Single Cell Group
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October 17, 1997

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Dr. E. M. Stuve:

The following is a proposal that states the objective for the Single Cell Group during the Fall Quarter of 1997. It includes information on what tasks the group and individual group members will focus on in order to reach this objective. A time table that displays when each task is to be completed is also included.

This proposal also includes information about past reasearch relating to this group along with literature from several references.

Information from the MSDS and the SOP relating to the Single Cell Group have been compiled and included in this proposal.

Sincerely,

Single Cell Group:

Michael Nguyen

Johnny Ferara

Timothy King

Seana Seraji

Single Cell Group

Proposal

October 17, 1997

Michael Nguyen
Johnny Ferara
Timothy King
Seana Seraji

Summary:

The single cell group is responsible for fabricating a fuel cell membrane which can produce a current density of 1.0 A $\cdot cm^{-2}$ at a cell voltage of 0.6 V. This fuel cell will power a small-scaled locomotive to be operated at the 1998 Engineering Open House. The first task is to measure the electrical resistance of the flow field plates (FFPs) which house the fuel cell membrane. During this quarter, the single cell group will determine whether the existing fabrication techniques are adequate and if the FFPs need to be reconstructed. The objective of the single cell group is to find out what is working correctly rather than what works incorrectly. A resistance test will be performed on the aluminum block that houses the serpentine pattern used in the flow of gases. An electrochemical cell will also be designed to determine the proton conductivity of the Nafion 117 (Dupont) membrane before and after the application of the platinum catalyst. The group's future tasks depend upon the results of this study.

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Introduction:

The Single Cell group is to determine proper catalyst formulation and preparation to achieve optimum performance of a single fuel cell. The design goal for a single cell is a current density of 1.0 A $\cdot cm^{-2}$ at a cell voltage of 0.6 V. Last year's effort produced a current density of 2.1 mA $\cdot cm^{-2}$ at a potential of 0.6 V. This year, the single cell group is required to achieve a current density of almost three orders of magnitude higher than the results from the previous year. This requires a tremendous amount of effort and dedication from each group member. More reliable flow field plates (testing apparatus) is crucial for us to know whether or not the problem lies within the membrane. This will identify whether more Nafion 117 (Dupont) membrane characterization is necessary.

The first task of the Single Cell Group is to determine what works rather than what does not work. The electrical resistance of the present aluminum flow field plates (FFP) will be measured. With the desired current density of 1.0 A $\cdot cm^{-2}$ at a cell voltage of 0.6 V, Ohm's Law gives us a resistance of 0.06 Ω. Thus the electrical resistance of the whole fuel cell needs to be smaller than this value in order to obtain the desired output. The resistance can be measured by setting up the aluminum block with a lead sheet in place of the catalyzed membrane, or the membrane electrode assembly (MEA). A power source, supplying a constant current of 1 A, will be attached to the different leads on the block. A voltmeter will be used to determine the potential drop at various locations on the block, and the resistance can be determined using Ohm's Law.

If the FFP's resistance has been determined to be greater than 0.06 Ω, we can conclude that a new design and/or even new construction material for the FFP is required. It will take some time to come up with a better design and to machine it to proper specifications. One choice for a material would be graphite. It offers less resistance when compared to aluminum, but it is much more difficult to machine due to its physical characteristics.

If the FFP's resistance has been determined to be smaller than 0.06 Ω, we can concentrate performing resistance tests on the membrane itself. An electrochemical cell will be designed with the role of testing the proton conductivity of the Nafion membrane both before and after the application of the platinum catalyst. There is no information of the age of the Nafion 117 (Dupont) membrane that has been used or how long it was made to last. The chemical or even physical properties may have changed. A comparison of the results from proton conductivity test on both the new and old Nafion membrane will determine if age is an issue. If age is an issue, it is important to start with a fresh membrane either through purchasing or generous donations from Dupont.

The membrane preparation calls for it to be saturated with water by boiling in aqueous peroxide solution. The current fabrication procedure for the MEA can be found in Appendix D. Tests needs to be conducted to examine the saturation degree of the membrane after this step. One method to determine the saturation degree of the membrane would be include measuring the weight of the membrane after each process step. The "bone dry" weight of the membrane will serve as a basis to determine the saturation degree of the membrane. This test will point out if the membrane saturation time needs to be extended, and if the membrane is kept properly saturated during the duration of its life.

Also during the membrane preparation process, application of catalyst ink onto Nafion membrane to obtain a consistent thickness presents a major challenge. During the previous year, a paint brush was used for this purpose. An airbrush will be used this year to apply the catalyst. The catalyst needs to stay on the membrane surface for a long period of time. The catalyzed membrane is called the membrane electrode assembly as mentioned earlier. The MEA will then be hot pressed and boiled in various solutions. The temperature and pressure of the press as well as the time duration for boiling are also parameters we might need to consider.

The inside surface of the FFP has been coated with Electrodag, a graphite solution, to prevent corrosion and thus reduce the resistance. However, adhesion of this thin graphite layer

on the aluminum is very difficult to achieve. The aluminum surface might need to be treated to provide better adhesion or an alternative solution of graphite needs to be used.

During operation, the cell needs to be maintained at ≥ 80 percent humidity at temperature of 80 °C. This "rain forest condition" requires a supply of saturated hydrogen and oxygen gases. However, an accumulation of water on the membrane's surface can cause "flooding". As a result, it is important to achieve a turbulent flow of gases which can entrain liquid water and remove it from the surface effectively. A turbulent flow requires a high value of the Reynolds number. However, this increases the potential for gas leaks and safety issues.

Design Problem and Proposed Work:

In order to solve the problem of low current density in the fuel cell, the root cause must first be found. The strategy that will be adopted to accomplish this is to examine every step in the process in hopes of finding the proverbial "weak link". The ultimate goal of the single cell group is to develop a test cell capable of achieving a current density of 1 ampere per square centimeter, at 0.6 volts. This is three orders of magnitude greater than any previous tests have produced. For this quarter, the challenge is to fabricate a membrane that will allow the cell to have an output of 25 mA at 0.6 volts.

Flow Field Plates Resistance Test (Seana and Johnny)

The first step is to determine if the currently used flow field plates (FFPs) are the cause of the poor performance of the cell. This will be accomplished by conducting resistance tests on the FFP apparatus. Figure 1 is a schematic of the set up of this experiment. The aluminum blocks will be assembled with a piece of lead (Pb) in place of the Nafion membrane. Lead was chosen because it is a relatively good conductor, but more importantly, it is softer than aluminum, and thus will not damage the serpentine flow field. This will then be connected to a power supply at a constant current of 1 ampere. A multimeter will then be used to measure the voltage at several points along the system as shown in Figure 2.

According to Ohm's Law,

$$V = I * R$$

where V represents the voltage difference and I the current. The resistance, R, of the system will be equal to the voltage reading from the multimeter, for the current will be held constant at 1 ampere.

After the resistance has been measured at various points on the system, other parameters will be examined. It is still to be determined if torquing the screws on the assembly will affect the resistance of the system. To eliminate this as a possibility, the resistance of the block will be measured at various torque levels.

The current electrical connections of the system are less than ideal. This will also be examined as a part of the resistance test. Various options for connections will be tested. Since the system is a low voltage high current set up, automotive grade equipment will be used.

Another possible factor in the resistance of the block is the coating on the serpentine pattern. Currently, a solution of 1:3 Electrodag and isopropyl alcohol, respectively, is applied to the surface of the serpentine flow field with a small artist's brush. The Electrodag is a graphite solution that is applied to the pattern to improve conductivity. The effects of altering the coating method, the thickness of the coating and the concentration of the solution will be examined. Also, alternatives to the Electrodag coating will be researched.

From the results of this test it should be apparent if the FFP's are the cause of the previous shortcomings of the fuel cell. Since the resistance is expected to be less than 1 ohm, it is not likely that this is the main problem of the fuel cell, but none the less, it is a variable that needs to be eliminated. Once this test had been conducted and the results analyzed, the Field Flow Plates and assembly can be deemed either satisfactory, or unsuitable for the fuel cell.

If it is proven that the FFP's are not the source of the problems, other possibilities will be contemplated. The quality of the membrane and the membrane processing are the next areas that will be explored.

Electrochemical Test (Timothy and Michael)

Power of the fuel cell is generated when the hydrogen proton diffuses through the proton exchange membrane (PEM) to react with oxygen at the cathode. The level of power output depends on various factors. The macroscopic factors are: efficient supply of hydrogen and oxygen at proper temperature and relative humidity, pattern of gas channels in the flow field plates (FFPs), electrical resistance of the FFPs, and mechanical force applied when bolting the two FFPs together. The microscopic factors involved revolve around the PEM itself.

The PEM used in the fuel cell locomotive project is a perfluoro sulfonated ionomer material called Nafion. This is the most common material for prototype applications because its properties are well understood.

It is vital that the Nafion membrane conducts protons well in order to have a continuous reaction at the cathode with oxygen. The proton conductivity of the currently used membrane is still questionable. To eliminate this uncertainty, it is necessary to investigate the proton conducting characteristics of Nafion.

One factor in the conductivity of the membrane is the sodium concentration in the Nafion. To test the proton conductivity, an electrochemical cell will be researched and built. Once an electrochemical cell is working, tests will be done on the conductivity of a unprocessed Nafion membrane. Next a membrane that has been converted to the sodium/thermoplastic form will be tested.

The steps to make the membrane thermoplastic involve boiling it with 1 M NaCl. This converts the Nafion from the hydrogen form to the sodium form. This is done to enhance the processing characteristics of the membrane. After processing, the membrane is then boiled in deionized water to convert it back to its original form. Because sodium impedes flow of current, it is necessary to dissolve as much sodium as possible during the deionizing step. Hence, the concentration of the sodium in the membrane needs to be determined in order to what percent

sodium remained. Ultimately, this test is to illustrate how conductive fluctuates as a function of processing.

This year, an electrochemical test will be performed to measure the resistance of the membrane. The apparatus for the test is shown in Figure 1. It consists of an H-shaped cell which holds 1M sulfuric acid solution. A Nafion membrane is positioned at the center of the horizontal section. A constant DC current (approximately 1 mA) runs through the platinum electrodes and dissociates the acid solution into hydrogen protons. The protons travel across the membrane as they would in a polymer electrolyte fuel cell and generate a voltage. The resistance will be measured by:

$$R = \frac{(V2 - V1)}{I}$$

V2: voltage across the chambers with membrane in place (V).

V1: voltage across the chambers with no membrane (V).

I: DC current (held constant at approximately 1 mA)

The membranes tested will be fabricated with different techniques to zoom in on the techniques that give the least resistance.

Various tests for this characteristic are presented in the literature. Among them is the four-electrode AC impedance test which was conducted last year by the single cell group [2]. However, the results from the test were inconclusive. Other possibilities are nuclear magnetic resonance (NMR), and other electromagnetic techniques such as fourier transfer interferometer resonance (FTIR) machine. The viability of these techniques will be discussed with both chemistry and material science professors. Another individual and group task includes keeping current on literature focusing on membrane assemblies and applications

Catalyst Application Techniques (Seana and Johnny)

Learning how to use the air brush in order to apply the electrocatalyst layer to the Nafion membrane is another task that needs to be accomplished. This will first involve finding an

appropriate laboratory where a circulated hood, and a nitrogen tank as compressed feed to the air brush is available. A good contact for this endeavor is Karen Fukuda since she has had some experience using this method of airbrushing. Other techniques of catalyst application will also be examined, such as sputtering coating, decal application.

Review of MSDS:

Chemicals we are concerned with include lead, isopropyl alcohol, hydrogen, oxygen, glycerol, tetra butyl ammonium hydroxide, hydrogen peroxide, Vulcan XC 72-R, Nafion solution from Aldrich, hydro sulfuric acid and sodium chloride. Lead can be poisonous if swallowed. Isopropyl alcohol is a potentially toxic chemical if swallowed or inhaled for long periods of time. In addition isopropyl alcohol should be used carefully as it may be ignited by heat; its flash point is 11.7 ° C. Acute effects of high exposure to hydrogen include nausea and dizziness. If hydrogen is inhaled the person should be removed to fresh air immediately, and a physician should be called if major symptoms arise. If oxygen is inhaled the person should be kept alert and a physician should be called. When exposed to glycerol it can cause headaches. Glycerol should not be mixed with hydrogen peroxide for a violent reaction can occur. This pertains only to a glycerol and H_2O_2 mixture and not other combinations such as glycerol and isopropyl alcohol. Tetrabutyl ammonium hydroxide is a relatively stable base which can cause severe irritation of the skin. In the case of hydrogen peroxide, it is unstable with heat or contamination. If inhaled drink plenty of water while skin contact does not pose problems. Vulcan XC 72-R in powder form can cause irritation to lungs. The hazards of Nafion solution at this point are unknown because a MSDS does not exist. Hydrosulfuric acid can cause skin burns if it exists in high molar concentrations. Sodium chloride is a highly stable chemical and poses no great health hazard.

Baseline Safety Assessment:

When operating or testing the single fuel cell, one should always be aware of the potential of hydrogen and oxygen mixing in the fuel cell stand. When working with glycerol, hydrogen

peroxide should not be mixed because a violent reaction can occur between these chemicals according to the MSDS for glycerol. Some basic safety precautions include wearing safety goggles and non-loose clothing. In addition group members should be aware of the location of the fire extinguisher, eye wash stand and sink. The potential for burns is mainly present when using the furnace and when operating the fuel cell stand. Attached to the stand are gas humidifiers which become extremely hot. In the near future we will be using lead wire to conduct resistance tests so this is a reminder to use appropriate gloves for lead and other harmful chemicals. When using the air brush make sure to use N2 as the compressed gas and not oxygen. In addition when preparing the ink powder (Vulcan XC 72-R) for the catalyst, proper facial mask needs to be worn.

List of Tables and Figures

Table 1: Preliminary Goals and Time

Week	Goals
1 (9/29 - 10/3)	Understanding of design problem and write preliminary proposal
2 (10/6 - 10/10)	Research and begin preliminary resistance test
3 (10/13-10/17)	Define goals and write proposal
4 (10/20-10/24)	Continue resistance test, practice catalyst applications, and research electrochemical H-cell
5 (10/27-10/31)	Begin making MEAs with revised techniques and start coordinating with the Systems Group to remodify the test stand to dead end hydrogen flow
6 (11/3 - 11/7)	Test current density on newly manufactured MEAs. Adjust fabrication techniques accordingly. Continue to research H-cell, and order material to build H-cell
7(11/10-11/14)	Make more MEAs with newly adjusted techniques. Write progress report
8 (11/17-11/21)	Investigate methods to quantify sodium concentration. Begin building the H-cell.
9 (11/24-11/26)	Continue building the H-cell.
10 (12/1 - 12/5)	Continue testing current density on MEAs with newly derived techniques.
11 (12/8-12/10)	Final Week

Table 2: Weekly Time Table:

The weekly meeting times, work times, and report writing times for each member in the Single Cell group are listed in Table 1 below:

Table 1: Time table for the Single Cell Group

Time	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday
7:30							
8:30		MN&SS		Meeting	TK,MN		
9:30	MN	MN&SS		Meeting			
10:30	MN	SS		Meeting			
11:30		SS		SS	TK		
12:30	Stuve			SS	TK		
13:30					TK		
14:30				SS	Stuve		
15:30				Meeting			
16:30		JF		Meeting	Meeting		
17:30		JF		Meeting	Meeting		
18:30				Meeting			
19:30							
2030							
2130							

Legend	
Meeting	All Single Cell group members meet to write and discuss progress. This also includes lab time.
JF	Johnny Ferara
TK	Timothy King
MN	Michael Nguyen
SS	Seana Seraji
Stuve	All Fuel Cell group members meet

Table 3: Group Organization:

Grp. Member	Address	E-mail/ Tel #	Emergency Contact
Johnny Ferara	13427 284th AVE. N.E. Duvall, WA 98019-6444	jtferara@u.washington.edu (425) 788-3028	Albert Ferara (Brother) (206) 885-3083
Timothy King	4240 8th AVE. N.E. Seattle, WA 98105-6039	tking@u.washington.edu (206) 632-7184	Sherry King (Sister) (206) 543-0200 (206) 632-7184
Michael Nguyen	743 N. 201 ST. Seattle,WA 98133	mn@u.washington.edu (206) 542-7309	Yvonne Bui (Friend) (206) 244-8141
Seana Seraji	5824 NE 75th #D103 Seattle, WA 98115	sserajii@u.washington.edu (206) 729-6061	Setty Seraji (Sister) (206) 526-9540

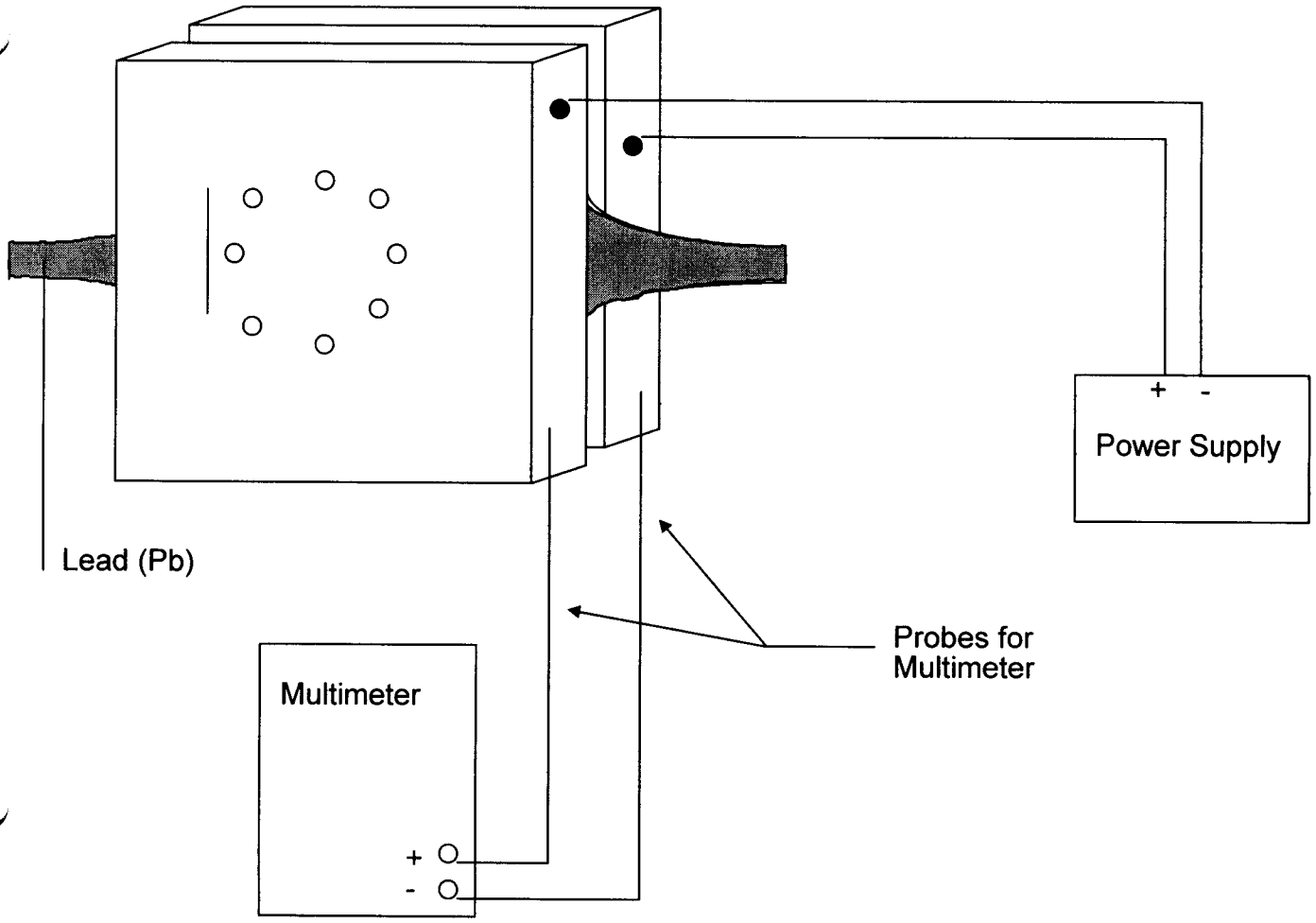

Figure 1: Schematics for the FFPs' resistance test.

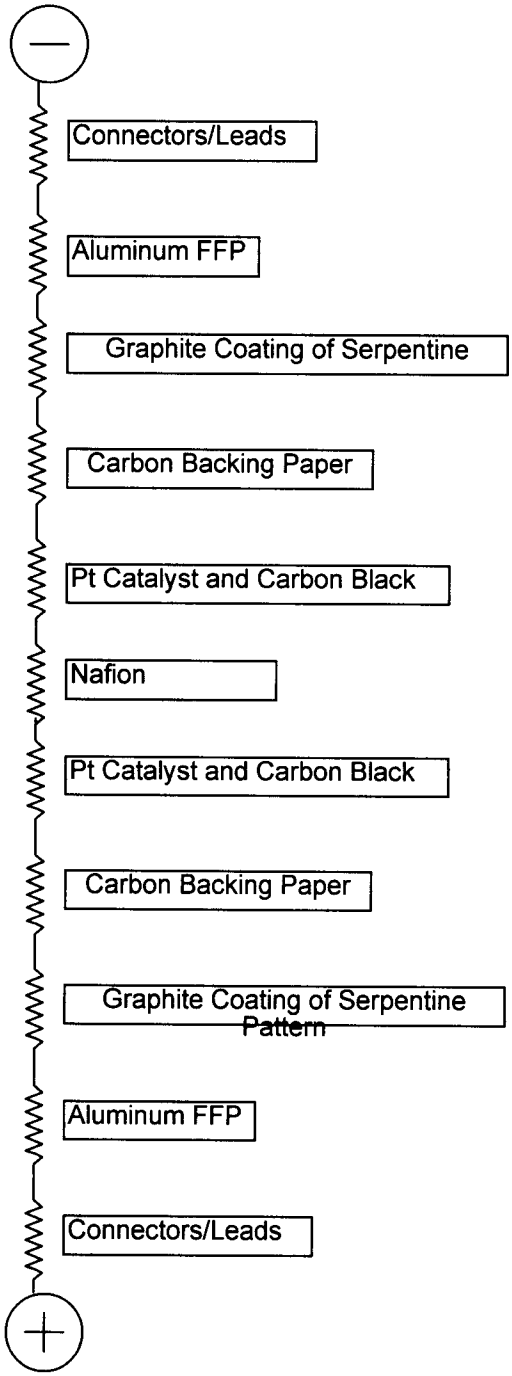

Figure 2: Resistor Train of the fuel cell.

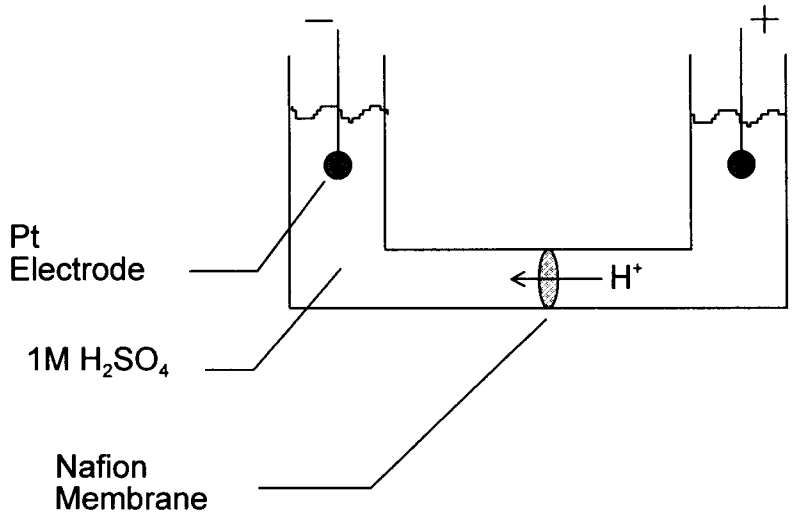

Figure 3: H-Cell Electrochemical Test for Proton Conductivity.

References:

1. Wilson, M.S., Valerio, J.A., Gottesfeld, S., "Low Platinum Loading Electrodes for Polymer Electrolyte Fuel Cells Fabricated using Thermoplastic Ionomers," Electrochim Acta. 40 (1995) 355-363.

2. Bailey, L, Ed Goldman, and Michael Nguyen, "Winter Quarter Final Report", 1997

Appendix A: Equipment Order List

The following list includes materials/equipment that should be ordered or obtained as soon as possible.

Description	Quantity	Recommended Vendor
Torque Wrench	One	Hardware store
NEW Macintosh Computer for electronic control purpose (minimum of 13" color monitor)	One	See Computer Lab Manager
Electrical wires	10 feet	Physics Store

Appendix B: Schematics of the Entire System and the Single Fuel Cell Test Stand

Note: Several valves will not be needed once we test our MEAs using dead-end hydrogen.

Blue = Hydrogen
Orange = Nitrogen
Purple = Hydrogen or Nitrogen
——— = Inlet Line
<> = Flame Arrestor

Red = Oxygen
Black = Oxygen or Nitrogen
Green = Water
- - - - - = Outlet Line
▷| = Check Valve

= Rotameter
= Pump
RV = Pressure Relief Valve

= Three-Way Valve
= %RH and Temp. Meter

Figure B-1: Legend for Figures B-2 and B-3

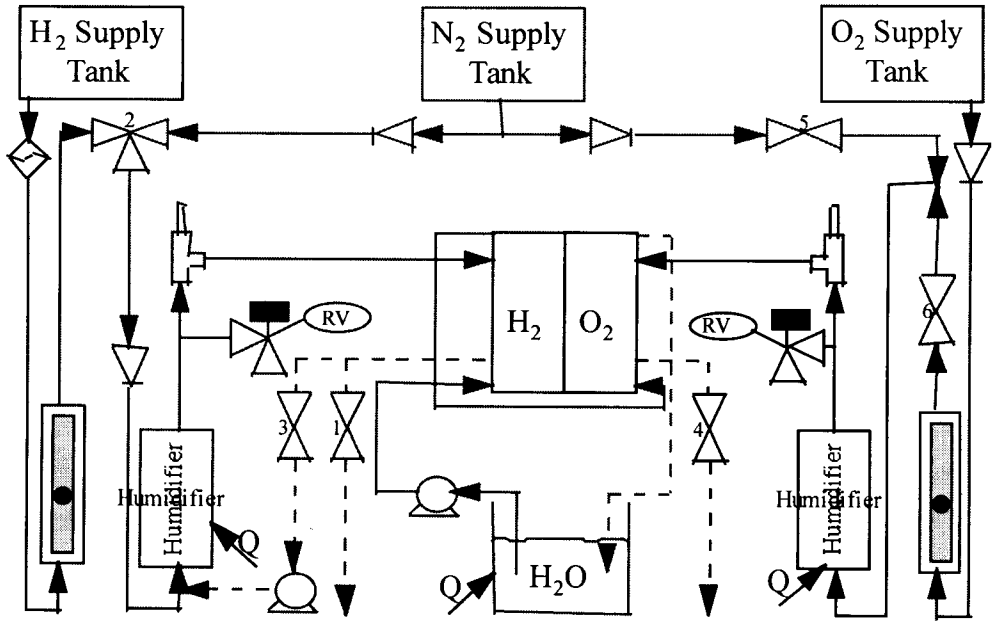

Figure B-2: Block Diagram of Fuel Cell Test Stand

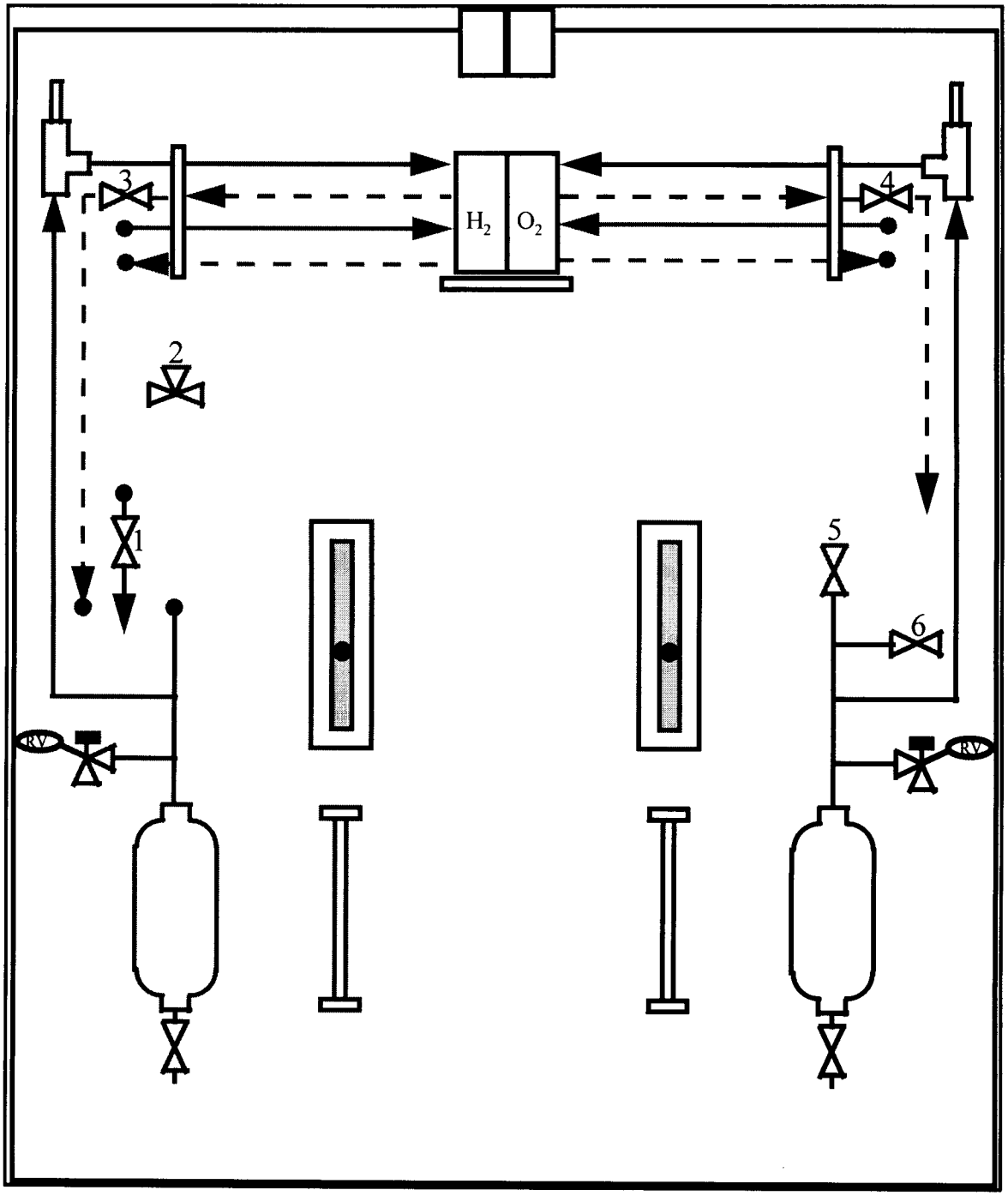

Figure B-3: Front of the Test Stand

Valve 1 = hydrogen purge; **Valve 2** = three-way hydrogen/nitrogen selection valve;

Valve 3 = hydrogen flow control valve; **Valve 4** = oxygen flow control valve;

Valve 5 = nitrogen feed for oxygen side; **Valve 6** = oxygen feed

Appendix C: Standard Operating Procedure for Test Cell and Stand

0. Emergency Shutdown Method

0.1 Runaway cell, immediately turn off main hydrogen and oxygen cylinder valves, making sure you cannot be burned from this action. If this is not possible, turn off gases with small valves on front of apparatus.

0.2 Flood both sides of gas delivery system with nitrogen gas.

0.3 Disconnect electrical power supply to band heaters and water supply system.

0.4 NOTE: Under no circumstance are hydrogen and oxygen gases to contact one another while at elevated temperatures or in the presence of the platinum catalyst.

1. Test Cell Assembly

1.1 Cell halves should be coated with ElectroDag and be in good condition. If either has been dropped or severely scratched, it should be checked to ensure that it can still function properly and then repaired.

1.2 O-rings are installed in each half, each should be retained in the gland even when the o-ring is .

1.3 A cut piece of Teflon coated carbon paper is placed in the recess in one of the halves, which has been suspended with the flow field facing up (the corner of a box works well for this).

1.4 A prepared membrane is then placed over the carbon paper, centering the electrode as well as possible on the flow area.

1.5 An identical piece of carbon paper is placed on top of the membrane, again centering as well as possible.

1.6 The second half of the cell is then brought down on this sandwich of parts. As it is brought down, it is imperative that the carbon paper find its way into and remain in the recesses above the flow fields. IF IT IS SUSPECTED THAT

THE CARBON PAPER IS NOT SEATED, DO NOT CONTINUE!!!
PROCEEDING WITH THIS MISASSEMBLY WILL RESULT IN
MEMBRANE DAMAGE AND A VERY DANGEROUS CONDITION.

1.7 After it has been made certain that the carbon papers are seated and the Nafion is making contact all around with the O-rings, bolt up can now be done. Using the nylon bolts, insert them all finger tight, then use the deep socket wrench only to tighten then in a circular pattern. Tighten to about 4 foot-pounds each (not very tight). Never use a wrench other than a box type or socket, as the nylon could be easily damaged.

1.8 After bolt up, unit is placed on the shelf of the test stand and all connections are made.

1.8.1 Attach feed gas tubes (these must connect to one of the center ports on the test cell.

1.8.2 Attach purge gas tubes to the other center ports.

1.8.3 Attach water feed and discharge lines to the outer ports on the test cell.

1.8.4 Connect resistive load to the cell.

1.8.5 Connect voltmeter to the cell.

2. Start Up of the System

2.1 Leak testing must first be performed.

2.1.1 With all valves closed, open main nitrogen cylinder valve 1/4 turn. Regulator pressure should not exceed 45 psi.

2.1.2 SLOWLY crack the valves feeding into the water bottles, one side of the cell at a time. This will charge the system through the cell. After charging, leave this valve open 1-2 turns.

2.1.3 Crack each exit valve for about 5 seconds to bleed air and charge entire system with feed gas. Then close these valves securely.

2.1.4 Close main cylinder valves tightly. A leak is now evidenced by a falling pressure reading on the high pressure gauge. If this pressure is falling faster than 100 psi/min., a substantial leak is present, and should be located/corrected. Correct the situation before continuing.

2.2 Disconnect gas supply to the test cell

2.2.1 Release gas pressure to both sides of the cell using the purge valves

2.2.2 Disconnect the gas supply lines to the cell

2.3 Start heating

2.3.1 Start water bath for heating of the cell, and turn on bottle heater VariACs to a setting of '60 V'. It may take up to 1/2 hour for substantial temperature rise.

2.3.2 Flow nitrogen gas through the system to heat the gas delivery lines (nitrogen will be vented to atmosphere at the cell gas supply lines).

2.4 When gas delivery lines reach the desired temperature and the nitrogen gas venting from the lines appears humid (collect it on a piece of paper), cell testing can begin.

3. Testing

3.1 Reconnect gas supply lines to the test cell and disconnect the exit lines from the cell.

3.2 Start heating/cooling water flow to the cell.

3.3 Allow the test cell to come up to the desired temperature while nitrogen gas flow continues.

3.4 NOTE: The membrane must remain humidified. Do not heat the cell to a higher temperature than that of the humidified feed gas!

3.5 Reconnect the exit gas lines to the cell.

3.6 Be sure that valve #3 is at least partially open and start the hydrogen recirculation pump.

3.7 Open the main valves on the hydrogen and oxygen supply tanks.

3.7.1 Adjust the pressure regulators to the desired pressure (less than 45 psig!)

3.7.2 BE SURE that the pressure on the nitrogen regulator is higher than that on the hydrogen or oxygen regulators. This ensures that there is no cross-flow of these gases through the nitrogen supply system.

3.7.3 Perform a second leak test as described previously in 2.1

3.8 Purge the nitrogen from both sides of the system using the two vent valves.

3.9 The cell should now be operational

3.10 Select different loads (resistance) to measure the voltage. Voltages are measured, and the resultant voltage versus current graph produced.

3.11 NOTE: Make sure water level and temperature in humidifier tanks is high enough to maintain good operation. This is one of the major problems with long term operation of the cell.

4. Shut Down of the system

4.1 Turn off hydrogen main cylinder valve, allow system hydrogen pressure to come to atmospheric pressure.

4.2 Turn off oxygen main cylinder valve, allow system to come to atmospheric.

4.3 Turn off heating units, water bath and bottle heaters.

4.4 Purge both sides of the system with nitrogen

4.5 Turn off volt meter.

4.6 Allow unit to cool sufficiently before handling

4.7 Disassemble cell, inspect components as desired.

5. Storage of the Test Cell

5.1 Unit should be stored disassembled with membrane faces secure from any form of damage

5.2 Over time, the ElectroDag coating may need to be replaced. This should be done with all components removed from the blocks, save the plugs in the water jacket. Follow the procedure appearing in previous works.

5.3 Membranes should be stored under deionized water in labeled containers. If it is necessary to store more than one in the same container, then they should all be discernible in some way. A suggested method is to cut very small notches into the periphery of the membrane, the part falling outside the sealing area.

5.4 Dry membranes should not be placed into the test cell. When put into use the membrane will swell. Only fully saturated membranes should be installed.

6. Other Notes

6.1 Carbon papers are cut using cookie cutter and nylon block in the press. Only run the press to 1500 psi, this is sufficient to cut through the paper. In the future, a new cutter will be made from a more suitable material (steel) once the correct size is known.

6.2 Nafion discs are cut from DRY stock with cutter an nylon block in press. This time around 2000 psi should be used. Don't worry if they appear small, upon saturation with water it will grow to the appropriate size.

6.3 It is unknown at this point the actual longevity of this cell. The ElectroDag coating is supposed to render the areas treated quite inert to this type of environment, but it is unknown whether or not sufficient coating is being achieved.

Appendix D: Fuel Cell Membrane Fabrication Procedure

The procedure followed during the course of our research is basically a reproduction of the procedure outlined in the paper by Mahlon, Valerio, and Gottesfeld; "Low Platinum Loading Electrodes for Polymer Electrolyte Fuel Cells Fabricated Using Thermoplastic Isomers" Electrochimica Acta, Volume 40, 1995, pp355 - 363 [1]. To add clarity, it is given below. Preliminary results using this procedure were very encouraging. However, reproduction of these results has been problematic. The attached plot details our initial findings.

Preparation:

Nafion Membrane

Materials:

- Nafion 117 membrane (MW = 1100, 0.18 mil thickness)
- 3% aqueous peroxide solution
- 1 M sodium chloride solution

Procedure:

- Cut membrane to fit the test apparatus, taking into account the fact that the Nafion will swell when hydrated
- Boil in a 3% by weight aqueous peroxide solution for 1 hour
  - performed to clean the membrane and saturate it with water
- Boil in approximately 1 M aqueous sodium chloride solution for 1 hour
  - performed to ensure complete conversion to the sodium form

Catalyst Ink

Materials:

- Nafion solution from Aldrich, 5% solubilized Nafion by weight
- Platinum catalyst
  - 10 % platinum by weight on carbon black Vulcan XC 72-R, the Electrosynthesis Company

- Tetrabutyl ammonium Hydroxide solution (TBOH), 1 M from Aldrich

- Glycerol, from Aldrich

- Isopropanol

Procedure:

- Combine Nafion solution and platinum catalyst material (1:3 Nafion to platinum ratio by mass) in a small flask

- Add isopropanol and glycerol until desired consistency is achieved

- Sonicate a minimum of 2 hours (longer is preferable)

- Add a 25 to 50% excess of the TBOH solution

  - TBOH must react with each sulfonate group in the Nafion solution.

- Knowing the amount of Nafion present, its molecular weight (1100), and the molecular weight of TBOH, the correct amount can be calculated.

- Sonicate a minimum of 2 hours

Membrane Assembly:

Materials:

- Small oven

- Airbrush

- Catalyst ink assembled above

- Copper gaskets from 2.75" Conflat flange

- Small screw-type hose clamps

- Lab press with heated platens

- Previously prepared Nafion Membrane

- Two Teflon sheets, slightly larger than the membrane

Procedure:

- Preheat small oven and press plated to 150 ^{o}C

- Clamp Nafion membrane between gaskets, centering it carefully

- At this point, we have two options, both of which have utilized successfully

    1. Apply the catalyst ink onto the membrane surface using the airbrush.

    2. Place two to three drops of Nafion solution on the membrane, followed by an application of the catalyst ink.

NOTE: The addition of Nafion solution prior to the catalyst application appears to aid adhesion of the ink.

- Hold assembly in the small oven and allow it to dry
- Remove membrane from clamp assembly
- Press membrane between Teflon sheets
- Press membrane and Teflon at 150 ^{o}C and 30 ATM for five minutes
- Remove the membrane and boil in 0.5 M Sulfuric Acid solution for 2 hours
- Boil in deionized water for 2 hours
- Allow to cool