

R&D on an Ultra Thin Composite Membrane for High-Temperature Operation in PEMFC

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Executive Summary

Fuel cells offer the best clean and efficient alternative to conventional power generation technologies. Proton exchange membrane fuel cells (PEMFC) are under active development for portable, transportation and small stationary applications. For PEMFC to be commercially viable, the performance of critical components, proton exchange membrane in particular, needs to be improved. This study is directed at developing a novel high-temperature polymer electrolyte membrane for PEM fuel cells for building applications. An extensive literature survey has shown that a new cost-effective higher-temperature ($>100^{\circ}\text{C}$) electrolyte membrane, whose proton conductivity is less sensitive to water content change, is needed to improve CO tolerance, to mitigate the water management issue, to accelerate the electrode reactions and to reduce or eliminate the need for fuel cell pressurization.

The overall objective of this program is to develop PEMFC for operation at $100\text{-}140^{\circ}\text{C}$. The approach is to develop composite membranes consisting of a mechanical support and of high-temperature proton conduction phases. The developed membrane is a Teflon-solid superacid-Nafion[®] composite. The Teflon phase provides the mechanical strength for the ultra thin membrane. Solid superacids are effective high-temperature proton conductors and moisture retainers. The Nafion phase serves as the binder in addition to acting as a proton conductor itself. Phosphotungstic acid (PTA), zirconium hydrogen phosphate, superacidic nano-oxides and zeolite were the solid acids evaluated.

Nafion[®]-Teflon-phosphotungstic acid (NTPA) composite membranes were first developed. The ionic conductivity of the NTPA membranes was significantly better than that of the Nafion[®]-Teflon (NT) membranes (without PTA) at high temperatures. Single-cell testing identified that the performance loss at elevated temperature was mainly due to cathode performance loss, resulting from the high ionic resistance and loss of catalyst utilization in the cathode catalyst layer. An "improved electrode structure" had increased proton conduction and catalyst activity within the cathode catalyst layer. An MEA (membrane-electrode assembly) with this cathode achieved $\sim 200\text{mV}$ performance improvement over commercially available MEAs, reaching $\sim 450\text{mV}$ at $400\text{mA}/\text{cm}^2$, 120°C , and atmosphere pressure. However, endurance testing revealed that the NTPA membrane mechanical durability needs further improvement. It was found that PTA's tendency to leach out limits its long-term effectiveness. The best performance of $\sim 600\text{mV}$ at 120°C and $400\text{ mA}/\text{cm}^2$ was achieved with an advanced MEA incorporating an alternate non-leachable superacidic nano-oxide high-temperature proton conductor (Figure E1). This constitutes a $>300\text{mV}$ or 100% improvement over the performance level at the beginning of this program. This advanced MEA achieved 500h endurance at 120°C , an at least five-fold improvement over commercially available MEAs. Under pressurized condition, $>750\text{mV}$ performance was achieved (Figure E2). The advanced MEA has been scaled up to 300cm^2 for full-area cell use with reasonable performance and resistance at 120°C . All the performance and endurance objectives for this project were successfully met.

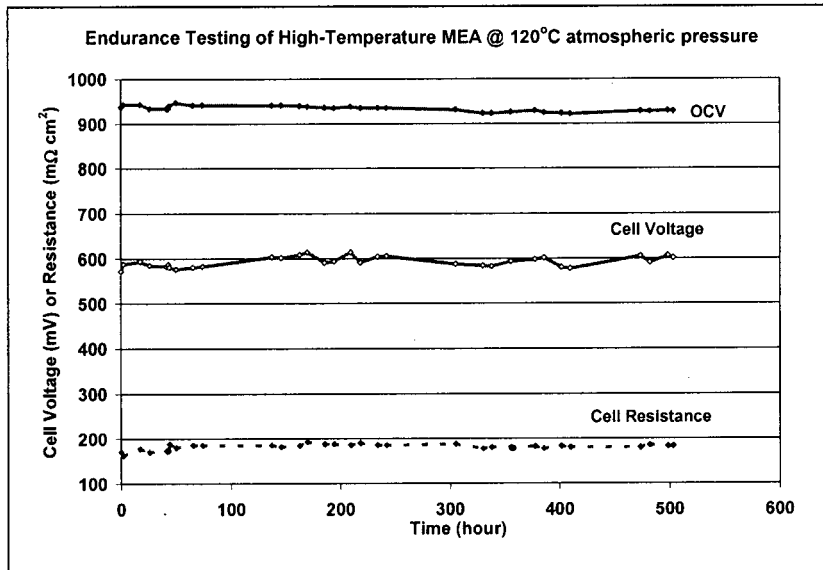


Figure E1. 500h stable 600mV endurance at 400mA/cm² achieved.

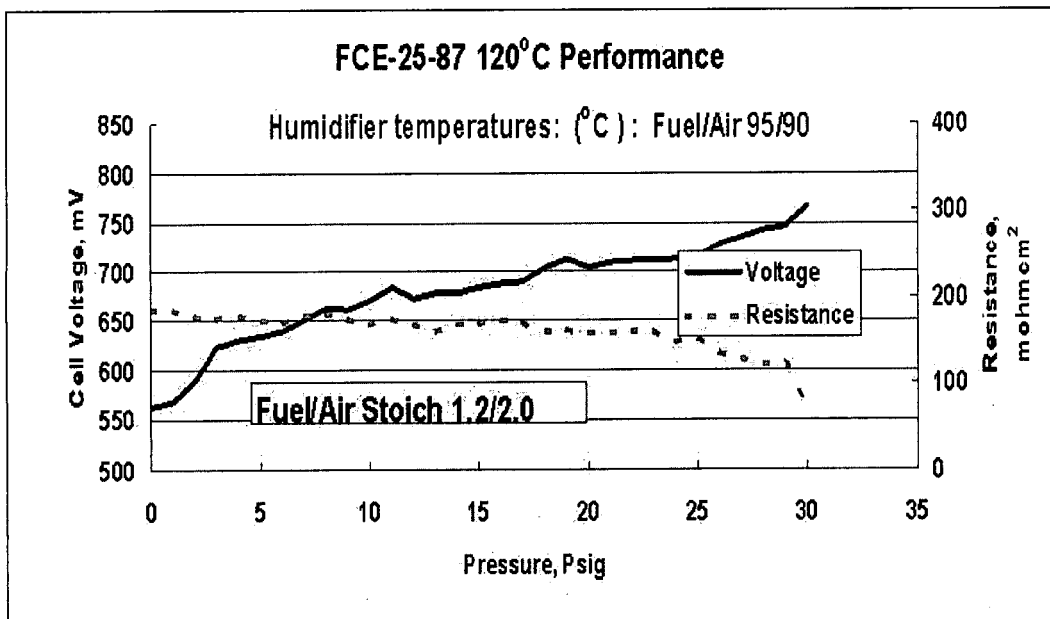


Figure E2. > 750mV performance at 400 mA/cm² can be achieved at 30 psig.

OVERALL OBJECTIVES

This study is directed at developing novel high-temperature membrane for PEM fuel cell for building applications. The overall objective is to operate PEM fuel cell at 100-140°C to improve CO tolerance, mitigate water and thermal management challenges, and to increase cathode kinetics. The approach is to develop composite membrane consisting of mechanical support and high-temperature proton conduction phases.

SPECIFIC TECHNICAL PROBLEM

Fuel cells offer the best alternative to conventional power generation technologies. They are inherently more efficient as compared to heat engines, resulting in improved fuel economy. Fuel cell power plants will be able to meet the more stringent emission standards anticipated for the future since they have reduced CO₂ emissions and do not emit air pollutants such as SO_x, NO_x and unsaturated hydrocarbons as produced from the combustion of fossil fuels.

For fuel cells to be commercially competitive, issues such as cost, size, and functionality need to be addressed. The overall goal of fuel cell for building application is to have atmospheric-pressure natural gas fueled PEMFC system with >35% HHV efficiency, >100°C operation for cogeneration, simple construction, reliable >40,000h life, and low system cost (<\$1,000/kW). For PEMFC to be commercially viable, the performance of critical components, proton exchange membrane in particular, needs to be improved.

At present, the state-of-the-art PEMFC is operated at <100°C. CO poisoning of its anode precious metal catalyst utilizing hydrocarbon feedstock is one major barrier for achieving the above goals. Fuel processing produces reformed natural gas containing significant amount of CO (about 5%). That level is reduced further in water-gas shift (WGS) reactors to ~2,000ppm [1]. However, baseline precious metal anode catalysts such as Pt cannot tolerate CO levels above only a few ppms due to strong chemisorption of CO. Intensive research to develop alternative anode catalysts, to reduce CO level by catalytic preferential oxidation (PROX) or by air bleeding has not satisfactorily resolved the CO poisoning issue [2].

Since the chemisorption of CO on anode catalysts is exothermic and therefore weakens considerably with temperature, another approach to mitigate the CO poisoning issue is to operate PEMFC at higher temperatures. Many other advantages can also be realized, such as faster electrode kinetics and higher quality waste heat for cogeneration. Operating at >100°C also mitigates cathode flooding by the liquid water reaction product, a technical challenge facing current <100°C PEMFC. Another important advantage is the greater ability to remove waste heat. With the current PEMFC systems, complicated cooling system design is needed to avoid overheating the stack. Such overheating can increase cell resistance considerably.

For the present state-of-the-art PEMFC, most developers use perfluorosulfonic acid (PFSA) polymer membrane such as Nafion[®] made by Dupont. Although this material meets the requirements in terms of strength, chemical stability, and ability to provide

relatively high-performing electrodes at $<100^{\circ}\text{C}$, it is deficient in terms of ionic conductivity and durability at $>100^{\circ}\text{C}$ and at low relative humidity (R.H.). Water in the Nafion[®] membrane is essential to facilitate proton conduction [1,2]. High-temperature operation tends to dry out the membrane. With insufficient water in the membrane, proton conduction by either hydronium ion migration or proton hopping is drastically reduced. Dried Nafion[®] is more permeable to gases, resulting in increased reactant cross-leakage. Furthermore, the loss of water embrittles the membrane, causing membrane cracking and poor electrode-membrane contact. For example, when a thin (50 μm thick) Nafion[®] membrane was tested to reduce the ohmic resistance, significant crossover of hydrogen was observed [3]. Such a thin Nafion[®] membrane is also mechanically weak and difficult to handle. In summary, high-temperature operation of the baseline Nafion[®] results in ohmic resistance and gas cross-leakage increases, and life and power generation efficiency decreases. Currently, many alternative membranes are actively being developed. A newer PFSA membrane manufactured by Dow has an equivalent weight lower than that of Nafion (i.e., a higher ion-exchange capacity), which increased its ionic conductivity and improved water retention, but its mechanical stability is poor [4-6]. The Dow membrane is expensive and has the same dry-out problem as Nafion[®]. Guzman-Garcia et al [7] and Büchi et al [8] prepared low-cost radiation-grafted copolymers. These membranes degraded and lost ion-exchange capacity during long-term use even below 100°C . Like Nafion[®] or Dow membrane, these materials, although less expensive, do not have high-temperature capability. Therefore, a cost-effective higher-temperature membrane is not yet available and needs to be developed.

Cell testing at FCE further reveals that the performance loss of the current Nafion[®]-base MEA increases sharply with temperature, mainly due to decreases of proton conductivity and catalyst utilization in the cathode. Therefore, in addition to the required membrane improvement, cathode catalyst layer also needs to be improved to achieve the above overall goal.

LITERATURE SURVEY ON HIGH-TEMPERATURE MEMBRANES

Proton-conducting mechanisms have been extensively discussed in the literature [2,9,10]. Extensive survey has been conducted in this program to update on high-temperature membrane development [9-29]. As shown in Figure 1, a useable proton conducting membrane material with desired proton conductivity comparable to fully hydrated Nafion[®] ($\sim 0.1\text{S/cm}$) between $100\text{-}140^{\circ}\text{C}$ is not yet available [9]. In this temperature range the liquid H_3PO_4 , the only material in Fig. 1 to have sufficient proton conductivity, can induce high cathode polarization due to a strong adsorption of the phosphate anions on Pt that disrupts oxygen reduction reaction. This is why phosphoric acid fuel cell is usually operated at much high temperatures (i.e., 200°C) to overcome the poor cathode kinetics.

Because useable $100\text{-}140^{\circ}\text{C}$ membranes are not yet available, many new high-temperature membranes are being actively developed. In summary, the reported membrane approaches include:

- Mechanical Support to enhance high-temperature mechanical strength
- Solid Proton Conductor/Super-Acid to enhance proton conductivity
- New High-Temperature Proton-conducting Ionomer
- Substitutes for Water in Nafion or other polymers to reduce humidity effect

Thermally stable porous polymer supports (PTFE, polytetrafluoroethylene; PBO, poly-p-phenylenebenzobisoxazole; PEO, polyethylene oxide), to improve membrane durability and strength, have been included in many composite membranes. One important consideration is to maintain the bonding between the support and the proton-conducting phase at high temperatures. A poor bonding can result in membrane cross-leakage increase. A present popular composite membrane is manufactured by Gore Associates [11]. This membrane can be made as thin as 25 μ m and consists of PFSA impregnated in a porous Teflon support. The Teflon support provides the strength. The proton conduction is provided by the PFSA phase. This combination provides a reduced internal resistance, ease of water diffusion through the membrane for self-humidification, reduced quantity of PFSA used (cost) and high mechanical strength. However, the ohmic resistance of this membrane still increases significantly with temperature due to the PFSA dry-out. Furthermore, separation between the PFSA phase (hydrophilic) and Teflon support (hydrophobic) may be adversely enhanced by the high temperature operation, resulting in the formation of many micro-cracks. Therefore, this membrane is not designed for high-temperature use.

The solid proton conductors/super-acid under development include:

PTA (Phosphotungstic Acid) $H_3PO_4 \cdot 12WO_3 \cdot xH_2O$

PMA (Phosphomolybdic Acid) $H_3PO_4 \cdot 12MoO_3 \cdot xH_2O$

STA (Silicotungstic Acid) $SiO_2 \cdot 12WO_3 \cdot xH_2O$

Sulfated Zirconia/Titania (S-Zr and S-Ti)

ZHP: $Zr(HPO_4)_2$ (Zirconium hydrogen phosphate)

Zeolite, Silica, $CsHSO_4$

These materials are brittle inorganics and cannot be easily formed into a flexible thin film. Therefore, they are generally incorporated into a composite structure containing more flexible polymeric ionomer phases. The conductivities of PTA, PMA and STA are comparable to well-humidified Nafion[®] at Low Temperatures. However, although to a lesser extent than Nafion[®], they also tend to lose water at high temperatures, with reduced proton conductivity. Water is still required to facilitate proton conduction in these materials. They also easily dissolve in liquid water; therefore, water condensation must be avoided. $CsHSO_4$ does not rely on water but on superionic phase proton hopping for proton conduction [10]. However, it is unstable and loses proton conductivity on the anode side. Other similar types of materials (phosphate-containing) are still at an early stage of development. ZHP, S-Zr/S-Ti, Zeolite, Silica do not dissolve in water but considerable modifications of their morphologies (nano-structure and well-dispersed) are needed to effectively enhance proton conductivity.

New high-temperature ionomers are in general sulfonated/phosphonated polymers, usually containing thermally stable aromatic polymer backbone, including:

Sulfonated Polyimide (SPI)

Bis(perfluoroalkyl) Sulfonyl Imide
Polyphosphazene
BPS (Polyarylene ether sulfone)
Sulfonated PEEK, PES, PPO
Phosphonic Acid Functionalized Trifluorostyrene (TFS)

They all still require water to facilitate proton conduction. So far, none of the above polymers under development have demonstrated sufficient high-temperature conductivity and durability at low R.H.

Because water dry-out is the main cause of proton-conductivity loss in many membranes at high temperatures, substitutes for water in the ionomers with high boiling-point proton-conducting liquid have been evaluated:

H₃PO₄ Doped PBI (Polybenzimidazole) [12]
H₃PO₄ Doped Polyoxadiazoles
H₃PO₄ Doped SPSF(Sulfonated Polysulfone)+PBI
Sulfuric, Phosphoric, Triflic Acids Doped PVDF Bonded Ceramic Powders (Alumina, Silica, Titania, Zirconia) [13]
BMITf or BMITF₄ Doped Nafion[®]
sPEEK+Pyrazole/Imidazole

These liquid (less volatile than water) impregnated membranes have sufficient proton conductivity at high temperatures; however, the liquids may decompose or adsorb at the Pt surface, resulting in high electrode polarization. Furthermore, the liquids may evaporate away slowly during long-term use, limiting their high-temperature durability.

Because no single membrane material so far is satisfactory at high temperatures, composite membrane approaches have been attempted. Many composite membranes are under active development, including:

PTA, ZHP/Nafion[®]/(Teflon)
PTA/Silica-PBI
Nafion/Ti Sulfonylphosphonate
Nafion, PFSA/Silica
Sulfonated PEEK+Phosphate/Oxide
PEEK or SPES+Zeolite
Sol-Gel Silica+PEO,PPO,PTMO,HMG+Solid Acids (PTA,MDP)
Pseudo PEO+STA
Zr-Phosphate+PTFE and Silicophosphate Gel Glass Composite
Sol-Gel Silane+PEO+HClO₄
Silicophosphate Gel Composite+Porous Alumina Support+HClO₄
PPSU+PBO
CsHSO₄+PVDF

So far, few composite materials have sufficient proton conductivity, comparable to a fully hydrated Nafion[®]. In addition, very little cell performance and endurance data is available. Srinivasan *et al.* [14-16] reported high temperature composite membranes prepared by impregnating various solid acids into the porous structure of Nafion[®] membranes. These membranes showed better cell performance at elevated temperature than Nafion[®] alone. However, no performance data under realistic ambient pressure with

low relative humidity was reported. In order to improve the high-temperature capability of Nafion[®], the incorporation of inorganic fillers has been found promising [17,18]. A survey of the use of composite membranes contains many references because of their use in catalysis [19]. These materials alter the structure of the Nafion[®] and assist in the Nafion's water retention, thereby enabling higher temperature operation. The addition of silica was reported to retain humidity [20]. A composite silica/Nafion[®] membrane was tested at 145°C [21]. Malhotra and Datta [22] have shown that the impregnation of PTA into Nafion[®] membrane pores allows moderately higher operating temperatures (120°C) at lower humidity levels.

Many of the membranes reported above were studied for their proton-conducting and mechanical properties. However, very little MEA performance data at high temperatures have been reported. The few reported performance data were usually obtained with unrealistic testing conditions (e.g., very high humidity or reactant stoichs). The electrode Pt catalyst loading level was also usually too high (such as using Pt black), not practical for commercial use. As will be discussed later, the cathode catalyst layer is the main MEA performance-limiting component (not membrane). The structure of the cathode catalyst layer can be illustrated as in Figure 2. In order for the Pt catalyst to be utilized, its surface needs to be accessed by the proton-conducting phases and reactant oxygen. Therefore, an ionomer of high proton conductivity is not sufficient to guarantee a high cathode performance. The acid groups (ionic clusters) of the ionomers also have to be situated right next to the Pt for the Pt surface to become available for the cathode oxygen reduction reaction (ORR). The water in the acidic ionic clusters is also needed to facilitate ORR [2]. Furthermore, high oxygen permeability is needed to reduce mass-transfer loss. The baseline Nafion[®] ionomer if well humidified has very high oxygen permeability. Any new proton-conducting phase in the cathode catalyst layer also needs to have the same high oxygen permeability. Therefore, the structure of the catalyst layer needs to satisfy the above considerations at high temperatures to achieve reasonable cell performance. Commercially available MEAs do not satisfy the above considerations at high temperatures, resulting in low cell performance. Therefore, cathode structure improvement development activity was also included in this program.

SPECIFIC PROGRAM OBJECTIVES

The overall goal of this program is to develop ultra thin composite membrane (< 75µm) capable of operation at 100-140°C. The MEA of a commercial building fuel cell needs to have the following properties:

- Membrane ionic area specific resistance less than $0.2\Omega\text{cm}^2$
- Improved humidification properties (minimal water transport and low hydration) and dimensional stability (low swelling)
- High mechanical strength
- Low gas permeability (less than 0.1% crossover of gases, $<4\text{mA}/\text{cm}^2$)
- Cell performance $>700\text{mV}$ at $400\text{-}500\text{mA}/\text{cm}^2$ and atmospheric pressure
- 40,000h life

Under this program, the specific objective is to develop ultra-thin composite high-temperature membranes/MEAs that can maintain >600mV cell performance at 400mA/cm² and 120°C for >500h.

TECHNICAL APPROACH

As discussed above, literature survey has showed that a useable proton conducting membrane material with desired proton conductivity comparable to fully hydrated Nafion[®] (~0.1 S/cm) between 100-150°C is not yet available [23-29]. A composite approach is adopted in this program because few single-component materials have all the desired properties. The membrane developed in this program is ultra-thin Teflon-solid superacid-Nafion[®] composite. Fine superacids with high proton conductivity were incorporated into the MEA (membrane and/or cathode). The fine proton-conducting additives included: PTA, sulfated zirconia, ZHP (Zr(HPO₄)₂), and several types of zeolites. The ionomer phase under study in the composite MEAs include Nafion[®] of various EW (equivalent weight) as well as low EW experimental ionomers (800EW). Nafion[®] phase serves as the binder phase in addition to acting as a proton conductor itself. The Teflon phase provides the strength for the ultra thin membrane. In order to improve the cathode performance, cathode ionic conductivity and cathode-membrane interface will also need to be modified. The overall technical approach is shown in Fig. 3.

Under this program, realistic Pt loading levels (<0.5mg/cm² per electrode) and test conditions (atmospheric, R.H.<40%, 1.2/2.0 H₂/air stoichs) are used for MEA evaluation. The MEAs were usually 5-25cm² active areas. Membranes and MEAs were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The hydrogen crossover current at high temperature (110°C) was measured using a "limiting current technique." Hydrogen saturated with water was fed to the cell anode as in a hydrogen fuel cell. However, the electrode which is ordinarily the cathode in a hydrogen fuel cell was only exposed to an inert atmosphere (nitrogen saturated with water in this study) and operated to oxidize the hydrogen crossing the membrane by controlling the potential (100mV to 900mV vs. the anode as a hydrogen reference electrode) by a potential scanning method. Membrane resistance in this study was measured by the current interruption or AC impedance technique. Cyclic voltammogram measurements of MEAs were carried out using the anode as the hydrogen reference electrode to determine the Pt surface area. AC-impedance technique was utilized to determine performance-limiting factors in the electrode layers.

The subcontractors UConn and Ion Power are performing the membrane development, fabrication and lab-scale cell evaluation. FCE, the prime contractor, is performing fabrication and lab/bench-scale cell testing. The overall program approach is shown in Figure 4. In addition to the membrane development in the original program plan, electrode improvement was also carried out.

TECHNICAL PROGRESS

High-Temperature Testing of Baseline Nafion MEA

The membrane Nafion[®] 101 membrane (25 μ m thick, 960EW), which is not designed for high-temperature use, was first tested at 120 $^{\circ}$ C to establish baseline. Indeed, the Nafion[®] 101 cells all developed crossover quickly (usually within 100 hours). Another non-reinforced Nafion[®]-base MEA (1100EW) was first tested for 4,000 hours, including 2,000h at 120 $^{\circ}$ C. The MEA contained no additives. The endurance results are shown in Figure 5 (under three conditions: A, B, and C). The cell performance at 60 $^{\circ}$ C is stable at 700mV for 2,000 hours (condition A). However, the cell performance at 120 $^{\circ}$ C dropped below 400mV (condition B), illustrating a significant cathode performance loss. The performance continued to decay due to membrane crossover increase. The cell resistance also increased sharply by 200% but remained stable. By increasing the relative humidity (R.H.) (condition C), the resistance decreased and the performance improved. However, the cell OCV continued to decay throughout the 120 $^{\circ}$ C testing. This test further confirms the necessity of improving high-temperature cathode performance and membrane durability.

A commercial reinforced MEA has an ultra-thin Teflon-supported membrane with coated catalyst layers (anode PtRu/C and cathode Pt/C). This commercial membrane was not designed to operate at temperatures above 100 $^{\circ}$ C or under low relative humidity (R.H.) condition. The total thickness of the MEA was about 50 μ m. Fig. 6 showed the cell polarization and ohmic resistance as a function of operating temperature, while maintaining constant reactant humidification temperatures. Therefore, the R.H. levels decreased with cell operating temperature. This decreasing R.H. caused MEA dry-out, resulting in increasing cell resistance and decreasing cell performance. The IR-free (ohmic-resistance free) cell polarization, which represents only electrode polarization, is shown in Figure 7. It revealed that the major cell performance loss with increasing temperature was mainly due to electrode polarization increase, not membrane ohmic resistance increase. The test demonstrates that for >100 $^{\circ}$ C operation, the catalyst layers as well as the membrane need to be improved.

Preparation of Composite Membranes

The procedure to prepare the reinforced membranes is by brushing a Nafion[®] solution onto the porous Teflon[®] matrix. The membrane was then hot pressed to increase the crystallinity of the Nafion[®] and to cause the Nafion[®] to plastically flow to seal any defects that could cause gas crossover. The reinforced membrane without any superacid additives is termed NT (Nafion[®]-Teflon).

This project initially focused on PTA superacid additive. The procedure to prepare the PTA-added Nafion[®]-Teflon-PTA (NTPA) membranes required some modifications. The phosphotungstic acid (PTA) anion is stable in aqueous media only at very low pH. To avoid the decomposition of PTA in a Nafion[®] solution containing water, the Nafion[®] solution used was prepared water-free.

Fabrication of Composite MEAs

MEAs consisting of membrane and two electrodes were fabricated by two techniques: 1) hot-pressing of commercial electrodes impregnated with Nafion[®] (hot-pressing technique), 2) direct application of the catalyst layers to the membrane (coating technique) and 3) decal method to transfer the cast catalyst membrane to membrane surface.

Hot-pressing Technique

Nafion[®] was first impregnated into commercial cloth E-TEK electrodes. The procedure was repeated until the desired Nafion[®] loading was achieved. Two Nafion[®] impregnated electrodes (anode and cathode) were placed on each side of the membrane and the assembly was hot pressed to fabricate the MEA.

Coating Technique

The hot-pressing technique has disadvantages that will be discussed later. An alternative coating technique was also developed. The electrode catalyst ink (PtRu/C anode and Pt/C cathode) was prepared by mixing a Nafion[®] solution with the catalyst. This ink was then coated onto the surface of the membrane. Two different approaches were used to control the structure of the cathode catalyst layer, a “baseline electrode structure” and an “improved electrode structure.”

Figure 8 shows SEM micrograph of typical membrane and MEA fabricated in this study. The total thickness of the MEA was only 50 μ m. The surface morphology of the Teflon[®] matrix, presented in Figure 8, shows that the pore size of the support matrix is about 0.5 μ m. The surface morphology of a NT composite membrane showed no “pin holes”.

Lab-scale Single Cell Testing

NT MEAs

It should be noted here that the hot pressing technique frequently caused cell shorting and cross-over due to the carbon fiber of the commercial cloth electrode penetrating through the ultra-thin membrane during the MEA preparation. Figure 9 showed cross-sectional microstructure of a hot-pressed MEA after cell testing, revealing shorting through the thin membrane by the conductive carbon fiber. Therefore, the coating technique is preferred for MEA fabrication.

The performance and resistance of the NT MEA, prepared by the hot pressing technique, in hydrogen over a temperature range of 80 $^{\circ}$ C to 120 $^{\circ}$ C under ambient pressure with the reactants saturated at 80 $^{\circ}$ C are shown in Figure 10. A significant reduction in performance and correspondingly a significant increase in resistance occurred with increasing temperature. Figure 11 shows that, based on IR-free cell performance similar to commercial Nafion[®]-base MEA, the membrane ohmic resistance is not major contributor for the loss in performance. Hydrogen crossover was also measured for this NT MEA at 80 $^{\circ}$ C. The hydrogen crossover current of this MEA was about 2 mA/cm², which is normal.

The resistance of the NT membrane as presented in Figure 10 at 80 °C is similar to that of the commercial ultra-thin membrane at similar operating conditions (Figure 6). However, the performance of this NT MEA is significantly lower. This suggests that the pressed E-TEK cloth electrodes are not able to provide the same cell performance as the commercial coated electrodes.

The performance and resistance of an NT MEA (by the coating technique) at 80°C under ambient pressure and with saturated H₂/air are shown in Figure 12. The performance of the sprayed MEA was slightly better than that prepared by the hot-pressed technique (Figure 10) although the cell resistance was similar. No cell shorting has been observed. The crossover measured at 80 °C was low at only about 4 mA/cm².

The IR-free performance of the NT MEA prepared by the coating technique plotted using semi-logarithmic coordinates (Figure 13) shows a Tafel slope at low current density of about 80mV/decade at 80°C, higher than the theoretical Tafel slope of 70mV/decade. The observed Tafel slope suggests that some diffusional losses or resistive losses are present in the cathode catalyst layer.

The coating technique was chosen as the technique to fabricate the NTPA MEAs. Since the development of high temperature MEAs was the main goal in this program, no further optimization on the NT MEA (not a high-temperature MEA) was performed.

NTPA MEAs

Phosphotungstic acid (PTA) is a strong solid acid and has as much as 29 associated crystalline water molecules per PTA if fully hydrated. Nafion[®] and PTA form a Lewis acid pair. The weaker acid should behave as a proton acceptor to increase the proton dissociation of the stronger acid, similar to the water-acid system. In addition, PTA is strongly hygroscopic, and this property should increase the retained water in the membrane to maintain good conductivity. The increase in the concentration of acid sites in the membrane due to the addition of PTA should also be beneficial as a Grotthus conduction mechanism, which occurs in a water-deficient environment when compared to migration, which requires large amounts of water.

Figure 14 compares the effect of cell temperature on the internal resistance for NT and NTPA MEAs with a water saturation temperature of 90°C. The beneficial effect of the PTA in reducing the internal resistance at the higher temperature is apparent. This increase of proton conductivity is still expected to increase the cathode polarization by about 50%, if such proton conductivity in the cathode has a similar increase.

The performance and resistance of the “baseline electrode structure” NTPA MEA prepared by the coating technique with a 40wt. % Pt/C cathode catalyst at 120°C are shown in Figure 15. The performance of the cell at 400mA/cm² is 0.408 volts. The MEA test parameters are listed in Table 1.

Figure 16 is the IR-free performance of Figure 15 plotted using semi-logarithmic coordinates. The Tafel slope at low current density is about 105mV/decade at 120 °C. The theoretical Tafel slope is 78mV/decade. The current ratio (oxygen/air) at a constant IR-free cell voltage at low current density (20mA/cm²) is about 3.5. In the absence of anode polarization and ionic resistance within the cathode catalyst layer, this current ratio would be expected to equal the ratio of oxygen partial pressure in the oxygen and air reactants (which is 4.8). Since the anode polarization is quite small, the lower current ratio indicates that significant ionic resistance is present in the cathode catalyst layers. The current ratio at 200mA/cm² is even lower at 2.4 indicating that the ionic resistance loss in the cathode catalyst layer increases with current density. Thus the ionic conductivity in the cathode catalyst layer needs to be enhanced for high-temperature operation. As a measure of the cathode catalyst activity, the current on oxygen at 0.8volts (IR-free cell voltage) is 26mA/cm². The hydrogen crossover current of the “baseline electrode structure” NTPA MEA is less than 2 mA/cm² at 110°C. Because the “baseline electrode structure” resulted in significant ionic loss, an “improved electrode structure” was developed to enhance the catalyst activity at the membrane-electrode interface.

The performances and internal resistance data of the “improved electrode structure” MEA h4 (defined in Table 1) under ambient pressure at 120°C using hydrogen as the fuel and oxygen or air as the oxidant are shown in Figure 17. The voltage of the NTPA MEA h4 at 400mA/cm² and 120°C was 0.465volts on air with a cell internal resistance of about 0.2Ω cm².

Figure 18 is the IR-free performance of Figure 17 plotted using semi-logarithmic coordinates. The Tafel slope on oxygen is about 83mV/decade between 20 mA/cm² and 60mA/cm². At 20mA/cm² for air, the current ratio was 4.0. When the h4 cell was operated at 200mA/cm², the current ratio was reduced to 3.2. These values were higher than the ratio for the MEA with the “baseline electrode structure” (as shown in Table 1). This suggests that the ionic resistive loss within the cathode catalyst layer of the “improved electrode structure” MEA h4 was less.

As a measure of the cathode catalyst activity with the “improved electrode structure”, the current at 0.8volts (IR-free cell voltage) on oxygen was about 50mA/cm². This is higher than the 26mA/cm² for the “baseline electrode structure” MEA. The higher activity for the “improved electrode structure” MEA h4 as well as the improved ionic conduction both contribute to the performance improvement over the “baseline electrode structure” MEA. The hydrogen crossover current at 110°C for this MEA is low at about 3.6mA/cm².

The IR-free performance of the “improved electrode structure” MEA h4 to above 800 mA/cm² shows significant curvature in the semilogarithmic polarization curves. Since curvature is also found in the linear polarization curves on air, some diffusion resistance for oxygen was present in this cell. The superior performance of the “improved electrode structure” MEA h4 compared to the “basic electrode structure” MEA was due to a slightly lower internal resistance, less ionic resistance in the cathode catalyst layer, and higher activity of the cathode catalyst. A comparison of the parameters discussed above

for the “baseline electrode structure” MEA and the “improved electrode structure” MEA h4 are summarized in Table 1.

Figure 19 summarizes the improvement of the performance achieved by NTPA. Significant improvement in performance (about 200mV at 400mA/cm²) and resistance (about 0.4Ωcm²) has been achieved by PTA impregnation and employing coated “improved electrode structure”.

Effect of introducing PTA into commercially available MEA

In order to study the effect of the proton conductivity in the cathode catalyst layer, a commercial MEA was impregnated by a solution of PTA. The performance and internal resistance of this cell at ambient pressure and 120°C using hydrogen and air saturated at 80°C was significantly improved with the PTA impregnation (Figure 20).

The improvement in resistance of about 0.1Ωcm² results in a performance improvement of only 20mV at 200mA/cm² current density. Since the performance improvement is about 100mV at this current density, the majority of the performance improvement can be attributed to the PTA-impregnated cathode. However, the IR-free cell voltage improvement still decreases with current density, indicating that further protonic conductivity improvement in the cathode is needed.

Effect of incorporating PTA into electrodes

PTA was added directly to the ionomer phase in the electrodes during the catalyst ink preparation. At 120°C, the Tafel slope on oxygen at low current density is about 110mV/decade and the current ratios (oxygen/air) for air at 20mA/cm² and 200mA/cm² were 3.2 and 2.8, respectively. These values indicate that ionic conductivity in the cathode catalyst layer is still high.

These current ratios are higher at 80°C, indicating that the electrolyte protonic conductivity within the cathode was increased by the lower temperature operation. A comparison of the performance and resistance at 120°C of this cell and the similar cell without PTA in the electrodes showed little performance improvement attributed to the addition of the PTA in the electrodes. The addition of PTA to the catalyst layers using this technique had little effect on the performance. Much of the PTA is suspected to have adsorbed onto the catalyst carbon supports and not enhanced electrolyte protonic conductivity. Therefore, the catalyst preparation method needs to be improved to uniformly disperse PTA and avoid PTA adsorption onto the Pt surface.

NTPA MEA h5 Cell endurance testing

Endurance testing results of the NTPA membrane hot pressed with commercial E-TEK electrodes are shown in Fig. 21. The cell performance, although low, was stable for about 30 hours. A new cell h5 (similar to MEA h4) with “improved electrode structure” was fabricated for endurance testing. The test parameters for h5 are listed in Table 1.

The initial cell voltage at $400\text{mA}/\text{cm}^2$ and 120°C was about 0.450volts on air with a cell resistance of about $0.22\Omega\text{cm}^2$, similar to the performance levels achieved by cell MEA h4. The Tafel slope on oxygen was about $90\text{mV}/\text{decade}$ between 20 and $50\text{mA}/\text{cm}^2$.

As a measure of the cathode catalytic activity, the current at 0.8volts on oxygen for h5 was about $41\text{mA}/\text{cm}^2$. This is less than the $50\text{mA}/\text{cm}^2$ measured for h4. The lower activity parameter with a higher platinum area factor indicates that the cathode catalyst is not being used as effectively in h5 compared to h4. This deficiency can also explain the higher Tafel slope on oxygen for h5 compared to h4.

The current ratio between the IR-free performance on air and oxygen is near or equal to the theoretical $4.8:1$ expected for a first order process with oxygen at all current densities, going from $4.0:1$ to $4.8:1$ for air at current densities of $20\text{mA}/\text{cm}^2$ and $200\text{mA}/\text{cm}^2$, respectively. This high value of current ratio for h5 indicates that little ionic resistance is present in the cathode catalyst layer. The hydrogen crossover current for h5 is fairly low at about $2.4\text{mA}/\text{cm}^2$ of hydrogen at 110°C .

The performance of h5 is similar to h4 at $800\text{mA}/\text{cm}^2$ (0.24volts compared to 0.23volts on air) with a significant straightening of the curvature in the semi-logarithmic polarization curves. This means that the diffusion losses have been reduced in h5 compared to h4. Since curvature also exists in the linear polarization curves on air, some diffusion resistance for oxygen remains in h5.

Endurance testing was then performed for this cell. The history of cell performance and internal resistance are shown in Figure 22. The initial performance using hydrogen and air at 120°C for this test was 0.409volts at $400\text{mA}/\text{cm}^2$, below the initial 0.450volts . After only 140 hours of operation, this voltage had dropped to 0.314volts , a loss of 95mV .

The initial open circuit voltage was 0.908volts , about the same as measured before the endurance testing. The open circuit declined to 0.85volts in 120 hours, for a loss of 58mV . The internal resistance slowly increased from $28.6\text{m}\Omega$ to $30.5\text{m}\Omega$, a change that accounts for a loss increase of only about 5mV .

The cell was diagnosed to determine the cause for the performance loss. The IR-free performance at the end of the endurance testing has been reduced during the endurance test by a nearly uniform voltage. The cause for this decline is clarified by the crossover test, showing a crossover rate about $78\text{mA}/\text{cm}^2$ compared to the initial value of only about $2.4\text{mA}/\text{cm}^2$. Therefore, the cell voltage loss with time can be mostly attributed to membrane crossover increase. The membrane ohmic resistance remains relatively stable. In conclusion, the mechanical durability of the membrane needs further enhancement.

Figure 23 showed another endurance result of an alternative MEA impregnated with $5\text{wt}\%$ PTA. The catalyst loading level was $0.5\text{mg}/\text{cm}^2$ for each electrode. The cell performance level was initially $>700\text{mV}$ at 95°C , with hydrogen and air humidified at 80°C . Raising the cell temperature to 120°C increased the ohmic resistance to $150\text{--}200\text{m}\Omega\text{cm}^2$ and decreased the cell performance to $400\text{--}450\text{mV}$ at $400\text{mA}/\text{cm}^2$ under

atmospheric condition. Although the membrane durability appeared better than the cell h5 (Fig. 22), the MEA life is still less than 300h and further durability improvement is still needed. Increasing the PTA content in the membrane and impregnating the cathode with PTA did not improve the MEA performance. Much of the PTA is suspected to have adsorbed onto the catalyst carbon support and not enhanced protonic conductivity.

Alternative Additives in Cathode

Due to the concern of possible leaching out of the PTA and adsorption of PTA onto the Pt surface during cell operation, other types of insoluble solid superacids were investigated. The main purpose of this series of evaluation is to evaluate the effectiveness of various solid superacid additives in enhancing cathode performance. The superacids included superacidic nano-oxide, ZHP (Zirconium hydrogen phosphate) and zeolites. ZHP and zeolite incorporation did not show any benefit in this study. Therefore, only the superacidic nano-oxide is discussed below.

The anode and cathode catalyst loading levels for all the MEAs with these additives were $0.4\text{mg}/\text{cm}^2$ each. The electrodes were applied to the membrane by a decal method. Figure 24 showed typical polarization curves of two identical non-PTA non-leachable superacidic nano-oxide-impregnated MEAs tested at 80°C under atmospheric condition. Good OCV of $>900\text{ mV}$ has been achieved. The cell resistances were all very low at less than $100\text{m}\Omega\text{cm}^2$. Slight increase of the ohmic resistance beyond $600\text{mA}/\text{cm}^2$ can be explained by the slight dry-out of the membrane near the anode side due to osmotic drag of water. The performance of all the experimental MEAs decreased with increasing temperature. About 600mV performance at 120°C had been achieved (Figure 25). The total cell ohmic resistance increased to about $170\text{m}\Omega\text{cm}^2$, corresponding to $\sim 45\text{mV}$ loss at $400\text{mA}/\text{cm}^2$. The anode polarization was measured while feeding humidified hydrogen to the both electrodes (Figure 26). The anode polarization was estimated to be less than 10mV at $400\text{mA}/\text{cm}^2$ and not temperature dependent. The cathode polarization (from OCV) was then found to be about 285mV at $400\text{mA}/\text{cm}^2$, clearly the dominant performance loss factor. The test demonstrated the need to further modify the cathode structure to achieve a higher performance. Diagnostic testing by AC-impedance showed an increase of ionic resistance in the cathode catalyst, explaining the decrease of the cathode catalyst utilization (Figure 27). In order to enhance the ionic conductivity in the cathode catalyst layer, a low-EW ionomer (800EW) was incorporated into the cathode. However, initial test did not showed cell performance improvement. Further detailed ionomer modification will be needed.

The effect of pressure was also evaluated. Increasing cathode backpressure decreased ohmic loss and increased cell performance. Increasing the cathode-side pressure not only increased oxygen partial pressure for faster oxygen reduction kinetics, but also decreased membrane ohmic loss. This result demonstrated improved back-diffusion humidification by the reaction product water from the cathode side. Figure 28 showed the effect of increasing the total cell pressure on the cell performance. At 30psig , a cell voltage beyond 750mV can be achieved at 120°C . The cell resistance also decreased with the cell total pressure, implying the benefit of a better membrane hydration by the reaction

product water at high pressures. The test concluded that to achieve the desired >750mV performance at high temperatures, pressurization is needed at this time.

Membrane durability is important for long-term commercial stack operation. The tests at FCE revealed lower EW membrane all developed crossover within 100 hours, accompanied by a decrease of OCV and cell performance. Figure 29 showed the endurance results of two MEAs. The cell #68 had a lower EW membrane and cell #84 had a higher EW membrane. The higher EW membrane showed better OCV and performance stabilities. About 500h stable endurance has been achieved. This represents a >200mV performance improvement over the baseline membrane tested for 4,000h (Fig. 6).

Bench-Scale Cell Testing

The superacidic nano-oxide impregnated MEA was successfully scale-up to bench-scale nominal 300cm² and evaluated in bench-scale single cells (Figure 30). 300cm² cell hardware was designed and fabricated for high-temperature cell testing. The cell performance at 80°C and 100% R.H. (Figure 31) was near 700mV with very little mass-transfer resistance at 400mA/cm². The cell resistance was very low at ~60mΩcm². At 120°C (Figure 32), the resistance increased to about 200mΩcm² that achieved the resistance goal. The performance was also reasonable. However, the edge seal area needs further optimization to reduce the crossover at the cell edges.

CONCLUSIONS

Extensive literature survey showed that new membrane and electrodes needed to be developed for high-temperature PEM fuel cell application. The performance of the MEAs prepared by the coating technique was better than that of the MEAs prepared by the hot-pressing technique. Nafion[®]-Teflon-phosphotungstic acid (NTPA) membranes were developed to enable higher temperature operation. The ionic conductivity of the NTPA membranes was significantly better than that of the Nafion[®]-Teflon (NT) membranes at high temperatures. Single-cell testing identified that the performance loss at elevated temperature was mainly due to cathode polarization increase, resulting from high ionic resistance and loss of catalyst utilization in the cathode catalyst layer. In order to improve the cathode performance, proton conduction and catalyst activity within the cathode catalyst was increased with the “improved electrode structure.” The “improved electrode structure” MEA performance at 400 mA/cm², 120°C, and atmosphere pressure with 90°C fuel saturation and 84°C air saturation using hydrogen as the fuel and air as the oxidant was 0.465 volts. This is a significant improvement over the initial 0.2-0.3V cell performance recorded at the beginning of the program.

Attempts to enhance cathode performance by impregnating with PTA have had only mixed results. Endurance testing also revealed the need to enhance the membrane mechanical durability. An advanced MEA has achieved a near 600mV performance at 120°C and 400mA/cm², an >300mV performance improvement over the cell performance level at the beginning of the program. The 500h endurance was also a significant achievement over any reported endurance. The MEA has been scaled up to 300cm² for

full-area cell use. The full-area MEA was tested in bench-scale cells, showing reasonable cell resistance and performance at 120°C. In conclusion, the goals set out for this project (resistance, performance, cross-over, endurance) were all achieved. The main performance accomplishments are listed in Table 2.

In order to achieve the required MEA commercial goal, further performance and endurance improvements are needed. The proton conductivity and catalyst utilization in the cathode needs further enhancement. The membrane mechanical strength needs enhancement to achieve the 40,000h life goal.

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Table 1. Comparison of Parameters for MEAs at 120 °C, with 3.4 stoich. H₂ saturated at 90 °C and air or oxygen saturated at 84 °C.

MEAs	Basic Electrode Structure	Improved Electrode Structure h4	Improved Electrode Structure h5 (Endurance Tested)
Performance at 400 mA/cm² on air (V)	0.408	0.465	0.450
Resistance (Ω-cm²)	0.22	0.20	0.22
Tafel Slope (mV/decade)	105	83	90
Current ratio for air at 20 mA/cm²	3.5	4.0	4.1
Current ratio for air at 200 mA/cm²	2.5	3.2	4.8
Current at 0.8 volts on Oxygen (mA/cm²)	26	50	41
Platinum area measured (m²/g)	195	265	429

Table 2. Performance overview at 120°C cell temperature

Performance parameter	Beginning of program	End of program	Ultimate goal
Membrane resistance at low hydration	~0.6 Ωcm^2	~0.18 Ωcm^2	<0.2 Ωcm^2
Gas permeability	>4mA/cm ²	<4mA/cm ²	<4mA/cm ²
Cell voltage at 400mA/cm ²	200-300mV	600mV	>700mV
MEA life	<100 hours	500 hours	40,000 hours
Cell size	25cm ²	300cm ²	250-300cm ²

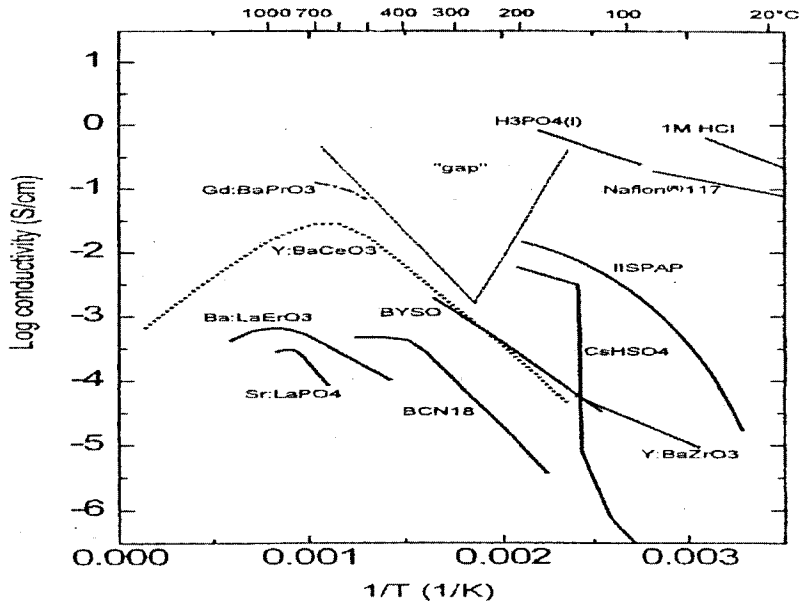


Figure 1. Literature survey showed no proton conductors available between 100 and 140°C with protonic conductivity similar to fully hydrated Nafion® [9].

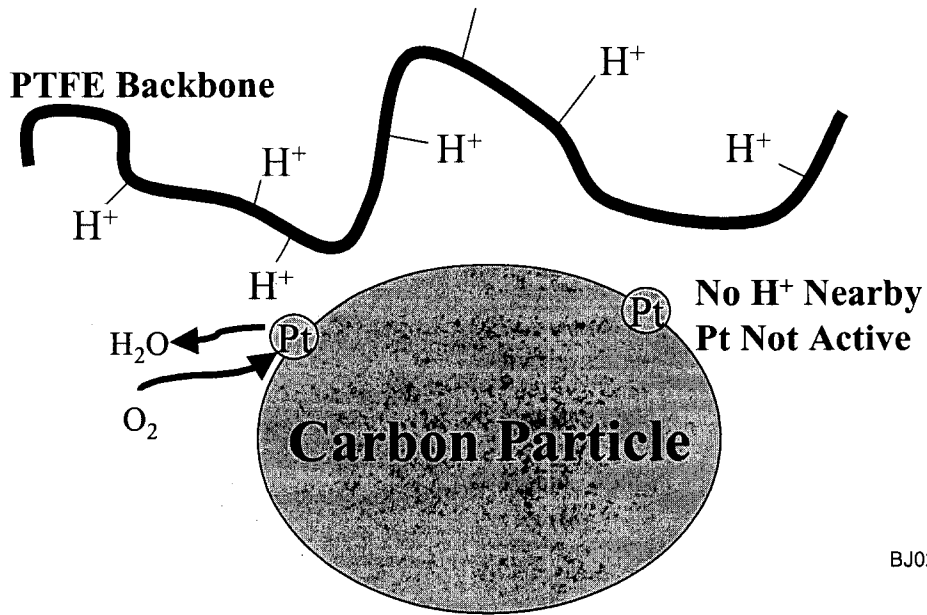


Figure 2. Schematics of cathode catalyst layer:
For Pt catalyst to be active and for a high cathode performance, high proton conductivity and oxygen permeability near the Pt surface are needed.

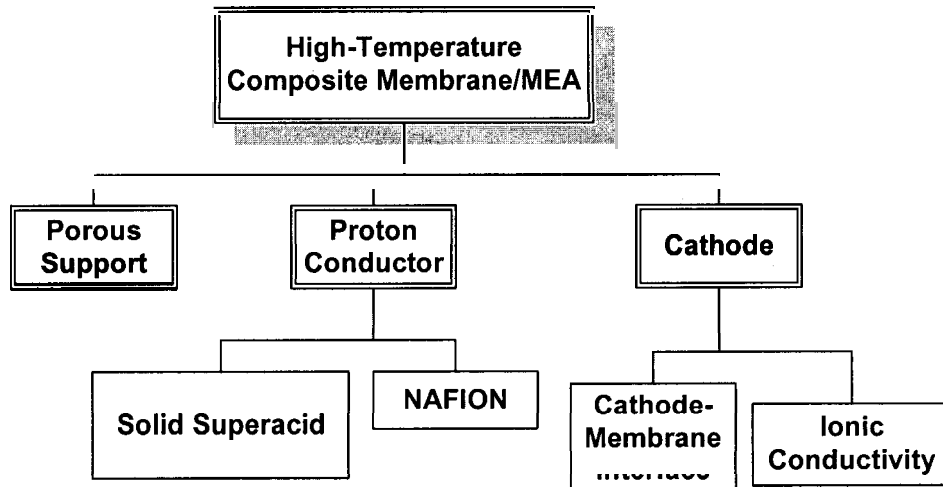


Figure 3. Overall Technical Approach:
Composite approach is adopted to enhance performance of cathode as well as membrane.

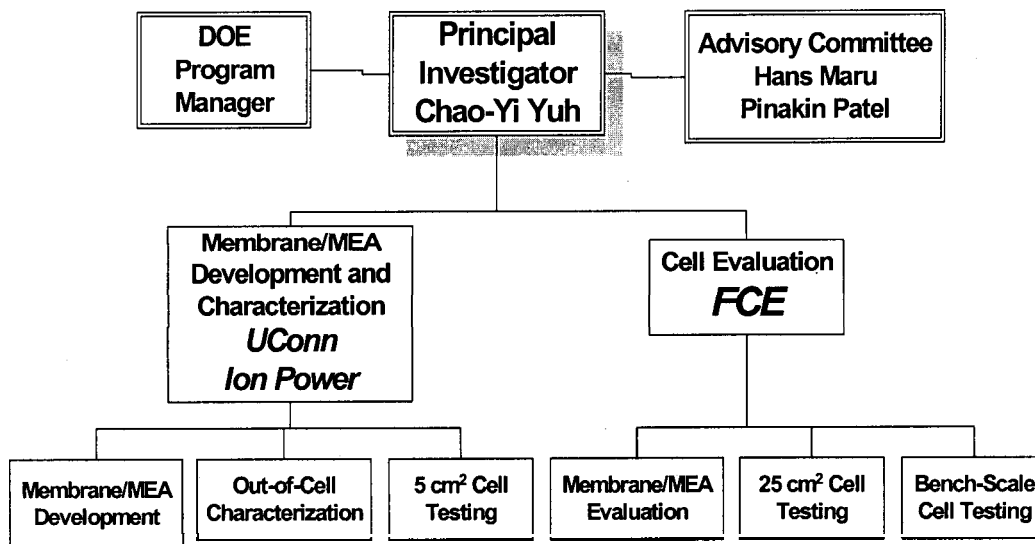


Figure 4. Overall Program Plan:
In addition to membrane development, MEA development and cell evaluation were also carried out.

FCE-25-3 Life Graph @400mA/cm²

Condition A °C : Cell 60, Fuel/Air Humi 60/61
 Condition B °C : Cell 120, Fuel/Air Humi 91/85
 Condition C °C : Cell 120, Fuel/Air Humi 95/95

Stoich: Fuel/Air: 1.2/2.0 Atmospheric

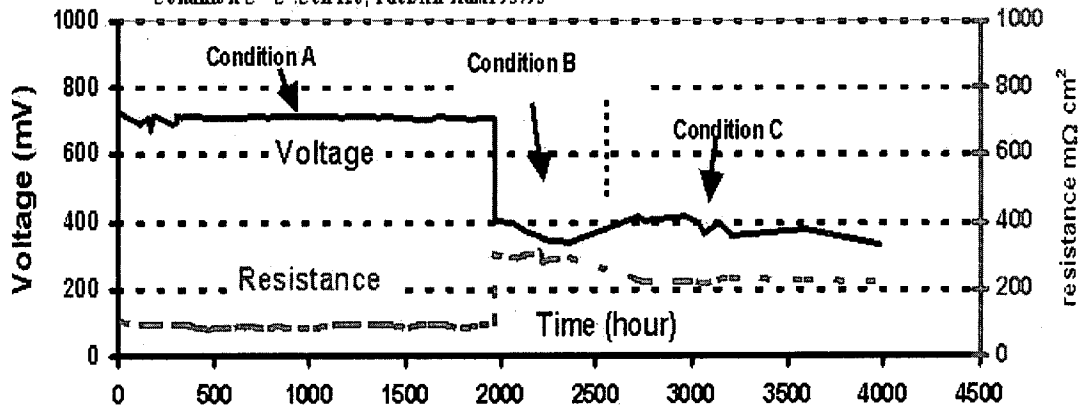


Figure 5. Endurance testing of Nafion-base MEA:
 The membrane durability and cathode performance both need substantial enhancement.

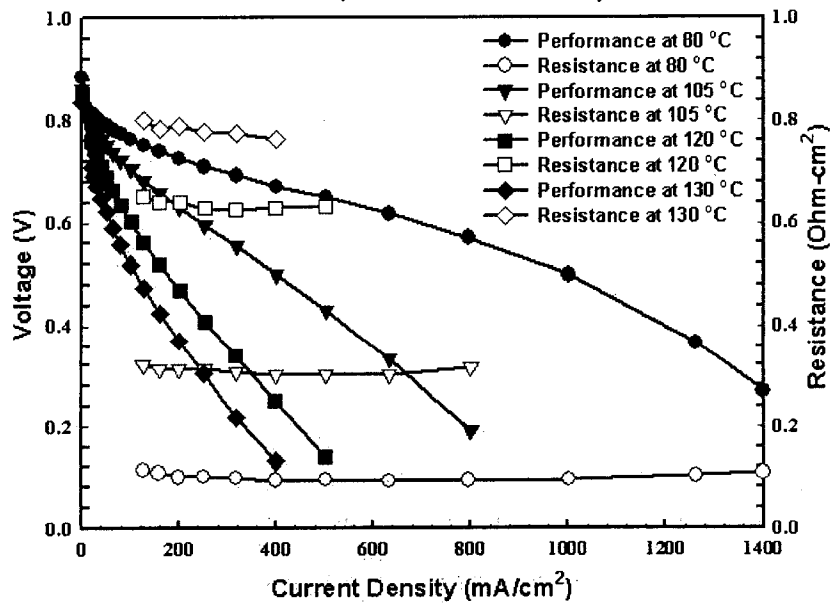


Figure 6. Significant increase of cell resistance and decrease of cell performance with temperature for a commercial reinforced PFSA-base MEA.

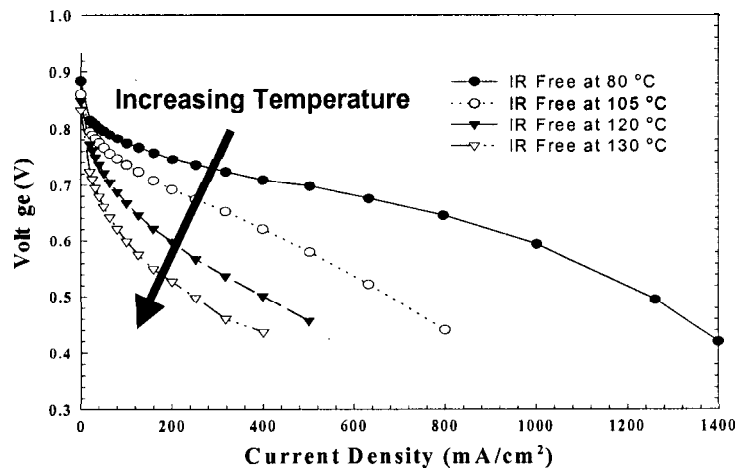


Figure 7. Significant increase of IR-free polarization loss with temperature for a commercial Nafion[®] base MEA.

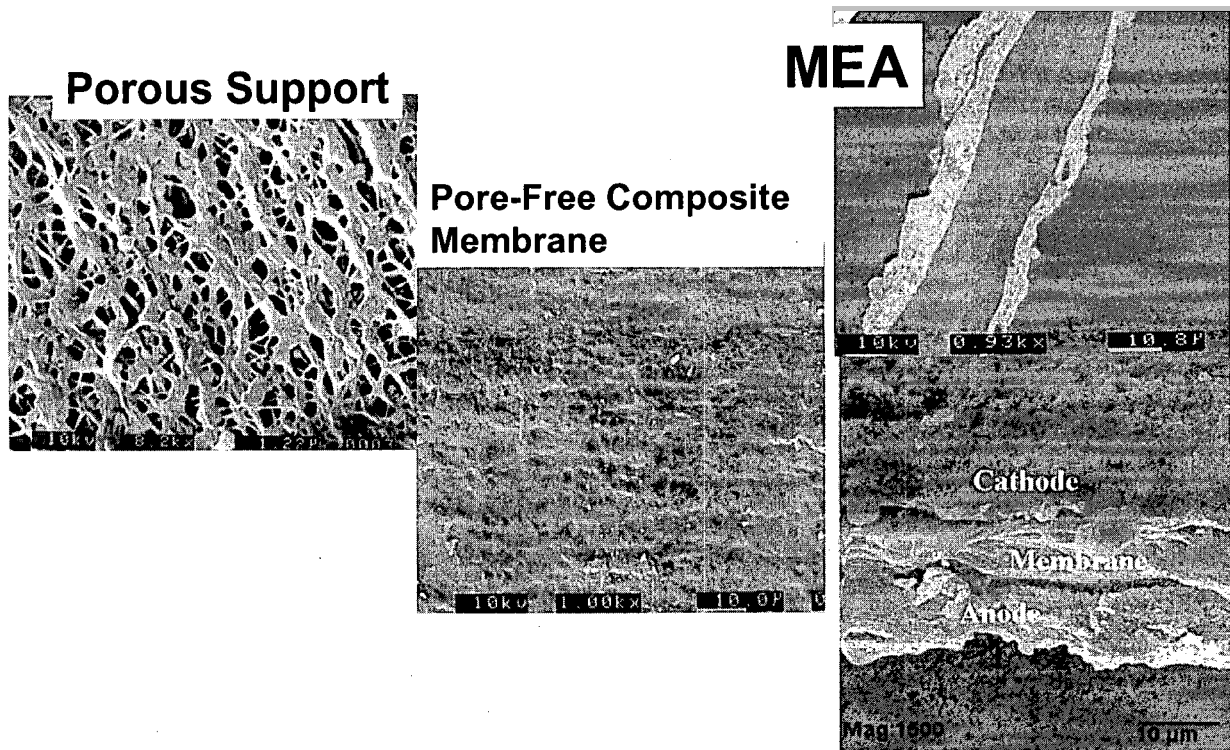


Figure 8. Advanced reinforced ultra-thin membrane and MEA were successfully fabricated.

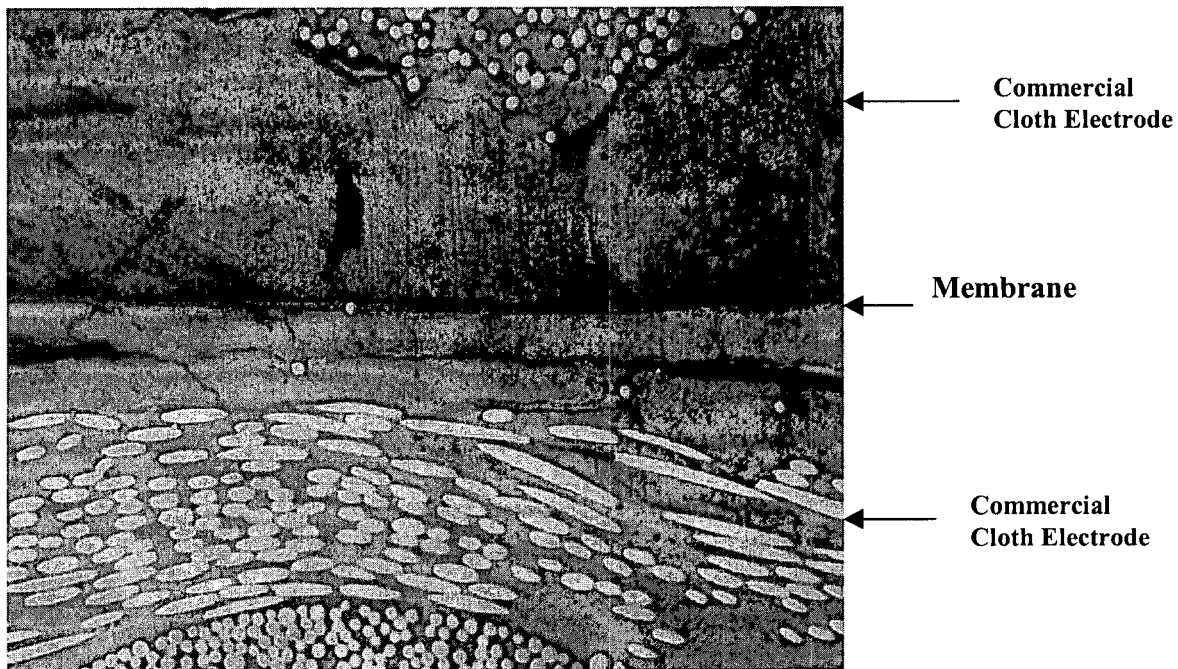


Figure 9. Carbon fiber from commercial cloth electrode penetrated the thin membrane and caused electric short.

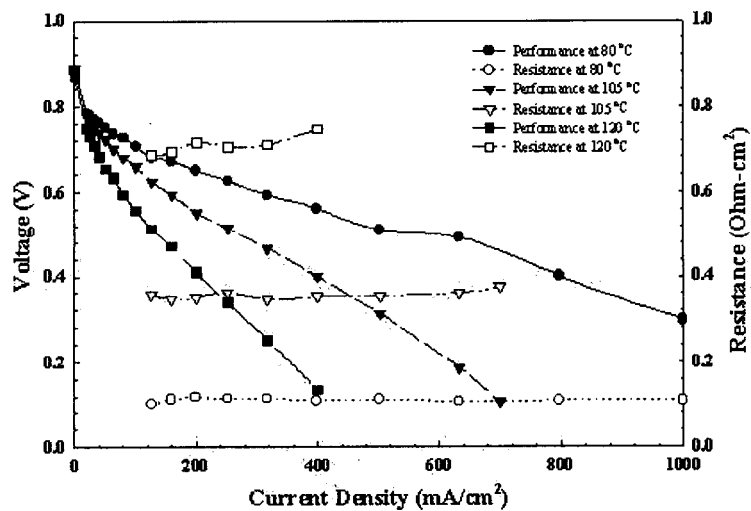


Figure 10. Effects of Temperature on Performance and Resistance of NT MEA Prepared with Commercial E-TEK Electrodes and by Hot-Pressing Technique (anode: 3.4 stoich H₂; cathode: 4.0 stoich air; humidifier temperature: 80 °C): Significant increase in cell resistance and decrease of cell performance with temperature.

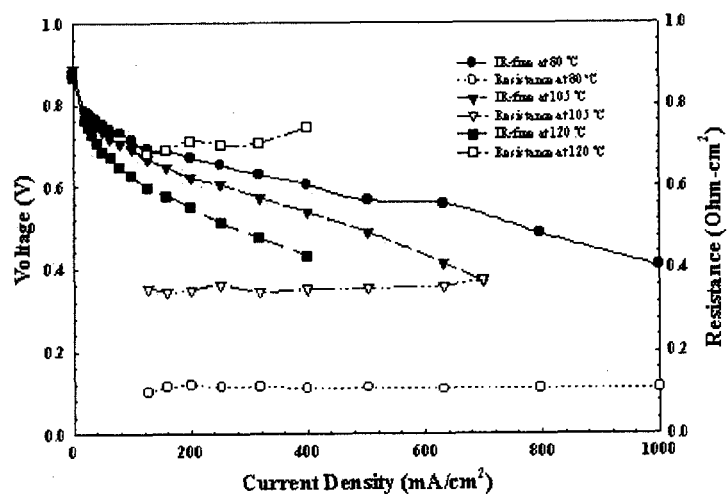


Figure 11. Effect of Temperature on IR-Free Performance of NT MEA Prepared with Commercial E-TEK Electrodes and by Hot-Pressing Technique (anode: 3.4 stoich H₂; cathode: 4.0 stoich air; humidifier temperature: 80 °C): The results showed increase of electrode polarization loss with temperature.

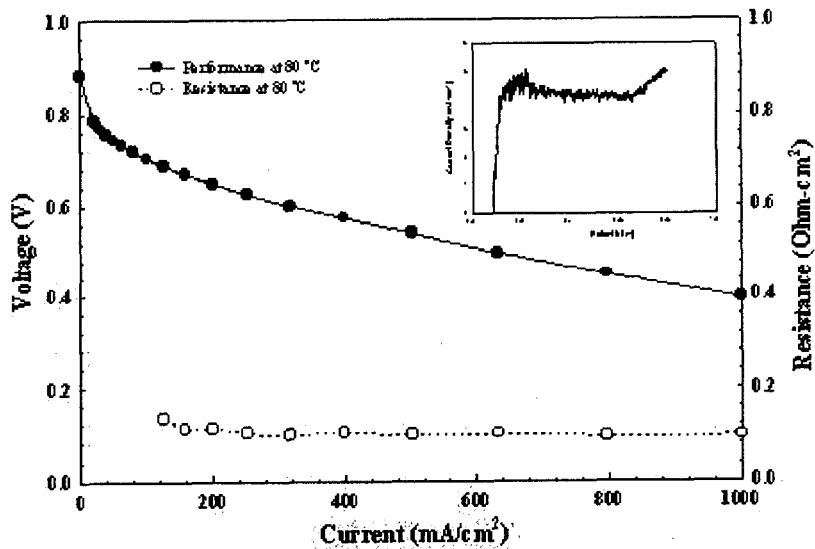


Figure 12. Performance and Resistance of NT MEAs Prepared by Coating Technique at 80 °C (anode: 3.4 stoich H₂; cathode: 4.0 stoich air; humidifier temperature: 80 °C).

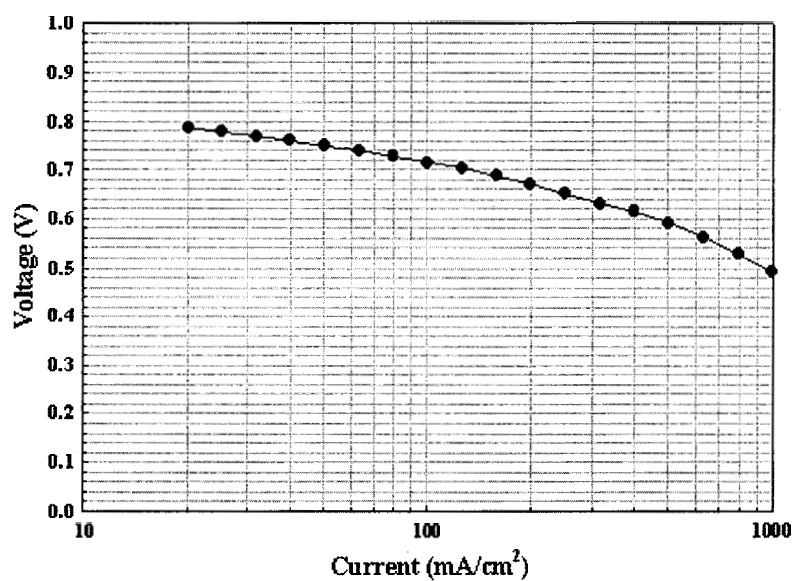


Figure 13. IR Free Performance of NT MEA, Prepared by Coating Technique, at 80 °C (anode: 3.4 stoich H₂; cathode: 4.0 stoich air; humidifier temperature: 80 °C).

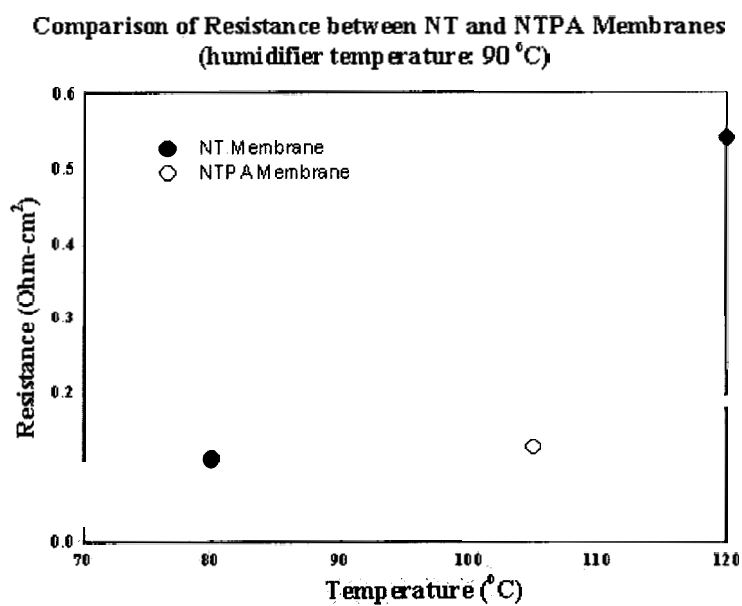


Figure 14. Comparison of Resistance between NT and NTPA Membranes at 120 °C (humidifier temperature: 90 °C): PTA additive maintains proton conductivity in the membrane at >100°C.

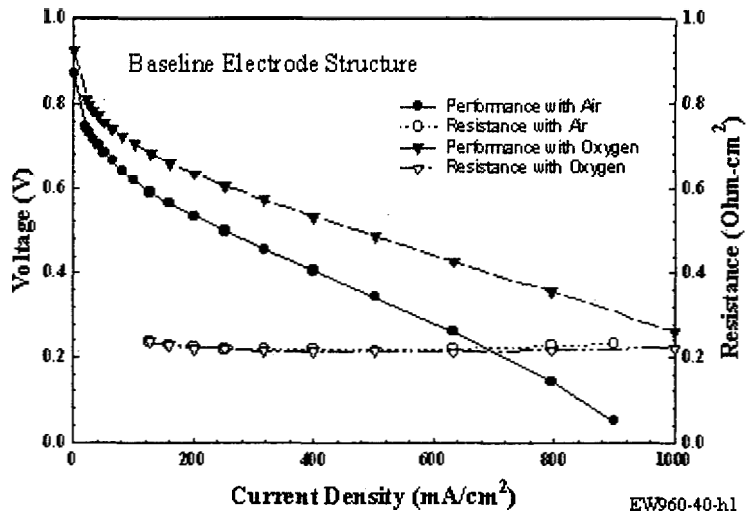


Figure 15. Performance and Resistance of NTPA MEA (baseline electrode structure) Prepared by Coating Technique at 120 °C (anode: 3.4 stoich. H₂ saturated at 90 °C; cathode: air or oxygen saturated at 84 °C): 0.4 V cell performance achieved with air at 120°C and 400mA/cm².

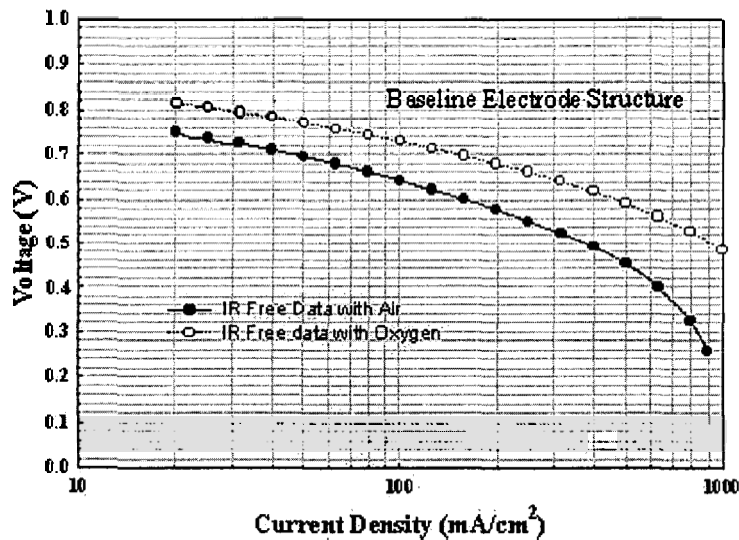


Figure 16. IR Free Performance of NTP MEA (basic electrode structure) Prepared by Coating Technique at 120 °C (anode: 3.4 stoich. H₂ saturated at 90 °C; cathode: air or oxygen saturated at 84 °C): The Tafel slope indicates significant ionic resistance in the cathode catalyst layer.

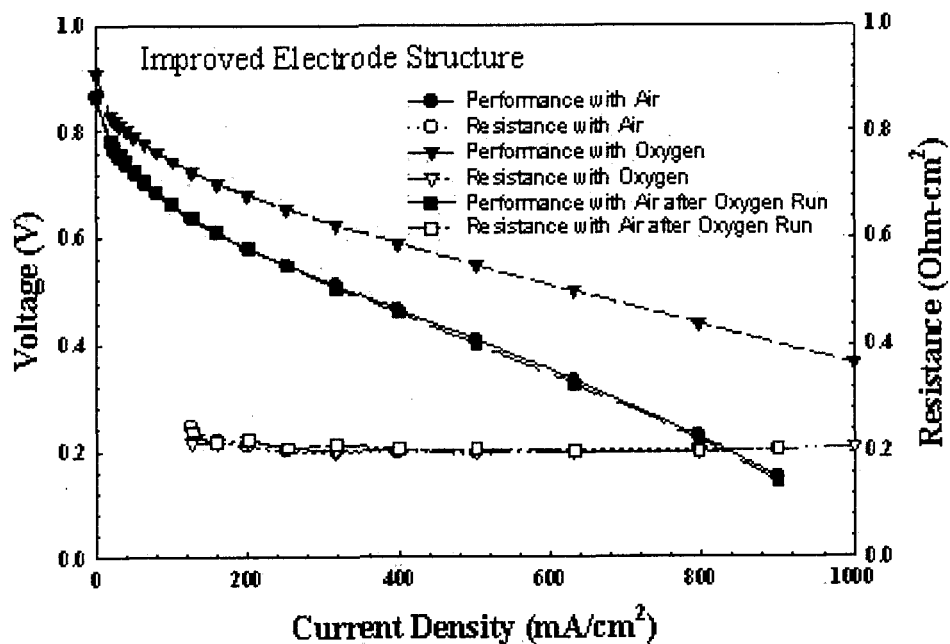


Figure 17. Performance and Resistance of the "Improved Electrode Structure" NTPA MEA h4 Prepared by the Coating Technique at 120 °C (anode: 3.4 stoich. H₂ saturated at 90 °C; cathode: air or oxygen saturated at 84 °C): 0.465 V cell performance achieved with air at 120°C and 400mA/cm².

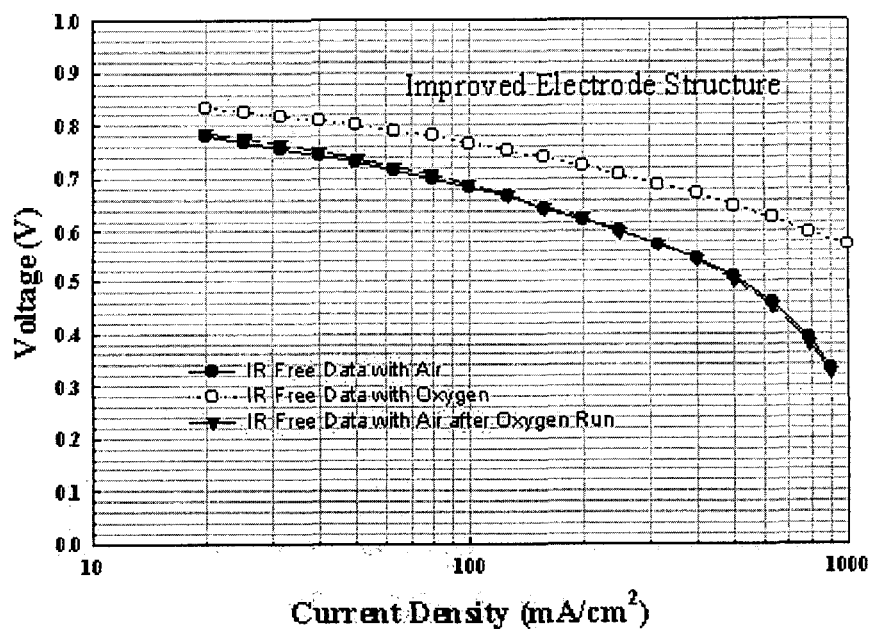


Figure 18. IR Free Performance of the "Improved Electrode Structure" NTPA MEA Prepared by the Coating Technique at 120 °C (anode: 3.4 stoich. H₂ saturated at 90 °C; cathode: air or oxygen saturated at 84 °C).

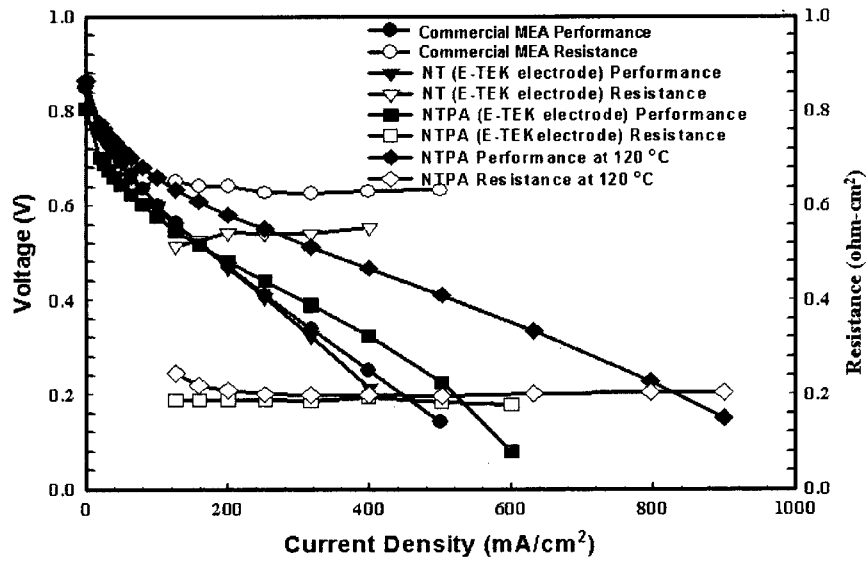


Figure 19. Significant improvement in cell performance has been achieved in this program by PTA addition: NTPA MEA had the best performance.

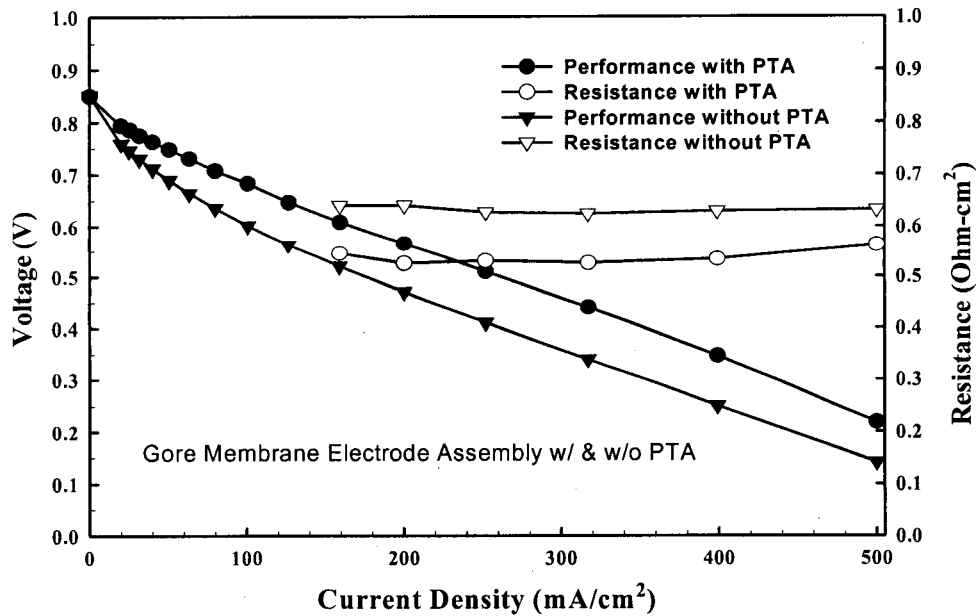


Figure 20. PTA impregnation reduced ohmic loss and enhanced cell performance at 120°C.

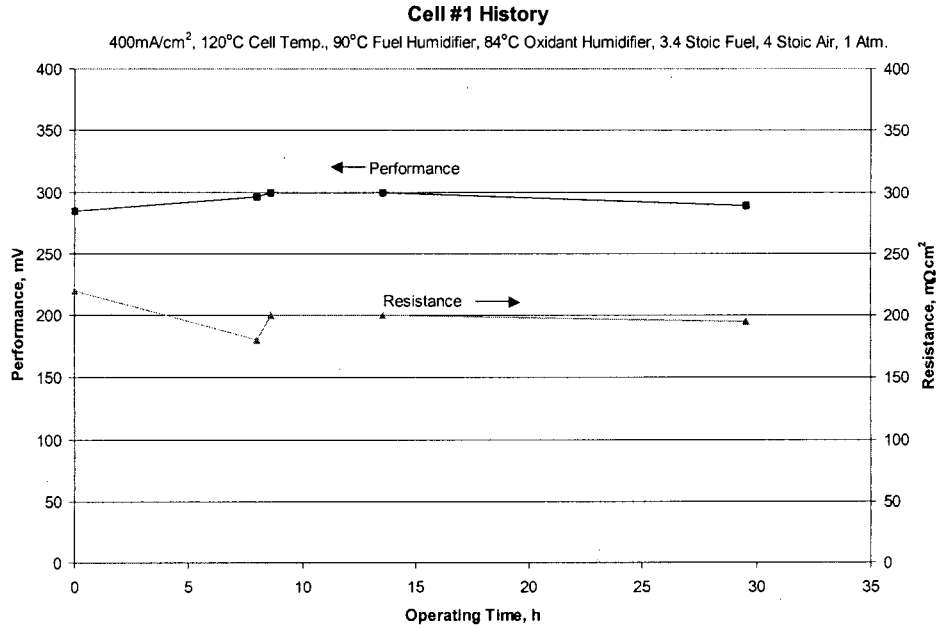


Figure 21. Stable cell performance at 120°C for 30 hours (NTPA membrane with hot-pressed commercial cloth electrodes).

h5 Performance History

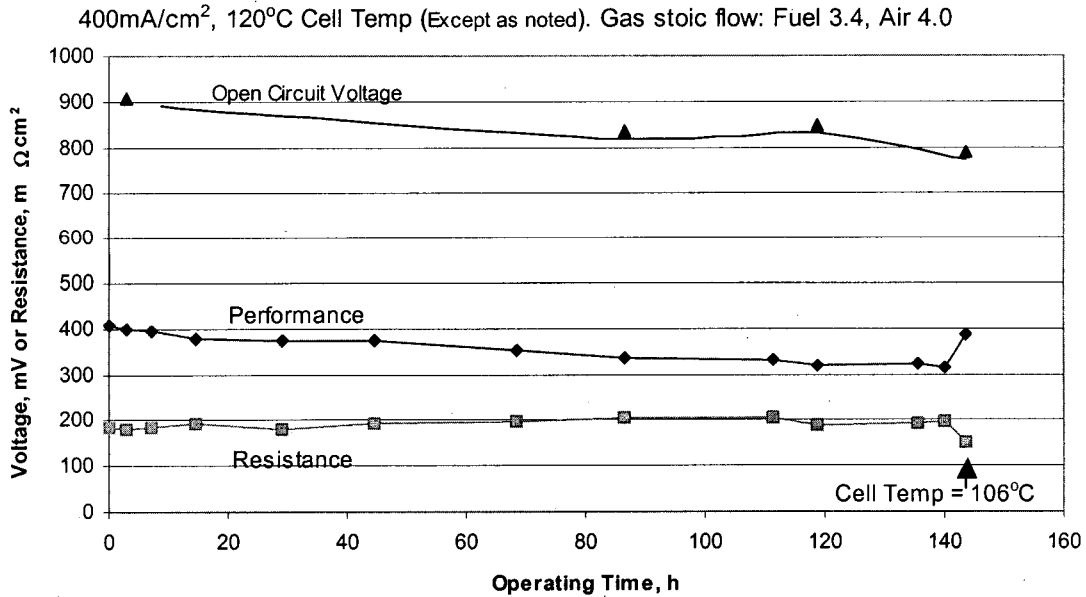


Figure 22. Endurance testing of MEA containing PTA impregnated membrane: Membrane crossover increased with time, indicating the need to enhance membrane durability.

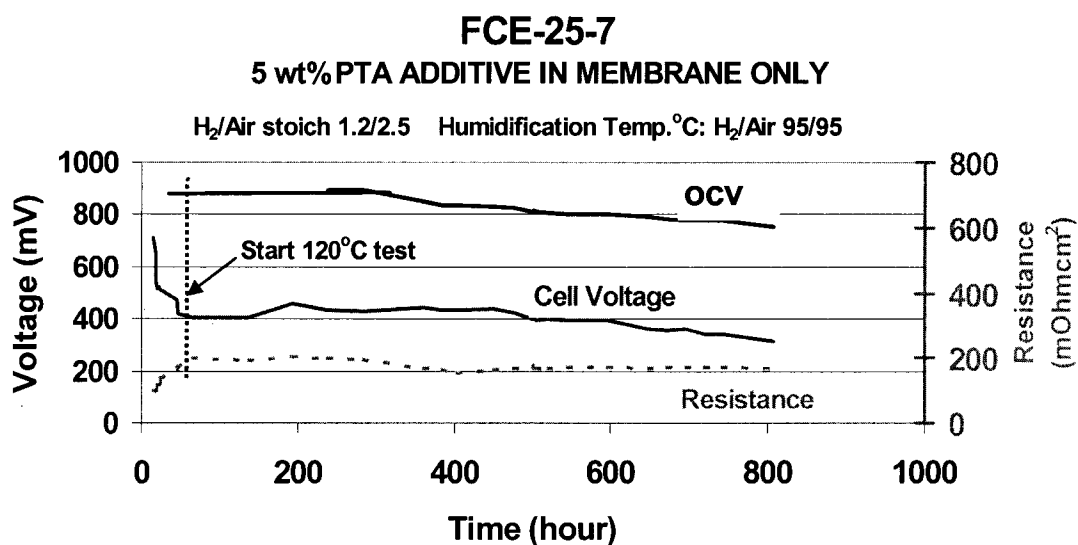


Fig 23. Endurance testing of MEA with PTA-impregnated membrane: Crossover developed after 300 hours as indicated by the OCV decrease.

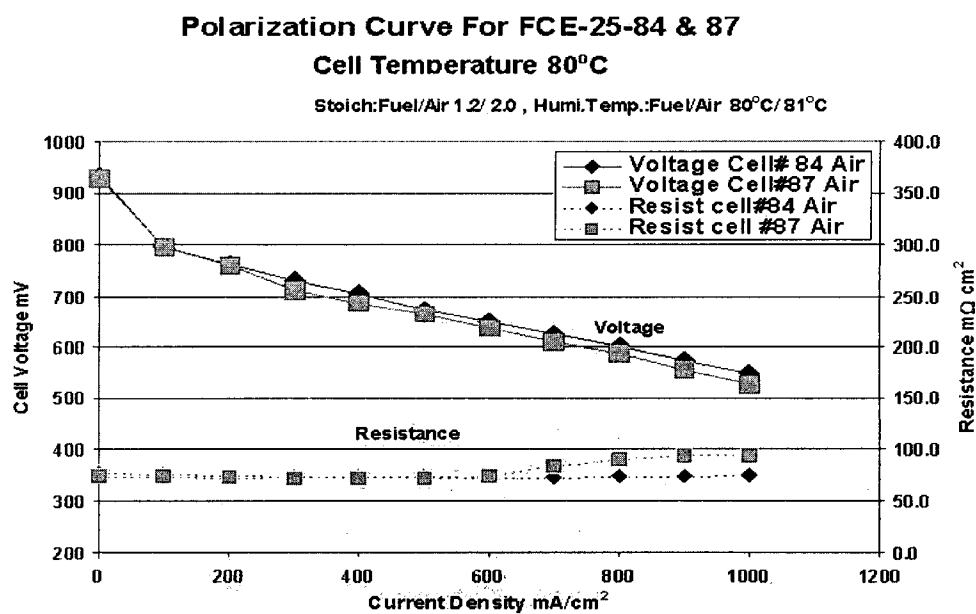


Figure 24. Atmospheric-pressure performance of two identical MEAs with solid superacids incorporated cathodes: Performance at 400mA/cm² was near 700mV.

FCE-25-84 MEA 37277B

Cell Temperature 120°C

Stoich: Fuel/Air 1.2/ 2.0 , Humidification Temperature: Fuel/Air 95°C/95°C

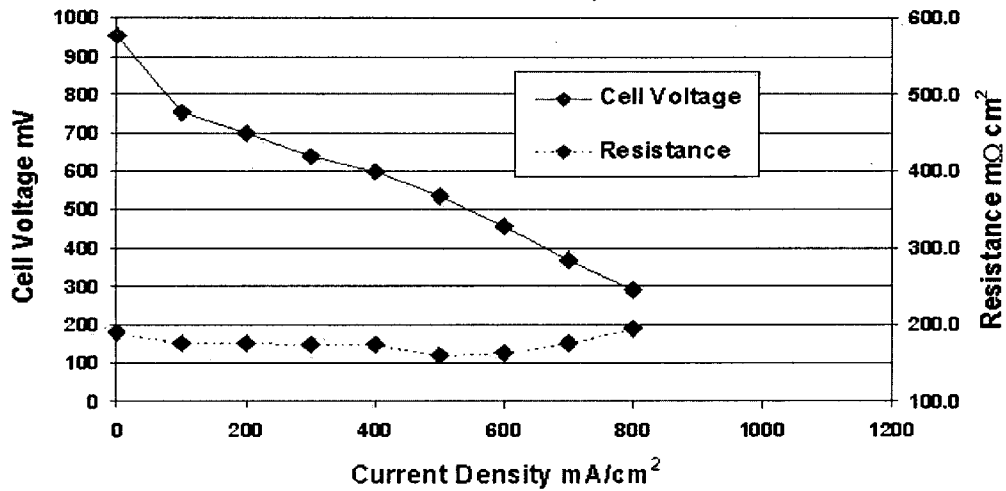


Figure 25. Near 600 mV atmospheric-pressure cell performance at 120°C and 400 mA/cm².

FCE-25-84

(iR-Free)

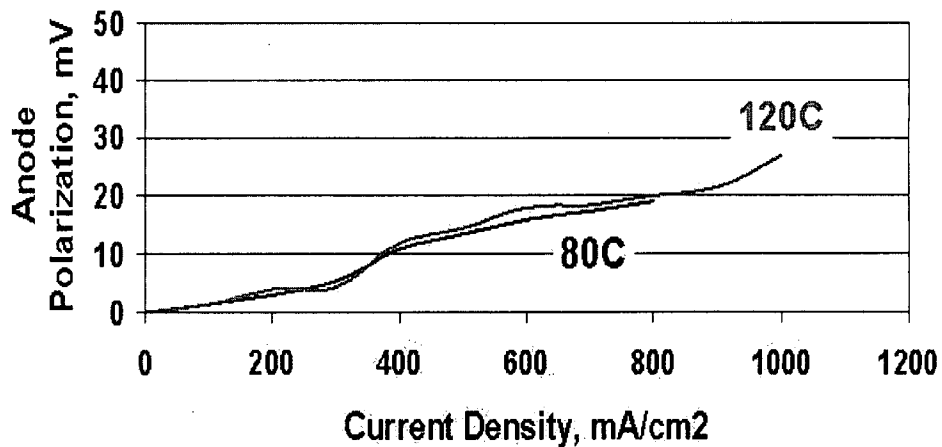


Figure 26. Anode polarization testing revealed low anode polarization loss at 80 and 120°C (even without superacid additives).

Fce-25-116 at 120°C

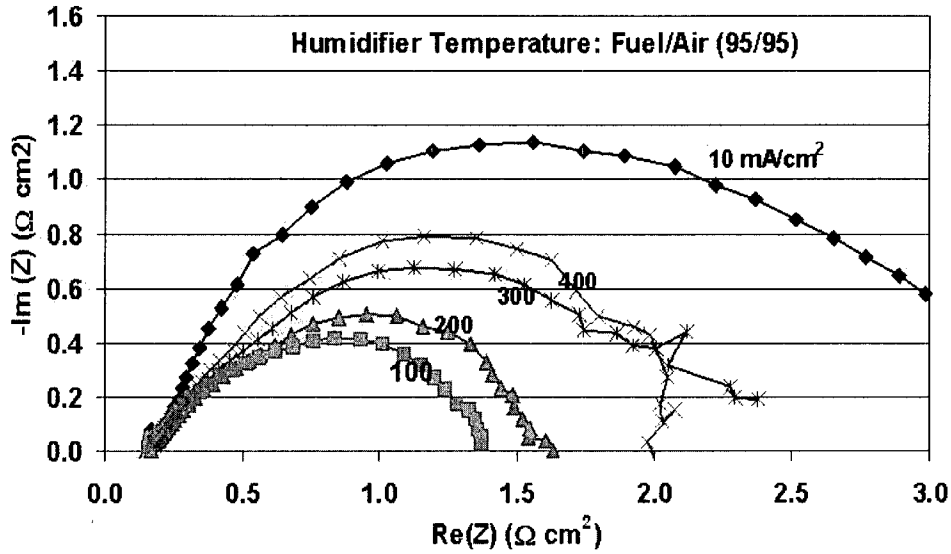


Figure 27. AC-Impedance of Superacidic Nano-Oxide Impregnated MEA: The cathode is under mixed kinetic-ohmic control at 120°C.

FCE-25-87 120°C Performance

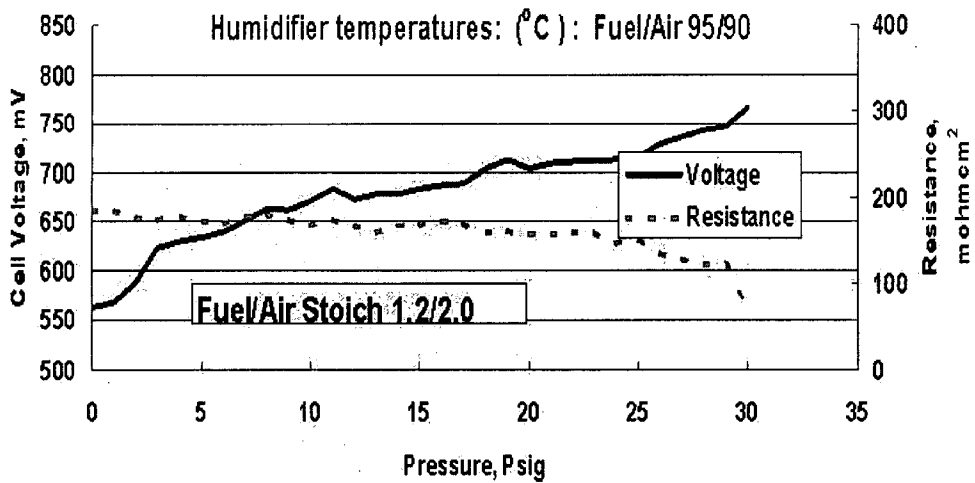
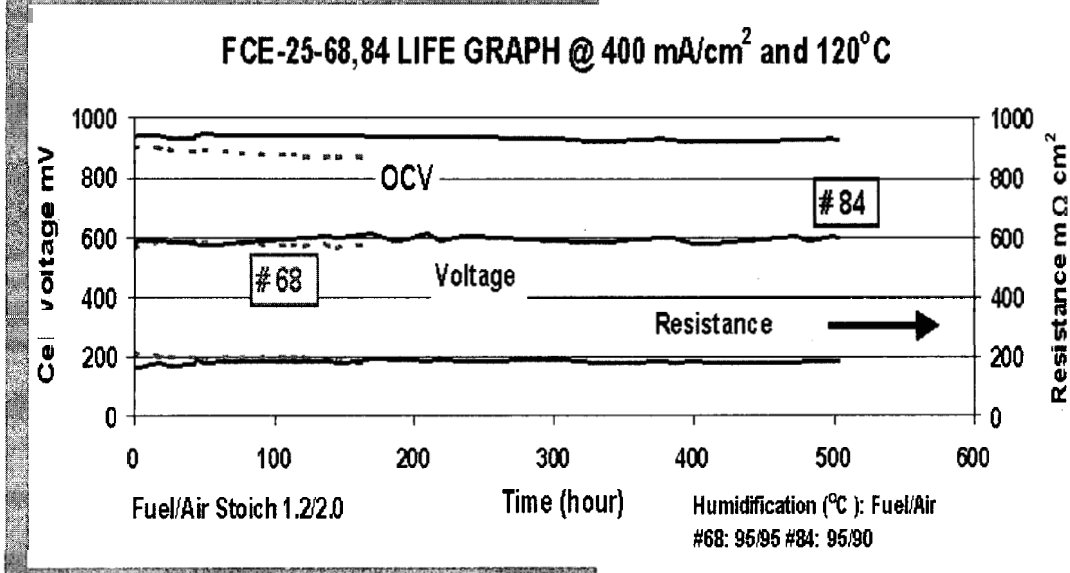
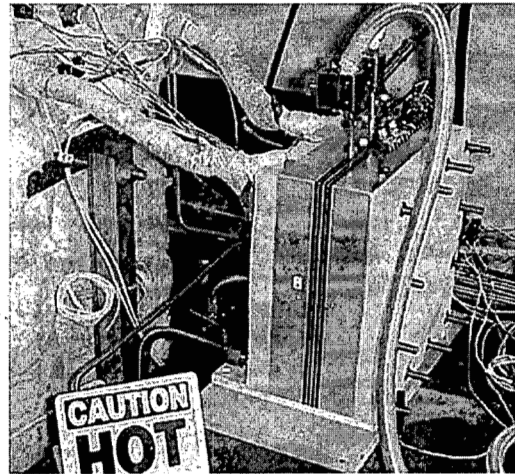


Figure 28. > 750mV performance at 400 mA/cm² can be achieved at 30 psig.



400mA/cm² achieved in cell# 84 (regenerated MEAs).



and evaluated in bench-scale cells.

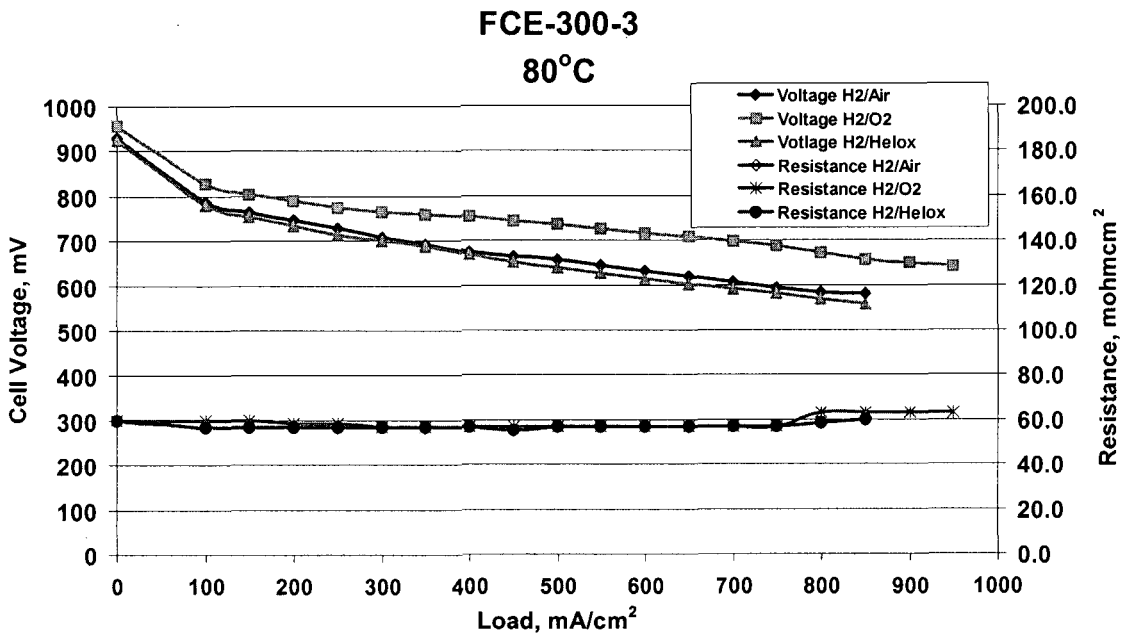


Figure 31. 300cm² cell performance and resistance at 80°C:
Good cell performance and resistance were achieved.

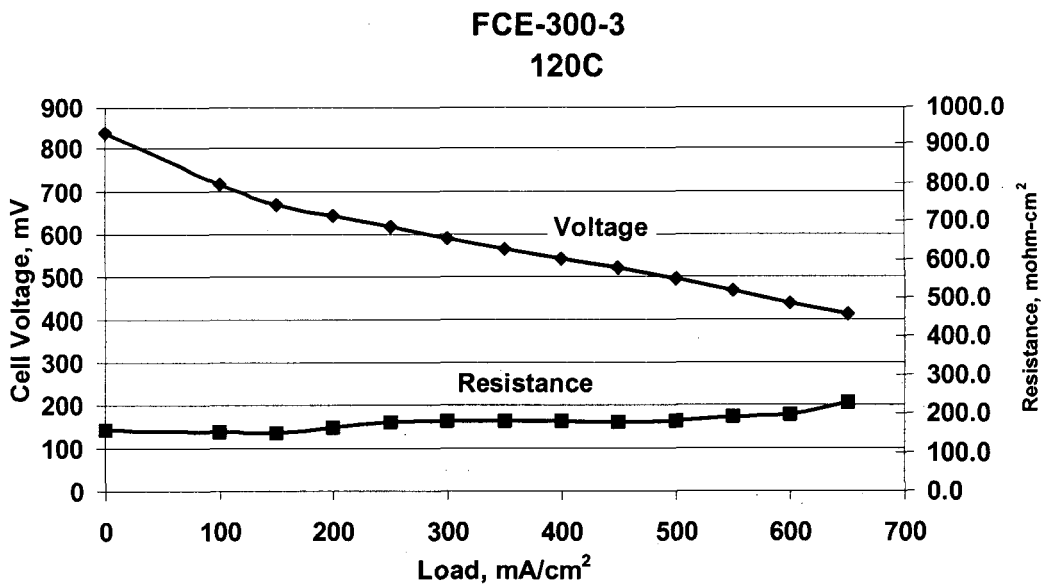


Figure 32. 300cm² Cell Performance at 120°C (H₂/O₂):
Reasonable cell performance and resistance were achieved.