

DE-FG02-04ER63821
Michigan Technological University

Project Title: Michigan Technological Center for Nanostructured and Lightweight Materials in the Department of Chemical Engineering (Phase II)

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Task 1 - Lightweight, Thermally Conductive Bipolar Plates for Improved Thermal Management in Fuel Cells (J. King and J. Keith)

Fundamental and applied research needs to be done to improve the potential for fuel cells to be used in stationary and transportation applications. The goal of this project is to develop lightweight composite materials to be used for bipolar plates within a fuel cell. Bipolar plates require high thermal and electrical conductivity, low hydrogen permeability, and good dimensional stability. On this project, Drs. King and Keith are developing new recyclable thermoplastic based materials using a liquid crystal polymer containing several different carbon fillers (carbon black, synthetic graphite, and carbon fiber) to meet the pertinent transport properties required for bipolar plates.

This seed project allowed Drs. King and Keith to pursue additional external funding. An NSF GOALI proposal was awarded for \$311,950 from May 1, 2005 until April 30, 2009 with Dana Corporation as industrial partner. Furthermore, this project has partially supported the research of three doctoral graduate students (Michael Miller, Rodwick Barton, and Rebecca Hauser), one MS student (Troy Tambling), and 50 undergraduate students. Twenty peer-reviewed publications have also been published, are in press, or are currently in review. These papers are listed below:

J. M. Keith, J. A. King, I. Miskioglu, and S. C. Roache, "Tensile Modulus Modeling of Carbon Filled Liquid Crystal Polymer Composites," *Polymer Composites*, in review.

R. A. Hauser, J. A. King, R. M. Pagel, and J. M. Keith, "Effects of Carbon Fillers on the Thermal Conductivity of Highly Filled Liquid Crystal Polymer Based Resins" *Journal of Applied Polymer Science*, in press.

J. A. King, T. M. Tambling, F. A. Morrison, J. M. Keith, A. J. Cole, and Rachel M. Pagel, "Synergistic Effects of Carbon Fillers on Rheology of Highly Filled Liquid Crystal Polymer Resins," *Journal of Applied Polymer Science*, in press.

J. A. King, J. M. Keith, O. L. Glenn, I. Miskioglu, A. J. Cole, R. M. Pagel, "Synergistic Effects of Carbon Fillers on Tensile and Flexural Properties in Liquid Crystal Polymer Based Resins," *Journal of Applied Polymer Science*, accepted, in press.

J. A. King, T. M. Tambling, J. M. Keith, A. J. Cole, and F. A. Morrison, "Synergistic Effects of Multiple Carbon Fillers on the Rheology of Liquid Crystal Polymer Based Resins," *Polymer Composites*, accepted, in press.

- J.A. King, R. L. Barton, R. A. Hauser, and J. M. Keith, "Synergistic Effects of Carbon Fillers in Electrically and Thermally Conductive Liquid Crystal Polymer Based Resins," *Polymer Composites*, in press.
- J.A. King, R. A. Hauser, A. M. Tomson, I. M. Wescoat, and J. M. Keith, "Synergistic Effects of Carbon Fillers in Thermally Conductive Liquid Crystal Polymer Based Resins," *Journal of Composite Materials*, **42**, 91-107 (2008).
- J. M. Keith, J. A. King, P W. Grant, A. J. Cole, B. M. Klett, and I. Miskioglu, "Tensile Properties of Carbon Filled Liquid Crystal Polymer Composites," *Polymer Composites*, **29**, 15-21 (2008).
- R. L. Barton, J. M. Keith, and J. A. King, "Development and Modeling of Electrically Conductive Carbon Filled Liquid Crystal Polymer Composites for Fuel Cell Bipolar Plate Applications," *Journal of New Materials for Electrochemical Systems*, **10**, 225-229 (2007).
- R. L. Barton, J. M. Keith, and J. A. King, "Electrical Conductivity Model Evaluation for Carbon Filled Liquid Crystal Polymer Composites," *Journal of Applied Polymer Science*, **106**, 2456-2462 (2007).
- E. Kunen, J. M. Keith, P. W. Grant, J. A. King, and F. A. Morrison, "FEM Calculations of Capillary Rheometer Flow for Carbon-Filled Liquid Crystal Polymer Composites," *Journal of Applied Polymer Science*, **106**, 433-438 (2007).
- J. M. Keith, J. A. King, K. M. Lenhart, and B. Zimny, "Thermal Conductivity Models for Carbon / Liquid Crystal Polymer Composites," *Journal of Applied Polymer Science*, **105**, 3309-3316 (2007).
- J. A. King, J. M. Keith, R. C. Smith, and F. A. Morrison, "Electrical Conductivity and Rheology of Carbon Fiber / Liquid Crystal Polymer Composites," *Polymer Composites*, **28**, 168-174 (2007).
- J. M. Keith, J. A. King, M. G. Miller, and A. M. Tomson, "Thermal Conductivity of Carbon Fiber / Liquid Crystal Polymer Composites," *Journal of Applied Polymer Science*, **102**, 5456-5462 (2006).
- M. G. Miller, J. M. Keith, J. A. King, B. J. Edwards, N. Klinkenberg, and D. A. Schiraldi, "Measuring Thermal Conductivities of Anisotropic Synthetic Graphite-Liquid Crystal Polymer Composites," *Polymer Composites*, **27**, 388-394 (2006).
- J. M. Keith, J. A. King, and R. L. Barton, "Electrical Conductivity Modeling of Carbon-Filled Liquid Crystal Polymer Composites," *Journal of Applied Polymer Science*, **102**, 3293-3300 (2006).
- J. A. King, F. Morrison, J. M. Keith, M. G. Miller, R. C. Smith, M. Cruz, A. M. Neuhalfen, and R. L. Barton, "Electrical Conductivity and Rheology of Carbon-Filled Liquid Crystal Polymer Composites," *Journal of Applied Polymer Science*, **101**, 2680-2688 (2006).
- M. G. Miller, J. M. Keith, J. A. King, R. A. Hauser, and A. M. Moran, "Comparison of Guarded Heat Flow and Transient Plane Source Methods for Carbon-Filled Nylon 6,6 Composites: Experiments and Modeling," *Journal of Applied Polymer Science*, **99**, 2144-2151 (2006).
- J. M. Keith, C. D. Hingst, M. G. Miller, J. A. King, and R. A. Hauser, "Measuring and Predicting In-Plane Thermal Conductivity of Carbon-Filled Nylon 6,6 Polymer Composites," *Polymer Composites*, **27**, 1-7 (2006).

J. A. King, M. G. Miller, R.L. Barton, J. M. Keith, R. A. Hauser, K. Peterson, and L. L. Sutter, "Thermal and Electrical Conductivity of Carbon-Filled Liquid Crystal Polymer Composites," *Journal of Applied Polymer Science*, **99**, 1552-1558 (2006).

The following conference papers were presented:

J. M. Keith, D. Chmielewski, H. Scott Fogler, and V. Thomas, "CACHE Modules on Energy in the Curriculum: Fuel Cells," 2008 ASEE Annual Meeting, Pittsburgh, PA.

H. Scott Fogler, J. M. Keith (speaker), and D. J. Chmielewski, "New CACHE Initiatives in Fuel Cell Modules," 2007 AIChE Annual Meeting, Salt Lake City, UT.

J. M. Keith (speaker), J. A. King, F. A. Morrison, T. M. Tambling, E. Kunen, R. C. Smith, A. J. Cole, P. W. Grant, "Rheological Measurements and Modeling of Carbon-Filled Liquid Crystal Polymer Composites," 2007 AIChE Annual Meeting, Salt Lake City, UT.

J. M. Keith (speaker), F. A. Morrison, and J. A. King, "FEM using COMSOL: Applications for Fuel Cells," 2007 AIChE Annual Meeting, Salt Lake City, UT.

J. M. Keith (speaker), "Engineering Education in Alternative Energy and Transport Phenomena," 2007 Chemical Engineering Summer School, Washington State University.

J. M. Keith (speaker), F. A. Morrison, and J. A. King, "Finite Element Method for Fuel Cell Fundamentals in the Undergraduate Transport Courses," 2007 ASEE Annual Meeting, Honolulu, HI.

R. A. Hauser (speaker), J. A. King, J. M. Keith, R. L. Barton, and M. G. Miller, "Thermal and Electrical Conductivity of Carbon/Liquid Crystal Polymer Composites For Fuel Cell Bipolar Plates," Society of Plastics Engineers Annual Technical Conference Proceedings, May 2007, Cincinnati, OH.

A. M. Tomson (speaker), J. M. Keith, J. A. King, and M. G. Miller, "Thermal Conductivity of Carbon Fiber / Liquid Crystal Polymer Composites," 2006 AIChE Annual Meeting, San Francisco, CA.

R. L. Barton (speaker), J. M. Keith, and J. A. King, "Development and Modeling of Electrically Conductive Carbon Filled Liquid Crystal Polymer Composites for Fuel Cell Bipolar Plate Applications," 2006 New Materials for Electrochemical Systems 6th International Symposium, Montreal, Canada.

J. M. Keith (speaker), K. C. Opella, M. G. Miller, J. A. King, G. D. Gwaltney, C. A. Green, J. S. Meldrum, and S. A. Bradley, "Engineering Education in Alternative Energy," 2006 ASEE Annual Meeting, Chicago, IL.

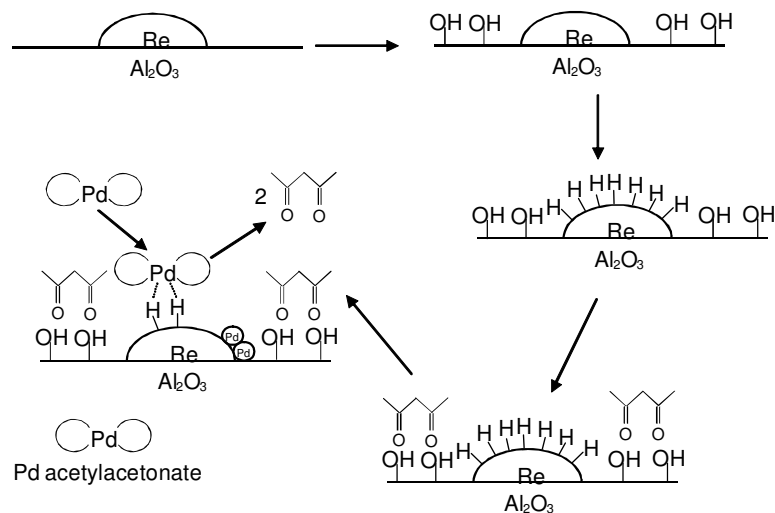
M. G. Miller (speaker), J. M. Keith, and J. A. King, "Graphite-Filled Liquid Crystal Polymer Composites for Fuel Cell Bipolar Plates," 2005 AIChE Annual Meeting, Cincinnati, OH.

Task 2 – Exploration of pseudomorphic nanoscale overlayer bimetallic catalysts (J. Holles)

Summary of Work Completed: During the reporting period, work has continued on developing methods to synthesize pseudomorphic overlayer bimetallic catalysts. The goal of this work is to develop high surface area supported metal catalysts with an overlayer of one metal on top of another metal. When the overlayer thickness is approximately one monolayer, the catalyst is expected to demonstrate electronic properties different from either of the two base metals.

While the benefits of this system has been demonstrated computationally and via single crystal high vacuum studies, the true demonstration of its usefulness requires successful application to high surface area supported metal catalysts.

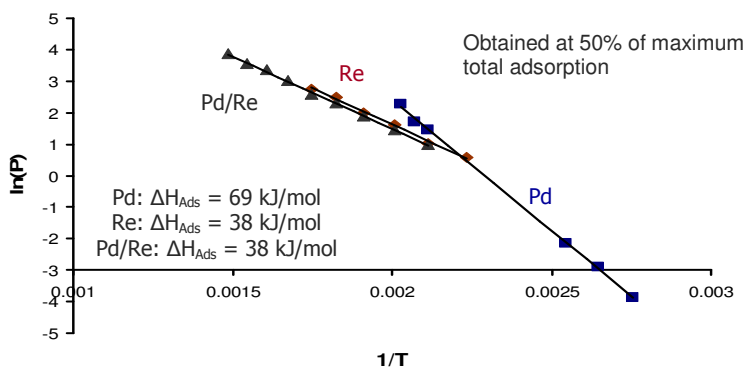
As a demonstration of the proof of principle, we have focused on a Pd overlayer on Re system for hydrogenation type reactions. To understand this system, we have synthesized and



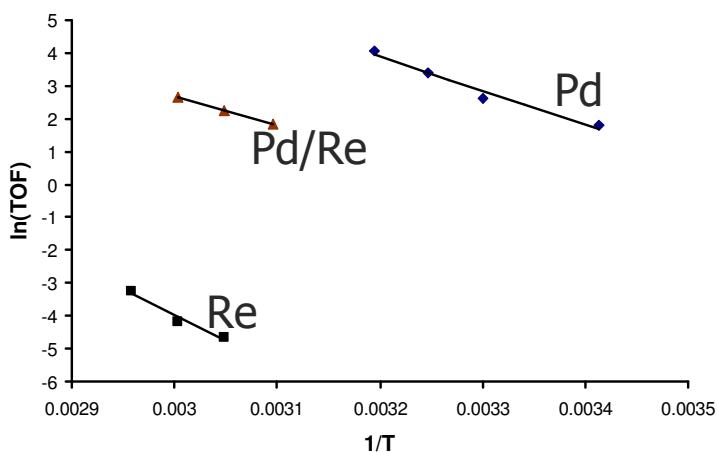
characterized pure Pd, pure Re, and a Pd/Re catalyst. The catalyst synthesis first deposits the base metal particle using any available technique. Next, the support surface is passivated by saturating it with hydroxyl groups. Then, a monolayer of hydrogen is deposited on the surface. Finally, the Pd precursor is introduced, reacts with the surface hydrogen and deposits the Pd. The main advantage of this method is that the surface reaction limits the overlayer deposition to one atomic layer.

All three catalysts were then characterized using hydrogen chemisorption and the ethylene hydrogenation reaction.

Hydrogen chemisorption studies were conducted by obtaining total and reversible isotherms. From this data, the active site density for each catalyst was determined and used to calculate turnover frequencies for the reactivity studies (below). Additionally, if the isotherms are



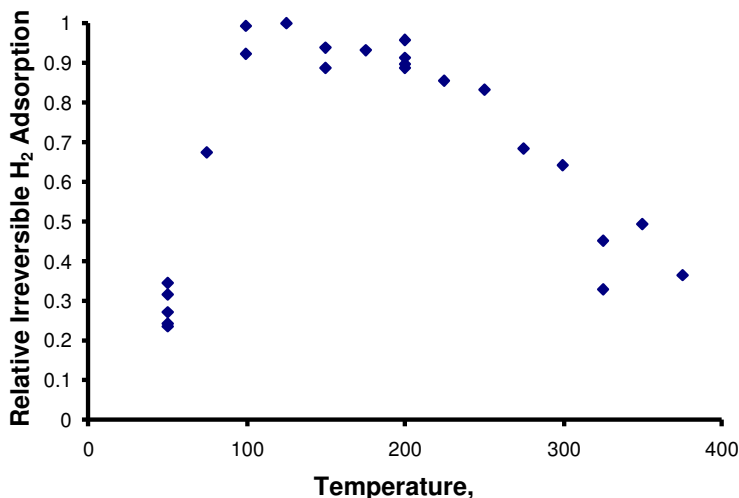
obtained as a function of temperature, the Clausius-Clapeyron equation can be used to calculate the isothermic heats of adsorption for the catalysts. The calculated heats of adsorption are clearly different for the Pd and Re base catalysts with the Pd/Re very similar to the Re catalysts. The Pd value also compares well with the value calculated by first principles quantum techniques.



The ethylene hydrogenation reaction was also used to compare the three catalysts. As expected, the Re was very unreactive. While the Pd/Re catalyst showed improved activity, it was still below the pure

Pd catalyst. Activation energies for the Pd/Re and pure Pd were 85 and 70 kJ/mol, respectively. The Re value was much lower at 30 kJ/mol. This indicates, together with the heat of adsorption data, that the Pd/Re catalyst is behaving as pure Pd dispersed on the Re. However, this result is not surprising considering that the Pd loading, by quantitative analysis, was only 2% of the loading necessary for a complete monolayer. Since the Pd loading was less than expected, we are focusing further work on improving the deposition technique.

Future Work: To further improve the Pd deposition, we have examined the hydrogen chemisorption. As mentioned in the synthesis procedure, chemisorbed hydrogen is the key to



depositing the overlayer on the base metal. As can be seen from the figure below, the quantity of hydrogen adsorbed is a function of temperature. The amount is initially low at room temperature, reaches a maximum around 100°C, and then decreases as temperature further increases. Thus future efforts will focus on performing catalyst synthesis around 100°C to maximize hydrogen coverage and consequentially, the ability for Pd to deposit.

Once the synthesis technique has been finalized, the the Pd/Re catalyst will be characterized as above using hydrogen chemisorption and reactivity studies. In addition, TEM using EDS will be used to demonstrate deposition of Pd on Re and not in separate particles. X-ray absorption spectroscopy (XAS) and Auger electron spectroscopy (AES) will also be used for characterization. Interatomic distances and coordination numbers from XAS will provide evidence for monolayer formation. Since AES is very sensitive to surface atoms, it will also indicate overlayer coverage. By sputtering the sample, AES can also provide depth profiles of the elements to demonstrate overlayer formation.

Finally, we are working to expand the use of these catalysts to other systems. Our target of choice is the Aqueous Phase Reforming (APR) of lactose to carbon monoxide and hydrogen gas (synthesis gas). Synthesis gas has a long history in the chemical industry and processes to convert it to many industrially important chemicals are already well established. Lactose is available as a byproduct from the cheese industry. Currently, the majority of this lactose is simply disposed of. Since the lactose is produced renewably, it is a carbon neutral source. In addition, the ability to convert a waste stream to a valuable product further emphasizes the potential for this process.

In laboratory scale batch reactor experiments, we have already shown that lactose can be converted to CO/H₂ via APR using a Pt catalyst. Current work by other researchers on APR has indicated that Pt overlayer on a Ni or Co catalysts would be ideal for this reaction. Therefore, we are currently synthesizing and characterizing Pt, Ni, and Co base catalysts along with a Pt/Ni

catalyst. The advantage of the Ni system is that it shows consistent hydrogen adsorption as a function of temperature. Thus, compared to the Re system, synthesis should be simpler.

Papers/Publications: "Directed Synthesis and Characterization of A Supported Bimetallic Overlay Catalyst," by M.P. Latusek and J.H. Holles presented to the AIChE Annual Meeting, November 2007, Salt Lake City, UT.

Students Supported: Michael Latusek (Ph.D. Student) and Robert Horner (NSF IGERT REU student).

Proposed Collaborative Efforts: Brookhaven National Laboratory (XAS studies), Virent Energy Systems, Madison WI (APR of lactose), NREL (APR of lactose).

Task III: Hybrid inorganic/organic polymer nanocomposites (M. Mullins and P. Heiden)

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 fuel cell electrolyte membranes that operate at slightly higher materials, and a new class of dielectric materials. Novel nanocomposite hybrid films have been prepared by a novel reactive method and the variables that affect the morphology and stability of the organic and inorganic phases have been investigated. The initial films possessed inorganic domains that were well dispersed in the organic film. Using the initial approach the inorganic domains ranged from 100 nm up to 450 nm depending on variables such as temperature, time, and catalysis. Further investigation and manipulation of these variables has been carried out and it has been shown that these inorganic domains can be reduced to a bimodal distribution of 30 ± 5 nm and 3-5 nm. The method used is flexible and permits the organic matrix composition to be varied significantly within the primary limitation being that one component must possess a large number of polar functional groups. Current work is focusing on understanding the process and investigating applications.

This work is still ongoing; however, an unexpected twist in our studies is that the new materials developed were found to be excellent for supporting tissue growth. In particular, we have used these materials to spin nanometer scale fibers that have shown a remarkable ability to guide and promote nerve cell growth. We believe that this discovery may be an important step towards the repair of central nervous system (spinal cord) injuries; which now leaves several thousand civilians and military personal crippled each year. A new collaboration in using these materials is now underway with the Johns Hopkins Medical School.

This project was seeded to allow the researchers to develop some expertise in the area and to permit the work to be continued with external funding. The project has progressed well. To date the results have been presented at two national meetings and produced two publications, with a third publication now in preparation. Proposals to obtain external funding have been developed and submitted this past year, with likely funding sources being DOE or DOD.

Publications

KenHeng See, Michael E. Mullins, and Patricia A. Heiden. A Reactive Core-Shell Nanoparticle Approach to Prepare Hybrid Nanocomposites: Effects of processing variables. *Nanotechnology*, Volume 16, Number 9, September 2005, pp. 1950-1959(10).

Presentations

“Morphology of Al₂O₃-Polyurethane-g-(Gelatin-g-PMMA) Nanohybrid Films prepared from Core-Shell Nanoparticles.” Ken Heng See, Patricia A. Heiden, and Michael E. Mullins. *Material Research Society*, Boston, MA , November 29-December 3, 2004. Presented by Mr. Ken Heng See.

“Reactive Core-Shell Nanoparticles: An Innovative Approach to Hybrid Nanocomposites.” Department of Chemistry Seminar, Michigan Technological University. September 2, 2005

Task 4 - Carbonaceous Monolithic Electrodes for Fuel Cells and Rechargeable Batteries (Rogers, Cornilsen, Chye)

Summary

In the past year, cycle-life tests of the MTU Ni-carbon foam cathode (patent pending) have been conducted in flooded cells. (The monolithic graphitic foams are impregnated with nickel oxyhydroxide active mass.) These have demonstrated competitive capacity retention and high cycle life at a variety of discharge rates required by consumer electronics and power tools. Replacement of the nickel metal current collector in Ni-MH (nickel-metal hydride) batteries with a lighter, cheaper, and readily available material, carbon foam, is expected to cut the weight of a rechargeable Ni-MH battery by up to 25%.

Proof-of-concept charge/discharge studies have demonstrated the potential of the MTU cathode for high rate discharge and have lead to funding from a Michigan Universities Commercialization Initiative (MUCI). Prototype coin cells with the MTU Ni-C foam cathode and an ultracapacitor anode will be constructed and evaluated. This anode is expected to make higher power cells possible. This cathode/anode combination will improve voltage and discharge rates making these more competitive with the newer Li-ion cells. Batteries and ultracapacitors based on the MTU technology would be inherently safer, without the fire hazard associated with Li-ion batteries.

Project Results

The following project highlights are a direct result of funding by the U.S. DOE Michigan Technology Center (MTC):

- Charged and discharged foam electrodes for 2000 to 3000 consecutive cycles, in laboratory flooded cells, which surpasses the reliability standard of 300 to 600 cycles for consumer electronics applications.
- Maintained stable capacities for more than 2000 cycles at a 1C-rate.
- Established optimal methodologies for active mass deposition and formation/activation.

- Performed x-ray diffraction and infrared spectroscopic analyses of electrodes as a function of state-of-charge to characterize the layer structure of the active mass. This is related to the stoichiometric composition.
- Conducted inelastic neutron scattering (INS) tests at the Intense Pulsed Neutron Source (IPNS) at Argonne National Laboratory (ANL) using the High-Resolution Medium-Energy Chopper Spectrometer (HRMECS) to study structural change as a function of state-of-charge of the active mass. Precursor structures also compared. Unique information about the proton positions is obtained from INS data, information not obtainable with other methods.
- Received funding from the Michigan Space Grant Consortium (MSGC) for a complementary effort, "Structural Characterization of Nickel Electrode Active Mass using Carbon Foam Impregnation; a Light Weight Ni-H₂ Battery Cathode." INS experiments at ANL were conducted.
- Acquired a companion project in January 2008, funded by the Michigan Universities Commercialization Initiative (MUCI), which will ascertain whether the MTU carbon foam electrodes are commercially viable. The MUCI proposal review panel consisted of technical experts, businessmen, and venture capitalists, external to MTU, who collectively viewed the MTU technology favorably.
- A U.S. Patent Application A1232707, Serial No. 11/182,891, was filed in 15 July 2005, "Nickel Hydroxide Impregnated Carbon Foam Electrodes for Rechargeable Nickel Batteries." Our earlier laboratory findings led to MTC funding for further scientific study, which in turn led to the pending patent on the electrode design. We are awaiting an office action by the USPO within the next 6 months, which we fully expect will lead to an approved patent.
- The MTC project has supported a Ph.D. student (M. Chye).
- An ACS poster was presented: Cornilsen, B.C., M. Ning, C. Baiz, T.N. Rogers, M.B. Chye, and A.I. Kolesnikov, "Inelastic Neutron Scattering Study of Hydrogen Positions in Nickel Hydroxide Battery Electrode Materials," Paper #28972, Poster presented at the ACS 37th Great Lakes Regional Meeting, Milwaukee, WI, May 31 - June 2, 2006.
- Poster presented: M. Ning and B. C. Cornilsen, "The Structural Relationship between α - and β -Ni(OH)₂," 106th Annual Meeting of the American Ceramic Society, Indianapolis, IN, April 19-21, 2004.

Future Work

At this stage of technology development, the scientific and technical challenges for commercialization are threefold:

- Design, fabricate, and test coin cell batteries directly comparable with commercial batteries;
- Optimize the composition and performance of the MTU technology by fabricating prototype electrodes and conducting cyclic charge/discharge experiments; and
- Study the Ni oxyhydroxide structure within the electrode at various states-of-charge, to optimize structure and electrochemical properties.

Future MTC Phase III funding for this project will complement the parallel MUCI commercialization effort by addressing (and optimizing) many of the performance and scientific issues that affect the technology's market acceptance.

Task 5 - Movement and Freeze of Water in Fuel Cell Electrodes (J. Allen)

A fundamental research project to characterize the transport of water within the porous electrodes of fuel cells has been started. This work is “enabling” research to provide data on the nature of capillary flow in unsaturated, anisotropic porous layers. A corollary to the water movement in the porous electrodes is the proposed fundamental research project on the freezing of water within the fuel cell electrodes. One of the primary factors influencing water movement in the porous layers of fuel cells is the contact angle; that is, the surface angle at the gas-liquid-solid intersection. During the last year a unique, automated analysis of contact angles on fuel cell diffusion media has been developed. The data obtained will be critical to the pursuit of more comprehensive research grants from government and industry sponsors.

Current methods for measuring contact angles include but are not limited to using a goniometer and finding the tangent line at the contact point either manually or through the use of a computer or taking an image of the drop profile and fitting the interface using a polynomial fit or a portion of a circle. These methods are particularly inaccurate when the solid substrate is not smooth as is the case with the fuel cell components. A more accurate method is to utilize the non-linear description of a drop surface to fit the imaged profile of the water drop. This required the development of a uniform backlight. Mr. Carlos Ubinas, an undergraduate summer research student from Polytechnic Institute of Puerto Rico, was employed to develop a Koehler Illumination source for the contact angle measurement project.

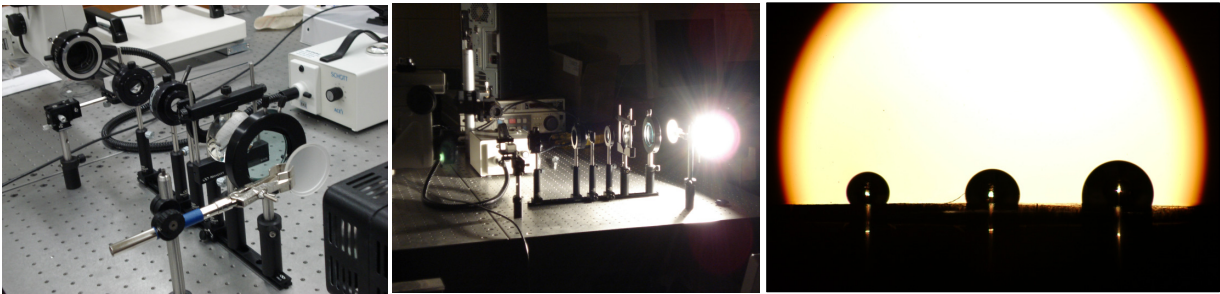


Figure 1. Koehler illumination setup and water drops on a gas diffusion layer.

A program, written in MATLAB, fits a drop interface to the Laplace-Young equation. The program requires an image of a drop profile with sufficient lighting and some properties of the fluid, such as density and surface tension. The program will then output the contact angle along with other information such as the bond number. There are four main steps in the program; Image Processing, Edge Detection, Curve Fitting, and Error Minimization. The original image can be RGB or gray scale and the image processing step converts the image into black and white. The black and white image is passed through a high pass filter which is then passed on to the edge detection step. The edge detection step scans the image pixel by pixel and stores the data points where the image changes from black to white or white to black. The stored data represents the drop interface which is then passed on to the curve fitting step. The curve fitting step fits the drop interface data to a polynomial curve. The polynomial curve fit is used in the error

minimization step. The error minimization step calculates the error between the polynomial curve and the Laplace-Young curve at every point for a given curvature. The curvature of the Laplace-Young curve is the variable that can be changed in order to minimize the error between the Laplace-Young curve and the polynomial curve. Once the error is minimized, the contact

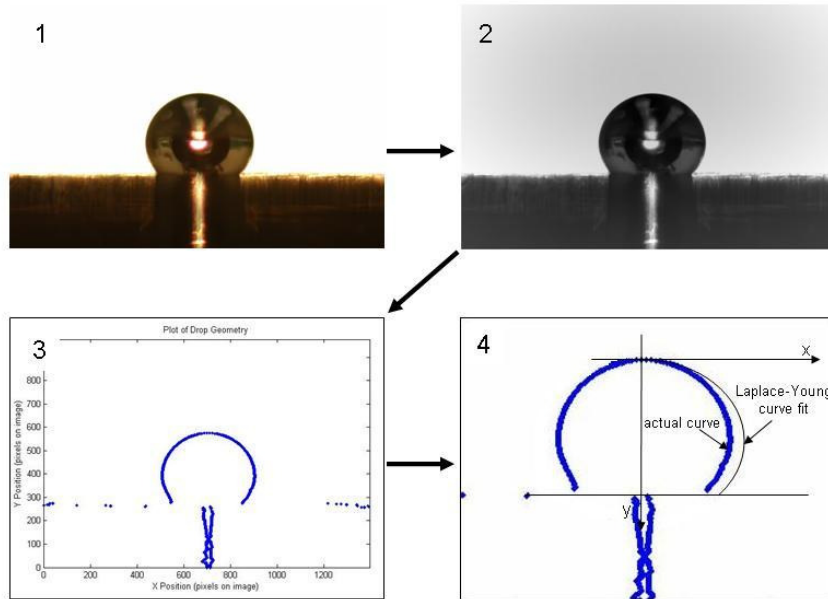


Figure 2. Sequence of steps to acquire drop image, detect drop edge and fit Laplace curve to determine the contact angle.

angle can be found directly by knowing the distance from the drop apex to the substrate surface or z-plane position.

Students Supported

A MS Graduate Student, Mr. Russ Stacy, was supported during the completion of this project. In addition, Mr. Carlos Ubinas, an undergraduate student from Polytechnic Institute of Puerto Rico, was supported for a summer internship to develop the lighting technique.

Publications and Presentations

The lighting and measurement technique will be presented by Mr. Russ Stacy at the 19th International Symposium on Transport Phenomena (ISTP-19) in Reykjavík, Iceland in August 2008. Contact angle measurement data with fuel cell components collected via this technique will be presented as a poster at the 2008 Gordon Conference on Fuel Cells in Rhode Island in July 2008.