2009 DOE Hydrogen Program

Center for Fundamental and Applied Research in Nanostructured and Lightweight Materials

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DOE Hydrogen Program

Project ID#: fcp_08_king

OVERVIEW

Timeline

- Project start date: July 1, 2008
- Project end date: January 31, 2010
- Percent complete: 60%

Budget

- Total project funding
  - DOE - $1.23 Million
  - Contractor - $352K
- Funding received in FY08: $139K
- Funding for FY09: $1.1 Million

Partners

- Dana Holding Corporation
- Clemson University
- Johns Hopkins University
- Michigan Technological University (Project Lead)

Barriers

A. Durability
B. Cost
C. Performance
D. Water Transport within the Stack
E. Thermal System and Water Management

Targets

- Fuel Cell Bipolar Plates:
  - Electrical Conductivity > 100 S/cm
  - Thermal Conductivity > 20 W/m·K
- Thermal cyclability in presence of condensed water
- Improved GDL performance, durability, and resistance to flooding
- Develop testing and characterization protocols and techniques
- Ability to tailor the properties of carbon foam supports to meet PEM fuel cell electrode requirements
- Ability to construct pseudomorphic overlayer catalysts, on carbon supports, that are designed to mimic conventional PEM anode catalysts
- Demonstrate the production of nanostructured polymeric membranes utilizing electrodynamic synthesis methods
- Develop enabling technologies for rapid production techniques

OBJECTIVES

This project involves fundamental and applied research in the development and testing of lightweight and nanostructured materials to be used in fuel cell applications and for energy storage.

Our research covers three areas, including:

Area I. Heat and Water Management

- Develop and test lightweight and nanostructured materials for fuel cell bipolar plates.
- Improve GDL performance and durability.
- Develop testing and characterization protocols and techniques for GDLs relative to water management.

Area II. Development of New Electrode Materials

- Develop graphitic carbon foams that will serve as current collectors and catalyst supports.
- Develop durable carbon-supported catalysts that have reduced weight and cost compared to conventional PEM anode catalysts.

Area III. Enabling Technologies for Membrane Synthesis

- Develop hybrid polymer membrane materials which are engineered at the micro and nanoscale.
- Demonstrate novel electrodynamic deposition methods (electrospinning and electrospraying) for a set of paradigm polymeric compounds in a membrane form.
- Identify candidate materials for membrane synthesis using these methods.

Area I. Heat and Water Management
RELEVANCE/APPROACH

RELEVANCE

- By 2010 to develop a 60% peak efficient, durable, direct hydrogen fuel cell power system for transportation at a cost of $45/KW, by 2015 , a cost of $30/kW.
  - Develop improved bipolar plate material
  - Improve GDL performance and durability
  - Develop testing and characterization protocols and techniques
  - Alleviate flooding and/or dryout of membrane through advanced understanding of GDL water transport

APPROACH

- Develop carbon filled thermoplastic bipolar plates that meet conductivity targets (electrical conductivity> 100 S/cm, thermal conductivity > 20 W/m·K)
- Develop environmental chamber for measuring GDL wettability at fuel cell operating temperatures in air and hydrogen; thermal, humidity and gaseous control
- Measure contact angles from 0 to 80^{o}C in air and in hydrogen
- Develop characterization method for GDL compression and damage resulting from compression
- Image GDL under compression
- Assess damage resulting from compression

Area I. Heat and Water Management
MILESTONES

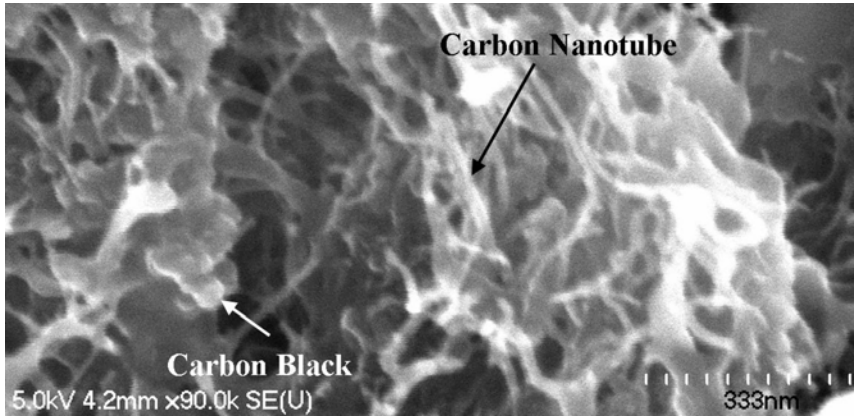
Milestones	Month/Year	Percent Complete
Compounded carbon nanotube/carbon black/synthetic graphite/ polypropylene conductive resins and molded test specimens for bipolar plate materials. Determined electrical conductivity, through plane thermal conductivity using guarded heat flow meter method and transient plane source test method, and rheological properties of each resin. Developed electrical conductivity models and through plane thermal conductivity models.	Nov-2008	100%
Complete in plane and through plane thermal conductivity testing and modeling on carbon nanotube/carbon black/ synthetic graphite/ polypropylene conductive resins using nanoflash test method.	Dec-2009	30%
Contact angle measurement apparatus and technique: There remains some development work on the humidity control for measuring the contact angle at elevated temperatures. A second-level thermostat is being constructed to maintain better humidity control and provide a sealed chamber for use with hydrogen.	Nov-2008	90%
Contact angle measurements on GDL samples: Contact angle measurements continue on the current GDL sample set.	Jan-2010	15%
Compression Fixture: The fixture is complete. A second set of springs will be ordered to improve the testing range for the stiffer GDL materials.	Nov-2010	98%
Compression-force measurements on GDL samples: Compression testing and imaging continue on the current GDL sample set.	Jan-2010	15%

Area I. Heat and Water Management

TECHNICAL ACCOMPLISHMENTS

Conductive Bipolar Plates for Fuel Cells

- Most Conductive Material: 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD carbon black/ 65 wt% Asbury Carbons Thermocarb TC-300 synthetic graphite particles/ 6 wt% Hyperion carbon nanotubes/ 26.5 wt% Dow homopolymer polypropylene H7012-35RN
  - Electrical Conductivity: obtained 91 S/cm (DOE target is 100 S/cm) via compression molding and 38 S/cm via injection molding
  - In Plane Thermal Conductivity: obtained 24 W/m·K (DOE target is > 20 W/m·K) via compression molding and 18 W/m·K via injection molding

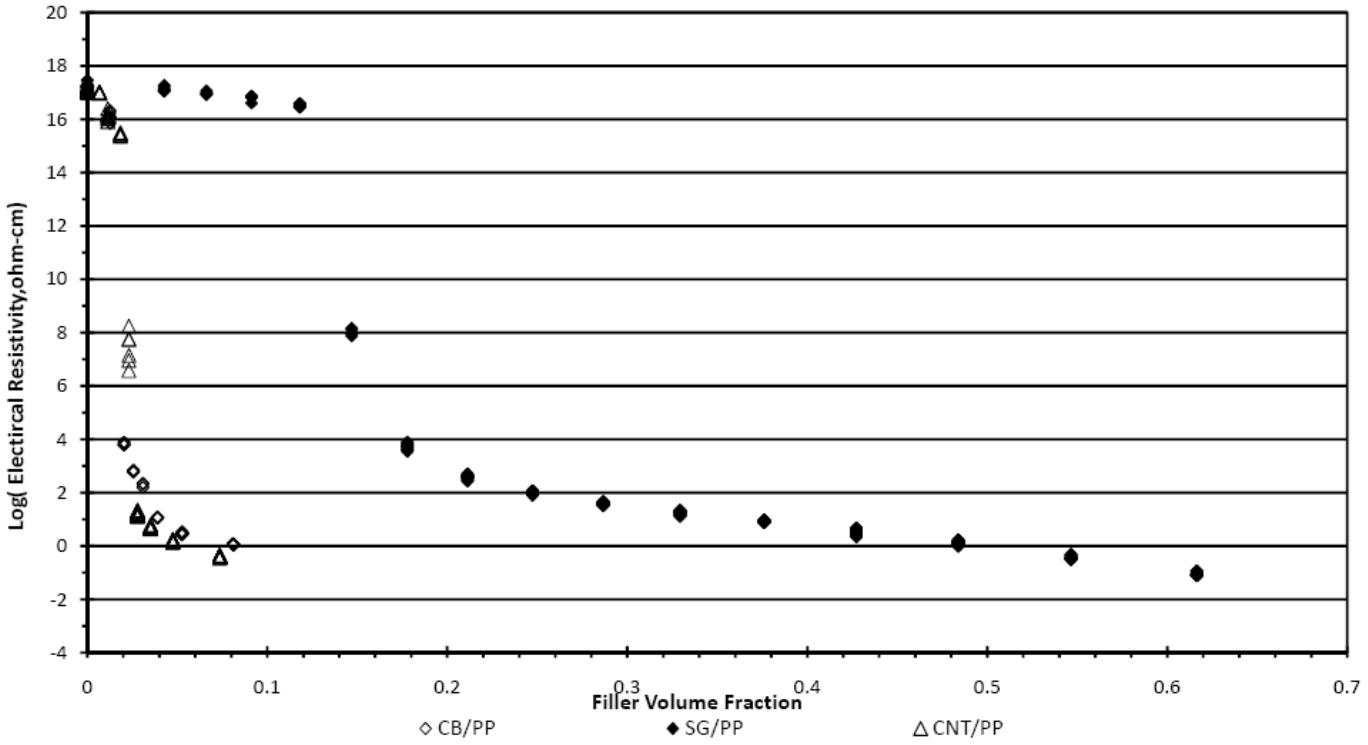


Area I. Heat and Water Management

TECHNICAL ACCOMPLISHMENTS

Conductive Bipolar Plates for Fuel Cells: Electrical Conductivity

- Percolation Threshold : Injection Molded Samples
  - Carbon Black : 1.4 vol%
  - Carbon Nanotubes: 2.1 vol%
  - Synthetic Graphite: 13 vol%

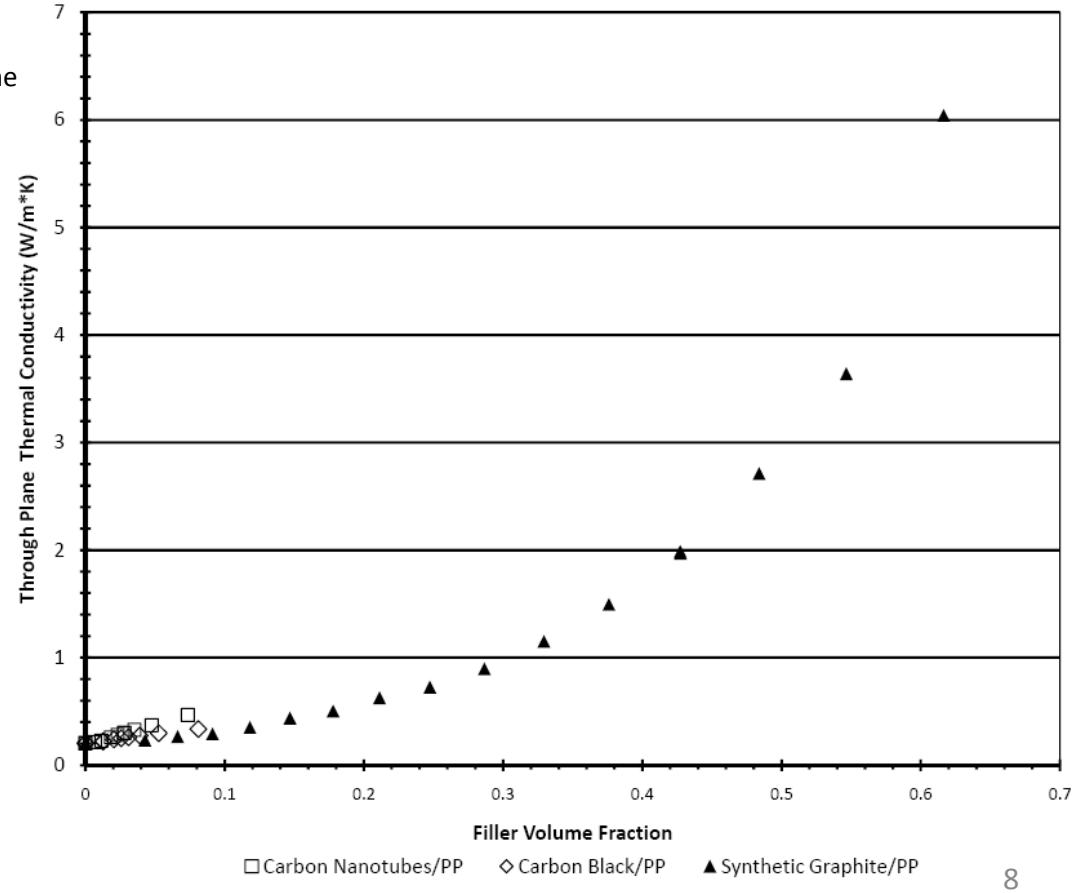


Area I. Heat and Water Management

TECHNICAL ACCOMPLISHMENTS

Conductive Bipolar Plates for Fuel Cells

- Through Plane Thermal Conductivity :
  Guarded Heat Flow Meter Method and Transient Plane Source Test Method: Injection molded samples
  - 15 wt% (8.1 vol%) Carbon Black /PP: 0.34 W/m·K
  - 15 wt% (7.4 vol%) Carbon Nanotubes/PP: 0.47 W/m·K
  - 80 wt% (61.6 vol%) Synthetic Graphite:/PP: 6 W/m·K

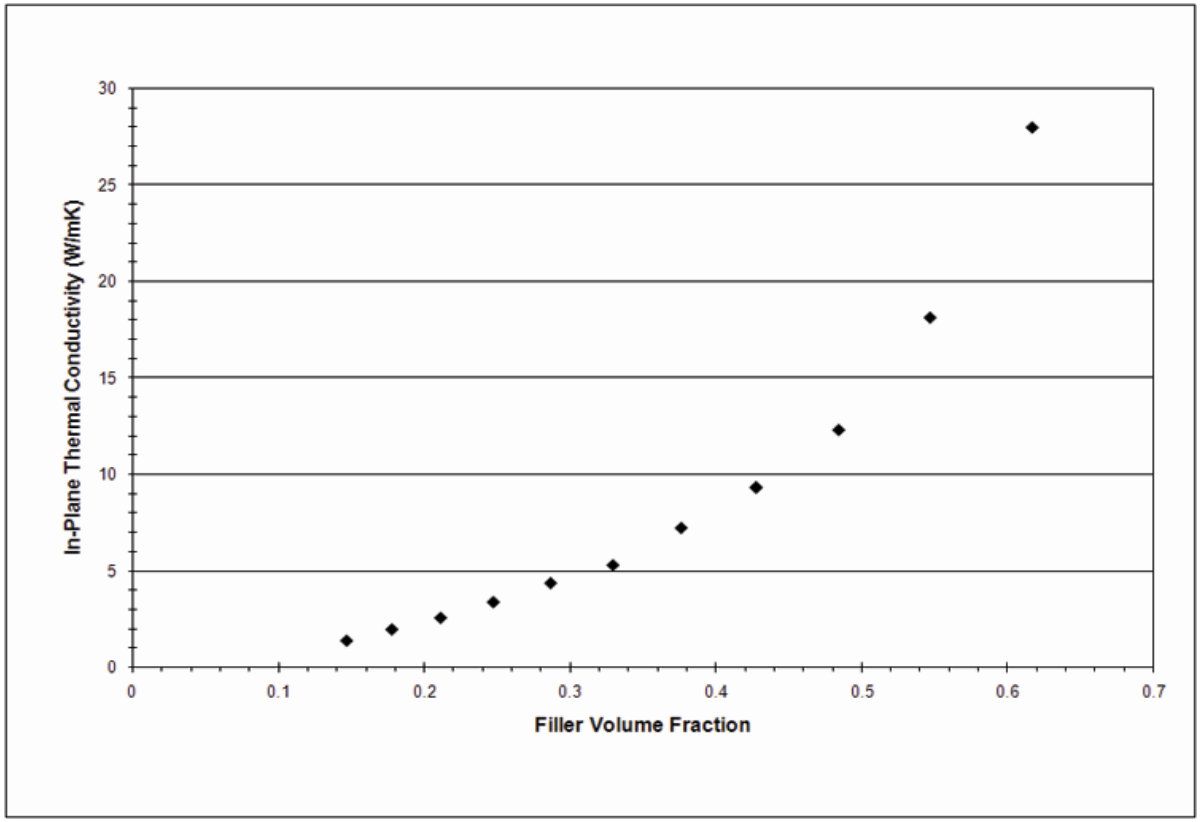


Area I. Heat and Water Management

TECHNICAL ACCOMPLISHMENTS

Conductive Bipolar Plates for Fuel Cells

- In Plane Thermal Conductivity: Transient Plane Source Test Method
  - 80 wt% (61.6 vol%) Synthetic Graphite/PP: 28 W/m·K



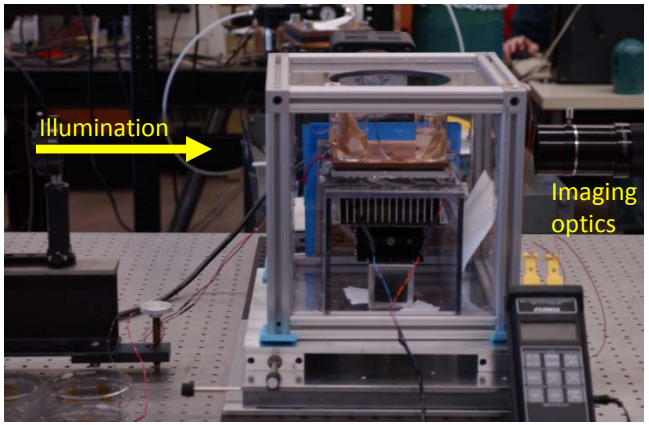
Area I. Heat and Water Management

TECHNICAL ACCOMPLISHMENTS

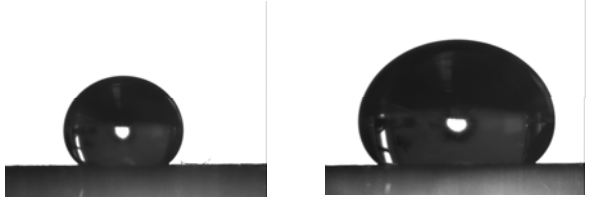
Movement of Water in Fuel Cell Electrodes

Technical Accomplishments

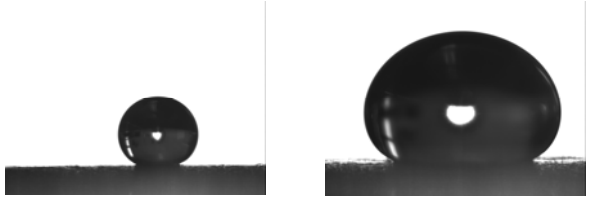
- Environmental chamber fabricated
  - Temperature control
  - Humidity control
- Contact angles exhibiting a size dependency; the larger the drops the larger the contact angle.
  - Likely causes are (i) drop deposition methodology or (ii) pinning effect for small drops which sample fewer pores than large drops
- Second level thermostat design is completed and fabrication started
- Analysis code completed
- Contact angle measurement error characterized
- Preliminary data indicates decreasing contact angle as temperature increases



Environmental chamber for contact angle measurements.



GDL 4. baseline with teflonation, $\theta \sim 140^{o}$



GDL 11. Toray with teflonation, $\theta \sim 155^{o}$

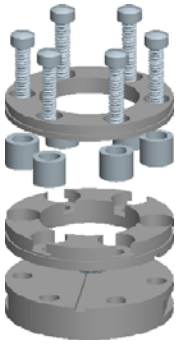
Area I. Heat and Water Management

TECHNICAL ACCOMPLISHMENTS

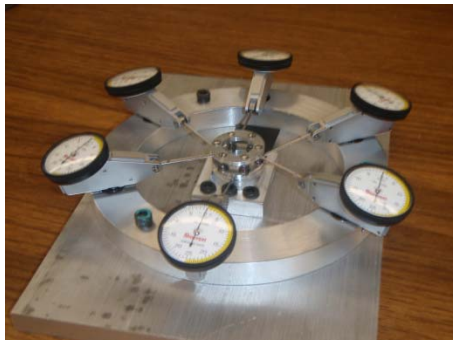
Movement of Water in Fuel Cell Electrodes

Technical Accomplishments (cont.)

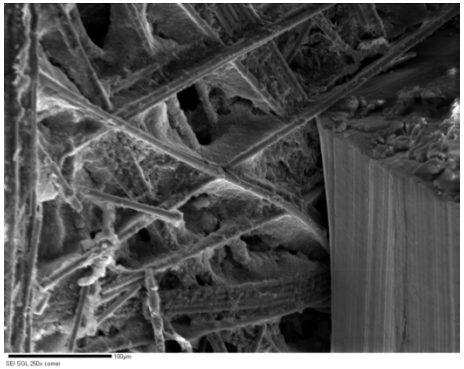
- Calibrated SEM compression fixture completed
- GDL imaging under incremental compression
- Obtaining stress-strain relationship for compression of GDLs under channels
  - Similar to that found in bipolar plates
- Compression range:
  - Up to 1600 psi based on the area of (4) ½ standard samples
  - Displacement resolution is 6.5 μm



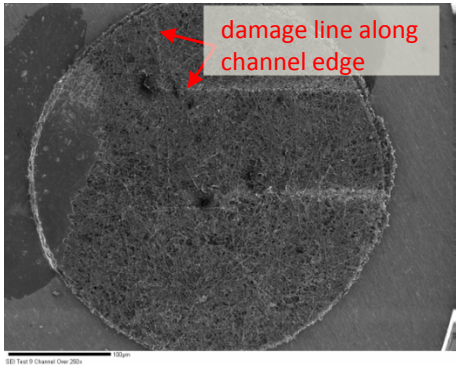
Sample holder



Setting compression on 6 GDL samples prior to SEM imaging.

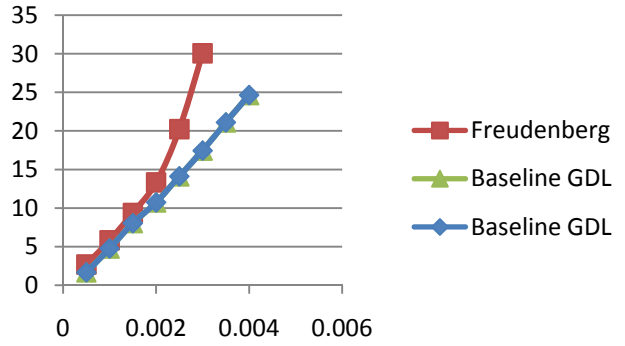


Baseline GDL under compression imaged at edge of channel



Baseline GDL post-compression showing damage

Force vs. Displacement



force (lbf) – displacement (in)

Area II. Development of New Electrode Materials

RELEVANCE

- Graphitic carbon can serve as a current collector and mechanical support for electrochemically-active mass and catalytic materials. A mesoporous carbon foam, in particular, offers an inexpensive, lightweight alternative to current electrode materials, and it can be machined to desired shapes. It is also tolerant of the corrosive environment within fuel cells. Both the electrical conductivity (degree of graphitization) and the porosity of the carbon foam are important for current collector and catalyst applications.
- A new type of hybrid battery based on a nickel-carbon foam cathode and a carbon supercapacitor anode may allow a lightweight battery system in fuel cell powered vehicles that allows temporary storage of excess generated electricity. The nickel-carbon cathode can be trickle-charged to store electricity and discharged rapidly when needed.
- Replacement catalysts for platinum are needed to reduce fuel cell cost. Synthesis of pseudomorphic overlayer bimetallic catalysts may reduce or eliminate platinum use in PEM fuel cells.

Area II. Development of New Electrode Materials

APPROACH

- Synthesize carbon foams of varying pore sizes at Clemson University using PAN precursors. Thermally induced phase separation (TIPS) of the polymer from the solvent phase is used to form the precursor material. The resulting foam is then pyrolized to produce a graphitic carbon foam. Goals are to: (1) increase the macropore/mesopore space to hold more active electrode mass , and (2) increase the accessible micropore surface area so that a supercapacitor anode can be created. The latter would allow charge storage through an electric double layer mechanism.
- Examine the electrochemical performance of carbon foam electrodes loaded with nickel oxide active mass by performing charge/discharge cycles in flooded and sealed cells.
- Measure the pore size distribution, surface area, and surface composition (via spectroscopic analyses) of candidate carbon foam electrode supports, both before and after electrochemical service
- Use surface passivation and controlled deposition reaction to synthesize a pseudomorphic overlayer bimetallic catalyst on a carbon support. The bimetallic combination is being chosen to mimic the heat of adsorption and catalytic activity of a Platinum active site on a PEM fuel cell catalyst.

Area II. Development of New Electrode Materials

MILESTONES

Milestones	Progress Notes	Planned Completion Date	% Complete
Microscopy and spectroscopic analyses of commercial Poco Graphite foam samples and MTU nickel-carbon electrodes	In progress	01/31/2010	40%
Making a graphitized carbon foam from a PAN precursor	In progress	01/31/2010	10%
Active mass deposition, forming, and cycling	In progress	01/31/2010	40%
Designing an electrochemical cell with a nickel-carbon working electrode	In progress	01/31/2010	80%
Synthesis and characterization of Pt/Ni Overlayer Catalysts	In progress	01/31/2010	30%
Extend catalyst synthesis procedure to a carbon support	In progress	01/31/2010	5%

Area II. Development of New Electrode Materials

TECHNICAL ACCOMPLISHMENTS

Synthesizing Graphitic Carbon Foams

- Porous, machinable current collectors and mechanical supports
- Pyrolysis of PAN-based foams in an oxygen-free environment at 1200°C; doping with silicon and boron to enhance properties
- Positive (cathodic) battery electrodes with a large macropore / mesopore space to hold electrochemically-active material
- Supercapacitor battery anodes with a large accessible micropore surface area (i.e., > 1000 m^2/g) for electric double layer charge storage
- Carbon supports for fuel cell pseudomorphic overlayer anode catalyst(s) that promote dissociation of diatomic hydrogen into constituent protons and electrons

Area II. Development of New Electrode Materials

TECHNICAL ACCOMPLISHMENTS

Studying the Structure and Morphology of Carbon Foam Electrode Supports

- Depositing and forming nickel oxyhydroxide active mass in a nickel-carbon foam working electrode
- Optimizing active mass deposition in the working electrode (efficient pore filling, accessibility to ion transport, electrical contact with the carbon support)
- Prelude work for future deposition of a nickel-based bimetallic overlayer fuel cell anode catalyst on a carbon foam support

Carbon foam cathode with deposited active mass

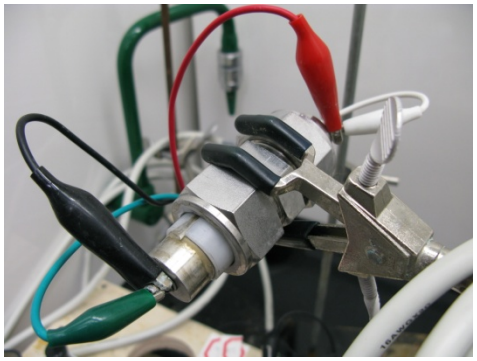
Optical image of carbon foam deposited with nickel oxyhydroxide (200X).

Area II. Development of New Electrode Materials

TECHNICAL ACCOMPLISHMENTS

Performing Battery Tests with Synthesized Electrodes

- Operating flooded and sealed cells with carbon foam electrodes
  - Nickel-carbon foam working electrode
  - Carbon foam or nickel foil counter electrode



Swagelok cell connected to a potentiostat for cycling

Swagelok cell component parts

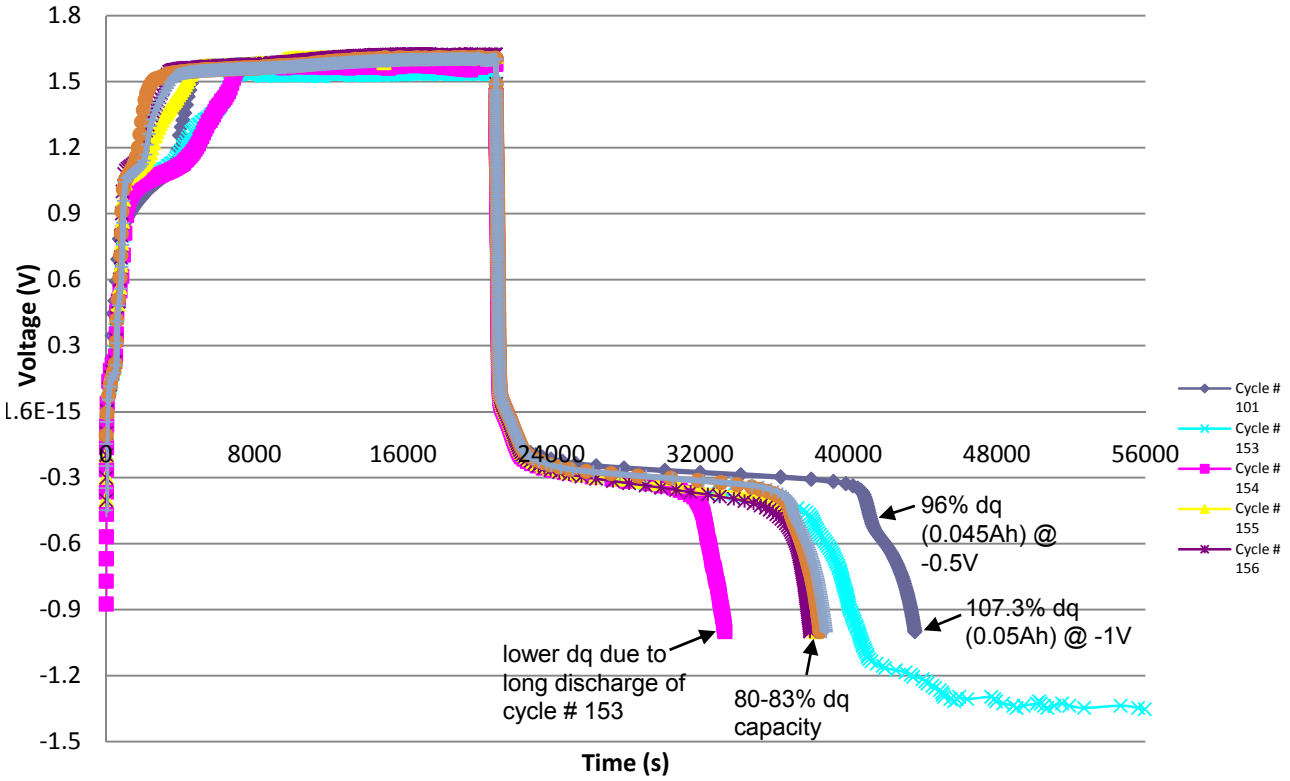
- Charge/discharge tests to determine cycle life and discharge capacity
- Experiments will be extended to future carbon-supported fuel cell electrode/catalyst prototypes

Area II. Development of New Electrode Materials

TECHNICAL ACCOMPLISHMENTS

Performing Battery Tests with Synthesized Electrodes

Electrode Charge/Discharge Cycles @ 0.2C-Rate

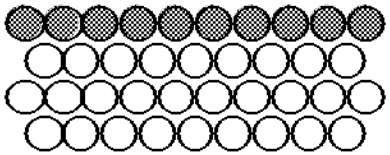


Area II. Development of New Electrode Materials

TECHNICAL ACCOMPLISHMENTS

Exploration of pseudomorphic nanoscale overlayer bimetallic catalysis for fuel cells

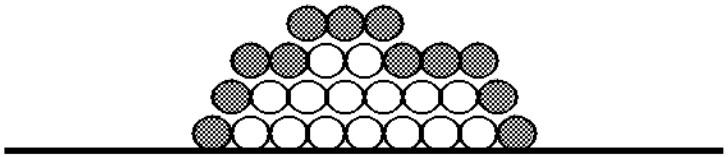
Single Crystal Pseudomorphic Overlayer



Metal A

Metal B

Supported Particle Pseudomorphic Overlayer



Oxide Support

- Structure results in unique properties:
  - Bonding strength can increase or decrease depending on the metal combination. Literature studies have determined precise values.
  - Bonding strength can be correlated with center of the d-band calculated computationally.
- Nomenclature: Base@Overlayer; e.g. Re@Pd is Pd overlayer on Re base

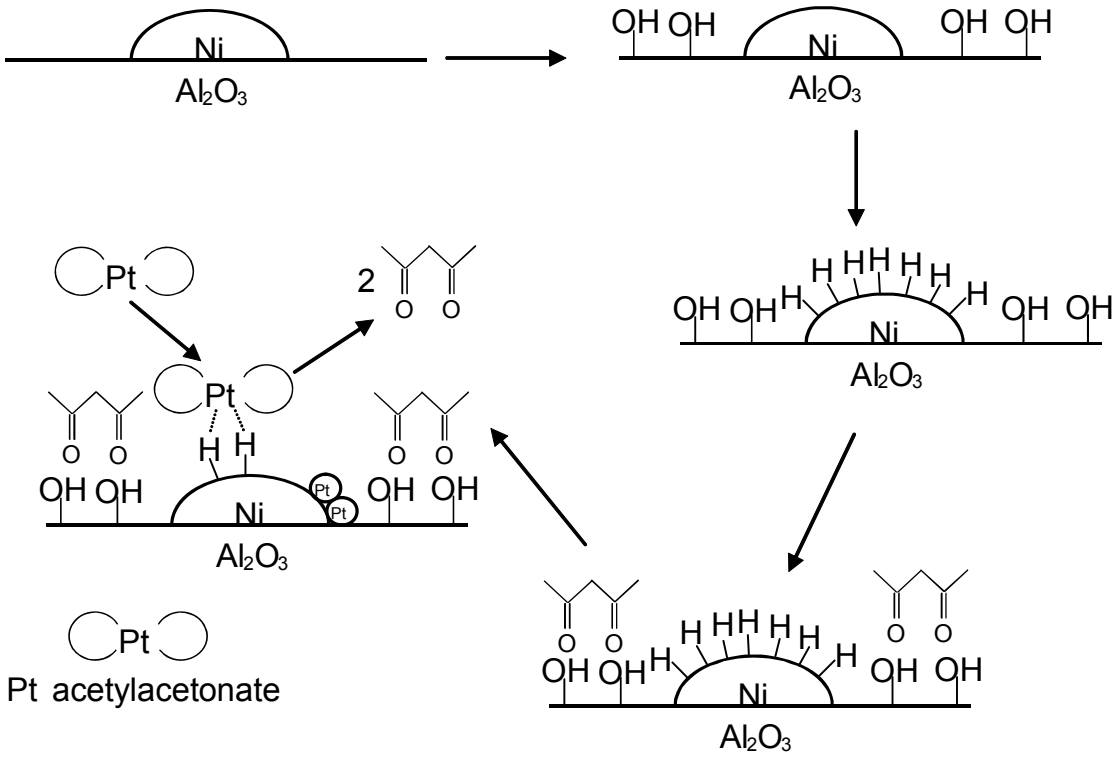
Area II. Development of New Electrode Materials

TECHNICAL ACCOMPLISHMENTS

Exploration of pseudomorphic nanoscale overlayer bimetallic catalysis for fuel cells

Reload Synthesis Procedure

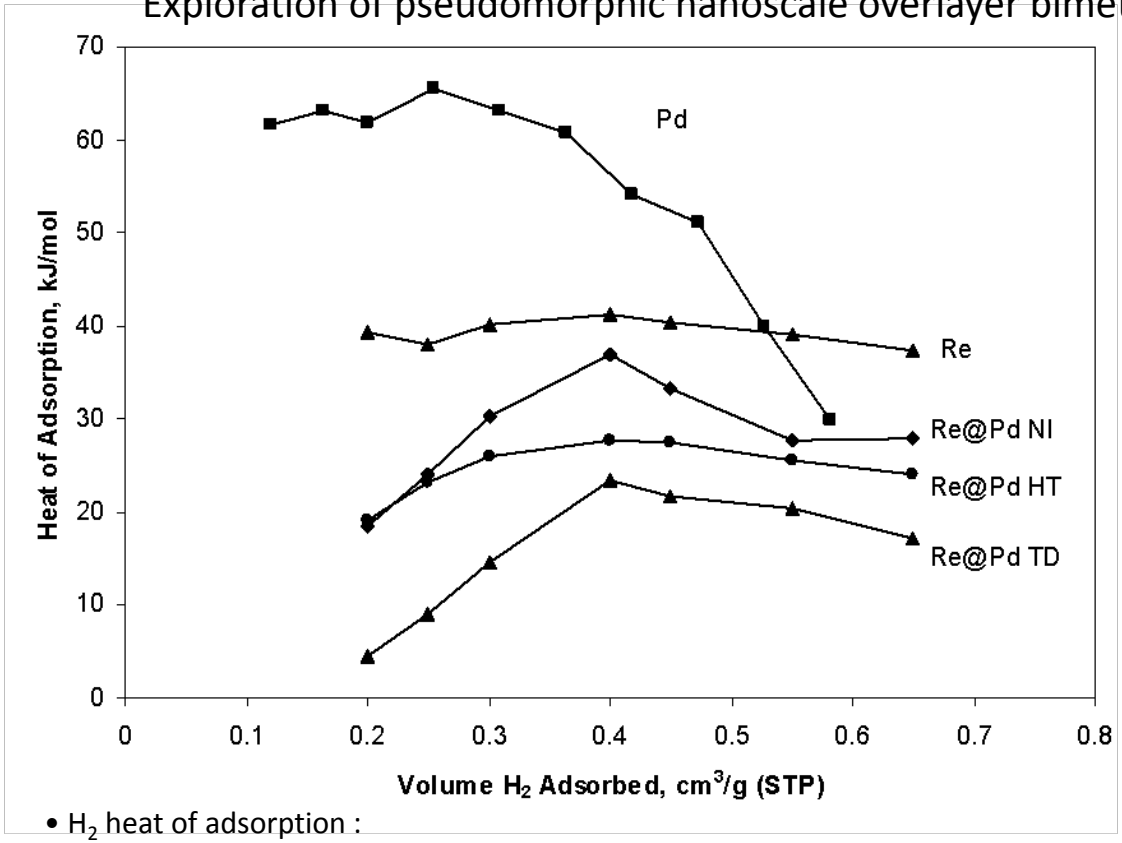
- Synthesis procedure focuses overlayer deposition:
- Hydroxylation and acetylacetone inhibit deposition on support.
- Surface reaction with hydrogen also direct deposition while only allowing one layer of coverage.



Area II. Development of New Electrode Materials

TECHNICAL ACCOMPLISHMENTS

Exploration of pseudomorphic nanoscale overlayer bimetallic catalysis for fuel cells



H_2 Heat of Adsorption

NI = no inhibitors (hydroxylation or acetylacetone)

HT = High temperature deposition

TD = deposition procedure repeated three times

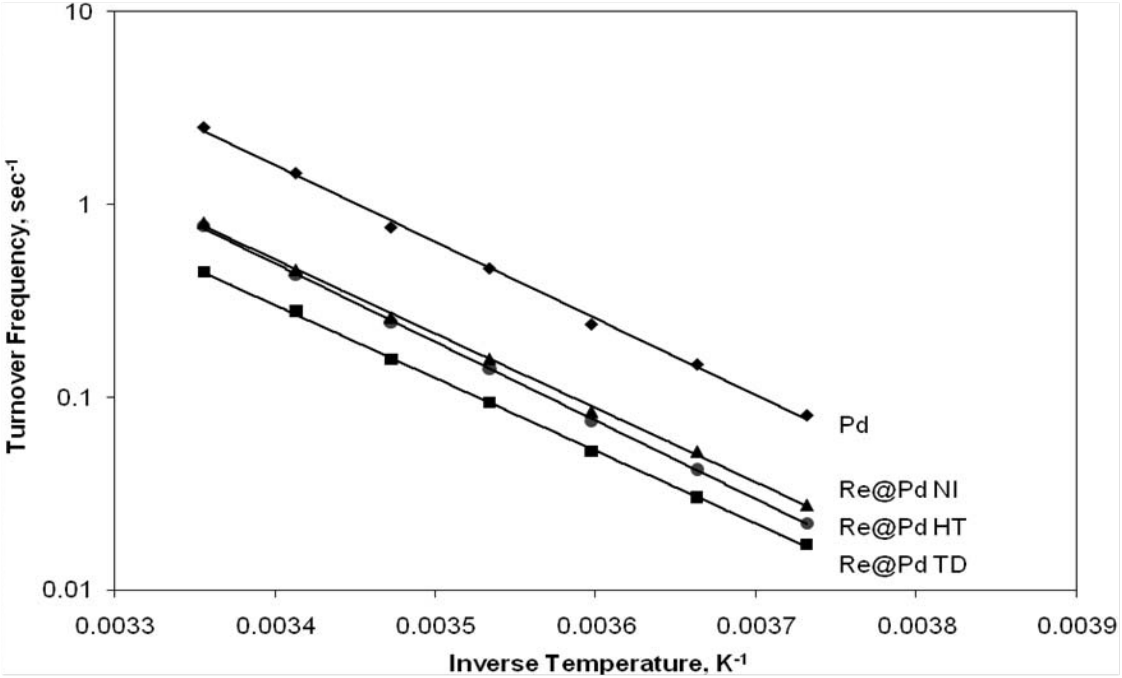
- H_2 heat of adsorption :
  - All three Re@Pd catalysts had decreased H2 adsorption strength compared to the base metal.
  - Re@Pd TD decreased heat of adsorption to approximately 20 kJ/mol at intermediate coverage.
  - Results consistent with literature first-principles computational studies.

Area II. Development of New Electrode Materials

TECHNICAL ACCOMPLISHMENTS

Exploration of pseudomorphic nanoscale overlayer bimetallic catalysis for fuel cells

Ethylene Hydrogenation



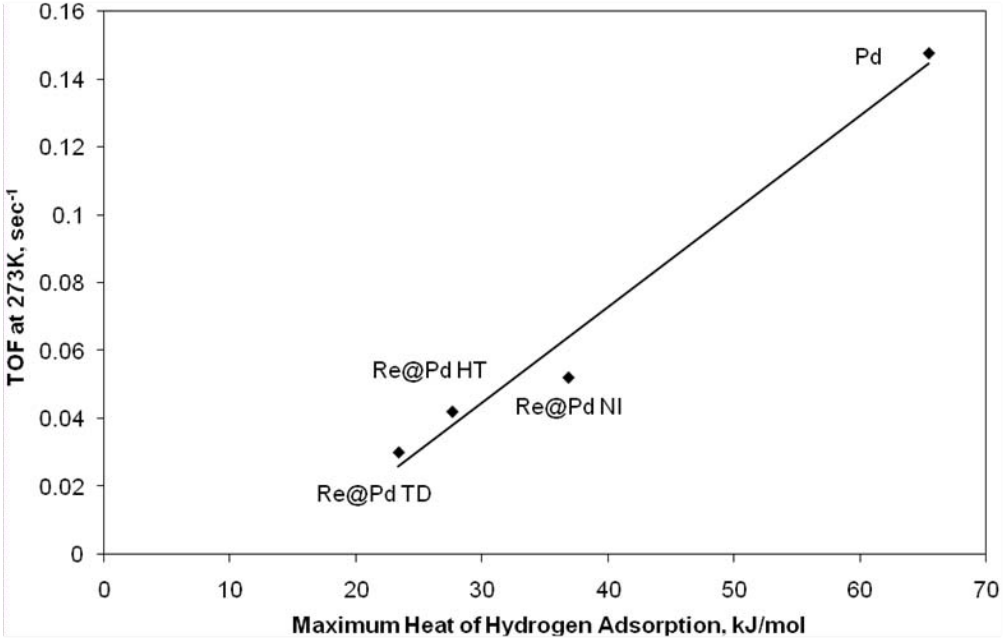
- Catalytic activity was compared using ethylene hydrogenation reaction:
  - Pure Pd was slightly more active than Re@Pd catalysts.
  - Apparent activation energy (~75 kJ/mol) are consistent with barrier for ethylene hydrogenation to surface ethyl (from literature).

Area II. Development of New Electrode Materials

TECHNICAL ACCOMPLISHMENTS

Exploration of pseudomorphic nanoscale overlayer bimetallic catalysis for fuel cells

Correlation of Activity and Heat of Adsorption



- Ethylene hydrogenation activity decreases as H_2 heat of adsorption decreases.
- Since H_2 surface coverage is low compared to ethylene; decreased binding strength results is lower coverage which consequently decreases reaction rate.
- Reload synthesis technique is capable of synthesizing catalysts whereby adsorption strength and reactivity can be precisely controlled.

Area II. Development of New Electrode Materials

TECHNICAL ACCOMPLISHMENTS

Exploring Alternatives to a Platinum-based Fuel Cell Anode Catalyst

- Extending recently developed synthesis method for bimetallic overlayer catalysts.
- Selecting bimetallic combinations with desired fuel cell catalytic properties
- Refining synthesis procedure to construct an overlayer catalyst on a graphitic carbon support

Area III. Enabling Technologies for Membrane Synthesis

RELEVANCE/APPROACH

RELEVANCE

- New technologies enabling the production of micro- and nano-structured polymeric membranes are needed to develop the next generation of fuel cell membranes that are engineered at the microscopic level.
- Hybrid polymer materials are promising for membranes that operate over a wider range of temperatures, with improved ionic conduction.
- Electrospinning and electrospraying are two techniques for producing these hybrid materials with a controlled micro- and nano-structure utilizing a wide variety of polymer and dopant materials. Highly anisotropic materials may be produced that could lead to improved mechanical and chemical properties for the membrane materials.

APPROACH

- Develop apparatus for continuous electrospinning of a single material fiber utilizing polymer solutions of a variety of types.
- Adapt the process to spin continuous fibers that are highly aligned utilizing a rotating electrode system.
- Develop a coaxial needle system to electrospin/electrospray core-shell fiber and particle of hybrid materials.
- Develop a novel pulsed DC electrospray apparatus top allow better control over the particle size distribution and particle shell thickness.

Area III. Enabling Technologies for Membrane Synthesis

MILESTONES

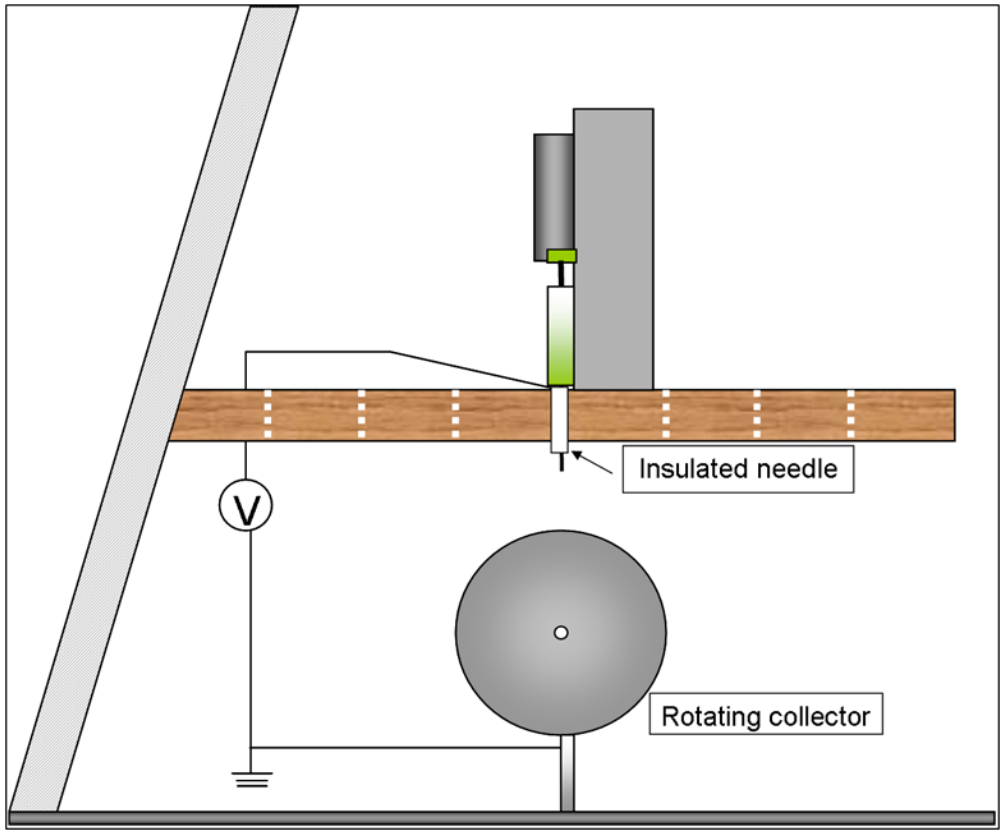
Milestones	Progress Notes	Comments	% Complete
Construct apparatus for continuous electrospinning	Completed	Undergoing testing using a single needle	100%
Adapt process to spin aligned fibers	Equipment completed	Further studies on control of fiber diameter underway	80%
Develop coaxial needle system for core-shell nanostructured materials	Equipment completed	Coaxial fiber and core-shell nanoparticle studies underway	50%
Develop novel pulsed electrospray	Equipment completed	Electronics testing underway	25%

Area III. Enabling Technologies for Membrane Synthesis

TECHNICAL ACCOMPLISHMENTS

Electrospinning Hybrid Polymer Composites

Electrospinning Apparatus



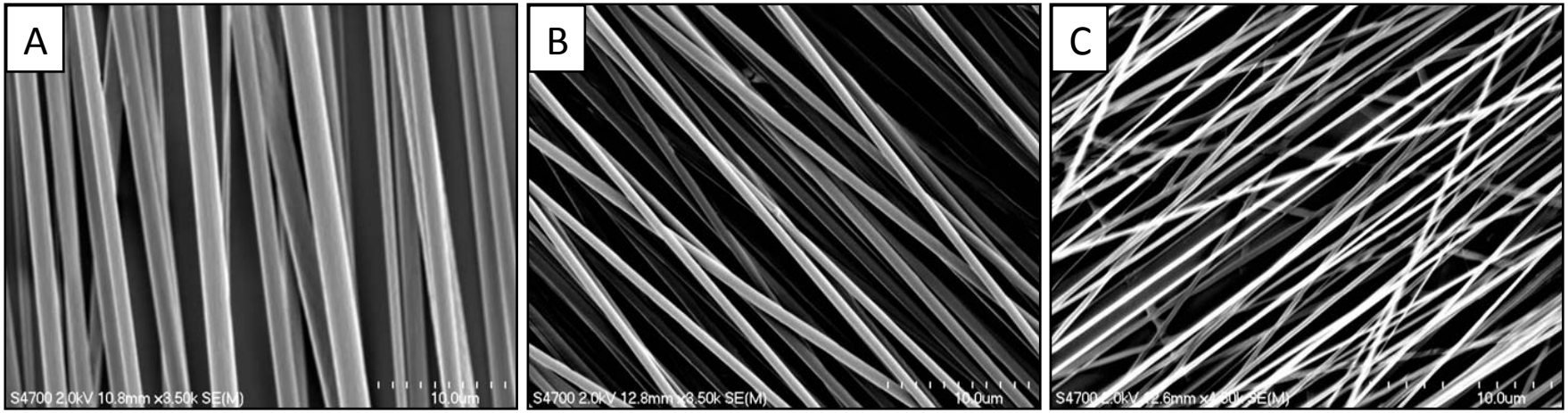
Area III. Enabling Technologies for Membrane Synthesis

TECHNICAL ACCOMPLISHMENTS

Electrospinning Hybrid Polymer Composites

Objective: Electrospin fibers of controlled diameter in a highly anisotropic manner.

Goal: Create three distinct variable diameter scaffolds by manipulating electrospinning parameters.



Generation of poly-L-Lactic acid (PLLA) fibers with varying diameters.
(A) Aligned fibers with diameters around 1-2 μm; (B) Aligned fibers with diameters around 800 nm; (C) Aligned fibers with diameters around 300 nm. Images obtained at 3500x using a SEM. Scale bar = 10 μm.

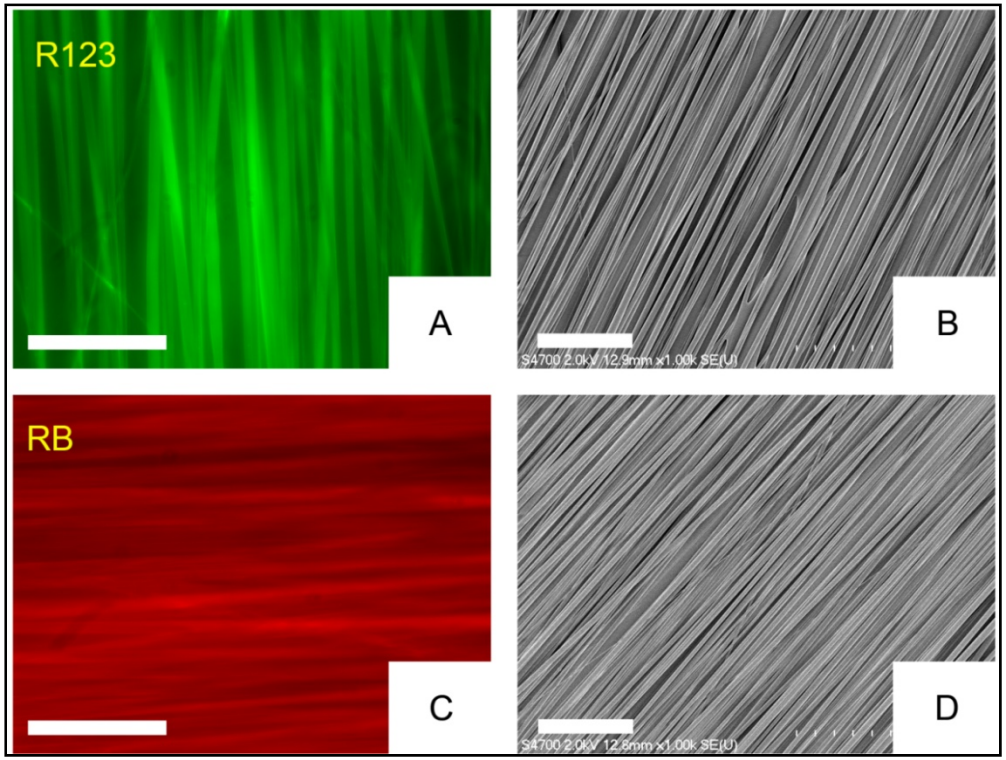
Area III. Enabling Technologies for Membrane Synthesis

TECHNICAL ACCOMPLISHMENTS

Electrospinning Hybrid Polymer Composites

Goal: Develop fluorescent, electrospun fibers by doping fluorescent dye into polymer solution without altering fiber alignment

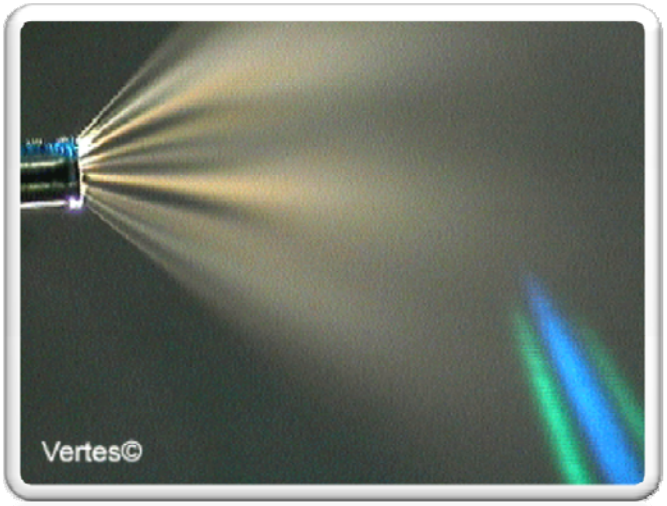
Fabrication of fibers doped with rhodamine fluorophors. (A) Fibers doped with Rodamine-123 (R123) under fluorescence microscopy; (B) SEM image of R123 fibers. R123 does not affect fiber alignment; (C) Fibers doped with Rhodamine-B (RB) under fluorescence microscopy; (D) SEM image of RB fibers. RB does not affect fiber alignment. Scale bar = 25 μm



Area III. Enabling Technologies for Membrane Synthesis

TECHNICAL ACCOMPLISHMENTS

Electrospray of Polymer Hybrid Materials



http://www.gwu.edu/~ipta/images/ElectrosprayPDA5.gif

- Simple one-step and economical technique
- Narrow size distribution
- Less coalescence
- Particles in submicron or micron scales
- Sphere and capsule particles

Area III. Enabling Technologies for Membrane Synthesis

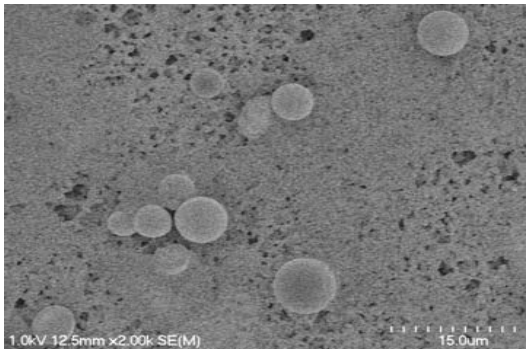
TECHNICAL ACCOMPLISHMENTS

Electrospray of Polymer Hybrid Materials

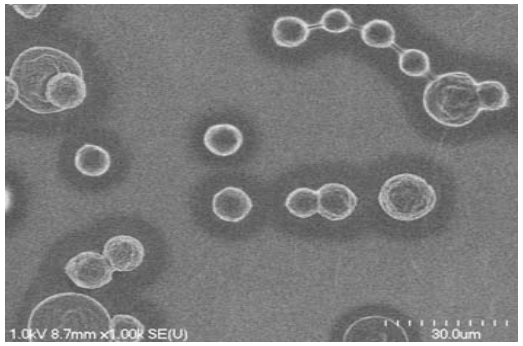
Past Work - Design

We focused on familiarizing the performance of single-capillary electrospray. This led to study the following parameters:

- Polymer concentration – varied from 1 to 4 wt./vol. %
- Organic solvent – 1,2DCE, DCM, DCM+Chloroform
- Additive – Tricaprylmethylammonium Chloride
- Feed flow rate – varied from 0.5 to 2.0 mL/hr
- Voltage applied – varied from 10 to 15 kV
- Spray head – 18 to 24 gauges; blunt and sharp tips
- Collector – distilled water, PVA, aluminum foil, dielectric medium



100 mg PLLA dissolved in 5 mL of 1,2-DCE. Sample was collected on Al foil at 1 mL/hr and 12.5 kV.



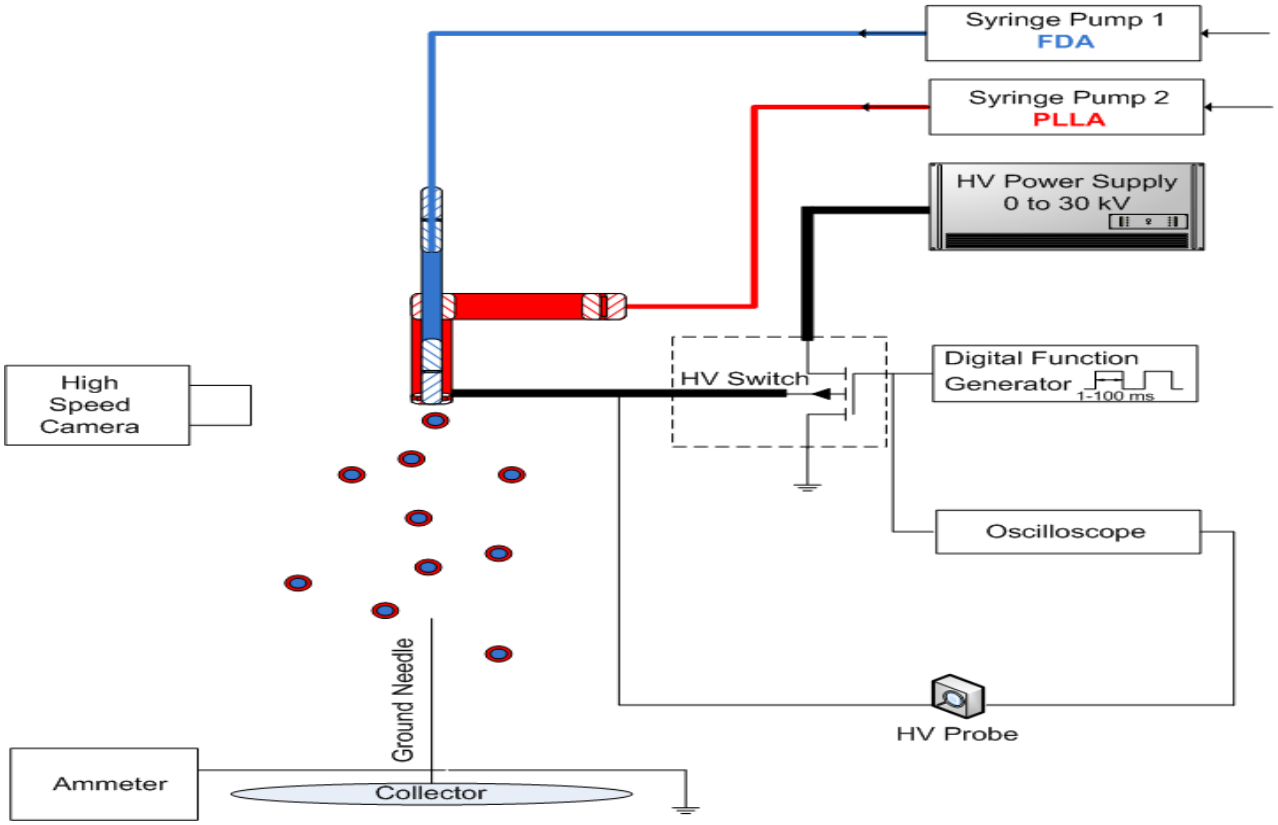
300 mg PLLA dissolved in 5 mL of DCM-Chloroform. Sample was collected in water + PVA at 1 mL/hr and 12.5 kV.

Area III. Enabling Technologies for Membrane Synthesis

TECHNICAL ACCOMPLISHMENTS

Electrospray of Polymer Hybrid Materials

Research Design



A schematic of pulsed dual-capillary electrospray setup.

Area III. Enabling Technologies for Membrane Synthesis

TECHNICAL ACCOMPLISHMENTS

Electrospray of Polymer Hybrid Materials

SCALING LAWS

- For liquids with low viscosity and conductivity [3]:

$$I \sim \left(\frac{\gamma^3 \varepsilon_0 K Q}{\rho}\right)^{1/4}$$

$$d \sim \left(\frac{\rho \varepsilon_0 Q^3}{\gamma K}\right)^{1/6}$$

where

γ: surface tension (N/m)

ε_0: vacuum permittivity ($A^2s^4/(kgm^3)$)

K: electrical conductivity ($A^2s^3/(kgm^3)$)

Q: flow rate (m^3/s)

ρ: density (kg/m^3)

I: emitted current (A)

d: droplet diameter (m)

Area III. Enabling Technology for Membrane Synthesis

TECHNICAL ACCOMPLISHMENTS

Electrospray of Polymer Hybrid Materials

Past Work - Conclusion

	Low	High
Polymer Conc.	No Particles	Particles+Fibers
Feed Flow Rate	Smaller Particles	Larger Particles
Voltage Applied	Larger Particles	Smaller Particles

- Co-solvent process offers an attractive alternative for particles formation.
- The particles became smaller and more spherical with the use of surfactant.

TECHNOLOGY TRANSFER/COLLABORATIONS

- Dr. Eve S. Steigerwalt, Technical Development Manager, Performance Plastics Center, Dana Holding Corporation, Paris, TN
- Dr. O. Thompson Mefford, School of Materials Science and Engineering, Clemson University
- RIT, GM: Visualization of Fuel Cell Water Transport and Performance Characterization Under Freezing Conditions, DOE DE-FG36-07GO17018
- MTU: Hydrogen Education Curriculum Path at Michigan Technological University, DOE DE-FG36-08GO18108
- MTU, State of Michigan: Fuel Cell Water Control System Prototype – Alternative Energy, Michigan Universities Commercialization Initiative (MUCI)
- A USPTO office action citing prior art relevant to the inventors' claims was received in March 2009, and preparation of a response is in progress. United States Patent Application No. 2006 0024583, "Nickel hydroxide impregnated carbon foam electrodes for rechargeable nickel batteries," filed 15 July, 2005, A. Singh, B. C. Cornilsen, M. E. Mullins, and T. N. Rogers (U.S. Provisional Application No. 60/588,108 filed July 15, 2004).
- Dr. Andres Hurtado, M.D., Kennedy Krieger Institute, Johns Hopkins University - In this project we have been developing new classes of materials that take advantage of the flexible properties of plastics combined with some of the chemical advantages of ceramic (inorganic) materials. The original object was to create better fuel cell membranes; however, an unexpected twist in our studies is that the new materials we developed were found to be excellent for supporting tissue growth.
- Technology and Commercialization Consultant: Mr. Ralph J. Brodd, President of Broddarp of Nevada, Inc.

FUTURE WORK

Area I. Heat and Water Management

- For carbon nanotube/carbon black/synthetic graphite /polypropylene materials for fuel cell bipolar plates
  - Determine through plane and in plane thermal conductivity using nanoflash method and develop models (FY 09, FY10)
  - Determine tensile and flexural properties and develop models (FY09, FY10)
- Four (4) publications in progress on contact angle measurements and compression experiments
- Complete contact angle measurements for current GDL set
- Drop size dependency
- Temperature dependencies
- Fabricate sealed, second-level thermostat for measuring contact angles in humid, hydrogen environment for range of temperatures
- Complete compression imaging for current GDL set

Area II. Development of New Electrode Materials

- Include silica nanoparticles during the carbon foaming process that can be etched out afterward with hydrofluoric acid or high pH solutions. This step will increase the porosity and the surface area of the materials. Our goal will be to produce a series of foams with varying pores sizes and morphologies for electrode applications.
- Produce boron-doped mesoporous/microporous carbon foams that have more oxidation-resistance and higher mechanical strength than bare carbon foams.
- Refine synthesis techniques to construct a pseudomorphic overlayer catalyst on the surface area of a carbon foam support.

Area III. Enabling Technologies for Membrane Synthesis

- Complete electrospinning studies on the control of nano-fiber diameter
- Utilize coaxial needle to produce dual materials fibers
- Complete development of an experimental setup for pulsed single-capillary electrospray
- Study the effect of pulse width, frequency, and amplitude on the particle size distribution produced
- Utilize the coaxial needle system for core-shell particle production
- Optimize the capsule size distribution and shell thickness
- Develop improved scaling laws for the particle and fiber production process

PROJECT SUMMARY

I. Heat and Water Management

Relevance:

- Develop improved bipolar plate materials
- Improve GDL performance and durability

Approach:

- Use combination of carbon fillers in thermoplastic to achieve desired fuel cell bipolar plate conductivity
- Measure contact angles in hydrogen and air to determine GDL wettability at fuel cell operating conditions

Technical Accomplishments:

- **Fuel Cell Bipolar Plates**: For 2.5 wt% Akzo Nobel Ketjenblack EC-600 JD carbon black/ 65 wt% Asbury Carbons Thermocarb TC-300 synthetic graphite particles/ 6 wt% Hyperion carbon nanotubes/ 26.5 wt% Dow homopolymer polypropylene H7012-35RN (DOE target of 100 S/cm and > 20 W/m·K)
  - Electrical Conductivity: Achieved 91 S/cm
  - Thermal Conductivity Achieved 24 W/m·K
- Fabricated environmental chamber; measured contact angles and characterized error; data indicates decrease in contact angle as temperature increases

Technology Transfer/Collaborations: Active partnerships with Dana, RIT and GM (DOE DE-FR36-07GO17018), Hydrogen Education Curriculum Path at Michigan Technological University (DOE DE-FG36-08GO18108), and State of Michigan: Fuel Cell water Control System Prototype- Alternative Energy, Michigan Universities Commercialization Initiative (MUCI)

Future Work:

- For carbon nanotube/carbon black/synthetic graphite /polypropylene materials for fuel cell bipolar plates
- Determine through plane and in plane thermal conductivity using nanoflash method and develop models
- Determine tensile and flexural properties and develop models
- Complete contact angle measurements for current GDL set for drop size and temperature dependencies
- Fabricate sealed, second level thermostat for measuring contact angles in humid, hydrogen environment for range of temperatures
- Complete compression imaging for current DGL set

PROJECT SUMMARY

II. Development of New Electrode Materials

Relevance: Porous graphitic carbon foams may serve as lightweight current collectors and mechanical supports for electrodes and catalysts. Pseudomorphic overlayer bimetallic catalysts deposited on a carbon surface may reduce or replace Platinum use in PEM fuel cells.

Approach:

- Synthesize carbon foams having varying levels of porosity by pyrolizing PAN-based precursors
- Fabricate and test carbon-supported electrodes and catalysts
- Construct overlayer catalysts tailored to have desired catalytic activity

Technical Accomplishment and Progress:

- Investigated electrode applications for porous, graphitic carbon foams
- Developed procedures for selecting and synthesizing bimetallic overlayer catalysts, on an alumina support, having desired catalytic properties

Technology Transfer/Collaborations:

- A USPTO office action citing prior art relevant to the MTU inventors' claims was received in March 2009, and preparation of a response is in progress. United States Patent Application No. 2006 0024583, "Nickel hydroxide impregnated carbon foam electrodes for rechargeable nickel batteries," filed 15 July, 2005, A. Singh, B. C. Cornilsen, M. E. Mullins, and T. N. Rogers (U.S. Provisional Application No. 60/588,108 filed July 15, 2004)
- Collaborating on carbon foam synthesis procedures with Dr. O. Thompson Mefford, Clemson University, School of Materials Science and Engineering
- Technology and Commercialization Consultant: Mr. Ralph J. Brodd, President of Broddarp of Nevada, Inc.

Proposed Future Research:

- Synthesize carbon foams with increased porosity and surface area
- Refine catalyst synthesis procedure to construct pseudomorphic overlayer catalysts on a graphitic carbon support

PROJECT SUMMARY

III. Enabling Technologies for Membrane Synthesis

Relevance: Develop new methods to produce nanostructured polymer hybrid membranes that will be needed for the next generation of PEMs.
Approach: Novel electrospinning and pulsed electrospraying devices have been developed to address this problem.
Technical Accomplishments and Progress: We have completed constructing and testing of the electrospray/electrospinning equipment for simple single polymer systems. We are in the process of adapting the systems to synthesize complex hybrid polymer/inorganic nano and microstructures in a membrane configuration.
Technology Transfer/Collaborations: The new fiber materials developed have been transferred to applications in the biomedical area in conjunction with Johns Hopkins University.
Proposed Future Research: To optimize the core-shell fiber and particle hybrid materials and their processing conditions in order to control the diameter, shell thickness, and materials employed. Future tests will be done with polymer shell materials with an inorganic core. Selection of paradigm materials relevant to PEM membranes will be employed in future work.

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Project ID#: fcp_08_king