for twenty-five electrode MEA cell
- Modeling of low-Pt electrode voltage losses
 - Code development for full automation of polarization curve and impedance data analysis
- DFT Non-PGM Catalyst Activity and Stability Screening
 - Calculations for full ORR pathway on down-selected sites
 - Code development for automation – DFT Monte Carlo search program anticipated to be completed before the end of the project



Proposed Future Work (FY'15 and beyond)

- Improve performance of 25 electrode MEA cell and determine if this cell is useful for resolving differences in high current density air performance
 - Utilize decal method rather than GDE method to fabricate future MEAs
 - Diagnose sources of cell voltage loss in combinatorial cell
 - Utilize characterization results for agglomerate structure of ink to improve electrode performance
- Utilize downstream collector electrode to determine extent of production of H_2O_2 during ORR and potential dependence of dissolution products from catalyst in deaerated electrolyte
- Deliver composition and heat treatment temperature of catalyst with highest activity catalyst to LANL for RDE testing, compare RDE and CFDE results
 - If half-wave potential exceeds the current status of 0.8 V with a catalyst loading of 0.6 mg/cm^2 , LANL will scale-up catalyst synthesis and fabricate and test MEAs utilizing this composition
- Catalysts – Non-PGM and low-PGM
 - High-throughput pyrolysis with *in situ* effluent analysis and X-ray absorption (XAFS) characterization of species
 - High-throughput/combinatorial XAFS characterization for metal species present in cathode catalyst layer and changes in species during ORR in fuel cell
 - Complete robotic synthesis of catalyst colloids of varying metal type, metal ratios, etc. and deposition on a variety of high surface area supports
 - Catalyst activity and stability characterization
 - Complete robotic fabrication of catalyst-ionomer inks and deposition of inks onto CFDE cell electrodes
 - Combinatorial characterization of ORR activity using CFDE cell with downstream electrochemical and on-line ICP-MS detection of dissolved catalyst components for stability characterization
 - *In situ/in operando* high throughput analysis of species formed, catalyst particle size evolution, and carbon agglomerate structure using XAFS and X-ray scattering



Proposed Future Work (continued)

- Electrodes/MEAs

- Complete robotic synthesis and characterization of agglomerate structure of catalyst-ionomer inks (light scattering of dilute inks; X-ray scattering of concentrated inks)
- Complete robotic fabrication of catalyst-ionomer decals and *ex situ* characterization of ionic and electronic conductivities of ink layers on decals as a function of T and RH
- Complete robotic characterization of porosity and diffusivity of catalyst-ionomer decals
- “High throughput” tomography with automated image analysis and output of structural parameters to cathode catalyst layer structure-transport-performance model



Summary

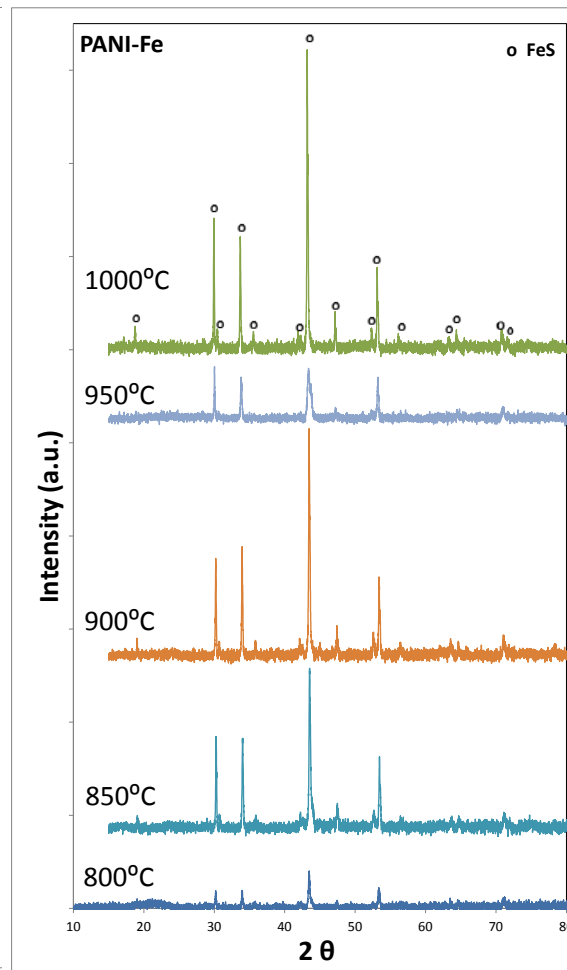
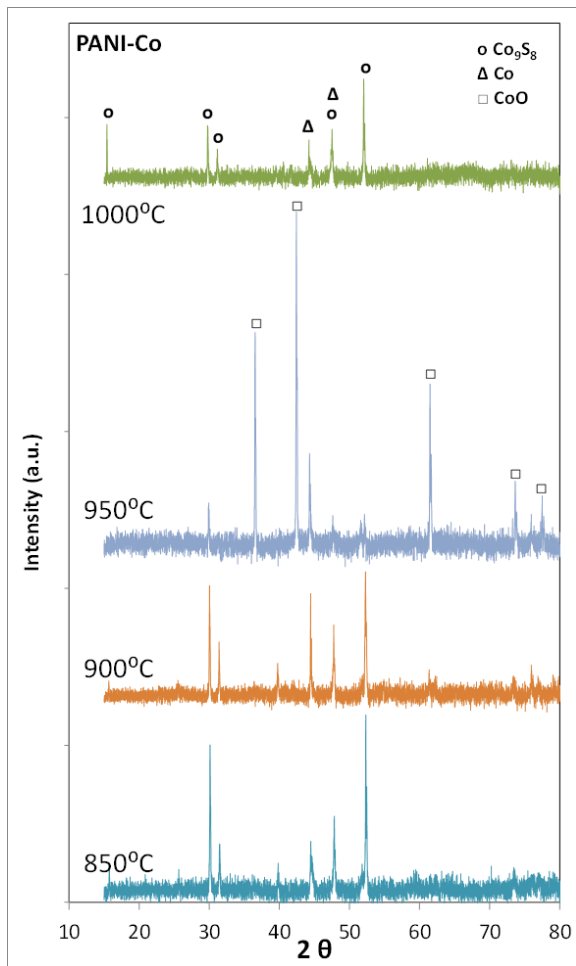
- A high throughput method was developed for synthesizing and characterizing high surface area (HSA) non-PGM ORR catalysts
 - Excellent agreement with phase compositions of single large batch catalysts was achieved
 - Continued development of high throughput grinding step is needed
- A combinatorial method/cell was applied to screening the ORR activity of HSA catalysts
 - Agreement was observed between the ORR activity trends from the HT hydrodynamic cell for the HT-synthesized catalysts and those from RDE tests of large, single batches of catalysts, validating the HT methods
 - Issues were observed with commercial cell which are being addressed with ANL cell
- A model was developed to rapidly analyze impedance and performance data providing mass activity, mass transport and kinetic overpotentials, and electrode resistances to aid in electrode design
 - Automation of data analysis is underway
- A DFT model was developed to screen non-PGM ORR activity and stability
 - Out of 22 sites analyzed, down-selected 8 most probable sites selected based on activity
 - Fe_2N_6 structure in addition to previously explored $\text{Fe}_2\text{N}_5(*\text{OH})$ are attractive target sites
 - Calculations ongoing for full pathway on down-selected sites
 - Full automation in progress – DFT Monte Carlo search program to be completed soon
- A high throughput method was developed for synthesis and deposition of powder-ionomer-solvent inks and fabrication of combinatorial MEA
 - Improvements are being implemented in fabrication method to improve electrode performance



Backup Slides



Phase composition of PANI-Co and PANI-Fe after first pyrolysis



- All PANI-Co-C samples contain Co sulfides and metallic Co, with the exception of 950°C sample (due to air leak and oxidation).
- PANI-Co-C pyrolyzed at 850°C contains the largest amount of Co_9S_8 . With increasing pyrolysis temperature, the amount of Co_9S_8 decreases.
- All of the PANI-Fe-C samples contain FeS.
- The amount of FeS increases with pyrolysis temperature.



Phase composition of PANI-Fe_xCo_y

- PANI-Fe₃Co's phase composition is reproducible within the same batch and in different batches, while PANI-FeCo₃'s is not.
- Polymerization step gave a very homogeneous powder/texture for PANI-Fe₃Co, contributing to the reproducible phase composition.
- Compared to all PANI-Fe_xCo_y, PANI-Fe₃Co's phase composition does not change with pyrolysis temperature (850-1000°C) indicating a stable compound is formed at this Fe to Co ratio.
 - Fe₃Co was reported as the most stable ORR catalyst of this class LANL (Zelenay, FC-003)
- Solid solutions of FeS and CoS were formed
- PANI-Fe is more crystalline than PANI-Co at a similar metal loading (due to combustion of carbon during pyrolysis, stability of metal sulfide phase...)
- At a pyrolysis temperature of 1000°C, the formation of a mixed CoFe sulfide phase is evident

