Observation of Magnetic Effects in Oxygen Reduction Electrocatalysis

A Thesis Presented

by

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to

The Department of Chemistry and Chemical Biology

in partial fulfillment of the requirements for the degree of Masters of Science

in the field of

Chemistry

Northeastern University
Boston, Massachusetts
August, 2009
OBSERVATION OF MAGNETIC EFFECTS IN OXYGEN REDUCTION ELECTROCATALYSIS

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ABSTRACT OF THESIS
Submitted in partial fulfillment of the requirements for the degree of Master of Science in Chemistry in the Graduate school of Arts and Science of Northeastern University, August 2009
ABSTRACT

Fuel cell polarization curves of a novel platinum alloy (Pt$_3$Co/C) catalyst displayed a unique mass transport signature unidentifiable with general fuel cell operation. Physicochemical characterization utilizing various methods was carried out to probe the properties of the catalysts and to compare with standard commercially available catalysts from ETEK. Magnetic measurements were also used to determine the properties and type of magnetic character the catalysts hold.

The intrinsic magnetic characteristics of the electrocatalyst in the fuel cell become apparent at relatively low operating pressures of oxygen; this is mainly due to the magnetic forces affected by concentration gradients. Other electrochemical experiments reveal the nature of the catalyst as one having high concentration of cobalt on the surface in the form of islands, which was probed with alkaline based cyclic voltammetry. SEM imagery confirms the particle sizes determined by XRD Scherrer particle size estimation. There is indication from the elemental analysis and XRD that the commercially available Pt$_3$Co/C electrocatalysts available from ETEK alloys are not truly the 3:1 Pt:Co atomic ratio they claim to be due to minor sublimation of cobalt and subsequent formation of cobalt clusters in the carbon support at high temperatures.

Future research is suggested in the form of new synthetic techniques and trials to achieve a more hard magnetic material which will further enhance the mass transport properties of the electrocatalyst. Other electrochemical experiments are suggested to determine the magnetic effects of other charged species relative to the oxygen reduction reaction (ORR).
ACKNOWLEDGEMENTS

I would like to thank my advisor, Dr. Sanjeev Mukerjee, for his encouragement, guidance and funding of this project while I was a graduate student in his research laboratory Laboratory for Electrochemically Advanced Power (LEAP) and the Northeastern University Center for Renewable Energy Technology (NUCRET).

I am very grateful to Dr. Sanjeev Mukerjee, Dr. David E. Budil, and Dr. Eugene Smotkin for their valuable time in review and advice in writing this thesis.

I would like to thank all of my research group members past and present as well as research colleagues. Special acknowledgement is given to Dr. Nazih Hakim, Dr. Lajos Gancs, and others who extended their assistance in this research and other associated research projects at Northeastern University. Many thanks to Chris Allen, Matthew Trahan, Thomas Arruda, Nagappan Ramaswamy, and Qinqgang He for their assistance with experiments and fruitful scientific discussion. To former students Dr. Joe Ziegelbauer, Dr. Wen Wen, Dr. Vivek Srinivasamurthi, and Dr. Craig Urian for their example.

My beloved parents Jane and Timothy Hult deserve a lot of thanks for supporting me in my academic and life pursuits. I thank them for their love, encouragement and understanding during this time in my life! I also thank my friends and my church for their support and prayers throughout the years.
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LIST OF ABBREVIATIONS

EIS – electrochemical impedance spectroscopy
EDX – energy dispersive x-ray
FC – field cooling
fcc – face centered cubic
fct – face centered tetragonal
HOR – hydrogen oxidation reaction
ICP-MS – inductively coupled plasma mass spectrometry
MEA – membrane electrode assembly
MHD – magneto hydrodynamics
NHE – normal hydrogen electrode
ORR – oxygen reduction reaction
PEM – polymer electrolyte membrane
RDE – rotating disk electrode
RRDE – rotating ring disk electrode
SEM – scanning electron microscopy
SQUID – superconducting quantum interference device
TFMSA – trifluormethane sulfonic acid, triflic acid
UPD – underpotential deposition
XRD – x-ray diffraction
ZFC – zero field cooling
/C – carbon coated
CHAPTER 1

Introduction to Fuel Cell Oxygen Reduction Electrocatalysis

In today’s advanced stages in the development of portable and low-temperature fuel cells, cost reduction has become the one of the primary focal points in order to commercialize fuel cells towards becoming competitive with the current battery technologies and internal combustion engines utilizing fossil fuels. One major cost factor is the Pt content of the electrode assembly that enables the most preferred cathode reaction, the oxygen reduction reaction (ORR), to occur with an acceptable rate. The ORR is known for its more sluggish reaction kinetics on Pt in comparison to the corresponding anodic reactions such as the electrooxidation of hydrogen or small organic molecules. ORR requires larger electrochemical surface area to match the current which the fuel cell anode reaction can provide, but any reduction of the Pt mass will decrease the overall fuel cell performance. According to the widely accepted particle size effect\textsuperscript{1-3}, when the Pt/C nanoparticles are the electrocatalyst of choice for use in ORR, there is a maximum in mass normalized activity (at approximately 4 nm) beyond which a higher dispersion (i.e. smaller nanoparticles) will not improve but rather hinder the performance of the reaction. Currently there are two approaches which seem viable for achieving this goal: (i) electrocatalysis coated with a few nm thick (high mass-specific surface area) thin Pt films\textsuperscript{4,5} and (ii) improving the kinetics with Pt alloys\textsuperscript{1,3,6}. While the first (i) approach is novel and still requires the surface morphology optimization, significant progress has already been made while pursuing the second approach (ii).

In a review published by Gasteiger et al.\textsuperscript{1}, they discuss the PEM fuel cell
electrocatalysis utilizing PtM binary and multi-metal alloys, in which researchers have reported 2-4 times enhancement of the surface specific activity of alloys as compared to pure Pt electrocatalysts. The mass-activity of these alloys is generally less than the mass activity of the state-of-the-art 50 weight % Pt/C electrocatalyst from Tanaka due to the generally larger particle size of the alloys (i.e. lower dispersion). This activity deficiency coupled with the higher tendency toward selective dissolution of the non-noble transition metals is the most likely reason why they have not yet been implemented in commercial applications. Several hypotheses have been given to explain the enhanced activity of Pt alloys over pure Pt. (See Ref.\textsuperscript{1} and references therein.) Briefly, they are: (i) Lattice contraction, (smaller Pt-Pt distance) for the alloys this favors the dissociative adsorption of \( \text{O}_2 \) (ii) Structure- or composition-sensitive inhibition of adsorption of anions including \( \text{OH}^- \) on the alloy nanoparticle surface (iii) Surface roughening of the alloys due to the selective dissolution of non-noble metal component(s) that increases Pt surface area (iv) Increased Pt d-band vacancy in the alloys that shifts the onset potential of the hydroxyl anion adsorption to higher electrode potentials and decreases the Pt-Pt distance depending on the electro-negativity of the alloying transition metals (v) Pt-skin effect; particles having a Pt-rich layer over the base metal core that is electronically modified to have a tendency to weakly adsorb hydroxyl anions. Despite the intensive research activity in this field, researchers probing the dominant mechanism(s) of activity enhancement have not yet reached a consensus on the matter. Among the possible mechanisms listed above, the some more recent studies\textsuperscript{7,8,9} strongly point out the decisive role of the extent and energy of the adsorption of hydroxyl ions on bi- or multi-metallic surfaces. Among the cited articles, Zhang and co-workers\textsuperscript{9} propose for the
first time an atomic-level phenomenon occurring on the alloy surfaces that is responsible for facilitating the kinetics of ORR. They have also attributed this phenomenon as a decrease in hydroxyl ion coverage at the Pt surface and explained that the lateral repulsion between OH\textsuperscript{−} and atomic oxygen adsorptives on neighboring transition metal elements which have different OH\textsuperscript{−} affinity. Considering the marked difference between the adsorption energy of a chemical bond and strength of the dipole-dipole interactions, this explanation from Zhang and co-workers does not answer the question of why the oxidation of Pt occurs at higher overpotentials for the alloy electrocatalysts;\textsuperscript{10} especially, since the OH\textsuperscript{−} species adsorb on the alloying element first and are likely to be localized further away from Pt compared to pure Pt surfaces. Therefore, Pt oxidation should be facilitated based upon structural reasons which are in contrast to the experimental observations. Nevertheless, some structural deformations in the OH\textsuperscript{−} adlayer of alloys can be conceivable due to the local variations in affinity towards OH\textsuperscript{−} adsorption on a heterogeneous surface, but those deformations should occur in opposition to the repulsive lateral interactions between adsorbed surface species.

This experimental work investigated the possibility of whether the magnetic repulsion of diamagnetic hydroxyl anions by the electrocatalyst components or particles possessing net magnetic moment are able to destabilize (and dislocate) the hydroxyl anions on Pt sites, and also what possible influence the electrocatalysts’ intrinsic magnetic properties have on the ORR, if any. Several possible effects of the permanent magnetic field (B) on the ORR activity have been recognized and are investigated in detail. These effects are briefly discussed here from the aspect of electrochemistry. Electrochemical impedance spectroscopy (EIS) data suggest that a homogenous B parallel to the electrode surface and
varied up to 1 Tesla in magnetic field strength does not affect the charge transfer coefficient, or hence the electrochemical kinetics\textsuperscript{11}. On the other hand, magnetic field effects on mass transport and the surface concentration of ferromagnetic, paramagnetic or diamagnetic species is widely accepted and exploited in the electrodeposition of Cu and Co metals onto electrode surfaces oriented parallel to the applied magnetic field\textsuperscript{12}. First, Wakayama et al.\textsuperscript{13} demonstrated by using a simple half-electrochemical cell setup that a strong (< 0.1 T) external magnetic field can promote the ORR in fuel cells. This enhancement was explained by the magnetic attractive force toward the paramagnetic oxygen gas that has exceptionally large positive value of magnetic susceptibility ($\chi = 1.91 \times 10^{-6}$) due to the parallel spin of electrons. Later, some of the same authors proposed that an external magnetic field gradient\textsuperscript{14,15} or magnetized Nd-Fe-B particles embedded into the cathode layer of the fuel cell (B > 0.5 T)\textsuperscript{15} would exert a repulsive Kelvin force on the diamagnetic water. Therefore, the phenomenon creates more space for the oxygen to react and also increases the velocity of the gas. Aside from the magnetohydrodynamic (MHD) effects, another consequence of a magnetic field gradient can be an influence on the concentration of species at the electrode surface. If the surface of alloyed electrode (Pt with Ni, Co, or Fe) is oriented parallel or perpendicular to the magnetic field, the open circuit potential moves to positive or negative directions, respectively\textsuperscript{16}. While a parallel field gradient actually repels, a perpendicular one attracts paramagnetic species in the vicinity of the electrode. Another novel type of the magnetic electrodes belongs to the family of ferromagnetic nanoparticle assemblies. These materials are potential candidates as fundamental constituents of a broad range of applications such as ultrahigh-density magnetic data storage media or sensors. Many
investigations have focused on the preparation of FePt and CoPt nanoalloys\textsuperscript{17-20} since these magnetically hard materials have very high coercivity and can better resist the superparamagnetic state which occurs in pure Co nanocrystals at room temperature\textsuperscript{17}.

Here, we benchmarked in-house-prepared carbon-supported (C) 30 wt\% Pt\textsubscript{3}Co nanoparticles with intrinsic magnetic and electrocatalytic properties in comparison with 30\% Pt/C (E-TEK), and 30\% Pt\textsubscript{3}Co/C (E-TEK) electrocatalysts. The experimental Pt\textsubscript{3}Co/C sample was uniquely synthesized following the procedure described in Ref. \textsuperscript{21}, with the exception of using borohydride as the reducing agent\textsuperscript{22}. Due to the nature of preparation involving extremely fast heat-up to non-controlled high temperatures, it is possible that a portion of the crystal structure undergoes transition from the magnetically soft face centered cubic (fcc) phase to the hard face centered tetrahedral (fct) L\textsubscript{10} phase\textsuperscript{17}. Nevertheless, collective effects due to the dipolar interactions and/or the structure of the nanoparticle organization\textsuperscript{17} might also play role in their noticeable ferromagnetic behavior. With consideration to the aforementioned, the experimental Pt\textsubscript{3}Co/C sample is expected to have different bulk and surface physical properties as well as ORR activity than conventional alloys of the same atomic composition. Using the selected samples, conclusions of a comprehensive physical and physicochemical characterization will be correlated to ORR activity data collected by fuel cell testing and rotating disk and rotating ring disk electrode (RDE, RRDE) experiments. The rotating electrode configuration provides a unique way to probe the inherent ORR activity of powdered electrocatalyst samples by eliminating the mass transport issues. Although the thin film RDE and RRDE is a widely and routinely used method, there have been large discrepancies in the data reported throughout the
electrocatalyst community as pointed out in Ref. ¹. Some of the major discrepancies have been that (i) the published Pt alloying enhancement factors ranging from 2 to 25, (ii) the Pt particle size effect was discovered with RDE but has not yet been confirmed by fuel cell data, (iii) there is at least a factor of 2 times less mass between the electrochemical surface area-specific kinetic currents (i*m, i*s) and those extracted from FC testing.
CHAPTER 2

PEM FUEL CELLS – Advancements with magnetic electrocatalysts

2.1 Introduction

Electrochemical reactions affected by magnetic fields have been studied more in depth over the last decade. The effects of magnetic fields on electrochemical reactions have been divided into three main categories, namely; electrodeposition morphology, electrode kinetics, and mass transport. Primarily, the enhancement of electrodeposition has received significant gains by the introduction of magnetic fields, with enhancements noted as large as 100% for fields on the order of 1 Tesla\(^2\). Electrochemical kinetics has been altered by magnetic fields and has been studied in the form of the enhancement of metal deposition rates and in the alteration of corrosion rates\(^2\). Other work has shown that magnetic field gradients can influence the transport properties of paramagnetic ions in solution\(^2\).

To understand more about the way in which magnetic fields alter the conditions of these chemical systems, it is necessary to learn more about the characteristic nature of magnetic energy and the associated forces. There are five magnetic forces that have emerged as forces that play a role in inducing these magnetic phenomena. Of these forces, two are dependent on the magnetic properties of the electrolyte species, and three are dependent on the motion of charged particles in the system.

\[
\vec{F}_L = j \times B \quad \text{ (Eqn. 1)}
\]

The Lorentz force \(F_L\) (Eqn. 1), is the force acting on a point charge due to electromagnetic fields, where \(j\) is the current density and \(B\) is the magnitude of the magnetic field. This force has been attributed as one of the main magnetic forces that would induce convection in an
electrochemical cell. This would result in a repulsive or attractive force dependent upon the magnitude of the

\[ F_p = \frac{\chi_m B^2 \nabla C}{2\mu_0} \]  
(Eqn. 2)

The “paramagnetic gradient force” or “concentration gradient force” \( F_p \) (Eqn. 2) is a force arising from the paramagnetic molar susceptibility \( \chi_m \) of the diffusion layer due to the concentration gradient, \( \nabla c \), of ions present there, and \( \mu_0 \) is the vacuum permeability.

\[ F_B = \frac{\chi_m c B \nabla B}{\mu_0} \]  
(Eqn. 3)

The field gradient force \( F_B \) (Eqn. 3), in contrast with the concentration gradient force results from a deviation in the magnitude of the magnetic field and concentration is held constant. This force can be absent depending on the nature of the experimental setup but mainly due to the uniformity of the magnetic field.

\[ F_E = \sigma_d \frac{E_{||}}{\delta_0} \]  
(Eqn. 4)

The electrokinetic force \( F_E \) (Eqn. 4) is the force on the charged ions in the diffusion layer; where \( \sigma_d \) is the charge density in the diffusion layer, \( E_{||} \) is a non-electrostatic field parallel to the electrode surface, and then this force is normalized to the dimensions of the hydrodynamic boundary layer, \( \delta_0 \).

\[ F_M = \sigma v \times \frac{1}{B} \times \frac{1}{B} \]  
(Eqn. 5)

The magnetic damping force \( F_M \) (Eqn. 5) is related to the conductivity of the electrolyte. The reason that this force is involved is that it considers longitudinally directed flow with the
direction of B not being hindered; however, this leaves the transverse flow to be damped by an opposing force, $F_M$. In the work of Hinds et al., they note typical magnitudes for these magnetic forces ranging from $10^1$ N/m$^3$ to $10^4$ N/m$^3$ with the Paramagnetic force being the greatest at $10^4$ N/m$^3$ followed by the Lorentz force and the Electrokinetic force in the range of $10^3$ N/m$^3$, and the Field gradient and magnetic damping forces being in the range of $10^1$ N/m$^3$. The major consideration here is not only the magnitude of the forces, but also the directionality of the forces and how that will affect the experiment\textsuperscript{23,26}.

Seminal work on the use of magnets to potentially enhance the performance of proton exchange membrane fuel cells (PEMFC) has been carried out by Wakayama, Wang, and Okada\textsuperscript{13,14,27-31}. In their work they noted a circa 28\% increase of oxygen reduction reaction (ORR) Levich limiting currents on bare Platinum in 0.05M H$_2$SO$_4$ Rotating Disk Electrode (RDE) experiments utilizing a field of 0.56T. Later they incorporated magnetic microbeads into a PEMFC membrane electrode assembly (MEA) after conducting a numerical simulation of water management due to the presence of these magnetic microbeads.\textsuperscript{13,14,30}

The next logical step is to incorporate the magnetic characteristic into the catalytic material being utilized in the fuel cell. N.B. Chaure et al. from the Physics Dept. at Trinity College in Ireland developed a nanoarray electrode of CoPt nanowires having a magnetic field gradient upon magnetization in a field. They have used a modeling software calculation to determine the magnitude of the field gradient at the platinum surface of their nanowire array which is rather large at $10^5$ Tm$^{-1}$. This nanowire array electrode has shown enhancement of a model ORR system limiting current in alkaline medium by a factor of three\textsuperscript{24}. 
Beyond this micro/nano engineering-based fabrication route, we have developed PtCo nanoparticles that have an intrinsic magnetic field, which will be described herein. By bringing the magnetic characteristic onboard the catalyst, rather than applying an external field, or embedding separate micromagnets into the MEA, multiple goals will be achieved. First and foremost is increasing the mass transport limiting current (magneto hydrodynamics, MHD), potentially achieving improvements in electrode kinetics (a typical feature of PtₓCo alloying), and lowering the precious metal content by alloying with cobalt (cost reduction). It is yet to be determined what exact strength of inherent magnetic field will be necessary to provide a significant improvement in mass transport for traditional fuel PEMFCs. The focus is to find utility in the nature of these magnetic forces towards yielding just as a significant or greater improvements in mass transport as have been reported with the success of utility of magnetic forces in the electrodeposition applications.

2.2 Experimental

2.2.1 Electro catalyst Preparation

The experimental alloy electrocatalyst was synthesized in-house, with a target Pt:Co atomic ratio of 3:1 with the final metal loading of 30% weight percent on the Vulcan XC-72R carbon support. A brief synopsis of the synthetic procedure is that after the deposition of Pt onto the carbon support from an aqueous solution, the Co precursor was deposited onto the Pt using borohydride\textsuperscript{21}. This reaction is catalyzed by the pre-deposited Pt. The other electrocatalysts in used in these experiments were obtained from E-TEK PEMEAS. All samples were used as-received.
2.2.2 Fuel Cell Experiments

The single cell unit was purchased from Fuel Cell Technologies, and the experimental conditions were adjusted using a fuel cell test station built in-house. The membrane electrode assembly (MEA) utilizes a Nafion 112 membrane. E-TEK 30% Pt/C electrocatalyst at 0.5 mg/cm$^2$ platinum loading was employed for H$_2$ oxidation at the anode for all experiments. The cathode electrode was prepared using the E-TEK and the experimental platinum cobalt electrocatalysts using a total metal loading of 0.6 mg/cm$^2$. The cell was operated at 70 °C, and fed by 100 % relative humidified gases. The H$_2$/O$_2$, and H$_2$/air relative gas pressures were chosen to be 43/36 psi and 10/10 psi.

2.2.3 Physicochemical Characterization

2.2.4.1 XRD

XRD spectra of the powder samples were collected with a Rigaku powder diffractometer (D/max 2200) using the 46 kV, 20mA Cu K$_\alpha$ radiation. The x-ray diffraction patterns were taken with a step size of 0.02 degrees, and a step time of 10 seconds between 5° and 90° 2Θ angles.

2.2.4.2 ICP-MS

Inductively Coupled Plasma – Mass Spectrometer (ICP-MS) measurements were performed with a VG Elemental PlasmaQuad 2 instrument. A 0.01 g aliquot of each electrocatalyst was digested (refluxed) in 50 mL of aqua regia at 85 °C for at minimum of 5 hours; meanwhile water was added to keep the solution from running dry. The remaining carbon was removed via filtration of the acid concentrate through a filter membrane (d = 0.2
µm). This solution was then diluted to reach a concentration within the calibrated detection limits of the ICP-MS instrument.

### 2.2.4.3 SEM/EDX

Imaging and quantitative elemental analysis was performed on a Hitachi S-4800 Field Emission-Scanning Electron Microscope (FE-SEM) operating at 3 keV equipped with an Energy Dispersive X-ray (EDX) Analysis Attachment. EDX data were collected with a take-off angle of 30° at 20 kV in 120 seconds. Data were subjected to ZAF analysis and are element-normalized.

### 2.2.4.4 Cyclic voltammetry in alkaline media

For cyclic voltammetry (CV) experiments in alkaline media, Ar-saturated 1 M KOH solution was used as the electrolyte. The working electrode was prepared from the powdered samples by depositing and dispersing them from aqueous suspensions on polycrystalline gold (A = 0.64 cm²) giving an electrode with 50 µgPt/cm² for the Pt and Pt₃Co samples and equivalent loading of Co and carbon for the 30% Co/C and Vulcan XC-72R samples, respectively. A thin Nafion film (0.05 µm) was applied as a capping layer in order not to influence mass transport at the electrocatalyst layer. The reference electrode was Hg/HgO/1 M KOH (-0.1 V vs. NHE) and a Pt wire was used as a counter. The CV curves were recorded after a few electrode potential cycles for activation in the hanging meniscus position at a 20 mV/s scan rate.

### 2.2.5 Magnetic Susceptibility

Magnetic susceptibility measurements were taken on a SQUID Magnetometer (Quantum Design MPMS XL-5) enabling 5.0 T maximum field. The sample was placed into
an EverCool Dewar; its temperature was adjusted between 1.8 and 400 K. The relative sensitivity of detection is $1 \times 10^{-8}$ emu. The ZFC-FC experiments were performed as follows: the sample was initially cooled in a zero magnetic field to 4 K. With an applied field of 100 Oe, the magnetization was recorded as zero-field-cooling (ZFC) curve while the temperature increased. When the temperature reached 400 K, the sample was progressively cooled and the magnetization was recorded as a field-cooling (FC) curve. The hysteresis loop was obtained while keeping the temperature constant. The magnetization was recorded while the field was decreased from 1.5 T down to -1.5 T; then increased back thus closing the loop.

2.2.6 Half-cell Electrochemistry

Rotating disk electrode (RDE) and rotating ring disk (RRDE) experiments were conducted by using a Pine Instruments Company (USA) CPR and MSR rotators, respectively. The electrode potential was controlled using Eco Chemie Autolab PGSTAT30 bipotentiostats (Brinkmann Instruments, Inc., USA) that were equipped with SCAN-GEN analog scan-generator modules. Electrocatalyst samples were deposited from aqueous or alcohol-based suspensions onto the glassy carbon disk (0.20 cm$^2$ for RDE, and 0.25 cm$^2$ for RRDE) to give a desired loading of Pt, Co, or C, and capped with a thin Nafion film (0.05 µm)$^{32}$. For RRDE, the Au ring in the electrode allowed for a 37% collection efficiency (N) as determined using the hexachloroiridate(IV)/hexachloroiridate(III) redox couple$^{33}$ in 0.1 mM concentration. ORR experiments were performed in ultra high purity O$_2$-saturated electrolytes; and ultra high purity Ar was used to purge the solution for the O$_2$-free experiments. One molar (1 M) HClO$_4$ electrolyte solution was prepared by diluting the ultra-trace metal grade chemical (70%, Veritas double distilled, GFS Chemicals, Inc., USA) with Millipore water. One
Molar (1 M) trifluoromethane sulfonic acid (triflic acid or TFMSA) was prepared from the monohydrate after the in-house purification of the stock solution (Sigma-Aldrich, > 99.6 % purity) by at least three times repeated distillation and recrystallization. All experiments were conducted in a conventional three-electrode glass cell using a Pt counter electrode and NHE or Ag/AgCl (3 M NaCl) reference electrodes depending on the electrolyte. The ferricyanide/ferrocyanide and hexachloroiridate(IV)/hexachloroiridate(III) experiments were performed in 0.1 mM analyte solution prepared in 1 M KClO$_4$ electrolyte.

2.3 Results and Discussion

2.3.1 Fuel Cell Testing

Figure 2.1 presents reproducible polarization curves recorded at three separate gas pressures for PEMFCs that were assembled with the commercial E-TEK and the experimental Pt$_3$Co/C electrocatalysts, respectively. When pure oxygen was supplied at relatively high pressure to the E-TEK sample, there was no clear indication of a mass transport problem on the polarization curve recorded (H$_2$/O$_2$ 43/36 psi curve in Figure 2.1). Rather, the fuel cell still exhibited sluggish kinetics and ohmic resistance at high current densities. It is interesting to note that the experimental catalyst cannot handle equivalently large oxygen supply as the E-TEK sample (H$_2$/O$_2$ 43/36 psi curve in Figure 2.1). Moreover, according to the Nernst equation, the slightly lower open cell potential of the experimental catalyst electrode (0.95 V vs. 0.98 V) also seems to indicate that the sample is deficient of electrochemical active sites as compared to the commercial E-TEK sample.
When air is introduced in lieu of pure oxygen as the oxidant gas at the same pressure, the performance of the E-TEK and the experimental cathode-employing fuel cells become comparable, with even the experimental cell slightly outperforming the E-TEK cell. This change from pure oxygen to air is a ca. five times decrease in oxygen concentration which will change the operating conditions of the cell resulting in an earlier onset of mass transport issues for both electrocatalysts. In other words, the fuel cell is now relying on the availability of oxygen at high current densities. Since the experimental catalyst has significantly lower electrochemical surface area and supposing that the kinetic and macroscopic mass transport properties are similar, if not identical for both cathodes, this behavior must then be attributed to a higher surface concentration of oxygen at the experimental catalyst.

When air was supplied at low relative pressure (H\textsubscript{2}/O\textsubscript{2} 10/10 psi curves), the thermal energy of the gas is suppressed and other transport phenomena responsible for creating net reactant and product fluxes toward or from the electrode surface will become more evident. At this low pressure, it is expected that the availability of oxygen will be much lower resulting in mass transport limitations at even lower current density as is the case for the conventional alloy nanoparticle electrodes. Surprisingly, the experimental electrode polarization curve almost completely overlaps the curve recorded at 3.6 times higher air pressure. Given that a similar amount of oxygen is being reduced at 10 psi and at 36 psi, the diffusion conditions must be similar. Furthermore, this also means that a similar amount of water is being produced; therefore, it is unlikely that water management can be significantly different for the same electrode. Rather, it is noticed that there is likely another force or delivery mechanism playing a dominant role over the thermal energy of oxygen at this low
pressure thus creating a net flux of the gas to the surface with a similar rate to the random Brownian motion of the gas at the elevated pressure of 36psi.

2.3.2 Physicochemical Characterization

2.3.2.1 SEM

Scanning electron microscopy (Fig. 2.2) was used to determine average particle size and to determine what the arrangement of the catalyst particles were on the carbon supporting substrate. The commercial E-TEK Pt/C and Pt$_3$Co/C samples exhibit highly uniform particle distribution with very little agglomeration. The catalyst particles are approximately 4 and 5 nm respectively. The experimental Pt$_3$Co/C on the other hand exhibit some agglomeration and the clusters are comprised of particles sized near 6 nm.

2.3.2.2 ICP-MS

Inductively coupled plasma – mass spectrometry (ICP-MS) was utilized to confirm the elemental composition of the catalysts as well as their metal loading (in weight %) on the Vulcan XC-72R carbon support. As depicted in Table 2.1, the platinum loading on all samples is very close to the nominal weight percentage of 30%. The atomic ratios show that the ETEK Pt$_3$Co is actually closer to a 4:1 Pt:Co composition which is direct evidence that a smaller amount of cobalt is present in the nanoparticles; this result is in contrast to the experimental Pt$_3$Co having an atomic ratio much closer to the 3:1 Pt:Co ratio expected from the synthetic procedure.

2.3.2.3 EDX

Energy Dispersive X-ray Analysis (EDX) was used as the initial catalyst screening method to determine the Pt:Co atomic ratio of the alloyed nanoparticles. The EDX data for
the ETEK Pt$_3$Co/C and experimental Pt$_3$Co/C Pt:Co atomic ratio supports the analytical result from the ICP-MS (See Table 2.1).

2.3.2.4 Powder-XRD

As seen in the powder-xrd patterns in Figure 2.3, the (111) peak shifts towards higher values of 2\(\Theta\) indicates the extent of cobalt alloying relative to the pure platinum samples, since the crystal lattice will actually contract due to the substituting of the lower Z transition metals into the fcc lattice. Although the E-TEK Pt$_3$Co/C shows a higher extent of alloying in comparison to the experimental Pt$_3$Co/C, it is interesting to note that the E-TEK Pt$_3$Co/C has some indication of secondary phases that can be associated with cobalt or perhaps cobalt oxide impurities. These phases are not present in the experimental Pt$_3$Co/C. The average particle size of the 60% ETEK Pt/C is similar to the 30% ETEK Pt$_3$Co/C and 30% experimental Pt$_3$Co/C. Estimated particle sizes are summarized in Table 2.2. In-house prepared cobalt on carbon is plotted as well to show that the small peaks in the ETEK Pt$_3$Co/C are indeed separate cobalt phases.

2.3.4.5 Cyclic voltammetry in acidic and alkaline media

In Figure 2.4a, cyclic voltammograms of two similarly synthesized experimental Pt$_x$Co$_y$/C samples are shown in comparison with the 60% ETEK Pt/C in 1 M KOH. The experimental samples were 30% PtCo/C (1:1 atomic ratio) and 30% Pt$_3$Co/C. Figure 2.4 is useful in noticing the trends of the formation of speciation of the surface cobalt oxides formed when cycling towards higher working electrode potentials, and what species of cobalt oxides are reduced when cycling towards lower potentials. The alloy particles in contrast to the pure platinum particles have Pt-OH oxidation occurring at lower overpotentials which is
due to the presence of Cobalt in the \textit{fcc} lattice. Cobalt species occur before and after the Pt oxidation and reduction. The Co(OH)$_2$, CoO, and CoOOH do not show any insight as to the arrangement of Cobalt atoms on the surface of the catalyst; however the Co$_3$O$_4$ species is indicative of clustering of Cobalt into islands on the surface of the particle. After determining cobalt speciation, further qualitative analysis (Figure 2.4b) reveals that the Co$_3$O$_4$ peak is absent in the well alloyed ETEK Pt$_3$Co/C sample, where as it is quite prominent in the Exp. Pt$_3$Co/C sample. There is also some inhomogeneity in the ETEK sample because it has both the pure Pt-OH reduction near 0.15V and also the shifted reduction peak near 0.45V which may also explain the more Pt-like character of the H$_{UPD}$ region of the ETEK Pt$_3$Co/C.

2.3.4.6 RDE and RRDE results

In Figure 2.5, it can be shown that the ETEK Pt$_3$Co/C does in fact have an overpotential improvement over the conventional ETEK Pt/C. The experimental Pt$_3$Co/C displays slightly diminished kinetics; however, it is remarkable that a catalyst with much lower accessible Pt surface area is able to achieve the same limiting current levels as the ETEK samples. Although the ETEK Pt$_3$Co/C is clearly the kinetic leader of the three samples, the Exp. Pt$_3$Co/C has a higher mass-transport limited current than the ETEK Pt/C and Pt$_3$Co/C samples, which also has improved kinetics over the ETEK Pt/C sample. When normalized to the platinum electrochemical surface area, one would see that the Exp. Pt$_3$Co/C is the clear leader, having the lowest mass transport limitation. The RRDE measurements (Figure 2.5 inset) revealed that Exp. Pt$_3$Co/C (black) has improved peroxide yield properties over both the E-TEK Pt/C and Pt$_3$Co/C (red and green respectively).

2.3.4.7 Magnetic properties of the electrocatalysts
2.3.4.7.1 Magnetic susceptibility measurements

Magnetic measurements, performed on dry powdered samples of ETEK Pt$_3$Co/C nanoparticles supported on carbon, reveal that the catalyst nanoparticles have single domain magnetic structure and are superparamagnetic at room temperature, an example of which has been seen in literature $^{34-37}$. Temperature dependence of the magnetization of the ETEK Pt$_3$Co/C is shown in Figure 2.6A. A cusp can be seen in the ZFC magnetization curve. The maximum of the cusp is the blocking temperature $T_B$ of this sample, $T_B = 36$K. Below $T_B$ is the ferromagnetic regime and above it is the superparamagnetic regime. The hysteresis plot shown in the inset of the same figure, Figure 2.6A, shows neither coercivity field nor remanence magnetization; magnetic domains of the nanoparticles align freely with the field during the measuring time. Thermal energy at room temperature is sufficient enough to overcome the coercive field of the neighboring particles. In the ZFC magnetization curve, at low temperature, $T=4$K, the magnetization is the lowest because cooling at zero external magnetic field freezes the random orientation of the moments of individual nanoparticles.

When the external field is turned on, the moments of individual nanoparticles start to align with the external field to minimize the energy of the system. As the temperature increases, moments of nanoparticles gain more thermal energy to overcome the coercive field pinning the moments and consequently the total magnetization increases. At the blocking temperature, the thermal energy is comparable to that of the magnetic energy trying to align moments of individual nanoparticles with the external field; as a result, the total magnetization reaches a maximum. Then as the temperature increases the total magnetization decreases. The data in the FC curve shows that the magnetization increases as the temperature decreases as
expected since the thermal energy is decreasing while keeping the external magnetic field constant. What is important for our discussion in this paper is the magnetic behavior of the Exp. Pt$_3$Co/C sample as compared to the ETEK sample. For Exp. Pt$_3$Co sample, Fig. 2.6B shows almost the same general behavior of the magnetization in both the ZFC and FC curves to that of the ETEK Pt$_3$Co/C. The ZFC has a maximum near the blocking temperature of ETEK Pt$_3$Co/C and the magnetization increases as temperature decreases in the FC curve. But there are two distinct features worth noting in the magnetic behavior of the Exp. Pt$_3$Co/C: 1) the ZFC cooling peak is broader causing a shift of the blocking temperature; the blocking temperature of the Exp. Pt$_3$Co/C is 52K, $T_B = 52$K. The broadening of the peak is due to a larger size distribution of the nanoparticles$^{38, 39}$. This is in agreement with what the XRD and SEM data in Table 1. 2) The hysteresis loop of the Exp. Pt$_3$Co/C sample shows a coercivity field, $H_C$, of 72 Oe. This means unlike the ETEK Pt$_3$Co/C there is a remnant magnetization at room temperature. The thermal energy at room temperature is not able to completely randomize individual moments of the nanoparticles.

2.3.4.7.2 Lorentz Force Experiment

After determining the bulk magnetic characteristics of the samples it was of interest to see if this phenomenon was reliant upon the catalytic properties of the samples or was truly a function of an inherent force being applied to the solution phase. The hexacyano ferrate anion has been historically used in electrochemistry as a one-electron system to probe the diffusion coefficient of an analyte. Unlike ORR which relies on specific catalytically active platinum surface sites, this redox couple is not catalyst-specific and as such utilizes the complete surface area of the carbon supported catalyst and the glassy carbon substrate in this
experiment as the electroactive area. Given that the surface areas between the samples are essentially equal, one would anticipate the reduction curves obtained to have the same Levich currents \((i_L)\) and to have the same kinetics unless there is a deviation due to an inherent property of the sample.

**Levich Current (RDE):**

\[
i_L = (0.620) \, \text{nFAD}^{2/3} \omega^{1/2} \nu^{-1/6} C
\]

Looking at the rotating disk electrode (RDE) reduction curves collected in the potassium hexacyano ferrate analyte it is evident that the E-TEK Pt\(_3\)Co/C and the Vulcan X72-R carbon black have nearly the same Levich current \((i_L)\); however, the Levich current \((i_L)\) of the experimental Pt\(_3\)Co/C is not nearly as high. A smaller Levich current \((i_L)\) suggests that there is less reactant arriving at the surface of the electrode to be reduced. Could there be a force repelling the analyte from reaching the surface? This is likely governed by the Lorentz Force, which as described earlier in Eqn. 1 is related to the motion of charged particles in the presence of a magnetic field,

**Lorentz force:**

\[
F = q(E + v \times B)
\]

where \(F\) is the force, \(E\) is the electric field, \(B\) is the magnetic field, \(q\) is the charge of the particle, \(v\) is the instantaneous velocity of the particle. Seemingly the Levich current expression has been modified by M. Uhlemann, et al. where they suggested the need to include a magnetic field dependency for use with magnetohydrodynamic and magneto convective conditions\(^{25}\).

\[
i = 0.0969nF \varepsilon_0D \left( \frac{1}{\mu \eta} \right)^{1/3} \left( \frac{\partial \chi}{\partial c} \right)^{1/3} \left| \Delta c \right|^{1/3} \left| \frac{dB}{dz} \right|_{z=0}^{1/3}
\]

Modified Levich Current:
This experiment was then repeated utilizing potassium hexachloroiridate as the analyte. Potassium hexachloroiridate is a more ideal one-electron redox couple as compared to potassium hexacyanoferrate which suffers from the formation of the Prussian blue complex. For the iridate ions, essentially the same reduction lineshapes are recorded for the carbon support and both electrocatalyst samples indicating that the charge transfer takes place essentially on the complete exposed surface area of the electrocatalyst including that of the carbon support. This also points to the fact that the weak ferromagnetic behavior does not influence the transport of the charged IrCl$_6^{2-}$ species. The same current levels are measured for the carbon support and the ETEK electrocatalyst in a solution containing the same concentration of Fe(CN)$_6^{3-}$ ions. Since the Lorentz force, which exists between the magnetic field parallel to the electrode surface and the moving charge particle, is governed by the charge on the ionic species, it is likely to be a greater force for the ferrate anion than the iridate anion. This potentially alters the trajectory of ions diffusing towards the electrode surface; hence, a smaller amount of analyte can be reduced at the electrode.

2.4 Conclusions

We are able to probe the magnetic characteristic of these electrocatalysts in multiple ways; both through typical SQUID magnetometer measurements to determine the magnitude and type of magnetic sample, but as well through the implementation of traditional electrochemical systems using RDE in the presence of two different redox couples.

The fuel cell exhibits sluggish kinetics and ohmic resistance at high current densities. It is particularly interesting to note that the experimental catalyst cannot handle equivalently
large oxygen supply as did the E-TEK sample (H/O \textsubscript{2} 43/36 psi curve in Figure 2.1). This observation can be associated with the SEM imagery and electrochemical data indicating that the Pt dispersion of the experimental sample has not yet been optimized. The experimental method of synthesis is likely the cause for the increase in the size of nanoparticles and the interparticle separation as compared to the high temperature bulk synthetic method employed by ETEK. Also to consider is the magnitude and directionality of the electromagnetic forces present on the microscopic level at the surface of the electrode. At higher oxygen supply, considering the paramagnetic force (Eqn. 2), as the concentration gradient is becomes normalized throughout the system, \( F_P \) is minimized; this is one of the more dominant forces present with large concentration gradients.

The physico-chemical characterization yielded very interesting insight into the nature of the bulk and surfacial properties of the electrocatalysts. We note that the ETEK Pt\textsubscript{3}Co/C alloy had a Pt:Co ratio closer to 4:1 as compared to the targeted 3:1 which the experimental Pt\textsubscript{3}Co/C catalyst sample achieved. This may be due to sublimation of the cobalt at high temperatures in the industrial scale production method used by ETEK. The cyclic voltammetry in alkaline media displayed a more well-alloyed surface for the ETEK Pt\textsubscript{3}Co/C sample, whereas the experimental Pt\textsubscript{3}Co/C had indicators of higher cobalt surface concentration, both evident in the H\textsubscript{UPD} region and the Co\textsubscript{3}O\textsubscript{4} region indicating islands of cobalt on the surface of the nanoparticles. RDE and RRDE electrochemical measurements showed a clear mass transport advantage although no apparent kinetic improvement over the commercial ETEK alloy. The experimental alloy also displayed an improvement in reducing
the peroxide generation. The mechanism and role of the magnetic characteristic of the catalyst in reducing the peroxide generation will need to be determined.

Further work is necessary in the synthetic route to more hard magnetic catalyst. Some direction can likely be taken by analyzing the synthetic methods of CoPt utilized in hard-disk media research. The closer these alloys approach hard magnetic $fct$ structures, the more improvement in mass transport should be evident. Future RDE experiments are also required to investigate whether other charged species pertinent to ORR (in particular OH$^-$) are affected by magnetic forces. In these experiments, elemental surface processes such as hydrogen- and CO-electrooxidation, and ORR will be compared for the selected electrocatalyst systems. The analytes are dissolved diamagnetic (H$_2$, CO) or paramagnetic (O$_2$) gaseous molecules, while OH$^-$ anions act as promoters and inhibitors for HOR and CO electrooxidation, and ORR, respectively.$^2$

2.5 Acknowledgements

E-TEK PEMEAS – For supplying the electrocatalysts

Geolabs, Inc. – Braintree, MA for use of their ICP-MS
2.6 Table and Figures w/ Captions

Figure 2.1: H₂/O₂ and H₂/Air fuel cell polarization curves for two PEM devices, for which the MEA were assembled with (A) E-TEK 30% Pt₃Co/C (B) magnetic 30% Pt₃Co/C electrocatalysts on the cathode side. Other operating conditions are identical.
Figure 2.2: Secondary electron SEM images for (A) 30 % E-TEK Pt/C, (B) 30 % E-TEK Pt$_3$Co/C, and (C) 30 % M Pt$_3$Co/C electrocatalysts taken at 150,000-times magnification.
<table>
<thead>
<tr>
<th>Table 1: Elemental Analysis (ICP-MS &amp; EDX)</th>
<th>Weight Percent (%) (ICP-MS)</th>
<th>Atomic Ratio (ICP-MS)</th>
<th>Atomic Ratio (EDX)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample</td>
<td>Co</td>
<td>Pt</td>
<td>Total</td>
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<tr>
<td>ETEK Pt/C</td>
<td>N/A</td>
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<td>33.5</td>
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<tr>
<td>ETEK Pt\textsubscript{3}Co/C</td>
<td>2.3</td>
<td>29.6</td>
<td>31.9</td>
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<tr>
<td>Exp. Pt\textsubscript{3}Co/C</td>
<td>2.8</td>
<td>29.7</td>
<td>32.5</td>
</tr>
</tbody>
</table>

**Table 2.1:** Elemental Analysis - ICP-MS & EDX Results
Figure 2.3: Powder X-ray Diffraction Patterns of E-TEK Pt/C, E-TEK Pt$_3$Co/C, Experimental Pt$_3$Co/C, and Experimental Co/C.
**Table 1: Particle Size**

<table>
<thead>
<tr>
<th>Electro catalyst</th>
<th>XRD - Scherrer /nm</th>
<th>SEM Imagery /nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>ETEK 30% Pt/C</td>
<td>1.7</td>
<td>3</td>
</tr>
<tr>
<td>ETEK 60% Pt/C</td>
<td>4.6</td>
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<tr>
<td>ETEK 30% Pt₃Co/C</td>
<td>4.5</td>
<td>5</td>
</tr>
<tr>
<td>Exp. 30% Pt₃Co/C</td>
<td>5.9</td>
<td>6</td>
</tr>
</tbody>
</table>

**Table 2.2:** Average particle sizes were determined from both XRD Scherrer analysis and via SEM imagery analysis.
Figure 1: 50mgcm$^{-2}$ catalyst loading on Gold 9mm Disc in 1 M KOH at 20 mVs$^{-1}$
(a) ETEK 60% Pt/C (b) Magnetic 30% PtCo/C (c) Exp. 30% Pt$_3$Co/C

Figure 2.4: Alkaline Cyclic Voltammograms for determination of surface Co and Pt speciation and relative extent of surfacial alloying
Figure 2.5: Rotating ring-disk electrode data for oxygen reduction and peroxide formation experiments.
Figure 2.6: Field Cooling/Zero Field Cooling for Pt$_3$Co ETEK (A) & Exp. Pt$_3$Co (B) Magnetization curves for the E-TEK 30% Pt$_3$Co/C electrocatalyst as a function of temperature and magnetic field.
Figure 2.7: Ferricyanide and Iridate Experimental Data (Lorentz force exp.)
CHAPTER 3

Directions for Future Research

3.1 Electrocatalyst Synthesis

Further work is necessary in the synthetic route to more hard magnetic catalyst. Some direction should be taken to review the synthetic methods of CoPt utilized in hard-disk media research. The closer this research can come to attaining hard magnetic fct structures, the greater improvement in mass transport will be realized.

3.2 Mechanistic Study of Magnetoelectrochemistry

Future RDE experiments are also required to investigate whether other charged species pertinent to ORR (in particular OH\textsuperscript{−}) are affected by magnetic forces. In these experiments, elemental surface processes such as hydrogen- and CO-electrooxidation, and ORR will be compared for the selected electrocatalyst systems. The analytes are dissolved diamagnetic (H\textsubscript{2}, CO) or paramagnetic (O\textsubscript{2}) gaseous molecules, while OH\textsuperscript{−} anions act as promoters and inhibitors for HOR and CO electrooxidation, and ORR, respectively.\textsuperscript{2}

3.3 Magnetic Field Gradient Electrodes – Benefit of Directional Polatization

There is question of whether magnetic field gradients may also improve the water management issues that plague fuel cell MEAs. Directionally polarizing (parallel or perpendicular) the PEMFC MEA utilizing hard magnetic catalysts in a magnetic field may allow for discovery of the mechanism by which forces are playing greater roles in the enhancement seen at the low oxygen concentration operating conditions as well as the effective spatial geometry of the interaction.
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