

# Fuel Cells Science and Technology 2006

## SCIENTIFIC ADVANCES IN FUEL CELL SYSTEMS

Reviewed by Donald S. Cameron

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Following meetings in Amsterdam in 2002 (1) and Munich in 2004 (2), another in this series of conferences was held on the 13th and 14th September 2006 at the Turin Incontra Conference Centre, Turin, Italy, with the theme 'Scientific Advances in Fuel Cell Systems'. The conference series (3) alternates with the Grove Symposium (4, 5), with a more technology-oriented content.

Authors from around the world submitted oral papers and posters from which the programme was compiled. Organised by the Grove Symposium Steering Committee and Elsevier Science, the meeting attracted almost 300 delegates from universities, research organisations, and fuel cell component manufacturers. The 34 countries represented included Japan (44 delegates), Italy (42 delegates), Germany (39 delegates), the U.K. (31 delegates) and the U.S.A. (12 delegates). As well as 56 oral papers, there were six poster categories with 205 high-quality poster presentations. The conference consisted of six sessions, covering the following subject areas:

- membranes
- systems analysis and applications
- component modelling and characterisation
- electrocatalysis
- fuels
- fuel cell and stack technology.

The latter two categories attracted sufficient papers to occupy two sessions each. Since the topic of fuel cells covers such a wide area, for this review only papers involving use of the platinum group metals (pgms) have been selected.

### Grove Medal

For the first time, the Grove Medal was presented at a Fuel Cells Science and Technology conference, rather than at the Grove Fuel Cell Symposium. The 2006 recipient was Shimshon Gottesfeld for his sustained high-quality scientific

and technical contribution to the field of fuel cells, combined with his drive and vision to commercialise new fuel cell technology, building on his research. Gottesfeld was Vice President and Chief Technology Officer of MTI Micro Fuel Cells Inc from December 2000 until May 2006, and prior to this he led the Fuel Cell Research Program at the Los Alamos National Laboratory (LANL) for more than 15 years. He is now a consultant with Fuel Cell Consulting.

In a Plenary talk, Gottesfeld focused on some of the important milestones along the paths of polymer electrolyte fuel cell (PEFC) and direct methanol fuel cell (DMFC) science and technology, culminating in a new polymer electrolyte DMFC concept developed at MTI Micro Fuel Cells Inc. These milestones include reducing the platinum loadings of solid PEFCs by an order of magnitude, by impregnating the catalyst with Nafion<sup>®</sup> perfluorosulfonic acid polymer (in 1986), fabricating electrodes by the application of catalyst/Nafion<sup>®</sup> 'inks' in 1992, and developing PEFCs capable of tolerating a concentration of 100 ppm of carbon monoxide in the hydrogen fuel by incorporating a small oxygen bleed into the fuel to oxidise the impurities on the anode (in 1998). The latest development is the Mobion<sup>®</sup> DMFC system, providing a high power density and high efficiency, up to 1100 Wh l<sup>-1</sup>, as compared with 200 Wh l<sup>-1</sup> for lithium prismatic batteries and 400 Wh l<sup>-1</sup> for cylindrical lithium batteries.

### Fuel Processing

Most fuel cells require a supply of reasonably pure hydrogen, and many projects exist to build small, portable reformers for fossil or renewable fuels. In his talk 'Fuel flexibility of bimetallic Pt-Ni system: Hydrogen production from LPG having different C3:C4 ratios', Ahmet Aksoy (Bogazici University, Turkey) explained that liquid petrole-

um gas (LPG) is one of the most promising hydrocarbon fuels for hydrogen production. However, the composition of LPG varies from country to country depending on local crude oil supplies and refinery processes. In this study of indirect partial oxidation of LPG over platinum-nickel on  $\delta$ - $\text{Al}_2\text{O}_3$  catalysts, the effects of temperature, steam:carbon ratio, carbon:oxygen ratio and residence time on the activity and the selectivity of the catalyst were varied. The results indicated that the Pt-Ni bimetallic system has high  $\text{H}_2$  production activity and selectivity in the temperature range 350 to 450°C, owing to the utilisation of the catalyst particles as micro heat exchangers, in which heat produced at platinum sites by oxidation catalyses the endothermic steam reforming reaction.

A more efficient use of coal was proposed by Gerardine Botte (Ohio University, U.S.A.), in her talk 'Hydrogen production from coal electrolysis'. In 1979 (6) a new means of coal gasification was discovered at the University of Connecticut, using platinum electrodes to electrolyse coal slurries at 25°C to generate pure streams of  $\text{CO}_2$  and hydrogen at the anode and cathode respectively, free of tar and sulfur compounds. The authors report that the reversible thermodynamic potential for the reaction is only  $-0.21$  V. This compares with the potential for conventional water electrolysis, which is theoretically 1.23 V, but in practice is rather higher than this. Effectively, the free energy change resulting from the oxidation of carbon lowers the voltage required for electrolysis. The  $\text{CO}_2$  product can be disposed of in a number of ways, while intermediate hydrocarbons which are also formed can be used to make byproduct oils.

One of the problems associated with the electrolysis is the low reaction rate. However, recently Ohio University has developed improved catalysts and improved operating conditions for the process. These include using higher operating temperatures (80°C), carbon fibre and titanium electrode support materials, and optimised platinum-iridium catalysts. The ratio of Pt:Ir is an important parameter, and these catalysts are more effective than platinum, while rhodium does not favour the carbon oxidation reaction.

In his talk entitled 'Fuel processing in integrat-

ed microstructured heat-exchanger reactors – current status and future perspectives', Gunter Kolb (Institut für Mikrotechnik Mainz GmbH (IMM), Germany) reported work on fuel processor components for systems rated from 100 W up to 100 kW, for a variety of fuels. The reactions take place inside heat exchangers, with exothermic catalytic combustion on one side and the endothermic reforming reaction on the other. Anode off-gas from the fuel cell is burned to provide the heat required. In their 5 kW-rated autothermal reactor for octane, which uses a rhodium catalyst, it is possible to obtain carbon monoxide impurity levels in the product hydrogen lower than 20 ppm. The reactors are also arranged for water injection to remove CO by the water gas shift reaction – the latter is facilitated by introducing a temperature gradient inside the heat exchanger, thus eliminating a second shift reactor. The estimated market for various applications is for 1000 to 10,000  $\text{y}^{-1}$  reformers for yachts and caravans, and 10,000 to 100,000  $\text{y}^{-1}$  for auxiliary power units for vehicles.

## Systems and Applications

The topic of pgm use was an important feature of a talk by Florian Finsterwalder (DaimlerChrysler AG, Germany) on 'Achievements and challenges in automotive PEM fuel cell stack development'. For vehicle use, fuel cells must operate at temperatures varying from sub-zero to preferably around 120°C to reject waste heat, in conditions with a wide range of humidity values. Between 1992 and 2006 there has been a linear reduction in the amount of pgms used on the electrodes for DaimlerChrysler vehicles, to a current value of 1.43  $\text{mg cm}^{-2}$  Pt, and a maximum power density of 0.72  $\text{W cm}^{-2}$ . Quoting U.S. Department of Energy milestones for 2010, there are targets for cost of U.S.\$30  $\text{kW}^{-1}$  for vehicle fuel cells, maximum power density of 0.8  $\text{W cm}^{-2}$ , and platinum metal loading of 0.3  $\text{g kW}^{-1}$  (implying a platinum loading of 0.24  $\text{mg cm}^{-2}$ ). Improved separators and membranes should enable the attainment of a power density of 2000  $\text{W l}^{-1}$  with durability of 4000 hours, and Finsterwalder is optimistic as to the industry meeting these targets for vehicle fleets numbering thousands.

The concept of a microchip with an integrated PEFC is being investigated at the University of Freiburg, Germany, and also Micronas GmbH, Germany, by Gerhard Erdler and coworkers. With a silicon chip as a basis, a palladium hydride anode is used in conjunction with an air depolarised platinumised cathode and a polymer electrolyte. The 4 mm square, 200  $\mu\text{m}$  thick palladium anode is charged with hydrogen, a 10 to 20  $\mu\text{m}$  polymer layer is applied by spin coating, and a platinum catalysed carbon cloth cathode is applied on top. Current collection is carried out using gold tracks. The generator is intended to power devices such as complementary metal oxide semiconductor (CMOS) chips used as body temperature monitors and intelligent 'BAND-AID<sup>®</sup>' adhesive bandages, which require less than 100  $\mu\text{W}$  for periods of up to one week.

## Membranes

Membranes play a vital role in the performance and durability of PEFCs. Improvements in this field were addressed by Eiji Endoh (Asahi Glass Co Ltd, Japan). Nafion<sup>®</sup> type membranes undergo a glass transition at around 80°C, whereas for adequate heat rejection, automotive use demands operation at 110 to 120°C, possibly under conditions of low humidity. A "new perfluorinated ion-exchange polymer composite" (NPC) developed by Asahi reduces the degradation rate by two to three orders of magnitude as compared with conventional membrane materials. This has been operated continuously for more than 4000 hours at 120°C and 50% relative humidity. Whereas the cell voltage fell from around 750 mV to 450 mV at 200 mA  $\text{cm}^{-2}$  during this time, under similar conditions, a conventional membrane electrode assembly (MEA), also with 0.6 mg  $\text{cm}^{-2}$  platinum loading on the cathode and 0.2 mg  $\text{cm}^{-2}$  on the anode, failed after the first 100 hours. The voltage decay was attributed to attack on the carbon support of the cathode catalyst by peroxide released during cell operation, the catalyst layer thickness having been reduced by 44% during this period from 18  $\mu\text{m}$  to 8  $\mu\text{m}$ . More oxidation-resistant cathode catalyst substrates are being sought to improve the durability of the system. Asahi are

seeking improved performance stability beyond 120°C using low surface area forms of their current Ketjen black carbon support, and hope that these, in combination with thinner membranes, will enable maximum cell current density to be increased from 1.6 to 2.0 A  $\text{cm}^{-2}$ .

## Electrocatalysis

The link between carbon stability and catalyst durability was highlighted by Sarah Hudson (Johnson Matthey Technology Centre, U.K.) in her talk 'An investigation into the effect of temperature on the stability of carbons and carbon supported Pt and Pt/Co alloy catalysts during 1.2 V potentiostatic hold regimes'. For automotive applications, catalysts must survive repeated potential cycling and exposure to high potentials during idling or open-circuit conditions. Excursions to high potentials during startup and shutdown can be expected 30,000 times during a 5000 hour fuel cell life. This equates to holding at 1.2 V for 100 hours in an accelerated durability test, and an interim target is sustaining this potential for 24 hours. Catalyst degradation can occur due to carbon loss by oxidation, while platinum surface area loss may result from particle agglomeration and also by dissolution and re-deposition as nanoscale Ostwald ripening.

Two carbons of surface area 850  $\text{m}^2 \text{g}^{-1}$  (C01) and 130  $\text{m}^2 \text{g}^{-1}$  (C03) were used to prepare three catalysts: 40% Pt on C01 with an electrochemical surface area (ECA) of 118  $\text{m}^2 \text{g}^{-1}$  Pt; 40% platinum-cobalt (Pt-Co) on C01 (ECA 24  $\text{m}^2 \text{g}^{-1}$  Pt); and 30% Pt-Co on C03 (ECA 26  $\text{m}^2 \text{g}^{-1}$  Pt). Electrodes were prepared and corrosion rates determined at 20, 40, 60 and 80°C. These indicate that at 80°C less carbon corrosion is seen for heat-treated carbons of low surface area than for commercial carbons of high surface area, for which carbon corrosion increases with rising temperature. The commercial platinum catalysts sinter during 1.2 V potentiostatic holds at 80°C, whereas the electrochemical area loss increases with rising temperature. However, Pt-Co alloy catalysts are stable to metal area loss at 80°C. Extrapolating these results to higher temperatures, for the commercial catalysts it is predicted that at 120°C there

will be significant carbon corrosion and loss of metal area, leading to low cell performance. However, for the carbon C03 of lower surface area, the corrosion rate appears to be independent of temperature. This is therefore the preferred carbon support for operation at higher temperature, in combination with the Pt-Co alloy.

An interesting means of screening catalysts rapidly was presented by Christopher Lee (Ilika Technologies Ltd/University of Southampton, U.K.) in his talk 'High throughput preparation and electrochemical screening of thin films for electrocatalysis in low temperature fuel cells'. Continuous thin-film binary and ternary alloys of varying compositions are prepared using controlled and simultaneous physical vapour deposition in a modified molecular beam epitaxy system. The alloys are deposited onto silicon wafers or 100-element electrochemical array electrode substrates, consisting of  $10 \times 10$  arrays of  $1 \text{ mm}^2$  gold electrodes, in which each electrode is connected to a gold contact pad on the periphery of the substrate. The compositions of individual electrodes can quickly be characterised using energy dispersive spectroscopy (EDS) and high-throughput X-ray diffraction (XRD) experiments. Rapid electrochemical characterisation can also be carried out on all 100 samples using specially developed cell hardware and software. For this purpose, the oxygen reduction reaction in pure oxygen-saturated 0.5 M perchloric acid at  $20^\circ\text{C}$  is used as the test reaction. Used to study the palladium-cobalt-gold ternary alloy system, it was found that the 50% Pd-25% Co-25% Au alloy system exhibits stable surface area. Adding gold to Pd-Co results in reduced activity, but this activity is still enhanced as compared with that of pure palladium catalyst.

As well as being susceptible to poisoning by carbon monoxide, low-temperature PEFCs are affected by carbon dioxide. In her talk 'Unravelling the complexities of  $\text{CO}_2$  tolerance at PtRu/C and PtMo/C', Andrea Russell (Southampton University, U.K.) explained that the performances of platinum-molybdenum/carbon catalysts were previously compared with those of platinum-ruthenium/carbon catalysts at  $80^\circ\text{C}$  in 40 ppm CO and 25%  $\text{CO}_2$ . In contrast to the trend in CO tol-

erance, the PtRu/C was found to have better  $\text{CO}_2$  tolerance than PtMo/C. Deactivation of the anode in the presence of  $\text{CO}_2$  is caused by the build-up of a CO-like poison. Two mechanisms have been proposed to explain the conversion of  $\text{CO}_2$  to CO under PEFC anode conditions: either a reversed water gas shift reaction or electrochemical reduction of  $\text{CO}_2$  can result in formation of strongly adsorbed CO species at the anode. Voltammetry indicates that at the PtRu/C catalysed anode, the reverse water gas shift reaction is dominant, while at the PtMo/C anode, CO is oxidised at a potential corresponding to the  $\text{Mo}^{+4/+6}$  redox couple, and the mechanism of  $\text{CO}_2$  poisoning proceeds *via* the electrochemical mechanism, leaving CO-like species adsorbed on the catalyst.

Robert Reeve (Defence Science and Technology Laboratory, U.K.) described their project to develop high-efficiency direct borohydride fuel cells for unmanned underwater vehicles (UUVs). These fuel cells combine high energy density with a  $\text{CO}_2$ -free exhaust for stealth. Sodium borohydride can provide up to  $5660 \text{ A h kg}^{-1}$  energy density, and can supply hydrogen when platinum or ruthenium is used as a decomposition catalyst, yielding  $948 \text{ kJ mol}^{-1}$ . Alternatively, direct electrochemical oxidation of borohydride can yield up to  $1273 \text{ kJ mol}^{-1}$ , and with oxygen- or hydrogen peroxide-depolarised cathodes, can provide up to  $1.64 \text{ V cell}^{-1}$  at open circuit. For the anodes, the highest energy density is provided by gold catalysts, since these do not catalyse the spontaneous decomposition of borohydrides, and for the cathodes, Pt/C catalysts are preferred. The 2 M sodium borohydride fuel solution is stabilised by the addition of 6 M sodium hydroxide, the alkaline conditions providing a tenfold reduction in the spontaneous decomposition of borohydride. Despite this, some decomposition still occurs during operation at high current density. This is attributed to pH changes within the pore structure of the electrode.

## Cell and Stack Technology

For fuel cell MEAs it is important to maximise the utilisation of pgms and thus reduce the platinum loading. In his talk entitled 'Application of

ink jet technology to fuel cell membrane electrode assembly production', A. D. Taylor (University of Michigan, U.S.A.) described a simple and elegant means of fabricating electrodes with very low loadings of pgms. By substituting catalyst ink solutions in ordinary office ink-jet printers, it is possible to deposit solutions with picolitre precision. Catalysts are mixed with solvents and Nafion® 5% solution to provide inks for the ordinary cartridges. By using colour cartridges loaded with different concentrations of pgms in solution (or even different catalysts), it is possible to build up multiple thin layers on membranes or electrode support materials. The technique produces extremely low loadings of catalysts, and for many applications it is necessary to use several passes of the printer to produce viable catalyst layers (four passes produced 21 µg cm<sup>-2</sup> Pt). However, the method offers a highly controllable means to deposit multiple layers, for example, with pure platinum on the membrane with gradations of Pt/C catalysts on the gas face.

A huge effort is in progress by leading electronics companies, as well as fuel cell developers, to commercialise DMFCs for mobile and remote off-grid applications. Peter Gray (Johnson Matthey Fuel Cells, U.K.) in his talk 'Leading performance DMFC catalysts and MEAs for portable and consumer electronics' reviewed the significant progress made in improving their performance and scaling-up manufacturing. As with other fuel cell systems, the stack incurs one of the largest component costs. To achieve cost savings, Gray outlined the options of reducing platinum metal loading, or improving cell performance at the same loadings. Compared with a saving of 30% from lower metal loadings, by tripling the power density to 150 mW cm<sup>-2</sup>, stack cost savings of 60% can be made, since the size and number of cells can be reduced.

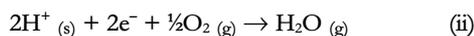
To produce a given performance at the end of the life of the device, costs can be pared by 20% by halving the degradation rate, or by 30% by reducing the decay rate to one third. Already, advanced MEAs have been demonstrated with half the degradation rate of baseline MEAs in accelerated stack durability testing. These

advanced MEAs are being developed using carbon-supported catalysts such as PtRu/C for the anode and Pt/C for the cathode, which together with improvements in MEA design and construction have doubled power densities to over 100 mW cm<sup>-2</sup>. Product lifetimes acceptable for consumer electronics applications have already been demonstrated, and MEAs are in production.

An interesting example of the catalytic properties of the pgms was illustrated by Sadae Yamaguchi (Chiba Institute of Technology, Japan) in his talk entitled 'Performance of one chamber type methane-oxygen fuel cell'. Natural gas or propane fuel are mixed with air, and fed to a solid oxide fuel cell (SOFC) with only one chamber held at 700°C. The anodes are coated with a selective catalyst (platinum, palladium or nickel) which enables them to carry out a partial oxidation reaction, generating CO, protons and electrons (Equation (i)):



The platinum, palladium or nickel coated electrodes are active for the anode reaction, but not for the cathode reaction, while gold or silver coated cathodes are active for oxygen reduction, but not for the anode reaction. Protons in solid solution are transported through the electrolyte to the cathode and are combined with oxygen to form water (Equation (ii)):



It was found that the power density of the cell was not affected by electrode cracking. This is an important consideration, given that differential thermal expansion is one of the fundamental problems of SOFCs. Due to the instability of ceria in low partial pressures of oxygen, yttria-doped barium zirconate was used as the electrolyte. However, current/voltage plots for palladium film anodes and silver film cathodes show poor results compared with those for conventional SOFCs. One reason for this was the 0.7 mm thickness of the electrolyte tile, which could be substantially reduced. A tendency to form carbon on the anode catalysts can be minimised by the use of 1% Ru/Al<sub>2</sub>O<sub>3</sub> oxidation catalyst adjacent to the

anodes. At 700°C with this catalyst 94% conversion of methane was obtained, of which 97% was converted to carbon monoxide. The system, although at an early stage of development, represents a simple engineering concept with considerable potential for improvement.

## Poster Exhibition

The poster section attracted 205 high-quality exhibits, a substantial proportion of which featured preparation or use of pgm catalysts for fuel processing, or fuel cell stacks. The geographical distribution of the authors provides an indication of the worldwide interest in fuel cells which is now apparent.

## Conclusions

Angelo Moreno (ENEA, Italy) summed up the conclusions from the conference in a final Plenary talk. For gas processing, there are still clear winners

among the several competing reactor types for producing hydrogen and the future appears very diverse. There is considerable interest in processing diesel and biodiesel for military and civilian applications.

The use of ink-jet printing for highly controlled application of catalysts to electrodes could present a means to reduce pgm metal loadings while retaining MEA performance. However, it is evident that there may be trade-offs to be made against performance if ultra low metal loadings are to be achieved. Low EU capital cost targets for fuel cells (€1000 to €1500 kW<sup>-1</sup> for stationary applications and €30 kW<sup>-1</sup> for automotive applications) are challenging, but ways are being found to meet these objectives.

The European Union Framework Programme is oriented mainly towards product research and development, while the fuel cell industry is moving towards demonstration programmes and commer-

## Poster Prize Awards

The posters were exhibited in six categories, and panels of judges shortlisted two from each category. The authors of shortlisted papers were then invited to defend their poster for the final selection panel. The prize winners were as follows:

### *Membranes*

'Inorganic/polymer composite membranes for DMFC', by G. Vaivars, R. Fakir, S. Ji, I. Sprinceana, Z. Wang and V. Linkov (University of the Western Cape, South African Institute for Advanced Material Sciences, South Africa)

### *Fuel Cell and Stack Technology*

'Effects of shut down process on the PEFC performance at sub-freezing condition', by N.-Y. Lim, G.-G. Park, J.-S. Park, Y.-G. Yoon, W.-Y. Lee, T.-W. Lim and C.-S. Kim (Korea Institute of Energy Research, Republic of Korea)

### *Component Modelling and Characterisation*

'Dynamic modeling and identification of a PEM fuel cell stack', by S. Philipps and C. Ziegler (Fraunhofer Institute for Solar Energy Systems, Germany); J. Niemeyer (Universitat Karlsruhe, Control System Laboratory, Germany); and J. O. Schumacher (Center for Computational Physics, Zurcher Hochschule, Winterthur, Switzerland)

### *Electrocatalysis*

'La<sub>0.6</sub>Sr<sub>0.4</sub>Co<sub>1-y</sub>Fe<sub>y</sub>O<sub>3-δ</sub> as a potential anode for IT-SOFCs: influence of the Co/Fe ratio on properties and reactivity', by F. Poletto, M. M. Natile, A. Galenda and A. Glisenti (University of Padova, Italy); and T. Montini, L. Derogatis and P. Fornasiero (University of Trieste, Italy)

### *System Analyses and Applications*

'An application for PEM fuel cell industrial plant', by G. Sibilis, A. Maggiore and D. Citelli (Nuvera Fuel Cells, Italy)

### *Fuels*

'An unconventional Au/TiO<sub>2</sub> PROX system for complete removal of CO from non-reformate hydrogen', by J. Steyn (AngloGold Ashanti Ltd, South Africa); G. Patrick and E. van der Lingen (Mintek, Advanced Materials Division, South Africa); and M. S. Scurrrell and D. Hildbrandt (University of the Witwatersrand, Johannesburg, South Africa)

cial development. The stack constitutes roughly one third of the purchase cost of a fuel cell system, while part of the overall efficiency, and a major proportion of reliability aspects are governed by the balance of plant, for example compressors, inverters, valves and sensors. These attract comparatively little attention in Europe, although lengthy trials in the U.S.A. and Japan have resulted in more effort being devoted to their efficiency and durability.

Small fuel cells, integrated into the electricity distribution grid at local or residential level, will need to be operated together for maximum availability and efficiency. This will need careful planning to protect the grid against individual fuel cells, and the cells against the grid. It will be necessary to operate the cells in unison to balance supply with demand so as to avoid shortages. Networked operation of linked residential fuel cells is already being demonstrated in Japan.

Many of the oral papers will be published in a special issue of the *Journal of Power Sources* in May 2007.

### References

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### The Reviewer



Don Cameron is an independent consultant on the technology of fuel cells and electrolysers. As well as scientific aspects, his interests include the standardisation and commercialisation of these systems. He is Secretary of the Grove Symposium Steering Committee.



*Fig. 1 The historic Italian city of Turin, also a major industrial centre famous for its motor manufacturing, provided the backdrop for Fuel Cells Science and Technology 2006*