Fuel cell techno-personal milestones 1984–2006

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Abstract

This paper is based on my award acceptance talk on the occasion of receiving the Grove Medal for Fuel Cell Science and Technology, at the 2006 Grove meeting in Torino, Italy. I chose to name the talk: "Fuel Cell Techno-Personal Milestones 1984–2006", trying to reflect on important milestones in the history of the science and technology of hydrogen/air and methanol/air polymer electrolyte fuel cells, in which I was fortunate to be involved for over 20 years.
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I started working in Los Alamos National Laboratory on a permanent basis in the summer of 1984. A group at Los Alamos was already involved at the time, albeit at a low level, with some, US DOE-funded investigations into the fundamentals of the poly[perfluorsulfonic acid] (PFSA) ionomer and the Pt/ionomer interface. The investigations were driven by interest in examining the potential application of a hydrogen/air fuel cell, based on such polymer electrolyte, as power source for transportation applications. The technology development plan at Los Alamos targeted basically conversion of a PEFC space technology, as developed earlier at GE and Hamilton Standard, to a terrestrial PEFC technology with attractive advantages including a system with no liquid electrolyte, fuel flexibility, high conversion efficiency and zero tail pipe emission.

Entry in the early 1980s into this new type of fuel cell technology for potential terrestrial applications was quite bold. The risks involved were actually summarized in a 1987 paper by Yeager and Appleby, that mentioned three severe "show stoppers" on the way from a space to a terrestrial polymer electrolyte fuel cell technology. These two authors highlighted three major barriers on the way to such transition:

(a) At least $10\times$ lowering of the Pt loading versus the 1980s state of the art PEFC was required to start making some economic sense in non-space applications. And this would have to be done while, at the same time, switching from neat O_2 used in the space technology, to an air fed cathode. It was recognized that development of electrodes which utilize the Pt catalyst better, was a challenge in the case of solid electrolytes like poly[PFSA]. Access of protons to any Pt crystallite removed more than a few nanometer from the proton conducting membrane surface is very poor when the Pt catalyst layer is held together and bonded to the membrane with PTFE, as in the case of the 1980s PEFC space technology. The important system simplicity achieved by elimination of any liquid electrolyte, carried with it a serious challenge: understanding what, if anything, can be done to provide sufficient protonic access to most the Pt catalyst sites distributed evenly in a catalyst layer 5–20 μm thick, while the only liquid in the system (de-ionized water) has no protonic conductivity.

(b) Not only the cathode needed to accommodate a diluted oxygen reactant (air) in terrestrial transportation applications, the anode needed to accommodate hydrogen of much lesser purity than the cryogenically stored hydrogen employed as fuel in the space PEFC technology. Hydrogen was expected to be generated from carbonaceous precursors, likely natural gas or methanol, and, consequently, difficult to obtain at carbon monoxide levels significantly lower than 100 ppm.

And at such partial pressure of CO, Pt anode catalyst surfaces will be largely covered by CO adsorbate at the typical temperature of PEFC operation (80 °C), thereby lowering strongly the rate of hydrogen oxidation at some given anode overpotential. Providing a radical solution for this "CO poisoning" challenge was critical for the implementation of PEFCs in transportation applications.

(c) Significant rise of the poly[PFSA] membrane resistance with cell current had been documented and was understood to be likely involved with insufficient hydration in, at least, some part of the ionomeric membrane when the cell is under current. The nature of this profile was not well understood, however, and no modeling tool for its quantification was available. Overall, the issue of effective "water management" in a PEFC, i.e., satisfying membrane hydration requirements under the complete dynamic range while avoiding excess liquid water in the cathode, was considered in the mid-1980s another tough nut to crack.

The general approach of the Los Alamos fuel cell R&D team was to address these challenges at two levels. At the "science level", each of the three problems was addressed by an effective description, i.e., by mathematical modeling employing the relevant physico-chemical parameters which, if unknown, were determined experimentally. At the "application level", practical technical solutions were provided that could be effectively implemented in the PEFC's membrane electrode assembly, the feed streams or some other fuel cell component, lifting to large degree the above three technology barriers. In reality, the practical technical solutions provided preceded the full understanding and quantification of any of the above problems. However, in such cases, subsequent detailed physical modeling enabled to fully appreciate all facets of the problem and, consequently, open the door for additional improvements based on further optimization of components and operation conditions.

A chronological description of the advancements at LANL in PEFC science and technology in the mid-late 1980s, should start from the success in demonstrating high PEFC performance on hydrogen and air with Pt loading of under 0.5 mg Pt cm^{-2} per electrode. The credit for this LANL team accomplishment between the late 1980s and early 1990s, is largely due to Raistrick and Wilson [1,2]. Raistrick was the first to impregnate a PAFC electrode from E-Tek, based on Pt/C catalyst at about 0.4 mg Pt cm^{-2}, with a Nafion solution, before pressing it onto a Nafion membrane to form the membrane electrode assembly [1] (such electrodes were supplied later by E-Tek, under the name "ELAT"—an acronym for Electrode a-la Los Alamos Technology). Filling the porous network of the Pt/C catalyst layer with recast Nafion, provided the continuous proton path from the membrane to, at least a significant fraction of Pt sites within the relatively thick (>20 μm) catalyst layer of such PAFC electrodes. Consequently, catalyst utilization was dramatically improved versus the Pt black/PTFE composite layers used in the space PEFC technology, enabling an increase by factor 10, or more, of the current density achieved at some overpotential per mg Pt cm^{-2}.

The next step in the same direction was made shortly later, with the technology developed by Wilson for application of an ink, made of a mixture of Pt/C, Nafion solution and some viscosity controlling additives, directly to the membrane [2].
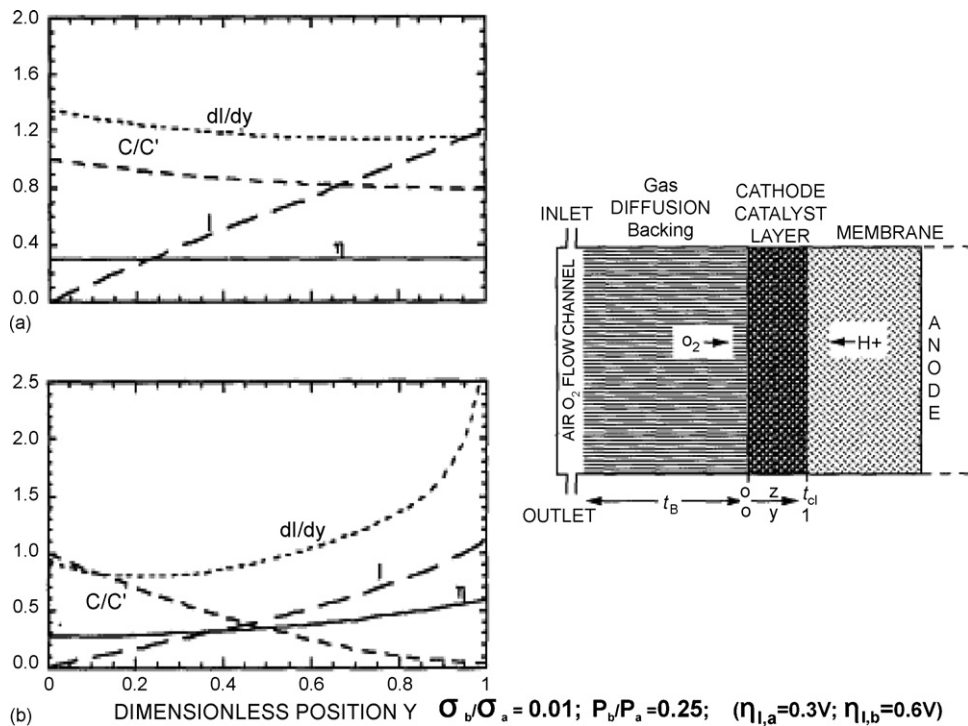

Fig. 1. Distributions, at current density of 1 A cm^{-2}, of electrode potential, reactant concentration and current generation in a PEFC catalyst layer, as result of limited transport rate of the gas reactant and/or the limited transport rate of protons. Two cases of reactant concentration and two cases of effective protonic conductivity in the catalyst layer are considered in these calculations (see Ref. [3]).

This development enabled the fabrication of significantly thinner catalyst layers (5–10 μm) of improved bonding to the ionomeric membrane. Catalyst utilization was thereby further improved significantly and a door was also opened to MEA manufacturing based on some established thin film fabrication techniques, such as screen printing, spraying or ink-jet printing.

The two practical steps described above, of using Pt|C||recast Nafion, composite catalyst layers bonded, or pressed to the ionomeric membrane to achieve good catalyst utilization, was followed by the first simulation of the combined effects of transport properties within such catalyst layers on the observed electrode current density [3]. Such simulation provided a first quantitative correlation between transport parameters and volume fractions of the ionic, electronic and gaseous reactants and products of the electrocatalytic process within the catalyst layer, and the net rate of the electrode process derived at some electrode overvoltage, given a uniform volume distribution of catalyst nano-crystallites and a local interfacial rate determined by a local overpotential and concentration. This was one of the first such simulations performed by Tom Springer of the Los Alamos fuel cell R&D team, who provided several seminal contributions to mathematical modeling of processes in polymer electrolyte fuel cells. Results of the 1993 paper that first showed distributions of overpotential, local rate of current generation and total current generated, all as function of location in the catalyst layers, are shown in Fig. 1. The figure elucidates how, with (in principle achievable) higher effective specific protonic conductivity and gas permeability within the catalyst layer, a cathode current density of $1 \, A \, cm^{-2}$ could be obtained at measured cathode overvoltage of 0.3 V (top left part of Fig. 1), whereas significantly lower transport rates of protons and gas molecules (bottom left part of Fig. 1), could result in a need of a prohibitive overvoltage of 0.6 V to achieve the same cathode current density. The specific interfacial rate constants for the electrocatalytic process itself at the Pt/recast Nafion interface, are assumed to be exactly the same in both cases (a) and (b) in Fig. 1 and the big difference calculated in electrode performance is thus all to do with limited access of protons and/or oxygen molecules to catalyst sites away from the membrane or away from the gas diffuser, respectively. Such early contributions to the understanding and quantification of the key characteristics of the polymer electrolyte fuel cell, provided a basis for a significant number of subsequent contributions, aiming at further refinements and insights. Fig. 2 shows, as example, very recent work by Mukherjee and Wang [4], where the distribution within
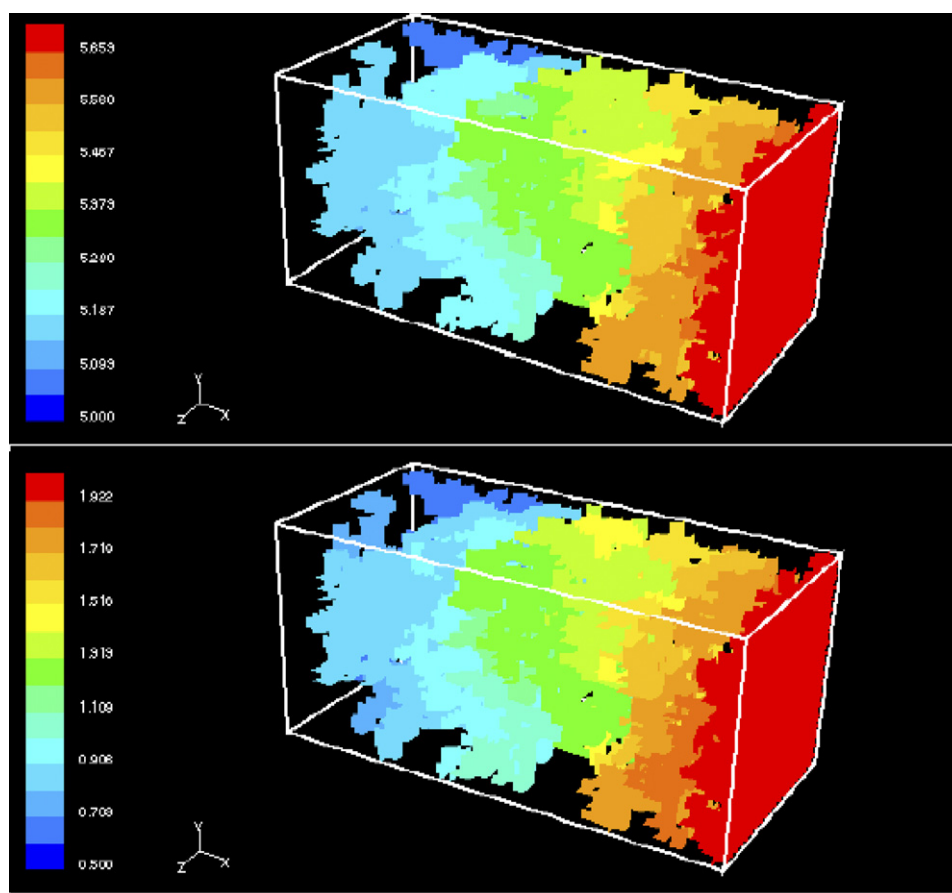

Fig. 2. Calculated three-dimensional contours of the oxygen concentration in a 10 μm-thick cathode catalyst layer of a PEFC, at $0.5 \, A \, cm^{-2}$ (top) and $1.5 \, A \, cm^{-2}$ (bottom). The drop in oxygen concentration across the thickness dimension of the catalyst layer is 1.2× in the top case ($J_{cell} = 0.5 \, A \, cm^{-2}$) and 4× in the bottom case ($J_{cell} = 1.5 \, A \, cm^{-2}$). The concentration of oxygen at the backing/catalyst layer interface (designated by red), corresponds in the top and bottom case, respectively, to 70% and 20% of the concentration of oxygen in air (from Ref. [4]). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of the article.)
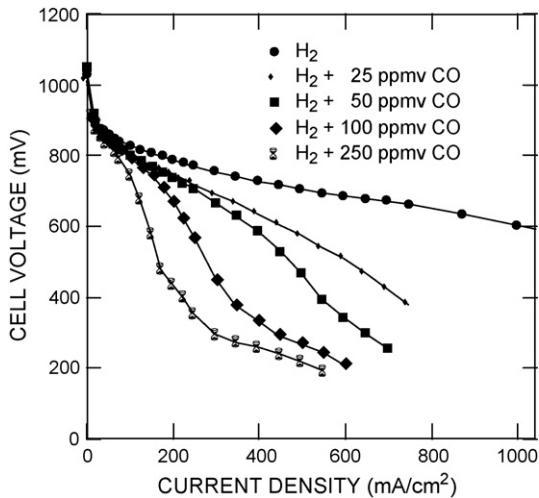
Fig. 3. PEFC polarization curves recorded at Los Alamos (1987) showing the effect of low levels of CO in the anode feed stream on the cell polarization curve.

the catalyst layer was evaluated in three dimensions, enabling to reveal the high effective tortuosity of the oxygen path in such catalyst layers. Such high tortuosity explains why relatively low effective transport parameters may be seen for the composite PEFC catalyst layer, even at high volume fractions of the relevant component. To be able to use in such modeling the proper interfacial rate constants for oxygen reduction, contributions by the Los Alamos team during the same period of time provided direct measurements of the ORR rate at a novel model system which very closely resembles the composition of the actual interface in a PEFC [5]. Such measurements also revealed the strong effect of the level of local humidification on the ORR rate at this interface.

This early example of substantial advancements in cell performance, combined with detailed mathematical description of cell characteristics in terms of the key physical parameters of electrode and cell, reflects well the approach adopted by the team at LANL to PEFC and, somewhat later, to DMFC R&D. In a similar way, the issue of severe anode catalyst poisoning by CO in a hydrogen/air PEFC in the presence of levels as low as 100 ppm CO, was addressed at both the practical remediation level and at the mathematical model level, where the model is based on the physical description of the relevant chemical and electrochemical processes. Fig. 3 shows polarization curves measured at LANL in a hydrogen/air PEFC back in the late 1980s and Fig. 4 shows simulated polarization curves which consider the rates of adsorption and desorption of CO and of hydrogen and the potential dependent rates of electrochemical oxidation of these molecules [6]. In that simulation, the dashed curves are simulated based on adsorption energy of CO which is not CO coverage dependent, whereas the solid curves are simulated based on adsorption energy of CO which falls linearly with CO coverage. The latter assumption is seen to give better fit to the observed dependence on Pco seen in Fig. 3. A sharp fall in the rate of hydrogen electro-oxidation (i.e., in anode current) in the presence of CO, is seen to occur at an ever lower cell current as the CO level (in hydrogen) increases

from 25 ppm to 200 ppm. The basic feature explaining such fall at some "critical current", is the confinement of the dissociative adsorption of dihydrogen to a small number of catalyst sites free of CO, as the coverage by CO of a Pt catalyst surface is very high at temperatures under 100 °C, even at a mole fraction of around 100 ppm in the gas phase. The anode overpotential required to electrooxidize the site-blocking CO off the surface of a Pt anode catalyst in the PEFC, is recognized from Figs. 3 and 4 as the strong drop in cell voltage needed to generate currents exceeding the "critical current". This drop in V_{cell} is seen to be around 0.5 V and the resulting penalty in cell conversion efficiency is obviously prohibitive. It can be understood, therefore, why such performance loss at 100 ppm CO was considered a serious impediment to implementation of PEFC technology in terrestrial applications, where hydrogen fuel derived from methane (natural gas), would be the most likely anode feed.

A radical possible solution to the issue of catalyst poisoning by CO was established in the late 1980s at Los Alamos [7,8], by revealing that continuous "air bleed" into the anode at a mol fraction of the order of 1%, can recover completely cell performance in the presence of CO levels around 100 ppm. The chemical processes at the anode catalyst during such air bleed, were subsequently explained by the following sequence:

- Catalyst site poisoning:

(a) 10 ppm CO at 1 atm $H_2 + Pt = Pt–CO_{ads}$
(b) also, 10 ppm CO at 1 atm $H_2 + Pt–H_{ads} = PtCO_{ads} + 1/2H_2$.

- "Cleansing" of the catalyst site by bleeding of dioxygen:

(a) $Pt–CO_{ads} + (1\%)O_2$ (at 1 atm H_2; 10 ppm CO) $+ Pt = Pt–O_{ads} + Pt–CO_{ads}$
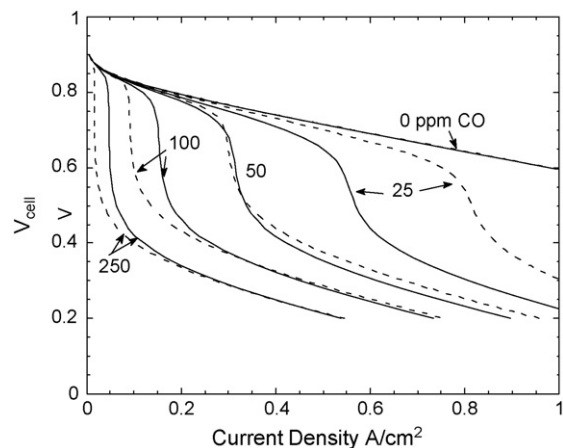(b) $Pt–CO_{ads} + Pt–O_{ads} = CO_2 + 2Pt$,

Fig. 4. Mathematical simulation of the polarization curve of a hydrogen/air PEFC in the presence of CO levels ranging between 25 ppm and 200 ppm. The model explained the appearance of a "limiting current behavior" from the availability of very limited number of CO-free sites for the chemical step of dissociative hydrogen adsorption. Better fit to experiment was obtained with coverage dependent adsorption energy of CO (see Ref. [6]).

with any oxygen beyond that consumed in the last process, reacting with hydrogen at CO-free Pt sites, according to

$$\frac{1}{2}O_2 + H_2 = H_2O$$
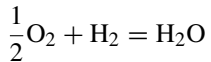
The validity and viability of this approach were questioned quite seriously at the outset. Skepticism originated, to large degree, from the unusual attempt to implement a heterogeneous chemical catalytic process, i.e., CO oxidation by dioxygen, into an anode where the electrochemical process of hydrogen electro-oxidation took place at the same Pt catalyst. Other questions raised had to do with cell heating effects caused by the air bleed (much lower in magnitude than the rate of overall heat generation in the cell brought about by the roughly 50% efficient conversion of fuel to electric power) and, more recently, to do with the development in understanding of generation of hydrogen peroxide during hydrogen/oxygen recombination at a Pt catalyst. It seems, however, that air bleed remains to date a useful tool for ensuring high performance in the presence of CO in the fuel feed stream to a PEFC anode, with the perceived weaknesses explained and some real challenges addressed. Historically, it was an important milestone in the wide adoption of the hydrogen/air (reformate/air) PEFC as a technology base for future power sources in terrestrial transportation, as well as in stationary power applications.

The last, but certainly not least significant chapter of PEFC science and technology development at Los Alamos between the late 1980 and the mid 1990s, was the detailed description of water distribution in the ionomeric membrane in a cell under current and the ability to explain, from it the observed rise in cell resistance at high current densities in PEFCs. Fig. 5 shows the results of modeling presented in a 1991 paper [9], of the water profile in a 175 μm-thick Nafion membrane in a PEFC operating at various current densities. The profile was modeled considering the opposite fluxes of water driven by (i) electro-osmotic drag of protons moving from anode to cathode and (ii) diffusion from cathode to anode driven by a water chemical potential gradient.

For this model to be predictive, it required detailed experimental determination of membrane water content as function of water activity and of the water level dependent diffusion and electro-osmotic drag coefficients in the membrane. The profile evaluated in a 175 μm-thick Nafion membrane with the boundary conditions defined by vapor saturated gas feed streams, is seen on the left part of Fig. 5. The water profile in the membrane projected at current densities around 1 A cm^{-2}, is seen to be highly non-uniform with the level of water near the anode projected at only 20% that of maximum membrane hydration level at the cell temperature, as established near the cathode. The impact of such water profile on the rise of membrane resistance with current density in a 175 μm-thick membrane, is seen in the top right side of Fig. 5. This model thus predicted quantitatively the observed rise of cell (membrane) resistance with cell current. Furthermore, it could be immediately seen from the same model (top right side of Fig. 5), that, if the membrane were to be thinned down, e.g., to 50 μm, the rise in membrane resistance with current will become insignificant, even at the high end of the current density range typical for PEFCs. The reason for that, is the much smaller degree of water level drop on the anode side of the thinner membrane, resulting basically from high water "back diffusion" rates enabled under smaller water level differential across a significantly thinner membrane. This elucidation of the importance of thinner poly[PFSA] membranes in facilitating water management in a PEFC [9], had a significant effect during earlier days of PEFC technology development, in substantiating the need for such thinner membranes if high power densities approaching 1 W cm^{-2} are to be achieved in PEFCs. Thinner membranes provided subsequently by DuPont and W.L. Gore and, more recently, by 3 M, allowed to achieve such high maximum power densities in PEFCs, removing the prohibitive rise in membrane resistance at high current.

Adding here a very recent, elegant complement to the early work done at Los Alamos, a recent effort at Tokyo Institute of Technology [10] is presented, that provided an image of the water distribution profile across a Nafion membrane in an oper-
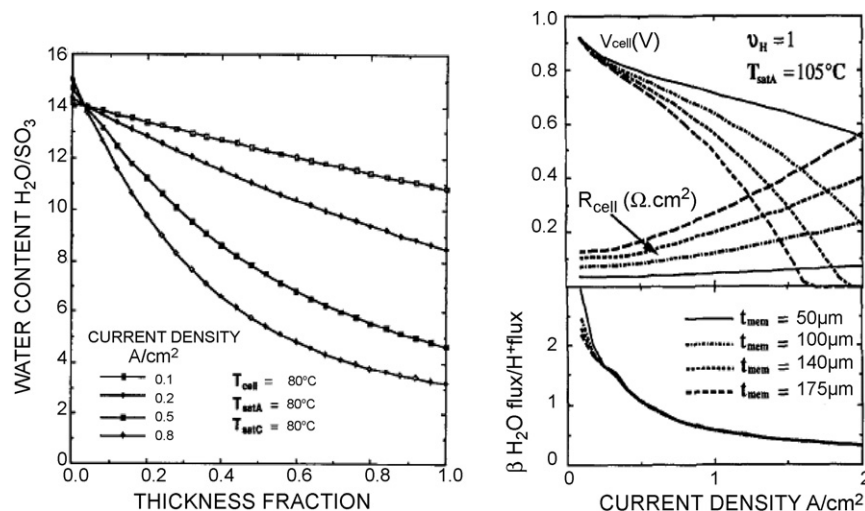

Fig. 5. Water profiles in a Nafion® membrane (left), R_{cell} and the ratio water flux/proton flux through the cell (right), at several current densities in an operating PEFC; cell and gas saturators are assumed to be both at 80 °C (from Ref. [9]).
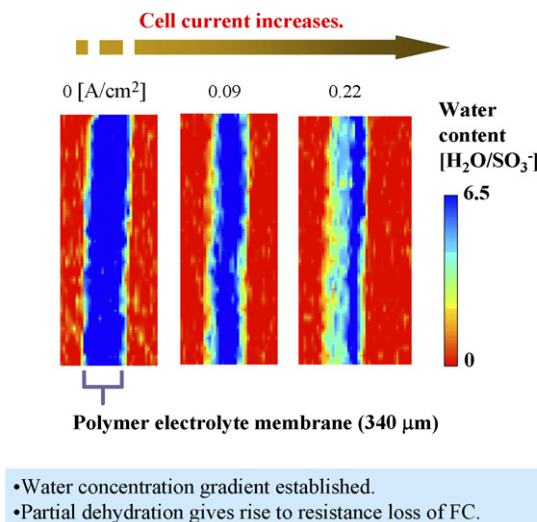
Fig. 6. MRI of water distribution in a PEFC at zero current and under two current densities. The cathode is on the right side of the MEA imaged (from Ref. [10]).

ating PEFC and its dependence on cell current. The images reported in [10] are shown in Fig. 6 which clearly reveals the rise in the steepness of the water profile with cell current, as directly imaged by MRI for a thick PEFC membrane (a 340 μm-thick membrane was used to accommodate the special resolution available with the imaging tool used; boundary conditions here are defined by dry gas streams on both sides of the cell).

Summarizing these seminal contributions to PEFC R&D by the Los Alamos team in the 1980s–1990s, I believe it is fair to say that the work of that team paved to significant degree the way to the vast increase in industrial interest and industrial involvement in PEFC technology. In the mid-1980s, the total R&D investment by Industry in PEFC technology, was a few million dollars per year (if that) and it increased by the mid-1990s to the level of 10^8 per year in several industrial development centers. I feel really privileged to have had the opportunity to work with in that team during that period of time.

Switching now to polymer electrolyte, direct methanol fuel cells (DMFCs), I like to preface this last part by saying that, as electrochemist specializing originally in electro-catalysis, the drive to develop fuel cell technologies based on direct conversion of a given fuel to electric energy, has been a preferred choice in terms of personal inclination. And this choice has been further amplified by the complexities and cost of elaborate fuel cell systems developed in recent years based on fuel processing upstream a PEFC stack. Methanol has always been the first carbonaceous molecule targeted for direct electrochemical oxidation and my first extensive involvement with DMFC work was at Los Alamos, during the project run in the 1990s by DARPA in a number of US national labs and industries, targeting advanced portable power sources for the military. At the outset of that project, one apparent serious barrier to implementation of portable DMFC technology, was a specific weakness of the polymer electrolyte fuel cell version. While having the highly attractive feature of no need of any liquid electrolyte and having demonstrated already high performance of H_2/air

PEFCs, the polymer electrolyte cell was considered almost unacceptable for methanol/air operation, because the permeability of methanol through Nafion membranes is high—equivalent of $100 \, mA \, cm^{-2}$ with 1 M methanol on the fuel side of the membrane. With loss of methanol by "cross-over" at a rate similar to that of anodic conversion of methanol at the anode catalyst, the cell efficiency was obviously projected to be too low. The main challenge perceived, was to come up with an alternative ionomeric membrane that would limit strongly the rate of methanol permeation and, at the same time, maintain good protonic conductivity. This challenge is still basically unanswered today, 15 years after the beginning of efforts to synthesize such a "DMFC alternative membrane", because the mobility of the proton and of methanol in the membrane both depend to very similar degrees on the water content and the porosity/tortuosity of the membrane. The different approach to this issue developed at Los Alamos, was to adhere to the "leaky", but available Nafion membrane and lower the rate of methanol cross-over in a DMFC under current by working at a cell current close to the anode limiting current set by the porosity/tortuosity of the anode backing [11]. This principle is demonstrated in Fig. 7 which shows the strong drop in methanol concentration next to the anode side of the membrane versus the concentration in the anode flow channel, when the cell current is a large fraction (e.g., 90%) of the anode (and cell) limiting current. This approach is helped, in fact, by the nature of the rate limiting step in the methanol electro-oxidation process: this step is zero order in methanol and, consequently, the interfacial (electrocatalytic process) rate at the DMFC anode is not reduced as the local concentration of the reactant drops strongly when the cell current is so close to the anode limiting current.

Having established at Los Alamos the viability of polymer electrolyte DMFCs based on state-of-the-art, poly[PFSA] membrane, it seemed that a cell with such membrane, using direct supply of aqueous methanol to the (PtRu catalyzed) anode and of air to the (Pt catalyzed)cathode, could be the basis for a first
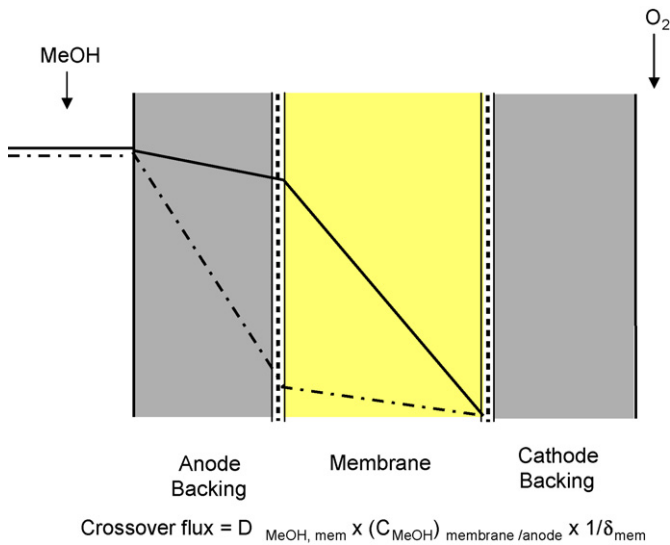
Fig. 7. Lowering the rate of methanol cross-over by operation at cell current close to the limiting current defined by the anode backing layer. Solid, MeOH concentration profile at open circuit; dashed, MeOH concentration profile near $J_{lim,an}$ (Ref. [11]).

DMFC power source product. I consequently accepted the offer of colleagues from MTI in Albany, NY, to start in early 2001 what became MTI Microfuel Cells. We targeted development of advanced portable power sources for consumer electronics as well as military applications, using as base line Los Alamos DMFC technology (year 2000 state-of-the art). Such portable power sources have had as goal an energy density advantage of 2–5× versus the most advanced rechargeable Li batteries. One central DMFC system challenge that attracted much attention on the way to defining a viable, miniaturized DMFC platform, was the issue of water management. In order to operate with no

water being carried as part of the miniaturized power system, some of the water generated at the DMFC cathode needs to be recovered and transported back to the DMFC anode, where it is used as a reactant in the anode process. Such water capture and back pumping to the anode around the cell, would make the DMFC system more complicated and bulky than acceptable for a battery-like, miniaturized power source. A battery-like power source has to be as passive as possible and as simple as possible to enable effective miniaturization and provide "transparency" to customers adopting such disruptive new technology.

The solution to the challenge of effective water management in a small-scale DMFC, was found by the team at MTI Microfuel Cells as result of the rule: "strong necessity is the mother of invention". Having been first through various early versions of miniaturized pumps and valves and facing the drawbacks of immature MEMS technology (2001–2002), the solution came from proper selection of the physical dimensions and the hydrophobic or hydrophilic nature of components of the unit cell itself. With optimized parameters for the above cell features and a proper selection of the cell operation design point, it could be demonstrated that all water required for the anodic process can be made to spontaneously flow through the thickness dimension of the cell, from the cathode to the anode. The scheme of this highly simplified DMFC platform (Mobion®) is given in Fig. 8. This enabled a tremendous advantage of being now able to supply the anode directly with 100% methanol fuel, operating with no need of any liquid pumping/recirculation around the cell. The cathode was also shown to be operable in passive mode ("air breathing"), thereby enabling a completely passive platform which is indeed much more "battery-like" than the classical DMFC system. This chapter of technology development at MTI Micro, was very rewarding as it showed that innovative modifications of the "heart" of the system, i.e., the basic composition and structure of the unit cell itself, can provide great simplifica-
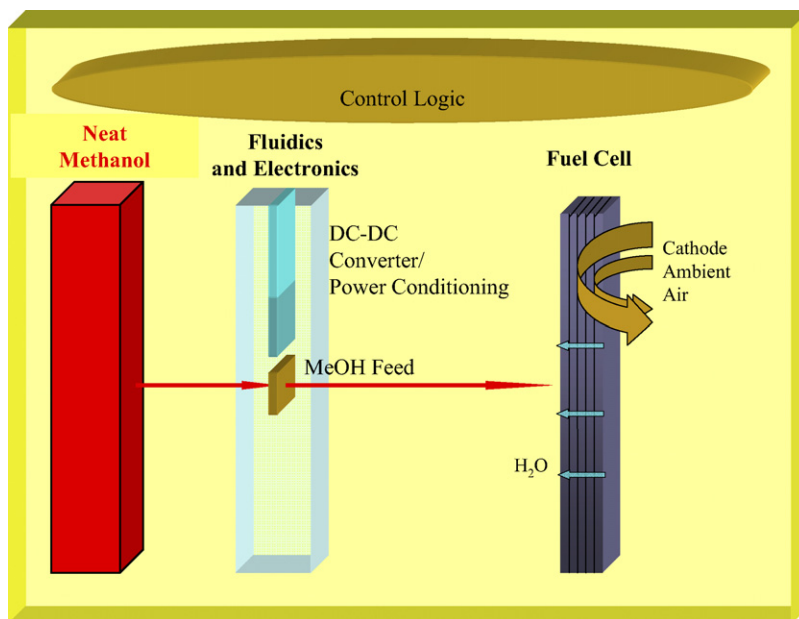

Fig. 8. Highly simplified DMFC system developed at MTI Microfuel Cells, Albany, NY, Mobion® based on direct feed of 100% methanol to the cell anode at controlled rate and elimination of any need of external water pumping.

Fig. 9. Air breathing DMFC power system including a flat cell array and methanol cartridge with 100% methanol (directly fed to the anode), integrated into the handle of a RFID tag reader.

tions of the overall system, replacing/saving major engineering efforts into auxiliaries. Demonstration of an early limited release product based on such passive operation of a polymer electrolyte DMFC, with 100% methanol supplied directly to the anode and the cathode breathing air, was late 2004. It is shown in Fig. 9. The methanol cartridge, the fuel cell and the controls were all introduced as part of the handle of a RFID tag reader (this RFID made by Intermec, Inc.) with no change to the external contours of the device. In a continuous discharge test, the energy harvested was 35 Wh per (100%) methanol cartridge, to be compared with the 8 Wh rechargeable battery placed ordinarily in the cylindrical part of the RFID handle seen in the photograph. The particular example in Fig. 9, is of a passive, air breathing DMFC system with the rate of (non-pumped) methanol supply to the anode the only system parameter under control.

Concluding my talk here, I hope this brief "scan" of highlights of fuel cell R&D activities during the last 22 years, conveyed some of the great personal satisfaction of being involved in those key steps of development of the science and technology of PEFCs and DMFCs. This interdisciplinary field of R&D presents serious challenges to developers of the technology, working typically as a team of scientists and engineers of various specialties and requiring very good level of communication and understanding across the team of all key issues and critical parameters. Such requirements are not always easy to meet, however when significant solid advancements are being made towards critical cost/performance targets, thereby opening the way to a new power source of ultra low emissions, high efficiency and superior energy density, the personal reward is very high indeed.

Note added in proof

A recent review covering PEFC and DMFC science and technology advancements updated to the mid 2000's, is scheduled to be available in 2007: S. Gottesfeld, "Polymer Electrolyte and Direct Methanol Fuel Cells", in the volume "Applied Electrochemistry" of the "Encyclopedia of Electrochemistry", A.J. Bard, chief editor (John Wiley, in press).

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