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13. ABSTRACT *(Maximum 200 words)*

Analytic Power has proven the feasibility of a mobile electric power unit in the form of a hydride fueled ion-exchange membrane (IEM) fuel cell stack. We have over 5 years experience building and testing IEM fuel cells. The power section of a 350 watt fuel cell stack weighs 4.65 pounds based on our five cell sub-stack component weights. The composite stack structure is fabricated from two components; a unitized flow field and catalyzed membrane. The lightweight unitized flow field concept was proven in the first three months of the contract. A single unit flow field weighs 0.155 pounds and can seal to 60 psi. The single cell catalyzed Nafion membrane exceeded our performance goal of 0.6 volts at 100 ASF. Stack performance points were 0.42 volts at 113 ASF and .75 volts at 96 asf.

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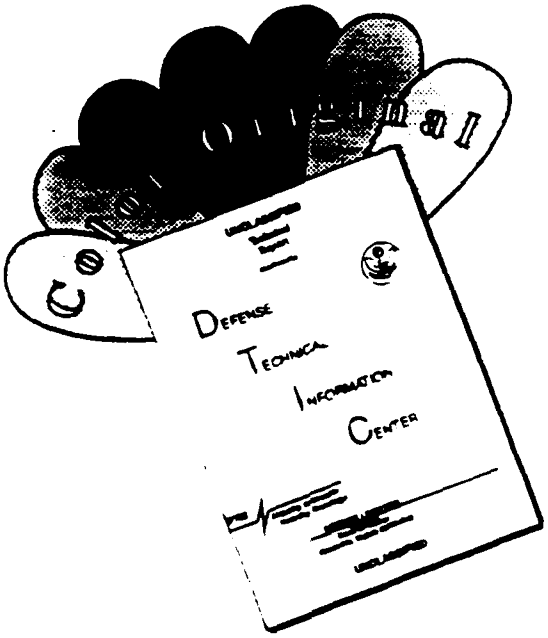
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INTRODUCTION

The purpose of the this program was to develop a small power source weighing close to five pounds and capable of supplying 300-350 watts of power. Current technology in this area is limited to batteries, solar panels and small gasoline engines. Each competitive power source has significant drawbacks. Batteries are limited by their relatively low energy density. Small gasoline engines are inefficient, noisy and unreliable. IC engines suffer from poor efficiency requiring a larg fuel supply. While gasoline has a high energy density, it also is highly flammable, has noxious exhaust fumes and has a high infrared signature. Solar power is unreliable and offers low power density.

Analytic Power has proven the feasibility of a mobile electric power unit in the form of a hydride fueled ion-exchange membrane (IEM) fuel cell stack. This unit operates with no moving parts at low temperatures (resulting in low IR signature), and produces water vapor as the only exhaust product. The fuel cell is a power source which combines the low maintenance of batteries with the light weight of gas engines and the cleanliness of solar power. A five cell sub-stack built and tested at Analytic Power is shown in the photograph below.

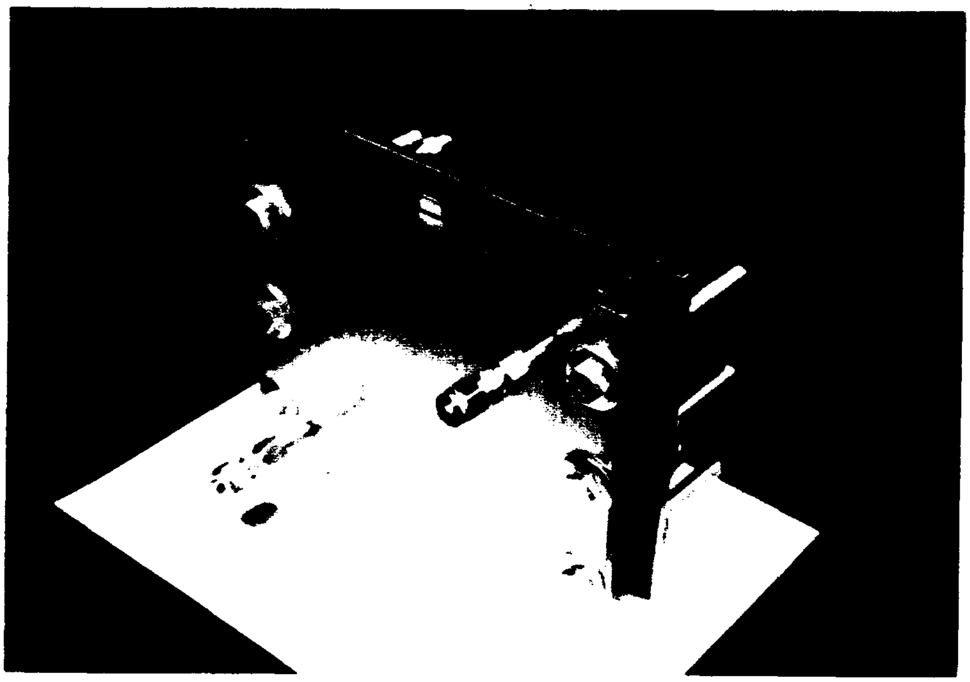

Fig. 1 ARO fuel cell power supply

PROGRAM OBJECTIVES

The contract objective was to design a cell stack capable of producing 350 watts of DC power at 5-6 lbs with an adequate water management system. We have come close to meeting all of our goals. We exceeded our performance goal of .6 volts at 100 asf. We identified the best membrane and catalyst loading to achieve our performance goal. Our full size stack weight is close to 6 pounds.

A further objective included building a 35 watt sub-stack in order to prove the capabilities of our lightweight technology and cell performance capability. Our sub-stack testing indicates a problem with water management and we will discuss this in detail in the conclusions and recommendations section.

CONCLUSIONS & RECOMMENDATIONS

New advances in catalyzation techniques, carbon composite technology and epoxy/adhesive seals have brought the cost and weight of our fuel cell stacks down considerably. We successfully demonstrated the capabilities of producing a lightweight, mobile, electric power supply. We achieved performance levels over .6 volts at 100 ASF. The power section of a full size 350 watt epoxy impregnated stack will weigh 4.65 pounds. This is based on the weight of components produced in this program. The end plate design is a limiting factor. G-10 (glass reinforced phenolic) and tie bolts raise the weight to near 7 pounds. Our testing identified a natural convection scheme for improved water management. Our finding are as follows.

FILM SEPARATOR TECHNOLOGY

Our original goal in designing this power supply was to remove all metal parts in the stack. The problems associated with metals in IEM fuel cells have kept these low end fuel cells out of the commercial market for decades. Simply put, most metal ions poison ion exchange membranes. Titanium with its limited aqueous chemistry is feasible but it is expensive, heavy and difficult to machine. It also oxidizes. Metals exposed to cell products oxidize, increasing resistance and degrading performance. Electroplating the surface with precious metal to inhibit surface oxidation pushes the cost over the affordable limit causing fuel cells to lose their competitive edge.

Our original thermoplastic separator plate design did not maintain a gas seal in most cases. Although, contact resistance was fairly competitive with its titanium alternative. The problem is the low glass temperature around 180°F. Most likely, the original stack was shorted when first placed on test. Shorting may have increased the stack temperature to the glass point of the thermoplastic. This is the reason we selected a thin metal separator to prevent crossover. We discuss this in detail under the bipolar plate heading.

The separator plate seals the gas but allows fiber contact between anode & cathode flow fields. Previously, we were limited to the epoxy outgassing temperature of 350°F since the flow fields must be epoxy impregnated prior to thermal bonding. If the frame can be sealed after bonding we may be able to use higher glass temperature films. This requires a different frame sealing technology which we will discuss in the following sections.

As an alternative, we may be able to eliminate the film and thermal bonding process completely. We've shown that epoxy filling the carbon paper perimeter seals gas. Epoxy filling the entire surface of a thin carbon paper sheet could provide the gas barrier. The fibers are already integral with the piece. Therefore, placing the anode and cathode flow field adjacent to the filled separator conducts electricity. The filled carbon sheet could be laminated with an adhesive frame to seal between the flow field/separator interface at the manifold port. Since the carbon epoxy surface is rough, the glue can displace into the surface ridges reducing dimensional differences. The adhesive layers do not require heat pressing. Resistance measurements should be taken to ensure resistances are not increasing. Cold pressing would significantly reduce fabrication time. However, the epoxy filling process takes time.

EPOXY IMPREGNATION

The epoxy impregnation technology is successful. However, there are limitations to its use. On the positive side, epoxy impregnation ensures a uniform thickness. The edge buildup problems were a result of the glue applied to the flat surface of the thermoplastic film rather than directly onto the porous carbon (refer to the unitizing process in the structural composition section). The epoxy filled edge is extremely strong since its integral with the carbon and not a separate piece. The seal has been designed and tested to 60 psi but it may hold even higher pressures.

The drawbacks are significant. The process of impregnating the surface neatly and accurately is extremely labor intensive. For high production volumes this would work against us in terms of time and money. Fabrication time may be reduced by vacuum impregnation. We weren't able to vacuum impregnate the thick cathode manifold with our existing equipment. With a different setup or a stronger vacuum it may be possible to impregnate the cathode more readily. Quality control is also difficult. The porosity and pore size of the carbon may differ and require varying amounts of epoxy to seal the anode frame. In this case a metering device may be useful in measuring fill quantities.

Also, carbon paper is a significant portion of the stack cost at almost 67%. The impregnation technology uses carbon paper in the inactive frame area increasing the stack cost.

EPOXY MOLDING

In a separate program at Analytic Power we investigated molding a solid epoxy frame in hopes of reducing fabrication time and carbon paper cost. The drawback are many. Replacing the porous rough surface of the carbon frame edge with smooth epoxy complicates the seal design. The adhesives used to seal are 1-2 mils thick. Without a surface to impregnate, the adhesive will increase the frame thickness causing centerline contact problems similar to our glue on film problem. This requires cutting down the frame edge by a mil or two. Its extremely difficult to hold these kinds of tolerances on an epoxy surface without expensive equipment. But the design requires an absolutely flat surface for sealing and anything less than a mil tolerance will probably not seal.

INJECTION MOLDING

An alternative is injection molding a frame around the active area of the carbon paper. We are investigating this as an alternative in our 25 watt commercial program. An injection molded piece, we are told, can hold these kinds of tolerances. The cost of the dies are significant at almost $2000 for a 3.75" x 3.75" aluminum die. In the long run the cost per piece and fabrication time reduction most likely make the unit price cheaper. It still remains to be seen whether this mil cutback in material will seal.

There are also some potential problems with an injected molded approach. Using a polypropylene material the cell edge will be weaker and may plastically deform under load. Injection molding relies on pressure rather than viscosity of the material to form the frame. With epoxy we used a very low viscosity fluid to penetrate and become integral with the carbon paper. The liquid polypropylene is pressure formed around the carbon paper. The highly viscous fluid will most likely not penetrate the carbon paper more than several mils at the edge. This weak connection may blow out under pressure. If so, minor modifications can prevent this from happening.

If injection molding is able to withstand internal pressures the ideal application is to injection mold the entire flow field at once. By first connecting the anode, separator sheet, hydrophyllic processed cathode and wetproofed electrode, one unit frame could be molded at once. This would eliminate any buildup problems and form a unit seal. The only sealing surface would then be the flow field membrane interface.

STACK CHANNEL LENGTH

By far the most important result of our sub-stack testing was the passive water management scheme. Forced convection which is not humidity controlled can dry out the membranes degrading performance. Humidity sensors are often quite large and would add significantly to stack weight. We know that passive management works on our small stacks. By redesigning our stack to reduce cathode channel length we cut down on the likelihood of blocked air flow. Using an electrochemical wetting process will also ensure the absorption of water beads into the flow field and away from the cathode surface. The increased heat release from increased power production should facilitate water evaporation from the cathode flow field. The design change only affects the length and width of the stack .

MEMBRANE FRAME

Our experience with membrane shrinkage and expansion indicates that with large active area cells a frame edge may eliminate the associated problems. We use this concept in our high performance 10 KW membranes. These membranes are designed to operate above 1000 ASF and over half a volt. The frame is a die cut mylar adhesive ring bonded to the membrane. Bonding the membrane to both sides of the flow field works well. Our sub-stack has been running intermittently for 2 months and no crossover has been encountered. However, adverse conditions such as extremely dry or cold environments may cause the membrane to shrink further from the manifold opening. Cold conditions often reduce the tackiness of some adhesives.

If the membrane extends to the stack edge it is important to cut the membrane immediately prior to placing it in the stack. Varying environmental conditions of heat and humidity will alter the distance between manifold holes. When this occurs the membrane is dipped in water and allowed to dry out until the manifold holes are aligned with the flow fields. This can take up to 15 minutes and must be continuously monitored. Catalyzed membranes should not be excessively handled since the catalyst can become contaminated. This is another reason to die cut just prior to stack fabrication.

IEM FUEL CELL OVERVIEW

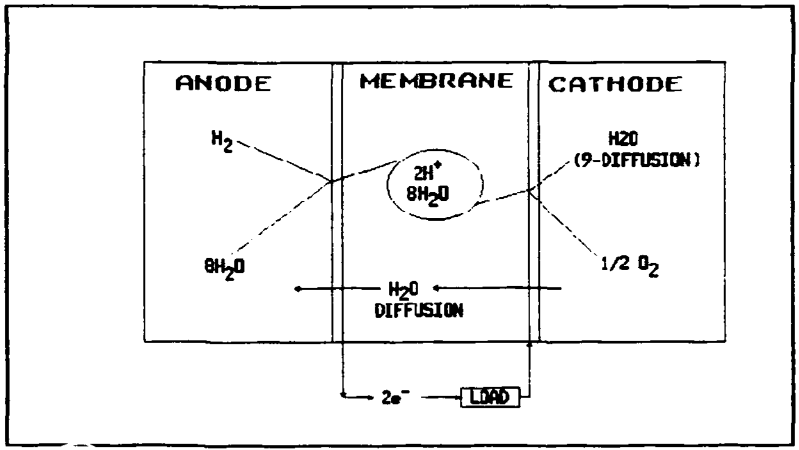

Fig. 2 IEM Fuel Cell

An IEM fuel cell is comprised of an ion-exchange membrane electrolyte sandwiched between two catalyzed electrodes. Fig. 2, above, shows an IEM fuel cell schematic. In a hydrogen/air cell, hydrogen at the anode reacts to form protons and electrons. Hydrated protons, comprised of up to 4 moles of H_2 per proton, migrate to the cathode. At the cathode, oxygen in the air is reduced and a mole of water is produced. The transport water diffuses back to the anode. Electrons from the reaction flow from the anode of one cell to the cathode of the adjacent cell in the stack. The DC current produced from the reactants is determined by Faraday's Law. The ideal voltage of the cell is given by the Nernst equation, (1), and is related to the hydrogen and oxygen partial pressures.

$$V=E_o-RT/nF*ln(r_p) \qquad \text{Eq. 1}$$

Where: E_o= Ideal voltage
R= Universal gas constant
T= temperature
n= equivalents/mole
F= Faraday constant
r_p= Pressure ratio

The actual voltage of the cell includes electrode and ohmic polarizations as shown in equation 2 below. The last term is the Tafel equation.

$$Vcell= Vnernst - (rho+anpol)*j - blog(j/j_o) \qquad \text{Eq. 2}$$

Where: Vnernst= Nernst potential
rho= resistivity (ohm ft^2)
anpol= anode polarization, (ohm*ft^2)
b= Tafel slope (volts)
j= current density, ASF
j_o= exchange current density

STRUCTURAL COMPOSITION

This section covers the structural composition of the fuel cell stack. It provides detailed explanations of the stack component materials and fabrication processes. The ARO stack is comprised of repeating packages of a unitized flow field (bipolar separator plate) and a catalyzed membrane cell.

UNITIZED FLOW FIELD

The crucial element in our stack is the porous carbon flow field material manufactured by Kreha Corp. of America. It's lightweight, almost 70% porous and exhibits low contact resistance due to the random fiber orientation. Unlike PAN type fibers which have laminar structures, KES-400 products are pitch based resin filled carbon papers. This carbon material is often used as oven insulation. The fiber orientation is important in that PAN fiber papers yield higher contact resistances.

Fig. 3 below shows an isometric projection of the unit cell. The unitized flow field is comprised of a flat anode flow field, a grooved cathode flow field, a bipolar separator and a cathode electrode. Removing the strength and rigidity of metal from this stack required a new approach to sealing. The following sections describe each component in the unitized flow field package.

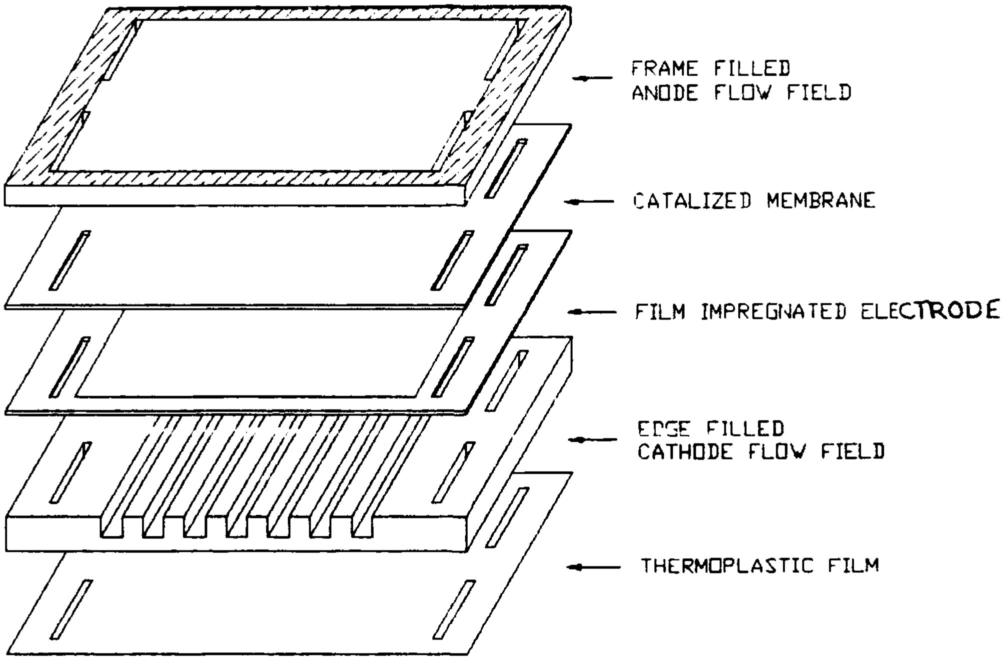

Fig. 3 Isometric projection of a unit cell

*************START CONFIDENTIAL PROPRIETARY INFORMATION**************

ANODE FLOW FIELD

The anode is fabricated from a 0.05 inch die cut piece of Kreha KES-400 carbon, see Fig. 4 below. A narrow dam of 3M-4060 film is thermo-compression bonded into both sides of the anode at the inner frame edge. This is done to control the flow of epoxy and prevent it from wicking into the active area. It also provides a clean edge around the active area. 3M's DP-270 epoxy/potting compound is brushed around the perimeter to seal the hydrogen within the plane of the anode. The excess epoxy is then wiped off to ensure a flat surface.

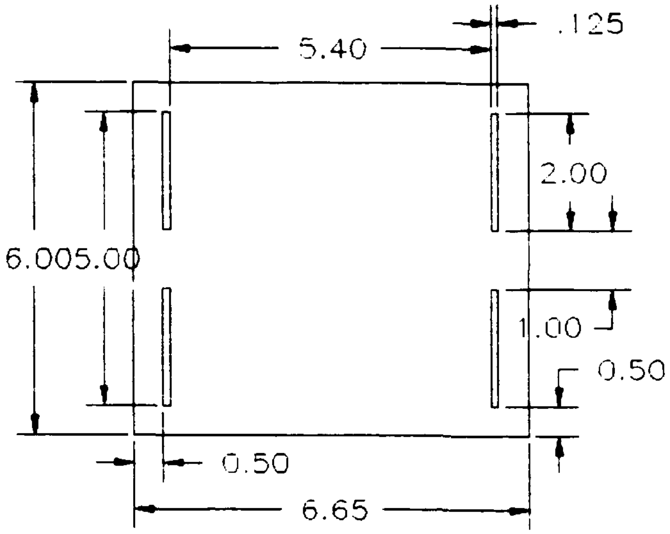

Fig. 4 Anode flow field

The full cure time for DP-270 epoxy at room temperature is 48 hours with a tack free time of 3 hours. Curing can be done in a 100 deg C oven in 30 minutes. Lower temperature curing for longer time periods is preferred since high temperatures cause the epoxy to flow more and could potentially cause a sealing loss. Care must be taken to ensure that the epoxy impregnated material does not touch any surface. In this case, the epoxy pools out of the carbon in that area and forms a surface ridge. Its important to allow the epoxy to fully cure before the bonding process. Otherwise it will boil and develop bubbles throughout.

CATHODE FLOW FIELD

The cathode is fabricated from a 0.130 inch thick piece of Kreha KES-400, grooved in the active region, see Fig. 5. A piece this thick with grooves is difficult to die cut with a standard die since liberating it from the die often causes the piece to crack. A clicker die (a die without a backing) can be used to cut this size but is quite a bit more expensive at approx. $1000 as compared to $100 for a standard steel rule die. Our solution was to fabricate a cutting template aligned within the grooves and use a razor blade.

The flow field is then processed to make it hydrophyllic (wettable). We investigated two methods to accomplish this task, electrochemical and flame treatment. The electrochemical treatment involves placing the flow fields in an acid bath and evolving oxygen off the surface. The cathodes are first submerged in distilled water in a vacuum jar to displace all of the air trapped within the pores. They are then placed in a .01 Normal sulfuric acid bath and connected to a 5 volt power source for 20 to 30 minutes. Afterwards, they are rinsed and baked in a 100 deg C oven for an hour.

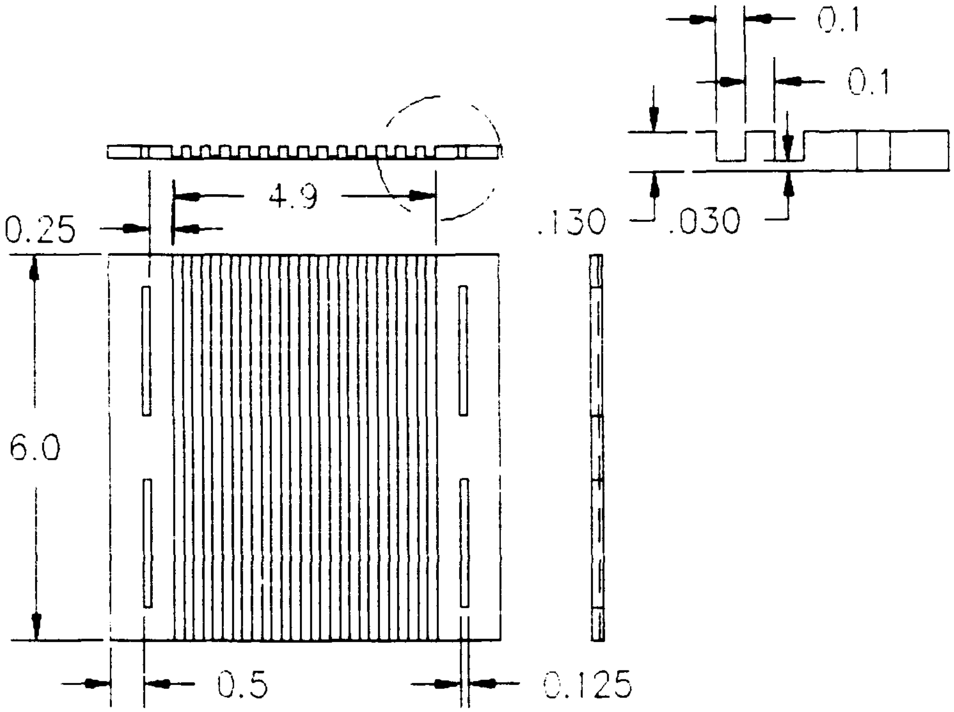

Fig. 5 Cathode flow field

The flame treatment is done with a propane torch. The piece is exposed to a blue flame until the surface becomes red. Although the flame treatment is faster and simpler its ineffective. This method only makes the very surface of the carbon wettable and it doesn't seem to last whereas the electrochemical method treats the whole thickness. Our small 25 watt commercial power supplies have employed this method successfully.

Cathode sealing was a challenge. Because of the 130 mil thickness, brushing on the epoxy is not enough to saturate the material. Although vacuum filling seems the ideal method we were unable to fully impregnate the KES-400. Sealing the cathode does not require filling the entire edge. Rather we are only concerned with sealing the manifold slots. The remainder of the cathode should be permeable to provide sufficient air flow and water absorption. We investigated potting the manifold slots with RTV (room temperature vulcanization) silicone rubber. Although it flows easily and readily saturates the KES-400, it seals poorly.

Rather than fill the carbon area with the epoxy, we completely fill the manifold slot leaving a slight meniscus. As the material dries it wets a small portion of the surrounding area along the entire thickness. A significantly higher amount of epoxy will wet the hydrophyllic carbon paper than the hydrophobic. The hydrophyllic properties and capillary forces, rather than gravity, cause the epoxy to wick into the surrounding pores of the carbon paper in order to seal the manifold port. This increases the likelihood of a continuous seal along the entire thickness of the cathode. The excess dried epoxy is machined out with a dremel tool leaving a small lining of epoxy around the manifold slot acting as the seal. The piece was tested to 50 psi and was able to hold pressure.

In a separate program at Analytic Power we explored the possibility of forming a solid epoxy shape rather than impregnate the surface to seal around the cathode manifold. This eliminates the lengthy machining step of drilling out the epoxy. Its possible with special molds but difficult to ensure the proper surface thickness.

BIPOLAR PLATE

Our original bipolar or separator plate was simply two sheets of thermoplastic film sandwiched between the anode and cathode flow field. During thermocompression bonding the fibers of the adjoining flow fields penetrate through the thermoplastic and provide the electrical connection. The film acts as a gas seal to prevent crossover from anode to cathode. Since the epoxy outgasses above 350 deg F, we were limited in our selection of thermoplastic film to one that bonds at about 300°F. 3M's 4060 film has a glass temperature of 180°F.

The 4060 film has several notable characteristics. It does not adhere to an epoxy impregnated surface. Most important however is that continued exposure to moisture and temperatures seem to cause it to weaken. We were successful at first in preventing gas crossover between anode and cathode but the prolonged exposure to water and pressure from leak testing may have weakened it. The unsupported film has no strength. In places where the cathode surface has a pit the film is unsupported. The pressure from the anode side could potentially break the film in that region causing crossover. This might explain why not all of the originally fabricated plates exhibited crossover.

As a quick solution we replaced the thermoplastic separator sheets with an aluminum foil sheet sandwiched between adhesive layers. The aluminum foil successfully prevents gas crossover. 3M's 9752 adhesive allows the fibers of the carbon paper to contact the aluminum foil and prevents metal ions from contaminating the membrane. Preventing exposure of the metal to reactants and products prevents oxidation of the metal which will increase its resistance.

ELECTRODE

The cathode electrode is needed only to support the membrane across the cathode flow field grooves. It is fabricated from a die cut piece of 0.016 inch flat Kreha HE-200 carbon paper. We also use Toray TGP-H-090 carbon paper which is 10 mils. We prefer the HE-200 because it has a lower contact resistance and it's slightly thicker. Thickness of the material becomes important depending on the sealing requirements. A thicker piece provides more substrate for the epoxy and adhesives that must impregnate the edge without buildup. Any buildup of materials at the stack edge will disturb the planarity of the components. This will usually result in increased contact resistance. Trying to press too much material into the surface will also cause the carbon paper to crack.

The electrode is wetproofed prior to edge sealing since the temperature requirements far exceed the limit of the epoxy. Teflonating or wetproofing the electrode causes water to bead up on the surface. This pulls the liquid product water away from the membrane surface preventing it from flooding and ensuring free catalyst sites to react with the oxygen in the air. These surface water beads are then drawn into the hydrophyllic or wettable cathode flow field.

The edge sealing is accomplished in much the same manner as the anode flow field. The electrode seal frames the active area edge surrounding the manifold holes to prevent hydrogen from traveling within the plane of the electrode. Before the epoxy is applied the wetproofing at the cathode edge must be burned off with a torch. Epoxy will not saturate a hydrophobic surface. 4060 film dams are bonded into both sides and DP-270 epoxy is used to fill the frame edge.

UNITIZING PROCESS

To make the unitized package the bipolar plate is placed between the anode and cathode flow fields. The electrode is bonded to the cathode surface with adhesive. We used a frame of 4060 film on the anode and electrode surface to create a flat sealing surface for the membrane. The package is thermo-compression bonded at 300 deg F for 10 minutes under a press pressure of 400 psi. In hindsight, it appears that the 4060 frames around the anode and electrode cause edge buildup increasing contact resistance. We will discuss these problems in the stack fabrication section.

**************END CONFIDENTIAL PROPRIETARY INFORMATION*************

A photograph of a unitized flow field is shown below.

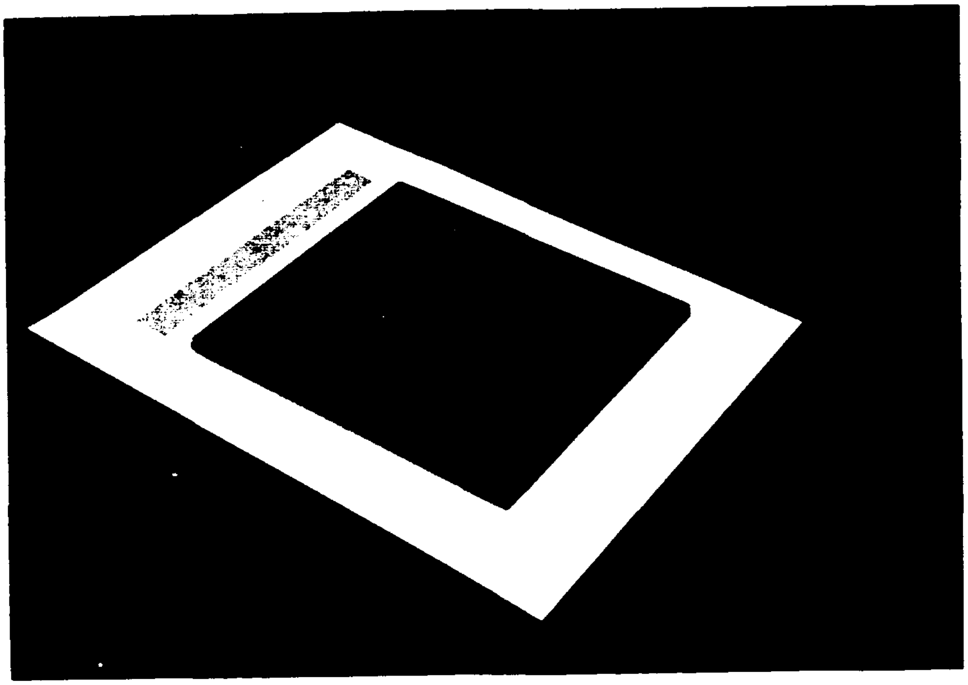

Fig. 6 Unitized flow field

CATALYZED MEMBRANE

During the contract we explored several different membranes and catalyst fabrication techniques. The membranes tested include Dupont's Nafion 117 and Dow membrane. Since performance and resistance values between the two were close the Nafion membrane was chosen for its low cost of $\$80/ft^2$ as opposed to Dow at $\$200/ft^2$.

A good deal of work has been done recently at Analytic Power towards developing fuel cells made with directly catalyzed ion exchange films. This technology has been under development at Los Alamos National Laboratory for some time now and we have been working very closely with LANL in this effort. Cells made with this technology use about 3 to 5% of the amount of platinum used in standard 4 mg/cm2 platinum black electrodes while still achieving similar performance.

Analytic Power's standard cell fabrication method involves teflon bonding catalyst to the anode and cathode electrodes and bonding the electrodes to the membrane. The catalyst loading used in this method is approx. .6 mg/cm^2 as opposed to the catalyzed membrane loadings of .2 mg/cm^2. The real savings however is not the catalyst cost but rather the reduced fabrication time to catalyze the membrane directly.

Two methods of application, direct and transfer catalyzation were tested. The fabrication methods are as follows.

**************START CONFIDENTIAL PROPRIETARY INFORMATION*************

DIRECT CATALYZATION

The process used to fabricate directly catalyzed film involves applying a catalyst ink to the electrolyte film and evaporating the solvent. The ink is composed of 20% Pt on carbon catalyst, liquid Nafion solution, water and glycerol. The weight ratio of catalyst to dry polymer in the Nafion solution is 3:1 and the ratio of carbon in the catalyst to water to glycerol is 1:5:20. This mixture is thoroughly stirred and the correct amount is weighed out in order to apply the desired platinum loading to the film. Analytic Power has been using .2 mg Pt/cm2, but LANL has reported similar results using only 0.1 mg/cm2. The ink is then brushed onto the dried film and baked in a 140 deg C oven until the solvent has completely evaporated. Extra coats can be applied in succession until the desired catalyst loading has been achieved. After catalyzation, the film is boiled in 3% H_2O_2 and heated in 1 N nitric acid to oxidize any remaining solvents and restore the film to the hydrogen form.

TRANSFER CATALYZATION

In the transfer catalyzation process, an ink containing catalyst and electrolyte polymer is evaporated on sheets and pressed onto an electrolyte film. The catalyst ink is composed of 20% Pt on carbon catalyst mixed with a 5% solution of Nafion polymer dissolved in alcohol. The optimum ratio of catalyst to Nafion in the ink has been experimentally determined to be 3:2. This results in an ink that is 1.4% Pt by weight.

After the ink has been mixed, the amount needed to produce the desired catalyst loading on the electrodes (usually 0.2 mg Pt/cm^2) is weighed out and spread with a brush onto sheets of glass reinforced Teflon that have been cleaned with acetone. The ink is brushed on in the same pattern as the finished electrode will appear. The sheets are then placed under infrared lamps until the alcohol from the ink has completely evaporated.

A sheet of electrolyte film is then sandwiched between the two sheets of Teflon. This sandwich is pressed at 300°F and 50 atm of pressure for 90 seconds. The sandwich is then removed from the press and the Teflon sheets can be peeled off, thereby transferring the catalyst layers onto the electrolyte film. The completed cell is then boiled in H_2O_2 to oxidize any remaining hydrocarbons and soaked in hot acid to restore the full ion exchange capacity to the electrolyte. The cell is then either boiled in water or thoroughly dried, depending on the application.

****************END CONFIDENTIAL PROPRIETARY INFORMATION***************

Our original set of cells were fabricated using direct catalyzation. This method is easier since the catalyst doesn't need to be pressed onto the membrane which adds a step. However, we believe applying the solvent directly to the membrane makes it more susceptible to pinholes resulting in gas crossover. The membranes were also weaker and ripped easily.

At first we were successfully able to patch the pinholes we discovered. However, each successive time the stack was placed on test new pinholes would arise. We fabricated a new set of membranes using transfer catalyzation. These were catalyzed in the active area only. The new membranes are much tougher than the original membranes and appear resistant to pinholes. The added toughness allowed us to remove the glued cells in tact from the unitized flow fields to process them in acid.

Four membranes were fabricated using glycerol in the catalyst ink like the ink used in the direct method. Since evaporating the glycerol is a lengthy process we fabricated the fifth cell without using glycerol as described above. This cells performance is somewhat higher than the other four membrane cells and will be covered under the performance testing section.

CURRENT COLLECTORS

The endplate collectors were fabricated out of G-10 (glass filled phenolic) plastic and brass, see Fig. 7. Three sheets of G-10 were punched out using the cell and flow field dies and held together with mylar with adhesive on either side. A brass current collector with a tab is placed in the G-10 frame and a non-unitized flow field is glued to the surface encasing the brass. The tab protrudes from the frame for the electrical connection rather than using the end plates as conductors.

STACK FABRICATION

Stack assembly is relatively simple since the unit cell consists of only two parts, the unitized flow field and the cell membrane. Successive plates and membranes are stacked together on an alignment jig through the manifold slots. The stack is placed between two sheets of 1/16th inch silicone rubber to seal between it and the end plates. A complete materials breakdown including specific cost and weight is shown in TABLE 1 on the following page.

Six tie bolts are placed along two opposing sides of the stack. The stack was placed under 3,000 psi press pressure which is 6000 lbs. force and the bolts were tightened. The resulting contact pressure is 150 psi assuming flat uniform contact. The resistance under load varies daily but is approximately 150 milliohms.

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IEMFC - FUEL CELL STACK CHARACTERISTICS       TABLE 1                          DATE  1/29/92
TYPE 3.0 FUEL CELL (ARO TECHNOLOGY)                                          ARO/CAT MEMBRANE
ANALYTIC POWER CORPORATION                                              FILE: CELL\IEMFC\STACK30
*********PROPRIETARY****************PROPRIETARY****************PROPRIETARY*****************PROPRIETARY********
+========================+=======================================+===================================+
|POWER              350  WATTS   STACK LENGTH          6.44  IN     |TOOLING COSTS                      |
|VOLTAGE             18  VOLTS   STACK WT.             7.38  LB     |          $4,603                   |
|CELL VOLTAGE       0.6  VOLTS   STACK COST          $2,499         |SPARES COST       SPARES        10%|
|CURRENT DENSITY    108  ASF     SPECIFIC COST       $7,139  $/KW   |           $250                    |
+------------------------+---------------------------------------+-----------------------------------+
|EDGE (5.0 x 5.1)   5.1  IN    (  W X L)=    6.65    6.00  IN     |NUMBER OF STACKS                  2|
|ACTIVE AREA       25.5  IN2   (    63.9%)                          |MATERIALS                    $4,997|
|FRAME AREA        14.4  IN2   (    36.1%)                          |TOOLING                      $4,603|
|TOTAL AREA        39.9  IN2   (   100.0%)                          |SPARES                         $500|
|No. OF CELLS        30            CAT LOADING          0.2  mg/cm2 |        TOTAL               $10,100|
+========================+======================+==================+==================+================+
|                        |                      |     WEIGHT       |                  |                |
|CELL COMPONENTS MATERIAL|  THICK. SPEC.WT. # PCS.| TOTAL    TOTAL  |  COST     COST   |    COST        |
|                        |   MILS  (g/pc)       |  (lb)     (%)    |   (%)     ($)    |    BASIS       |
+------------------------+----------------------+------------------+------------------+----------------+
|MEMBRANE        NAFION  |     7     4.7     30 |  0.310           |           $497   | $85.00 $/FT2   |
|                3M-9752 |     3     1.2     30 |  0.077           |             $3   |  $0.70 $/FT2   |
|                  TOTAL |     7     5.85       |  0.387    5.24%  |  20.01%    $500   |                |
+------------------------+----------------------+------------------+------------------+----------------+
|ANODE ELECTRODES    CAT |     1     0.2     30 |  0.011           |            $84   |    $17 $/GM    |
|                 TFE 30 |           0.1     30 |  0.004           |             $0   |  $0.12 $/GM    |
|                LIQ NAF |           0.0     30 |  0.002           |            $14   |    $19 $/GM    |
|                  TOTAL |     1     0.25    30 |  0.017    0.23%  |   3.91%     $98   |                |
+------------------------+----------------------+------------------+------------------+----------------+
|CATHODE ELECTRODES  CAT |     1     0.2     30 |  0.011           |            $84   | $17.00 $/GM    |
|                 TFE 30 |           0.1     30 |  0.004           |             $0   |  $0.12 $/GM    |
|                LIQ NAF |           0.0     30 |  0.002           |            $14   |    $19 $/GM    |
|                 HE 200 |    10     4.3     30 |  0.285           |           $177   |    $13 $/FT2   |
|                3M-4060 |     5     1.3     30 |  0.088           |             $5   |     $1 $/FT2   |
|                  TOTAL |    11     5.90    30 |  0.390    5.29%  |  11.19%    $280   |                |
+------------------------+----------------------+------------------+------------------+----------------+
|BIPOLAR FLO FIELD       |                      |                  |                  |                |
|       ANODE    3M-270  |    50     7.6     30 |  0.501           |             $3   |     $5 $/LB    |
|               KES-400  |    50    14.7     30 |  0.976           |           $396   |    $57 $/FT2   |
|               SUB TOT  |          22.31       |  1.476           |           $398   |                |
|       CATHODE  3M-270  |   130     1.2     30 |  0.079           |             $0   |     $5 $/LB    |
|               KES-400  |   130    35.9     30 |  2.374           |         $1,098   |   $169 $/FT2   |
|               SUB TOT  |          37.08       |  2.453           |         $1,099   |                |
|       INTERFACE 3M-4060|     5     3.7     30 |  0.244           |             $8   |  $1.00 $/FT2   |
|                  TOTAL |   180    63.1        |  4.173   56.57%  |  60.24%  $1,505  |                |
|                        |  -----               | -------  ------- | -------  ------- |                |
|                        |  5.94 in    SUBTOTAL |  4.967   67.33%  |  95.36%  $2,383  |                |
|                        |   198 mil   per cell |  0.166    2.2%   |            $79   |                |
+------------------------+----------------------+------------------+------------------+----------------+
|ENDPLATES     FIBERGLAS |   250   233.9      2 |  1.031           |            $50   |    $25 $/PC    |
|TIE RODS          STEEL |         104.3      6 |  1.379           |            $66   |    $11 $/PC    |
|                        |  ----                | -------  ------- | -------  ------- |                |
|                        |   500 SUBTOTAL       |  2.410   32.67%  |   4.64%    $116  |                |
+------------------------+----------------------+------------------+------------------+----------------+
|TOTAL                   |                      |  7.378           |         $2,499   |                |
+------------------------+----------------------+------------------+------------------+----------------+
*********PROPRIETARY****************PROPRIETARY****************PROPRIETARY*****************PROPRIETARY********
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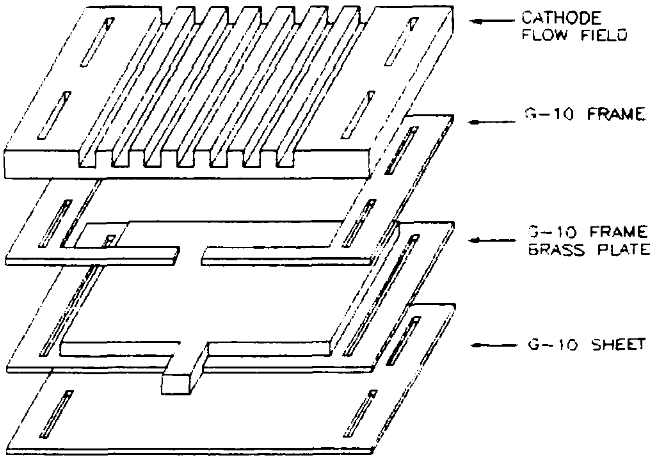


Fig. 7 Current collector

Several problems were apparent upon building the stack. The center of the stack was unloaded and the end plates were deflecting. The epoxy, film and glue used to seal and bond the flow fields together builds up the edges of the unitized plates. When successive cells are stacked up this added dimension causes the center of the stack to unload.

To reduce the high contact resistance from the electrode edge buildup we placed carbon paper shims in between the electrode and the cathode flow field grooves. This not only reduced the contact resistance but aided in sealing. The sealess design relies on absolute component planarity. The cathode end current collector contains a 10 mil sheet of Toray carbon paper. The unitized flow field closest to the anode end of the stack contains a sheet of 3 mil Toray paper. The 3 mil carbon paper is approx. the dimension of the unit cell edge buildup. The 3 mil sheet must be placed in position prior to unitizing since it has no strength and would crush trying to slide it in place later.

Exacerbating the problem is the deflection of the end plate. The center is 10 - 20 mils higher than the edges. This forced us to use a bulky follow up system to load the centerline of the stack. We placed two unistrut bars horizontally across the middle of the stack. This reduced the resistance significantly. After leak testing was complete we placed 4 C-clamps at each corner of the stack near the center load plate. This reduced the resistance again by half.

A third cause of edge buildup and contact problems is the added glue layers at the frame edge. Due to membrane contraction, discussed in the following section, we had to glue both sides of the membrane to the adjoining surfaces. As mentioned previously, the 4060 film thermal bonded to the anode and electrode frames works against us in this case. The thermoplastic flows into and fills the rough surface of the flow field in the frame area. Any material placed on top of the film causes buildup. If we knew glue would be necessary on the anode and electrode side we would not have impregnated the surface with thermoplastic as well. The added glue layers almost doubled the resistance.

STACK WEIGHT & SIZE

To produce 350 watts with an active area of 25.5 in^2 at a performance level of .6 volts and 100 ASF we will consume 0.024 lbmoles/hr of hydrogen (see equation 3 below).

$$\dot{n}_{H2} = \frac{.04112 * P_G}{V_C} \qquad \text{Eq. 3}$$

where:

P_G = Gross power,KW
V_C = Cell volts
$\dot{n}_{H2}$ = H_2 consumed,lbm/hr

This is also equal to:

$$\dot{n}_{H2} = .04112\ E\text{-}3 * J * A_e * N_c \qquad \text{Eq. 4}$$

where:

J = current density, ASF
A_e = Cell area, ft^2
N_c = Number of cells

Solving for N_c yields a cell number of 33 at this performance level. Increasing the current density to 110 ASF, 350 watts requires 30 cells. At 0.155 lbs. per unit cell (based on component part actual weights), a 30 cell stack excluding endplates would weigh 4.65 lbs.

The power generation portion of the stack has an acceptable weight. Reducing the follow-up system will require a substantial portion of a Phase II program. We've considered Commercial Carbon Composite (C3) end plates manufactured by Fiber Materials Inc. The material weight for two endplates is 2.5 pounds. This brings the stack weight to over 7 pounds. The problem with C3 is the cost. At $131/per plate for an 8" X 6.5" plate, the cost is $363/$ft^2$. Performance improvements which we've achieved with our single cell testing, .7 volts at 110 ASF, would reduce the cell number to 26 dropping the stack weight to 6.5 pounds. A better solution is to identify a less costly material and an improved end plate design.

The overall size of the unitized plate is 6.65" x 6.0" with an active area of 5.1" x 5.0", see figures 4 & 5. The thickness of the plate is about 0.2 inches. Assuming a 30 cell stack the power section dimension is 6.65"L x 6.0"W x 6.0"H. The current test endplate are 8.0" x 6.5" x 0.25" stainless steel. Adding an additional 0.5 inch to the total stack height. These are test plates only. At a weight of 8 lbs. and 1 lb. more for tie bolts, the end plates are almost twice as heavy as the entire fuel cell stack. A lighter weight material will most likely be thicker since stainless steel has a very high Young's modulus of around 30 million. A possible alternative are fiberglass end plates. The additional weight is just over a pound and the total cost is only $50.00. This fiberglass has a Young's modulus of elasticity of 2-4 million.

TESTING

Our work included individual cell and flow field testing as well as stack leak, resistance and performance testing. The following sections will describe all of the experimentation and results of those tests.

LEAK TESTING

A significant amount of time was spent pressure testing the stack. There are several potential areas where leaks can occur. These areas include leaks through the epoxy seal frames; crossover through the separator plate; membrane pinholes; membrane shrinkage; cathode groove edge electrode deflection and misalignment. We investigated, encountered and cured each one of these leaks.

ELECTRODE GROOVE EDGE LEAKS

Although the electrode is impregnated with epoxy and thermoplastic film it has very little strength. The electrode is unsupported every other 0.1 inches when it traverses a cathode air channel. One side of the membrane is supported uniformly by the anode flow field surface and the opposing side must counteract the load in order to seal around the membrane. The electrodes are not strong enough to support the load and gas escapes in the area where the cell and electrode are unsupported. Gas leaks of this nature are seen along the cathode groove edge. Gluing the membrane to the anode surface cured this problem since only the hydrogen side of the cell is pressurized. This eliminated the stack perimeter leaks.

BIPOLAR SEPARATOR LEAKS

Many of our 4060 film separator plates leaked during stack submersion and the stresses of pressure and load testing. Gas leaks are evidenced by bubbles rising through the center cathode channels. Our solution to this problem was to replace the thermoplastic with an aluminum sheet sealed between acrylic adhesive. This was a quick fix only since time was limited. Our current commercial fuel cell development stacks use 1 mil stainless steel shim stock separator. The important factor with a metal separator sheet is that it be completely sealed from exposure to stack reactants and products. Metal ions that dissolve in the product water poison the membrane causing performance degradation.

MEMBRANE PINHOLES

Membrane pinholes are usually a rare occurrence. Our first set of cells however, had an abundance of pinholes, see Fig. 8. Membrane pinhole leaks can be confused with plate crossover leaks since the result of both is a low open circuit potential. Even a small amount of hydrogen exposed to the cathode side of the membrane significantly degrades the cell voltage. The transfer catalyzed membranes we are currently testing appear to be pinhole free. However, as a precaution we purge the stack with nitrogen prior to placing on test. This eliminates any oxygen that may be present on the anode side.

MISALIGNMENT

One of the aluminum separator unitized flow fields was slightly misaligned in the press. The resulting leak was exhibited by bubbles through the stack tie bolt edge and through the first cathode channel. Since the anode manifold was misaligned with the cathode manifold, the hydrogen travelled through the anode manifold hole and out around the cathode manifold hole where there is no epoxy. The cathode is sealed around the holes only. Epoxy was used to fill the exposed part of the cathode sealing surface. Leak testing indicated a seal was maintained.

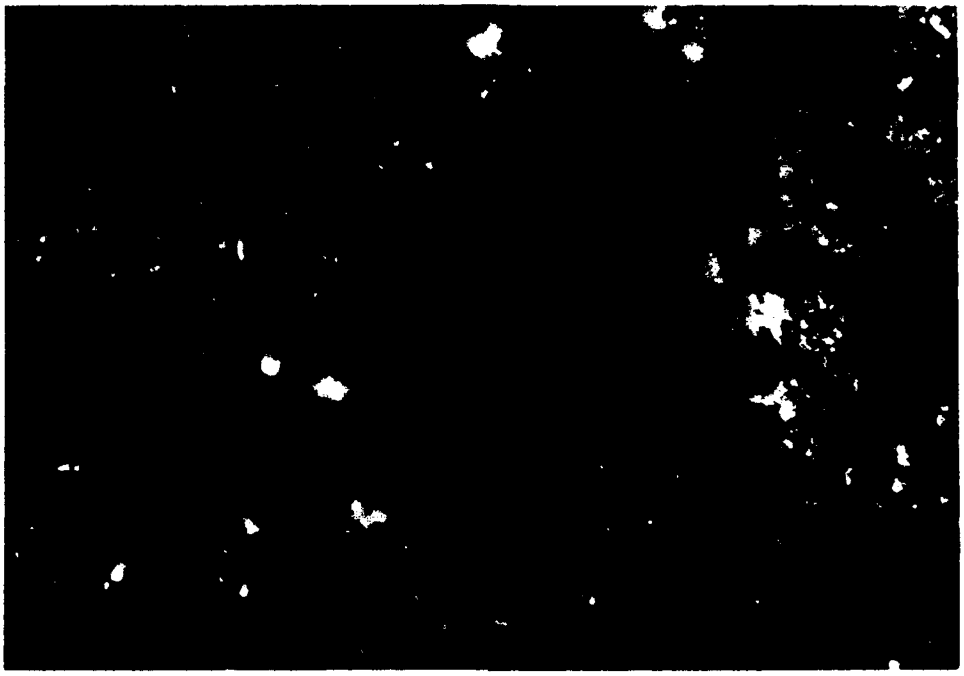


Fig. 8 *Photomicrograph of a* pinhole

MEMBRANE SHRINKAGE

During leak testing, the stack edges and often the entire stack is submerged in water. This causes the membranes to expand considerably. Subsequently, when the stack dries out the membranes shrink considerably. Membranes are very sensitive to ambient humidity and should be die cut immediately prior to stack fabrication.

Many of the membranes pulled away from the manifold inner edge. This exposes the anode hydrogen space to the cathode groove area which is open to the environment. Since the membrane was not blocking access to the cathode side, gas diffused through the cathode channel edge rather than reacting at the membrane surface. The result is the same as a membrane pinhole but the gas only escapes through the first cathode channel at the manifold edge. To prevent a sealing loss from membrane shrinkage, we were forced to glue the membrane to the cathode side as well as the anode. The result is a solid unit stack that cannot be disassembled without some damage.

MEMBRANE EXPANSION

As mentioned above, the membrane greatly expands when wet almost 2 inches wider than the cut dimension. Membranes expand asymmetrically depending on the orientation of the roll. Since they are glued at the frame, expansion then results in a bubble in the active region. Care must be taken not to build or tear down the stack when the membranes are very wet. The electrode can crack in the area where it is unsupported, such as across the cathode groove. If this occurs the membrane bulges out into the groove space from the anode gas pressure and it also blocks off the cathode channel to natural convection. Under a stack sealing load the membranes only expand slightly in the plane of the surface.

RESISTANCE TESTING

FLOW FIELD RESISTANCE

We tested a spare unitized flow field for contact pressure limit and resistance. Although Kreha KES-400 has a 400 psi contact pressure limit the grooves on the cathode reduce the surface contact area. Therefore, the total force limit on a 39.9 in^2 piece is 8000 lbs. instead of the almost 16,000 for a flat surface. We subjected the piece to a force of 9000 lbs. which resulted in a characteristic 45° shearing of several of the cathode bars. Figure 9 shows a graph of contact pressure vs. resistivity. The resistance difference between 9,000 lbs. and 6000 lbs. compression is only 10 milliohms. To avoid shearing we used a stack loading of 300 psi yielding a resistivity of 3.6 $ohms*cm^2$. The contact resistances are comparable with Analytic Power's titanium plate technology at a 400 psi level. The ARO technology has a somewhat higher resistance.

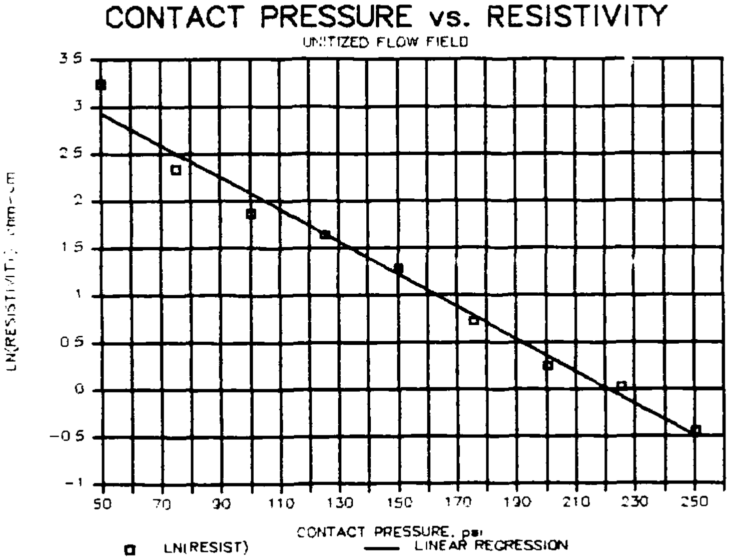


Fig. 9 Resistivity Plot

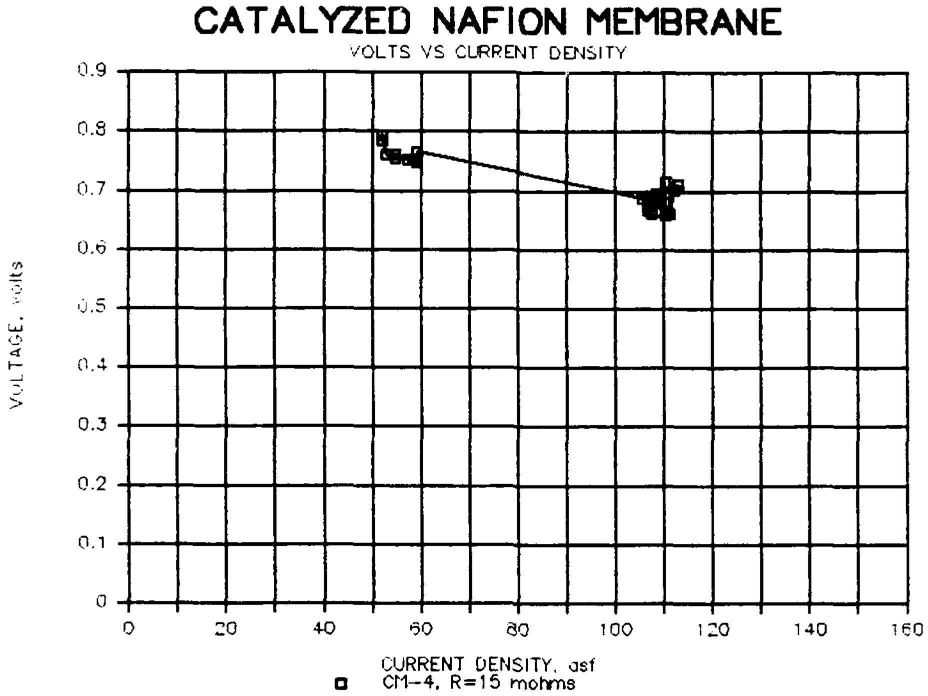
STACK RESISTANCE

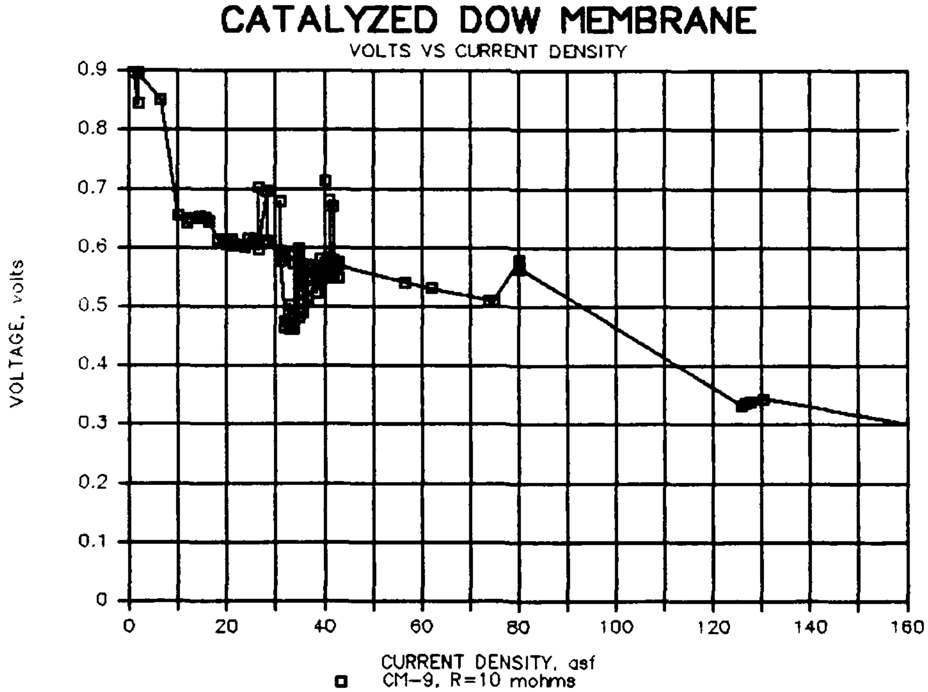
Our initial stack resistance measurements were orders of magnitude too high at 3.5 ohms. These measurements were taken on our five cell sub-stack. To calculate the theoretical resistance we determined the membrane and flow field resistivity. Nafion 117 is 7 mils thick and has a resistivity of 0.2 $ohms*cm^2$. The theoretical membrane resistance for five cells is 3.88 milliohms. The individual flow field resistivity at the stack compression force of 6000 lbs. is 3.6 $ohms*cm^2$. This yields a total flow field resistance of 84 milliohms. The resulting theoretical stack resistance is 88 milliohms.

The high contact resistance is the reason for the bulky end plate follow up system. Reducing the contact resistance requires a different end plate design and eliminating edge buildup. With the added compression of the cross bars and clamps the measured stack resistance drops to 150 milliohms.

Contributing factors to the resistance include the possible exposure of aluminum resulting in surface oxidation. During adhesive lamination of the aluminum the adhesive sometimes adheres to the paper backing rather than the aluminum surface leaving surface holes. There are laminating machines which prevent this from occurring. The other metal surface in the stack is the current collector. During leak testing water seeped into the G-10 adhesive bonded frame and corroded the brass surface. The slight curvature in the brass tends to weaken the glue when uncompressed allowing water to enter. Under compression the current collector flattens and the adhesive maintains the seal.

PERFORMANCE TESTING





SINGLE CELL PERFORMANCE

The graphs above show the single cell performance of a catalyzed Nafion 117 membrane and a catalyzed Dow membrane. The performance goal of .6 volts at 100 asf was exceeded with these cells. The single cell testing rig is a "best case" environment for membrane testing. It is designed to eliminate stack and system interference problems. Its used as a baseline for membrane performance. Since both cells exhibited similar performance we decided to choose the Nafion 117 for its low cost. Dow membrane tends to be fragile whereas Nafion is quite tough.

We used a .2 mg Pt/cm^2 catalyst loading employing the direct catalyzation method for this testing. The Nafion 117 cell has achieved operation at .71 volts & 110 ASF on H_2 and air at 90°F with 100% H_2 utilization and reactant pressures of 2 atm. The Dow membrane achieved operation at 0.7 volts and 130 ASF at the same operating conditions.

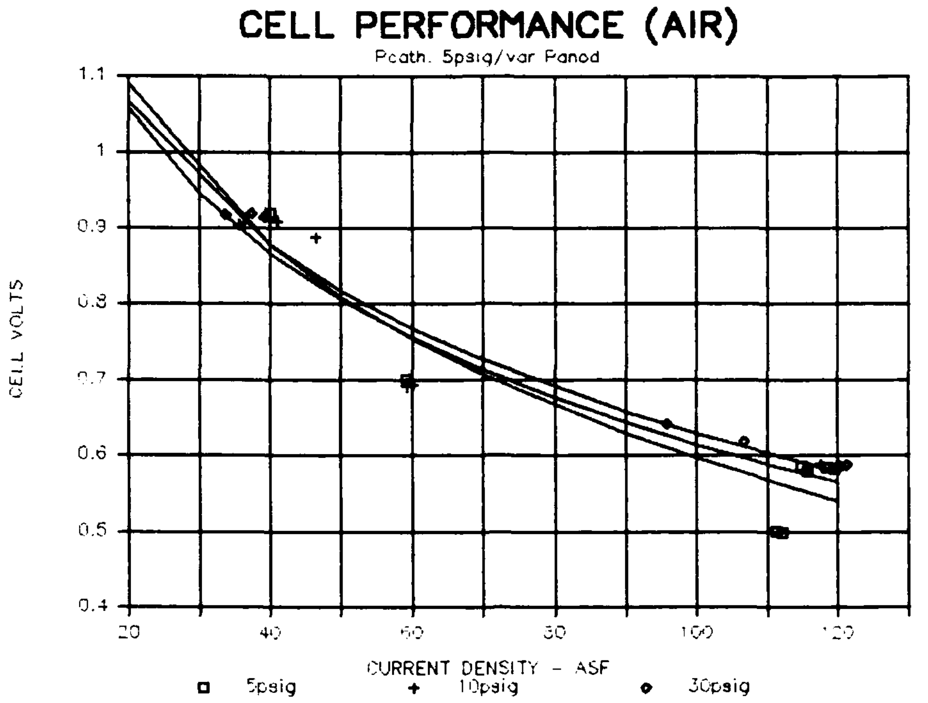
Single cell pressure testing included 5,10 & 30 psi hydrogen in combination with air and with oxygen. Although the operating conditions of the stack are ambient air we tested pressurized air and oxygen to obtain complete performance results of each membrane type.

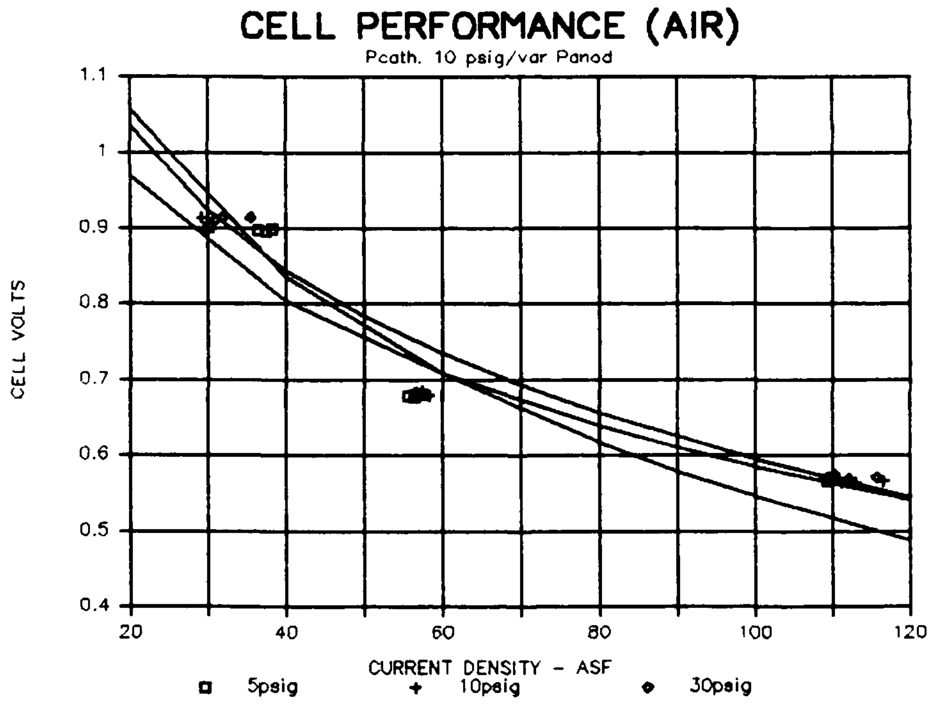
The single cell test catalyzed membrane has a resistivity of 14 milliohms and is approx. .009 inches thick (including catalyst layer) with a cell area of 6.25 in^2. As expected, cell performance is higher with higher partial pressures of hydrogen. Performance was also somewhat higher with the oxygen cathode. However, the air cathode exceeded our performance requirements of .6 volts and 100 asf and should be sufficient for this application. Figures 10-12 on the following pages show three variations in performance with anode pressure at each cathode air pressure. Figures 13-14 show the same performance variation with an oxygen fed cathode. The final graph, Figure 15 depicts the oxygen gain, comparative performance with an oxygen fed cathode vs. an air fed cathode.

STACK PRECONDITIONING

When a stack is first put together the performance is usually less than expected. This is because the membranes require a period of time for conditioning. Depending on the membrane type the conditioning can take hours or days. Teflon bonded electrodes heat pressed to the membrane require several days to reach maximum performance. The catalyzed membrane cells sometimes require only hours of conditioning for low temperature applications.

Performance degradation can be counteracted by conditioning the cell stack. The anode side of a fuel cell is relatively problem free. Most stack performance losses are cathode related. If the cathode catalyst becomes contaminated the catalyst can be electrochemically cleaned. Exposing the anode to hydrogen and connecting the stack to a large power source will raise the cathode potential and burn off contaminants (such as alcohols from the membrane surface). Alternately, by running the stack under high load and blocking off the air intake the cathode potential will drop and any oxides formed on the catalyst will be removed.


CELL PERFORMANCE (AIR)
Pcath. 5psig/var Panod
CELL VOLTS
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
20
40
60
80
100
120
CURRENT DENSITY - ASF
5psig
10psig
30psig


CELL PERFORMANCE (AIR)
Pcath. 10 psig/var Panod
CELL VOLTS
1.1
1
0.9
0.8
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20
40
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80
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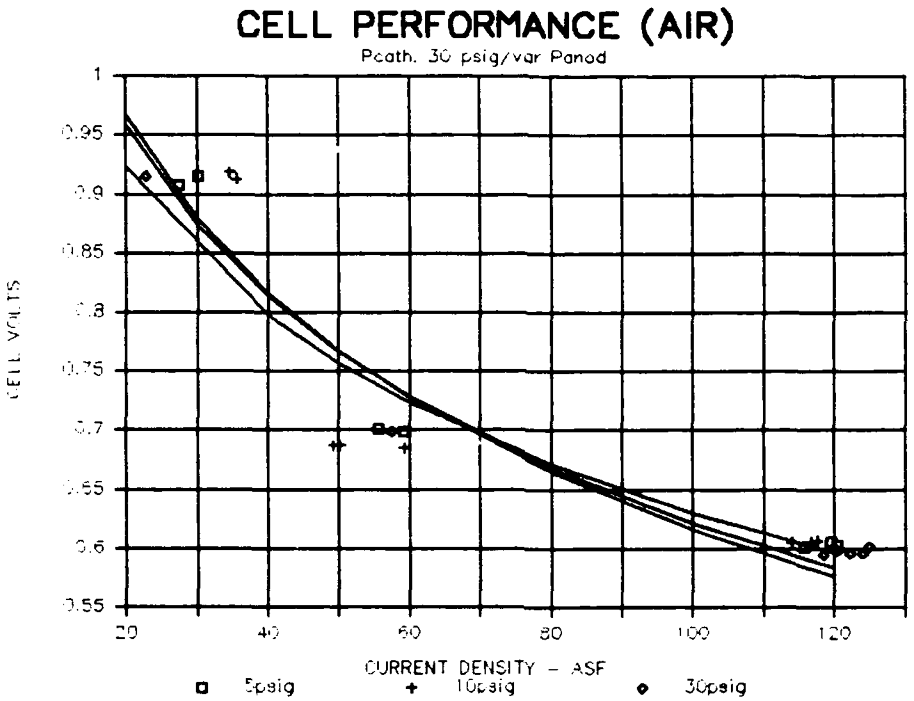
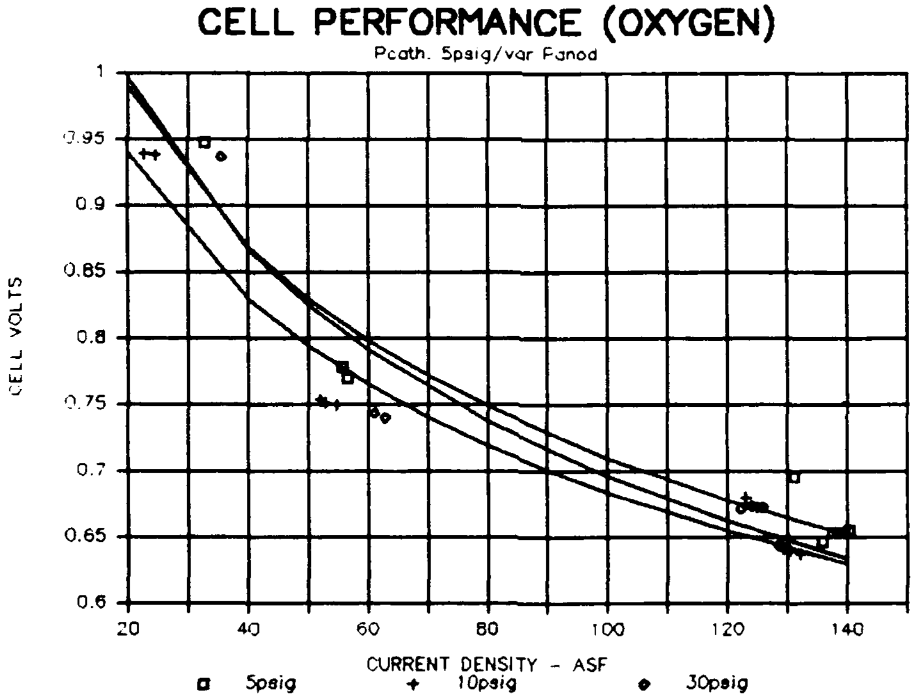
120
CURRENT DENSITY - ASF
5psig
10psig
30psig



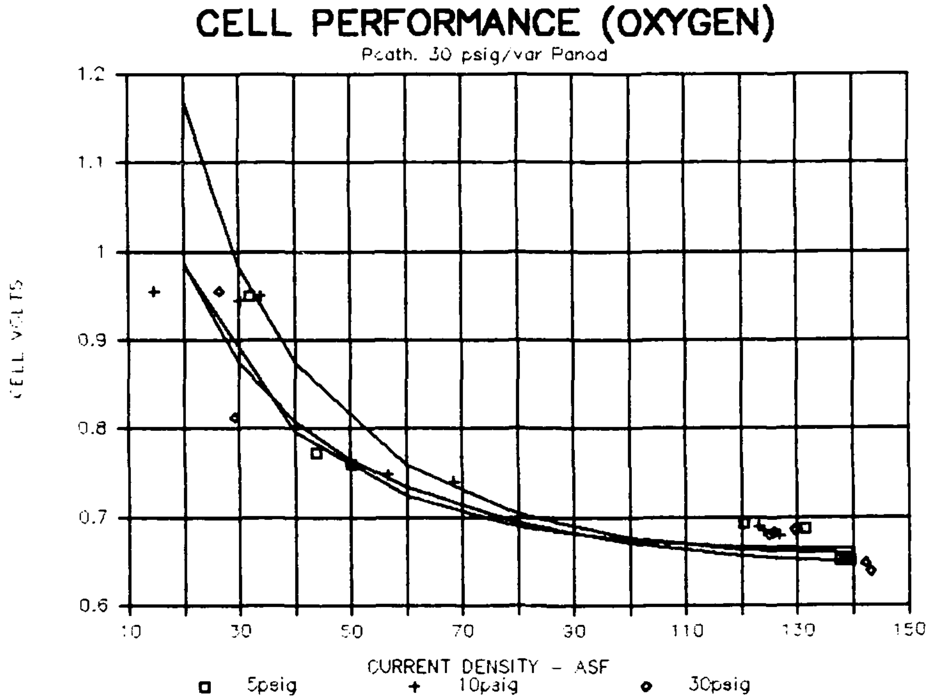
CELL PERFORMANCE (AIR)

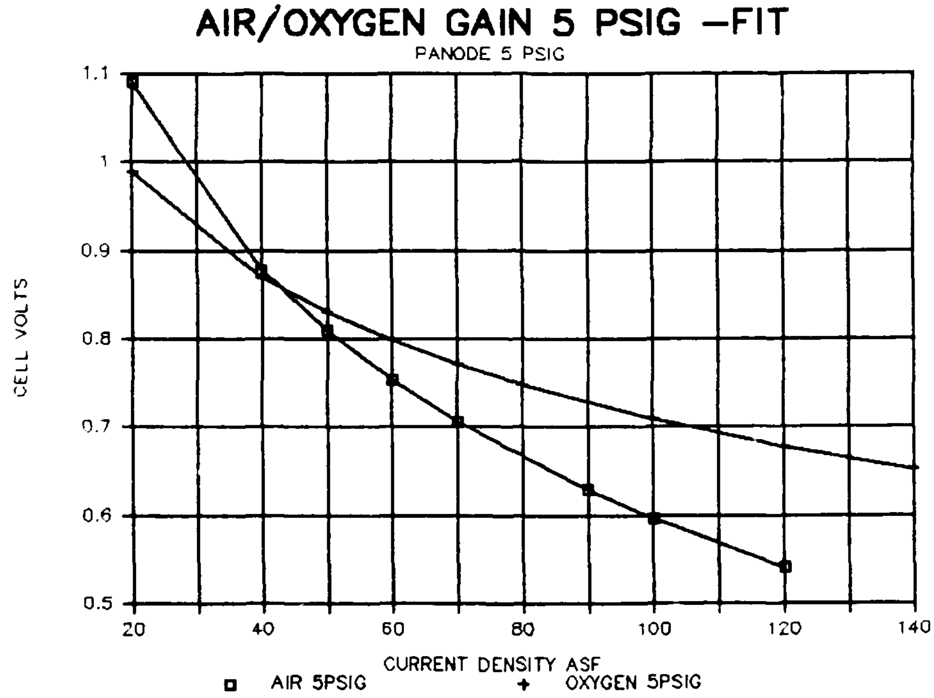
Pcath. 30 psig/var Panod



CELL PERFORMANCE (OXYGEN)

Pcath. 5psig/var Panod





Often during thermal cycling the tie bolts lengthen and the follow up system on the stack will loosen. This usually results in a drop in performance due to higher resistivity or sealing loss. With traditional silicone seal technology we have found that it usually takes about 3 days of heating up the stack and tightening the tie bolts to permanently set it. The silicone rubber will plastically deform until it takes a permanent set. We still see this phenomenon with the sealess design but not as pronounced. The only contributing factor in the ARO stack is the steel tie bolts. We have explored several different follow up systems to eliminate the tie bolts but have not yet been successful.

Once the stack is conditioned and sealed it no longer needs attention. Mostly stacks like to be run because the membranes remain hydrated. When the membranes completely dry out it takes time to recondition them.

STACK PERFORMANCE

The current stack performance plots are shown below in Figures 16 & 17. The first graph shows our best performance with forced convection. The second graph shows the performance with natural convection. We have discussed the use of forced convection in the conclusions and recommendations section. We have encountered several problems with the stack but have managed to exhibit reasonable performance levels. Our highest stack current density was 113 ASF with a cell voltage of 0.42 volts. The stack has produced at best 27 watts. The next closest performance point was 96 ASF at a cell voltage of 0.75 volts which is above our performance goal.

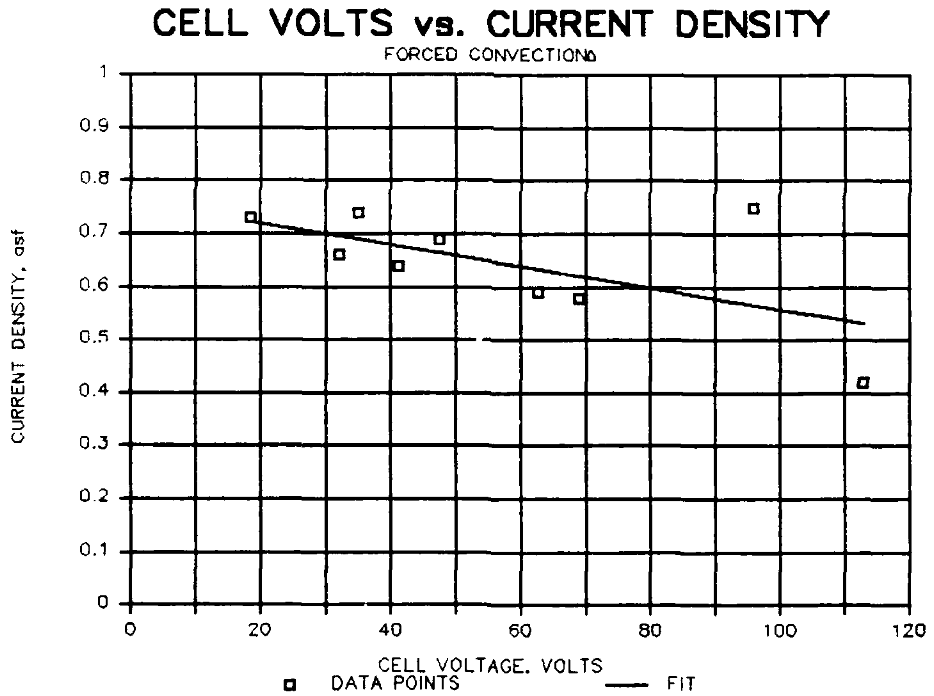


Fig. 16

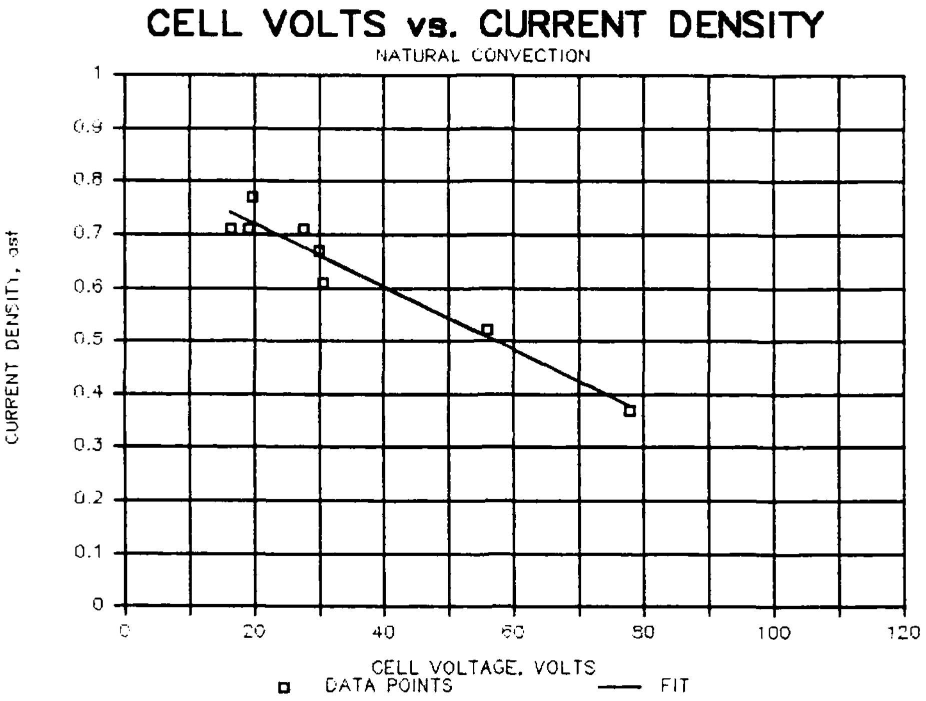


Fig. 17

The theoretical open circuit potential for this sub-stack is 5.3 volts. We have seen open circuit potentials as high as 5.25 volts. However, the open circuit drops to 4.9 volts. We have seen this effect with our 25 watt stacks. There is usually a 5% to 10% drop in open circuit within the first minute. Since each cell exhibits high open circuit potentials there are no gas crossover leaks. If a cell has a high open circuit but low operating voltage it usually means its not getting enough hydrogen at the anode or oxygen at the cathode. Often this is due to flooding.

During stack testing we placed an oxygen gas line underneath the cathode manifold opening. Performance was somewhat improved. The stack voltage at the same current density increased by 0.5 volt increasing power output 26%. The oxygen gain is more significant with a pressurized cathode. The Nernst potential rises using oxygen over air since nitrogen is not acting as a diluent.

Minor stack modifications

The stack has several different parts in it. One of our aluminum unitized flow fields leaked through the epoxy seal and could not be repaired. We substituted a 4060 film separator unitized plate that did not crossover. The hydrophyllic treatment on this cathode flow field was done electrochemically and the electrode is a non-wetproofed 16 mil sheet of Kreha HE-200. Although, the cathode absorbs water better the electrode is probably flooding. Since it was not teflonated the water does not bead up on the surface preventing the cathode from readily absorbing the beads. This is evidenced by the almost zero voltage during high load operation. The interesting phenomenon is during forced convection the membrane next to this wettable electrode, cell #5, is often the highest cell voltage. In this case the flooding works to our advantage since forced convection often dries out the membranes.

There is also one membrane, cell #4, that is different than the others and is outperforming them in most cases. The cell was fabricated without glycerol in the catalyst ink solution to speed drying time. This cells open circuit and initial performance levels exceed the others. When the stack is up to temperature close to 140°F , cells #3 & #4 are usually very close.

Active & Passive Water Management

High performance on this stack can only be achieved with forced convection. This isn't to say that passive heat and water management is not feasible, just not with this sub-stack. Our problem arises from flow field flooding. The first set of cathode flow fields were fabricated using the electrochemical treatment method for hydrophyllic processing described previously. The aluminum separator flow fields used in the current stack were fabricated from flame treated cathodes. As a result the flow fields are not properly wicking the water away from the membrane surface since the flame method doesn't seem to work effectively. Not only are the active catalyst sites blocked to reactants but the air channels are also blocked due to water beads. Basically the stack is not getting any air from natural convection.

We use an air compressor to purge the water beads from the cathode channels. This eliminates close to 1/8 cup of liquid water after several hours of operation at high load. Once the water is purged, placing a common hair dryer about 2-3 inches above the stack forces air through the cathode channels. After five minutes however, the hot air dries out the membranes and performance degrades as expected. If the membrane water level drops too low the resistance increases.

With forced convection we are able to reach current densities at or above 100 ASF. However, our stack voltage levels are still a bit low. Hence the power levels of 27 watts rather than the expected 35 watts. The low voltage levels are most likely a function of all the phenomenon described previously including metal oxidation, contact resistance, cathode polarization and flooding.

ENDURANCE TESTING

We left the stack running overnight several times at low and high load. However, the flooding problem degrades performance significantly in only one hour. As expected, leaving the stack on low load levels reduces the flooding problem. Left on overnight at low load the cathodes eventually fill with water. An air compressor is not necessary to clear the channels. Blowing through the stack will clear the water droplets. Doing so instantly revives the performance.

ANODE FLOODING

Flooding takes place at the anode. The dry hydrogen feed gas causes a water concentration gradient across the membrane. This causes the liquid product water to back diffuse through the membrane into the anode space. The ARO stack design is a flat anode flow field. In the past the anode flow field was grooved. Our flooding problems indicate that grooving the anode may be necessary. The liquid water is drawn into the KES-400 material. The micron sized pores act as a capillary wick holding the water against the surface of the catalyst and blocking hydrogen access.

With a grooved anode design an electrode is needed on the anode side to support the membrane. This adds another part and another seal interface. Eliminating the cathode flooding problem should be done before any design changes to the anode side. This will indicate whether the flooding problems are at all interdependent. It is much simpler to use a flat anode and it yields a stronger unitized flow field.