Premium Act

New Energy World JTI
fuel cells & hydrogen for sustainability

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D 6.2 – Conclusions of the characterisation conference

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D 6.2 – Conclusions of the characterisation conference

An international Workshop on *"characterization and quantification of MEA degradation processes"* was organised on September 26-27, 2012 in Grenoble at CEA – Minatec. The purpose of this workshop was to show the contribution of different characterisation methods for better understanding and quantification of the degradation mechanisms of PEMFC components. The workshop booklet is presented in the next pages.

Around 50 people attended the workshop, people coming either from industrial or academic institutions (see the participant list at the end of the booklet). Five speakers, internationally recognized, have been invited to present their work:
- **Atsushi Ohma** from Nissan Research Center in Japan,
- **Karren More** from Oak Ridge National Laboratory in USA,
- **Hans Bettermann** from University of Düsseldorf in Germany,
- **Laetitia Dubau** from LEPMI-University of Grenoble in France
- **Arnaud Morin** from LITEN CEA-Grenoble in France.

Nineteen other presentations were given by the other participants of the workshop.

Presentations were related to physical, chemical and electrochemical methods leading to direct or non-direct information on the degradation of MEA structure and properties.

Many presentations focused on the degradation mechanisms of both the active layers and the membrane. They gave a good overview of the state of the art of research on these two subjects.

Many other presentations showed different techniques and strategies developed in order to localise, across the MEA surface area (in plane or through plane) or between the different MEA in a stack, the zone where the MEA degradation is the most severe.

All these presentations gave rise to fruitful discussions and probably will emerge on new collaborations between the participants.

The workshop ended with the visit of the Nano-characterization Platform at Minatec in CEA-Grenoble during which discussions continued. Our invited speaker, Karren More who is group leader of the Microscopy Group in the Oak Ridge National Laboratory in USA, was particularly interested in this visit.
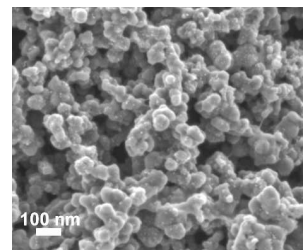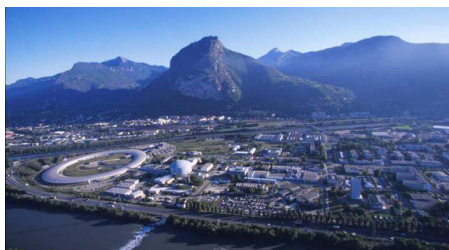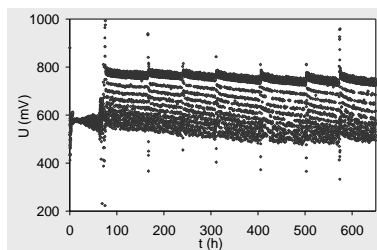
"Characterization and quantification of MEA degradation processes"

September 26-27, 2012 in Grenoble, France

Invited Speakers:

- **Atsushi Ohma** (Nissan Research Center, Japan):
  "Study on Microstructure of Catalyst Layer and its Impact on Performance and Degradation"

- **Karren More** (Oak Ridge National Laboratory, USA):
  "Application of Advanced Microscopy Methods to Understand MEA Materials Degradation"

- **Hans Bettermann** (University of Düsseldorf, Germany):
  "*On-Line Gas Detections from PEM Fuel Cell Flow Fields by Raman Measurements and Electric Arc Emission Spectroscopy"*

- **Laetitia Dubau** (LEPMI, Grenoble University, France):
  "*Durability of "Pt₃Co/C Cathode Electrocatalyst during Long-term Real PEMFC Operation"*

- **Arnaud Morin** (LITEN, CEA-Grenoble, France):
  "What about the link between chemical degradation of membrane and PEMFC durability? The example of S-PEEK Membrane"

Visit of the Nano-characterization Platform at Minatec:

Electron Microscopy   X-Ray Analysis   Magnetic Resonance   Ion Beam Analysis   Surface Analysis   Near field

"Characterization and quantif ication of MEA degradation processes"

September 26-27, 2012 in Grenoble, France

PROGRAMME (1ˢᵗ day)

September 26, 2012		
9:00	**Atsushi Ohma** Nissan Research Center, Japan	**Study on Microstructure of Catalyst Layer and its Impact on Performance and Degradation**
9:40	**Nicolas Caqué** Axane, Sassenage, France	**Multidisciplinary approach of MEA degradations in commercial PEMFC systems**
10:05	**Madeleine Odgaard** IRD Fuel Cells, Denmark	**Degradation Studies of PEMFC**
10:30	**Coffee break**	
10:50	**Karren More** Oak Ridge National Laboratory, USA	**Application of Advanced Microscopy Methods to Understand MEA Materials Degradation**
11:30	**Mathias Schulze** German Aerospace Center, Stuttgart, Germany	**X-ray Photoelectron Spectroscopic Investigations of Degradation Processes in low temperature Fuel Cells**
11:55	**Denis Bona** Electro Power Systems Spa, Torino, Italy	**Methods to Detect and Mitigate Catalyst Support Carbon Corrosion in PEMFC**
12:20	**Lunch**	
13:45	**Hans Bettermann** University of Düsseldorf, Germany	**On-Line Gas Detections from PEM Fuel Cell Flow Fields by Raman Measurements and Electric Arc Emission Spectroscopy**
14:25	**Erich Gülzow** German Aerospace Center, Stuttgart, Germany	**Current Density Measurements for *in situ* Studies of Aging Processes of PEMFC**
14:50	**Sönke Gössling** ZBT GmbH, Germany	**Long-term Test of PEMFC with a Dry Cathode Supply and Daily EIS Analyses**
15:15	**Samuele Galbiati** Politecnico di Milano, Italy	**Experimental Study of the Degradation of Polybenzimidazole-based Membrane Electrode Assemblies Doped with Phosphoric Acid**
15:40	**Coffee break + Poster session**	
16:30	**Jacob Lindner Bonde** IRD Fuel Cells, Denmark	**Degradations Studies of DMFC**
16:55	**Fausto Bresciani** Politecnico di Milano, Italy	**Experimental Methodology to Perform and Analyze DMFC Degradation Tests**
17:20	**Sébastien Rosini** LITEN, CEA-Grenoble, France	**Individual Cell Degradation during Fuel Cell Stack Operation**
17:45	**Sylvie Escribano** LITEN, CEA-Grenoble, France	***Ex-situ* electrochemical analyses of PEMFC electrodes after *in-situ* ageing tests**

PROGRAMME (2nd day)

September 27, 2012		
9:00	**Laetitia Dubau** LEPMI, Grenoble University, France	**Durability of Pt$_3$Co/C Cathode Electrocatalyst during Long-term Real PEMFC Operation**
9:40	**Pawel Gazdzicki** German Aerospace Center, Stuttgart, Germany	**FTIR and Raman Spectroscopic Investigations of Degradation Processes in PEMFC**
10:05	**Laure Guétaz** LITEN, CEA-Grenoble, France	**Influence of the Local Conditions on the Catalyst Degradation Mechanisms during Fuel Cell Operation**
10:30	**Coffee break**	
11:00	**Arnaud Morin** LITEN, CEA-Grenoble, France	**What about the link between chemical degradation of membrane and PEMFC durability?** **The example of s-PEEK membrane**
11:40	**Eddy Moukheiber** LEPMI, Savoie University, France	**Investigation of Ionomer Structure through its Dependence on Ion Exchange Capacity (IEC)**
12:05	**Gilles De Moor** LEPMI, Savoie University, France	**Detection of MEA's Flaws in PEMFC: *in situ* Relaxometry Combined with *ex situ* Infrared Imagery**
12:30	**Lunch**	
14:00	**Laurent Gonon** INAC, CEA-Grenoble, France	**Chemical Stabilization of Polymer Electrolytes used in PEMFC**
14:25	**Lorenz Gubler** Paul Scherrer Institut, Switzeland	**Insights into the Degradation Mechanism of Radiation Grafted Fuel Cell Membranes**
14:50	**Zhuoxiang Zhang** Paul Scherrer Institut, Switzeland	***In Situ* Characterization of Styrene-based Radiation Grafted Membranes with Nitrile Groups in Fuel Cell**
15:15	**Eddy Moukheiber** LEPMI, Savoie University, France	**The Essential Work of Fracture (EWF) of PFSA membranes: a way to understand MEA degradation**
15:40	**Coffee break**	
16:00	**Visit of the Nano-Characterization platform at Minatec**	

Invited Speakers

Study on Microstructure of Catalyst Layer and its Impact on Performance and Degradation

Atsushi OHMA

Nissan Research Center, Nissan Motor Co., Ltd.
1, Natsushima-cho, Yokosuka-shi, Kanagawa 237-8523, JAPAN

The biggest issue that must be addressed in promoting widespread use of fuel cell electric vehicles (FCEVs) is to reduce the cost of the fuel cell system. Especially, it is of vital importance to reduce platinum (Pt) loading of catalyst layers (CLs) in the membrane electrode assembly (MEA) of a proton exchange membrane fuel cell (PEMFC). In order to lower the Pt loading of the MEA without sacrificing its performance and durability, apparent kinetics of CL and mass transport in CL are to be enhanced as well as durability against Pt dissolution and carbon corrosion etc. However, it is a big challenge to characterize and quantify microstructure of CL.

In this presentation, examples of study on the microstructure of typical CLs at Nissan are introduced. Also the relationship of the microstructure to the performance and durability of the CLs is discussed.

Application of Advanced Microscopy Methods to Understand MEA Materials Degradation

K.L. More, D.A. Cullen, M. Chi, and K.S. Reeves
Oak Ridge National Laboratory, Oak Ridge, TN, USA

Polymer electrolyte membrane (PEM) fuel cell performance degradation can be directly attributed to the stability and durability of individual material constituents comprising the membrane electrode assemblies (MEAs), including the electrocatalyst, catalyst support, recast ionomer, polymer membrane, and gas diffusion-microporous layers (GDL/MPL), particularly associated with the cathode catalyst layer. The structural and chemical changes of these MEA constituents are being quantified via advanced electron microscopy methods to more fully understand the specific degradation mechanisms contributing to performance loss, with the ultimate goal being microstructural and compositional improvements to enhance durability. Research efforts at Oak Ridge National Laboratory have been focused on the high-resolution characterization of as-fabricated (fresh) fuel cell materials (individual constituents and/or materials incorporated in fresh MEAs), and these fuel cells subjected to accelerated stress tests (ASTs) designed to degrade specific MEA components, and field-aged MEAs. These studies are used to establish critical processing-microstructure-performance correlations and to elucidate the individual materials changes contributing to measured MEA degradation, performance loss, and failure. Analytical microscopy methods have been used to elucidate the degradation of various carbon support structures, quantifying Pt loss due to migration, catalyst coalescence, and ionomer films. Understanding and quantifying the structural and compositional changes of the materials comprising the MEA during electrochemical-aging will allow for the implementation of processing changes and critical materials development that are required for optimizing PEM fuel cell durability and performance.

On-Line Gas Detections from PEM Fuel Cell Flow Fields by Raman Measurements and Electric Arc Emission Spectroscopy

Hans Bettermann
Institute of Physical Chemistry, Heinrich-Heine-University of Düsseldorf, Germany

Mechanical degradations of membranes as cracks or pinholes result mainly from local wetting and de-wetting cycles as well as from changes of local temperatures.
This contribution shows how early stages of such defects can be monitored on-line by optical spectroscopy. For this a new electric arc emission spectrometer was developed.

This method bases on the electronic excitation of molecules by electron impact within an electric arc. The excited molecules undergo an irradiant deactivation that leads to a light emission which is specific for each molecular species. The device placed in a by-pass at the end of the anode flow field enables the record of gases over a large range of concentrations starting from measuring pure gases to concentrations in the ppm range on a millisecond time scale.
The gas detector was used to record the intrinsic gas cross over and permeabilities of MEA materials on the anode side. Such perforations let nitrogen pass over from the cathode to the anode. Gas cross over was studied under selected conditions of fuel cell operations as for instance changes in the humidification of the anode and the cathode, stoichiometric factors, and electric loads. Long-term measurements on small stacks were applied to monitor early stages of membrane degradations. Since the detector can unambiguously detect nitrogen and hydrogen simultaneously it is considered to control recirculation modes of fuel cell operations.
In addition, locally and temporally occurring gas distributions within flow field channels were investigated by a multiple-fiber Raman spectrometer. This device permits the simultaneous record of gas compositions, liquid water and water vapor at several ports along a meander and shows how those species do change locally and which mutual interdependencies among them occur during operations.
Concluding, the coupling of these two approaches seems to be promising to achieve a concise image of gas compositions inside flow fields and offers a component for a comprehensive analysis of fuel cell operations.

Durability of Pt$_3$Co/C cathode electrocatalyst during long-term real PEMFC operation

L. Dubau[1], J. Durst[1], F. Maillard[1], M. Chatenet[1], L. Guétaz[2], J. André[3], N. Caqué, E. Rossinot[3].

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During the last decade, proton-exchange membrane fuel cells (PEMFC) have witnessed technology breakthroughs, which for the first time rendered fuel-cell powered applications a practical possibility. Bimetallic Pt$_3$M/C electrocatalysts (with M = Cr, Fe, Co, Ni, Cu) are now classically used at the cathode because of their enhanced oxygen reduction reaction (ORR) activity. However, an increased activity is meaningless if it cannot be maintained over time. The stability of a Pt$_3$Co/C-based cathode during long-term real PEMFC aging test (*ca*. 3500 h) is the focus of this talk. Regarding theoretical studies [1-3], Pt and M atoms possess different affinity to oxygenated species (atomic oxygen, water), which may destabilize the structure of the alloyed surface over time. Indeed, recent density functional theory calculations showed that the high oxophilicity of Co atoms drives their surface segregation and further leaching. We confirmed the instability of Pt$_3$Co/C in real PEMFC conditions [4]. In particular, we evidenced that the cathode potential strongly influences the changes of the nanostructure of Pt$_3$Co electrocatalysts over time [5]. By combining electrochemical and microscopic techniques, we propose a dissolution mechanism of the Co atoms. Interestingly, depending of the operating conditions, we will show how the Kirkendall effect can induce the formation of pure "hollow" Pt nanoparticles (Figure 1). Furthermore, we will correlate the fine nanostructure of the particles and the ORR activity.
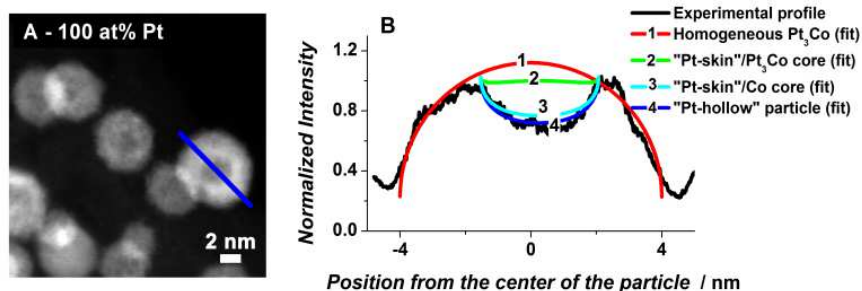

Figure 1. (A) Aberration-corrected atomic-resolution scanning transmission electron microscopy - high-angle annular dark field (HRSTEM-HAADF) image of "hollow" Pt nanoparticles formed in real PEMFC conditions, (B) experimental and theoretical normalized HAADF intensity profiles across an individual particle.

1. References

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[3] G.E. Ramirez-Caballero, Y. Ma, R. Callejas-Tovar, P.B. Balbuena, Phys. Chem. Chem. Phys., 12 (2010) 2209.
[4] L. Dubau, F. Maillard, M. Chatenet, L. Guetaz, J. André, E. Rossinot, J. Electrochem. Soc., 157 (2010) B1887.
[5] L. Dubau, F. Maillard, M. Chatenet, L. Guetaz, J. André, E. Rossinot, Electrochim. Acta, 56 (2011) 10658.

# What about the link between chemical degradation of membrane and PEMFC durability? *The example of S-PEEK Membrane*

Arnaud Morin[a], Pauline M. Legrand[b], Vincent H. Mareau[b], Laurent Gonon[b], Hakima Mendil-Jakani[b]

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Polyaromatic membranes, like sulfonated poly(Ether Ether Ketone) (sPEEK), used as polymer electrolyte for fuel cells offer a better thermomechanical stability than perfluorosulfonic membranes like Nafion (reference membrane) but a much lower chemical stability. Our goal was to understand the impact of fuel cell operating condition on the chemical stability of this alternative membrane and on the device performance and durability.

In a first step focused on the impact of operating conditions on the device performance, we observed when increasing gas stoichiometries, a much important decrease of the performances for sPEEK than for Nafion, as illustrated in Figure 1. Electrochemical Impedance Spectroscopy (EIS) was used to understand the origin of the performances drop observed for sPEEK[3]. This technique highlighted a very high spatial heterogeneity of hydration and therefore of performance. This has been ascribed to the water transport in the polyaromatic membrane which shows a slower water diffusion coefficient and a higher electroosmotic drag coefficient. Chemical degradation was not responsible for the fast drop of performances observed after few hundreds of hours of operation, as we demonstrated that performance could be recovered, at least in part, after rehydration of the membranes. However a non-reversible ageing was noticed through the slight decrease of the molecular weight observed for sPEEK 3/1.5, and the drop of the initial output voltage, not recovered after rehydration of the membrane.

In order to test the impact of chemical and structural modifications resulting from oxidative ageing (H_2O_2 can be produced during fuel cell operation) the second step of this study was focused on ex-situ ageing tests. These tests highlighted the correlation between a decrease of the sPEEK molecular weight observed during ageing and an improved swelling and conductivity of the modified membranes. In conclusion we have shown that a pre-oxidation of the membrane, using a H_2O_2 solution, improves sPEEK in-situ performances to such an extent that it could reach those of Nafion. This is also for us an evidence of the lack of influence of the bad quality of the interface between the membrane and the electrodes which do not stuck on each other, even after the chemical treatment.
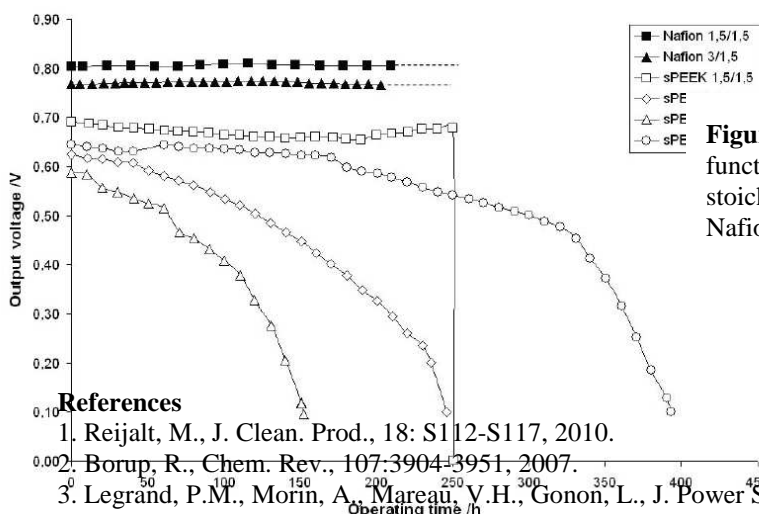
Figure 1: Evolution of output voltage as a function of operating time, for different gas stoichiometry configurations for both Nafion and sPEEK membranes

References
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Oral Presentations

Multidisciplinary approach of MEA degradations in commercial PEMFC systems

O. Lottin[1], N. Carême[1], J. Dillet[1], A. Lamibrac[1], G. Maranzana[1], J. Durst[2], L. Dubau[2],   F. Maillard[2], M. Chatenet[2], L. Flandin[3], C.Bas[3], G. De Moor[3], A.El-Kaddouri[3], E.Moukheiber[3], A. Montaut[4], S. Moutin[4], P. Rival[4], N. Caqué[5], E. Rossinot[5]

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MEA degradation appears as one of the most important problematic considering industrialization of PEMFC. Indeed, it is responsible for (i) performances losses leading to an increase of dihydrogen consumption, (ii) in most cases, the end of life of stack, and finally (iii) important cost of maintenance. As a consequence, the understanding of MEA degradation appears as a priority for the PEMFC industry in order to develop new durable materials and mitigation strategies to overcome their degradation.

In that frame, we have built a partnership which enables studying MEAs degradation within a global approach, the rationale for that being that (i) all MEAs components do degrade upon use and (ii) their degradations are often related to one-another, which (iii) therefore renders any survey of one component separately from the others insufficient. Furthermore, the degradation of most MEA components is highly linked to the operative conditions used in final systems, and as a consequence some degradations mechanisms are specific to each system. Therefore, a multidisciplinary approach is mandatory.

In this work, detailed post-mortem analyses of different commercial MEAs aged in test benches as well as in commercial systems have been carried out. Most ageing tests have been realised in the Axane's specific commercial hardware in order to observe the corresponding specific degradation mechanisms. Multi-scale techniques like membrane leak detection (using a thermal camera), physico-chemical characterisation of catalyst layers as well as local performances measurements were used and gave complementary results. Among the striking information is that degradations are not homogeneous according to the gas path: membranes appear more damaged near the air channels outlet; in the contrary, the cathodic active layer degrades more rapidly in the outlet part of H_2 channels. Furthermore different degradation mechanisms have been identified depending on system operative conditions (cathode potential, start/stop protocol and frequency). This work offers precious data to select MEAs and MEAs components, optimise the hardware geometry and adapt operative parameters to specific applications.

Degradation studies of PEMFC

Madeleine Odgaard and Jacob Lindner Bonde

IRD Fuel Cells

Durability is one of the most important issues for commercialization of PEM FC's based stationary and portable applications. Cost, reliability and functionality are also important commercial factors.

IRD participates in a Danish fuel cell based μCHP project that was initiated in 2006 and estimated to continue until the end of 2012. IRD has constructed three versions of a hydrogen fuelled μCHP based on PEM FC (Fig. 1). Units have already been in field test for one year (2008-9) in ordinary single family houses. 35 units are presently being installed for the final field test. Fuel cells integrated into systems is often subjected to various operating conditions that may differ significantly from those found in a laboratory environment and fuel cell degradation can here become a serious problem. The understanding of the failure mechanisms and performance degradation of the various MEA and stack components is therefore upmost important.

The presentation will include the status and latest results from the development and durability test of MEAs, cells and systems.

	2006/7: α-μCHP	2008/9: β-μCHP	2010/12: γ-μCHP
All units: Nominal power: 1.5 kW$_{AC}$			
Efficiency (LHV):			
Electrical (H$_2$→P$_{DC}$):	49%	52%	47%
Electrical (H$_2$→P$_{AC}$):	43%	47%	44%
Combined efficiency:	75%	94%	94%
Ready-mode Power:	105 W$_{AC}$	40 W$_{AC}$	15 W$_{AC}$

Fig. 1 The three IRD hydrogen fuelled μCHP units aimed for pure hydrogen operation.

X-ray photoelectron spectroscopic investigations of degradation processes in low temperature fuel cells

Mathias Schulze

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The surface properties (chemical composition and structure of the surface) are important for the catalytic behavior and transport processes (hydrophilic-hydrophobic characteristic of the surface as well of the inner surface). X-ray photoelectron spectroscopy (XPS) allows to analyze the chemical composition of the surface region with an information depth of few nm, which determines the surface properties. Due to the degradation processes the fuel cell behavior and performance decreases caused by alteration of the fuel cell components. Therefore, surface analytic tools, which give information about the chemical surface composition, are a helpful tool to identify and investigate the degradation processes. From XPS measurements not only the element concentrations but also the different binding states can be derived.

With XPS the alteration of the catalyst composition or the oxidation of the catalyst can be investigated electrochemical stressing during fuel cell operation can be determined. The alteration of the catalysts leads to a change of the catalytic properties.
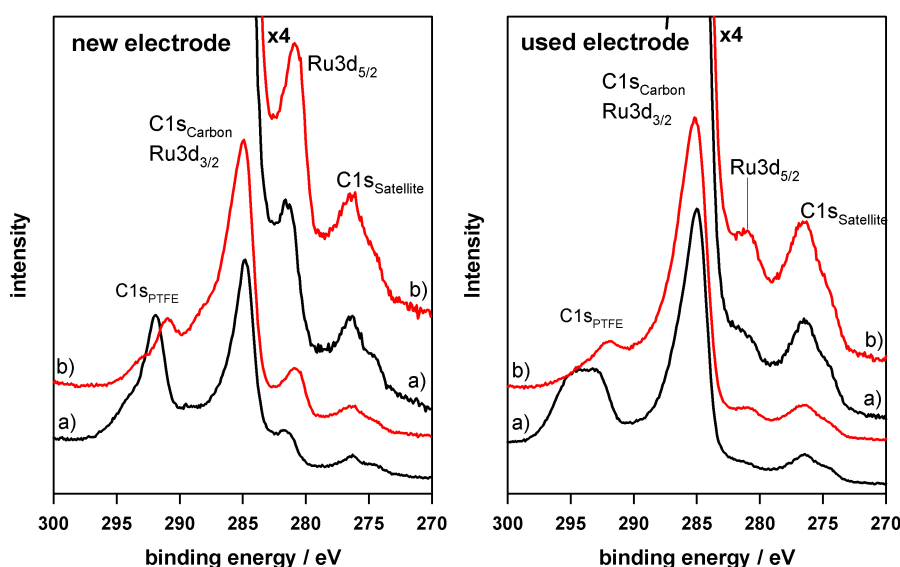
Fig. 1: XP spectra of a new and a used DMFC anode, a) before ion etching, b) after ion etching

The second example for XPS investigation for degradation processes in low temperature fuel cells is investigation of the decomposition of the polymers, which are used to create the hydrophobic properties of electrodes and gas diffusion layers. The decomposition of the PTFE and Nafion under electrochemical stressing can be observed in XPS measurements. The decomposition of the polymers leads to an alteration in the hydrophobic properties and consequently to a changed water balance in the cell.

Methods to detect and mitigate catalyst support carbon corrosion in PEMFC

Denis Bona, Dennis Curtin, Francesco Pedrazzo, Ilaria Rosso, Elena Maria Tresso

Electro power Systems Spa, Torino, Italy

Today, the state of art for PEMFC technology is the carbon black-based electrode layers, which are basically composed by agglomerations of Pt/C and ionomer, where the Pt facilitates the reaction, the carbon conducts electrons to the GDL, and the ionomer transfers protons to the membrane. The carbon blacks are characterized by high porosity, high surface area and good conductivity for electrons and ions [1]. So while the Pt/C catalysts ensure good **performance**, they lack **durability** and are subject to chemical attack under certain operating conditions. One of the most destructive concerns is **carbon corrosion**, which causes the catalyst support structure to degrade, leading to Pt dissolution and MEA failure [2], [3], [4].

Concerning the PEMFC durability, the catalyst degradation caused by carbon corrosion is likely a minor problem in stationary application or when the fuel cell performs with constant **operating conditions** during its lifetime. However, it is well-documented in the literature that if the fuel cell undergoes many startup – shutdown cycles, as in backup and automotive applications, this failure mode can become extremely relevant. Customer field data collected by Electro Power Systems confirms that the catalyst support carbon corrosion is by far the most relevant failure cause for backup applications.

This problem is extremely important in backup power applications because carbon corrosion leads to an irreversible and relatively rapid failure. Field-installed power systems which experience one or more cells with advanced catalyst degradation must be reported at the facility and the fuel cell stack scheduled for replacement. This leaves the site unpowered and unsafe from a power blackout for a certain time period.

The aim of this work is to offer a better theoretical understanding of the carbon corrosion mechanism, to define methods or techniques to monitor and quantify the degradation status, and to use the theoretical basis to define mitigation solutions best able to minimize the carbon corrosion.

This presentation will focus first on a review of the carbon corrosion mechanism with the aim to research the **fundamental cause** of this degradation mechanism. Carbon corrosion can occur under two different operating conditions. 1.When oxygen and hydrogen are mixed at the anode and no load is applied to the cell (during a startup or a shutdown), a **reverse current** is generated in certain parts of the cell leading to carbon corrosion of the cathode catalyst. 2.If the cell is generating a current and the anode suffers from fuel starvation (the hydrogen cannot reach the active area), both the anode and the cathode catalyst can be damaged by carbon corrosion.

The core discussion will review the most useful **detection methods,** together with a list and description of several demonstrated **mitigation strategies**. Some detection methods only can be applied practically in a laboratory setting, so the major interest is for methods that can be used directly *in situ*. Once the degradation mechanism is determined and explained, the most pressing concern is usually to establish a reliable mitigation strategy that will avoid or minimize carbon corrosion. Two different but probably complementary categories will be presented:
- Mitigation solutions adopting new more resistant materials
- System mitigation strategies reducing the carbon corrosion generation

Actual data from Electro Power Systems' experience with carbon corrosion will be presented and will support above mentioned discussion.

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Current density measurements for in-situ studies of aging processes of polymer electrolyte membrane fuel cells

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From the technical point the performance loss of fuel cells is the main problem of the degradation. From ex-situ measurement the various degradation processes can be identified and investigated, but the loss of performance cannot be determined from these measurements. For this purpose in-situ measurements are needed. At DLR a tool for current density measurements using printed circuit board (PCB) was developed [1]. The conducting area on the surface of the PCB is segmented (on the MEA side) and conducted via small resistance to the back side of the PCB. The small individual voltage drops over these resistance gives the information about the current density distribution, which can be measured and recorded on-line. The flow field will be machined directly in the PCB, so the PCB can be used as bipolar plate.
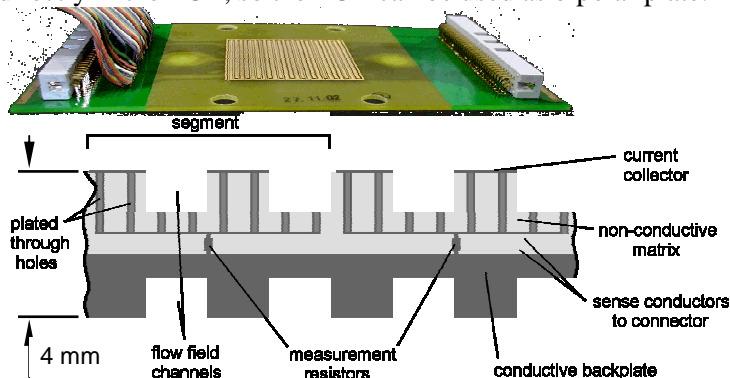
Fig. 1: Printed circuit board (PCB) for current density measurements (on top) and sheme of the PCB (bottom)

In the investigation of degradation the current density measurements allow to observe the local changes in the performance and to detect local differences in the degradation. Caused by the degradation the performance decreases and caused by inhomogeneity in the degradation also the shape of the current density distributions change.
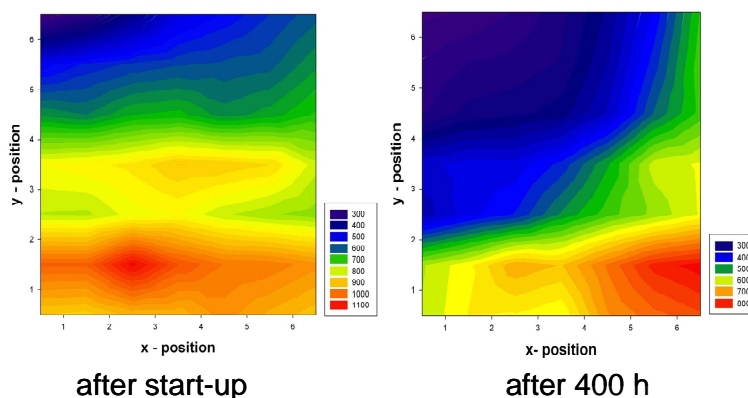

Fig. 2: Alteration of the current density distribution, left side current density distribution after start-up phase, right side after 400 operation at 600 mV

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Long-term test of PEM fuel cells with a dry cathode supply and daily EIS analyses

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In scientific publications long term performance of PEM fuel cell components is usually presented on the basis of highly sophisticated test environments. Nevertheless real life stability of the individual components is dependent on system architectures, media supply, operational strategies and further materials both of the fuel cell stack itself and all peripheral elements within the balance of plant.

The main focus of long-term tests at ZBT is the simulation of real operating conditions in portable and mobile applications. Therefore the tests are being performed without cathodic humidification and using anode recirculation of hydrogen. Furthermore for secured test results short stacks of 5 cells are used and always three short stacks are being tested in parallel (Figure 1). This allows verified results for long term tests e.g. comparing stack components (MEA, gaskets, bipolar plates) or operational topics like media supply independently from any outside influences.

This talk will give an overview on the design and iterative optimization of the implemented EIS (Electrochemical Impedance Spectroscopy) test architecture and its influence on the measured impedance spectra. The results of multiple test series, including continuous and various loads and start/stop cycles, are presented. A strong influence of start/stop cycles on the cell performance has been detected.

FIGURE 1 Long-term parallel operation of three short stacks.

Experimental study of the degradation of polybenzimidazole-based membrane electrode assemblies doped with phosphoric acid.

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The recent technology of polybenzimidazole-based high temperature polymer electrolyte fuel cells (PBI-based HT-PEFCs) doped with phosphoric acid provides a solution to some of the most critical features of the traditional Nafion-based low temperature PEM fuel cells. The higher operational temperature included in between 140°C and 200°C allows higher resistance to fuel impurities (CO up to 2%), easier and more effective heat recovery and simplified water management due to the absence of liquid phase. These features make this type of technology attractive for use in stationary heat and power cogenerative systems where the fuel is produced via the reforming of hydrocarbons.

While the achieved power density levels can be considered satisfactory, durability still represents a critical issue and contributes to its high cost and limited lifespan. To date, the operation of PBI-based HT-PEMFCs has been demonstrated for up to 18000h of steady state operation under reference conditions (T=160°C, i=0.2 A cm-2, pure hydrogen as fuel λH_2=1.2, air as oxidant λair=2) with a voltage loss of around 10%.

Nevertheless, it is widely recognized that some parameters play a major role on the lifespan of HT-PEMFCs, in particular fuel cell temperature, current density and reactants stoichiometry. Therefore further research is needed to clarify the effect of different operating conditions in order to individuate optimized operational strategies that may extend the life of the fuel cell. Furthermore clarifying how the single components are affected by the fuel cell operational mode is useful to understand the reasons of degradation and develop more stable materials.

This work reports the results of a long-term testing activity carried out on a PBI-based HT-PEMFC operated under steady state conditions changing temperature, current density and air stoichiometry. Aim of the reported experimental investigation is to evaluate the degradation rate of this type of fuel cell under test parameters of practical interest. Insight in the causes of degradation is obtained by means of dedicated measurement techniques such as Electrochemical Impedance Spectroscopy (EIS), Cyclic Voltammetry (CV), Linear Sweep Voltammetry (LSV) and polarization curves.

Degradation studies of DMFC

Madeleine Odgaard and Jacob Lindner Bonde

IRD Fuel Cells

The step from small-scale prototype manufacturing to real products for making fuel cells a commercial success is a major challenge. The direct methanol fuel cell system, directly converting methanol to power, water and CO_2 is one of the potential future substitutes for power generators. The potential applications range from stationary heat and power sources to mobile battery chargers or range extenders.

IRD is participating in several national and international projects ranging from MEA development to field test of DMFC systems.

Durability studies and impact of the various operating conditions on the catalyst stability and study of components integrity is mandatory to tailor the right approach leading to further performance enhancement. The presentation will include the status and latest results from the development and test of cells and systems and discuss durability and performance of the MEAs in commercial fuel cell applications. An example of a lifetime test of a DMFC MEA can be seen on figure 1.
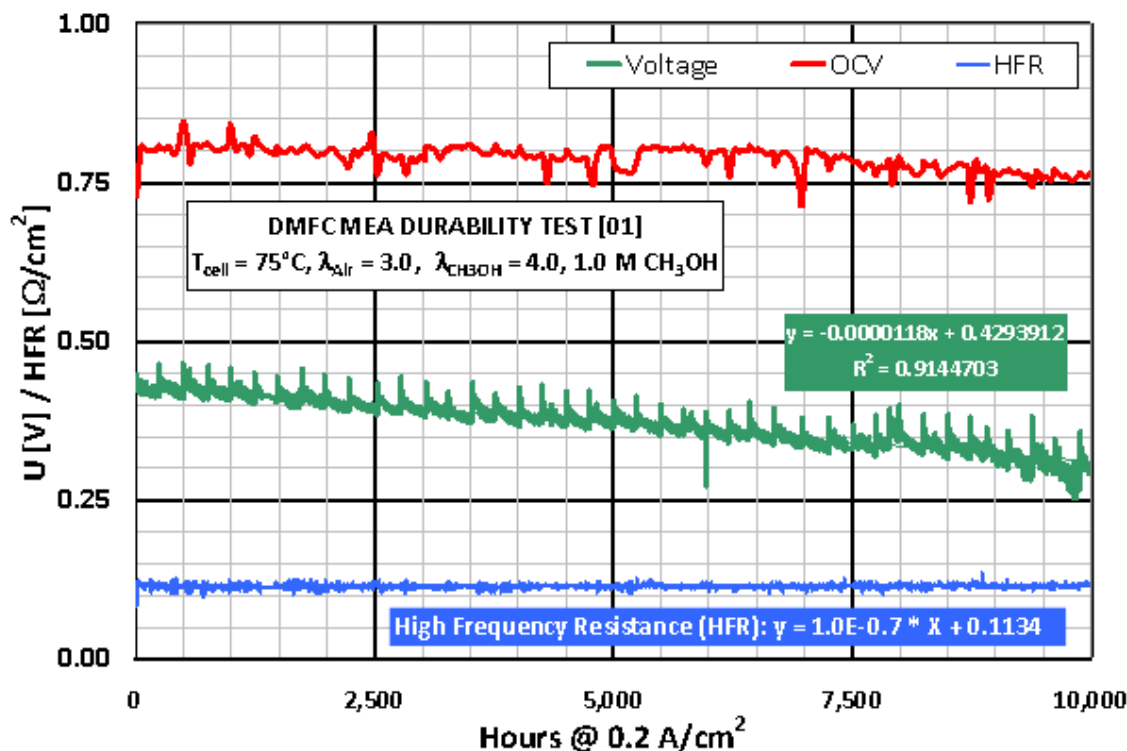
Figure 1: Results of an extended single-cell DMFC MEA produced by IRD.

Experimental methodology to perform and analyze DMFC degradation tests

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Direct methanol fuel cell (DMFC) is a promising energy source for portable electric

applications due to its low operation temperature, rapid start-up, and liquid fuel. At present, the degradation of DMFC is still one of the major concerns on the commercialization of this technology.There are several interconnected and complex phenomena that contribute to degradation. In literature there are few experimental investigations of direct methanol fuel cell degradation: they show that degradation has both temporary and permanent contributions but generally they do not quantify them nor estimate its uncertainty. No shared methods are available in literature, reducing the reliability of experimental results comparison. Moreover evaluating operating condition influence on degradation, the duration of the test is established a priori in an arbitrary manner, risking time waste or information loss.

The aim of this work is to propose a statistical method to characterize permanent and temporary degradation rates and that permits to define test duration considering the requirements of measurement accuracy and reliability. The proposed method can be applied to both steady state and cycling operation.

Moreover it is difficult to distinguish the contribution of both anode and cathode to the overall voltage loss because of the poor reliability of in-situ reference electrodes. In this work an experimental characterization of anodic degradation is also presented. Anodic overpotential is continuously measured supplying the cathode with hydrogen to serve as a reference electrode. Experimental tests show the presence of temporary degradation at the anode side, which can be recovered switching off the cell.

The above cited methodology allows quantifying degradation rates also in anodic operation and anode permanent contribution is comparable with the overall one

Keywords: DMFC, degradation.

Individual Cell Degradation during Fuel Cell Stack Operation

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In an international context of low-carbon energy development, Fuel cell systems are one of the most promising systems to convert chemical energy with high efficiency and low carbon emission. In spite of important improvements during the last decade, fuels cell system commercialization must pass through the enhancement of lifetime and reliability of stacks. Performances of PEM fuel cell stack are linked not only to MEA and bipolar plate manufacturing but to degradations induced by working point of the fuel cells stacks too.

This presentation deals with the degradation of performances occurring in fuel cell stack. In a first part, it focuses on the degradation induced by high active area utilization and as a consequence, by a discrepancy of gas concentration, of thermal and mechanical management all along stack surface area. In a second part, degradations of the elementary cells are studied and explained by a stacking effect and by fluidic, electric and thermal management along the stack.
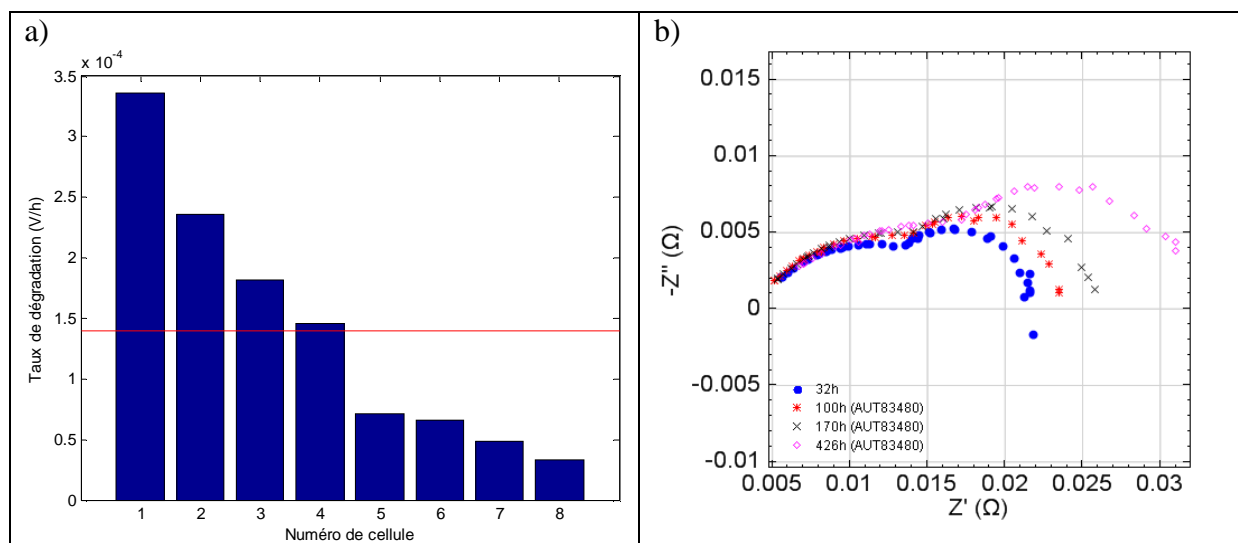
Figure 1: a) Degradation rate of individual cell voltage on a short stack. b) Evolution of stack impedance during operation on a short stack.

Ex-situ electrochemical analyses of PEMFC electrodes after *in-situ* ageing tests

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Increasing Proton Exchange Membrane Fuel Cells (PEMFC) durability as well as reducing their cost are two objectives to be reached for large scale development and systems commercialization. Work is conducted in different projects to check the durability and study the degradation of Membrane Electrode Assemblies following various load cycles profiles.

During fuel cell ageing tests, *in-situ* diagnostics are applied for both performance and components degradations analysis. It is clearly established that these diagnostics give averaged information on the electrodes and MEA status whereas the degradation is not homogeneous. In order to complete the *in-situ* results for a better understanding of the degradation, the local modifications of the microstructure are studied showing different mechanisms in different zones (*see L. Guétaz presentation & poster*).

In the worst degradation cases, particularly when the membrane is strongly damaged, it is not possible to get *in-situ* electrochemical data.

It has been decided to intend investigating *ex-situ* also the local electrochemical characteristics after ageing. The aim is to check the electrochemical status of samples extracted from the complete aged MEA, enabling to relate the microstructure modifications to their functional properties. A protocol has been applied to test particularly cathode samples in half-cell device. MEA samples are taken from aged MEAs and the anode is removed from the sample to obtain a half-cell (membrane plus cathode) for analyses. Typically, samples are selected in the air inlet or air outlet zones for MEAs tested in counter flow operation.

These aged samples are submitted to electrochemical diagnostics like cyclic voltammetry under nitrogen, oxygen reduction reaction test, carbon monoxide pollution.

Since samples are coming from aged MEAs and sometimes strongly degraded MEAs, the plots of the electrochemical diagnostics obtained have not, in several cases, the characteristic expected aspects but it has been possible to get additional information compared to *in-situ* data. Results have evidenced clear differences between new and aged electrodes; different modifications depending on the MEA zones (inlet/outlet), on the ageing test applied, on the MEA composition.

FTIR- and Raman spectroscopic investigations of degradation processes in polymer electrolyte membrane fuel cells

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Polymer electrolyte fuel cells (PEFC) are zero emission electrochemical energy conversion devices that allow the efficient use of hydrogen produced with renewable energy sources such as wind or solar power. Even though, PEFC are being investigated for decades (1), they are still not yet commercialized due to relatively high cost and unsufficient durabiliy. The life time of PEFC is thereby limited by numerous degradation processes (2), such as the loss of hydrophobicity of the gas diffusion layer (GDL) caused by dissociation of PTFE (Teflon®) polymers (3).

Vibrational spectroscopic methods represent an excellent approach to study chemical degradation processes in PEFC, and particularly to perform laterally resolved chemical maps and images of PTFE polymers in GDLs. In this context we present Fourier transform infrared spectroscopic (FTIR) as well as Raman measurements of the distribution of PTFE in membrane electrode assemblies (MEAs) and discuss the advantages and drawbacks of each method. Even though both methods are based on the excitation of molecule vibrations, they represent complementary techniques. For instance due to different selection rules Raman allows the direct access to PTFE as well as to carbon fibres in the GDL while with FTIR only PTFE can be detected. On the other hand, FTIR is less time consuming and non-destructive as compared to Raman. Generally, we use FTIR and Raman microscopy in order to perform large area overview chemical maps of entire MEA components ($25cm^2$) and small area ($1mm^2$) detailed investigations, respectively. Typical measurements depicted in Fig. 1 demonstrate the suitability of these techniques to study local degradation processes of MEA components as well as the spatial degradation along the flow field of a PEFC.
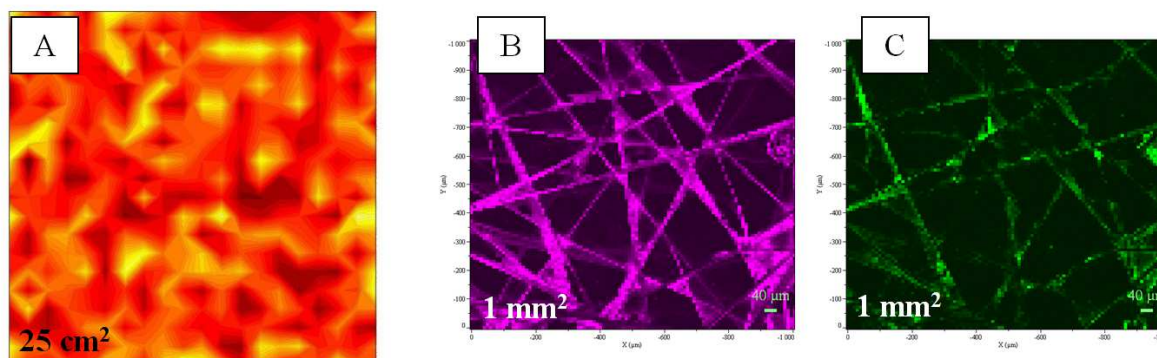

Figure 1: **(A):** Distribution of PTFE in GDL measured with FTIR-ATR (20 x 20 points). The colour scale corresponds to peak areas of C-F stretching modes (1030-1260 cm^{-1}); **(B):** Carbon (1577 cm^{-1}) Raman map (101 x 101 points) of a GDL; **(C)**: PTFE (730 cm^{-1}) Raman map of a GDL.

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Influence of Local Conditions on the Catalyst Degradation Mechanisms during Fuel Cell Operation

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In this study the microstructure evolution of membrane electrode assemblies (MEA) aged under different conditions - a constant load mode and a load cycling mode –, using a counter-flow single-cell, have been carefully analysed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The microstructure degradation has been studied in two particular selected regions; the first one is located near the air inlet and the second one near the air outlet [1].

Degradation of the MEA is not always uniform across the surface. Particularly, when the MEA is aged under load cycling operation, the degradation is more severe in the MEA region located near the air inlet. For the MEA aged under constant load mode, the degradation appears identical in the two regions (air inlet and outlet). In these different regions, two types of MEA microstructural evolutions have been observed (Figure 1). The first one, observed in the two analysed regions (air inlet and an outlet) of the MEA aged under constant load mode and in the air outlet region in the MEA aged under load cycling mode, consists of a well-dispersed monocrystalline spherical nanoparticles in the cathode and no particle formation in the membrane. This microstructural evolution is characteristic of the electrochemical Ostwald ripening mechanism and probably appears when the cathode potential remains at the level of the mean potential of the cell (0.6 to 0.9 V).

The second type of MEA microstructural evolution, observed for the MEA aged under load cycling operation in the zone located near the air inlet, consists in large particle agglomerates and carbon degradation within the cathode active layer, in addition to platinum precipitation inside the membrane. This microstructural evolution results from exposition of the cathode to a high interfacial potential. This high interfacial potential is explained by the reverse-current mechanism and is caused by the presence of oxygen at the anode. A significant oxygen crossover, that increases when the membrane is damaged, seems to be the main factor at the origin of this important catalyst degradation mechanism.

(a)

(b)

(c)
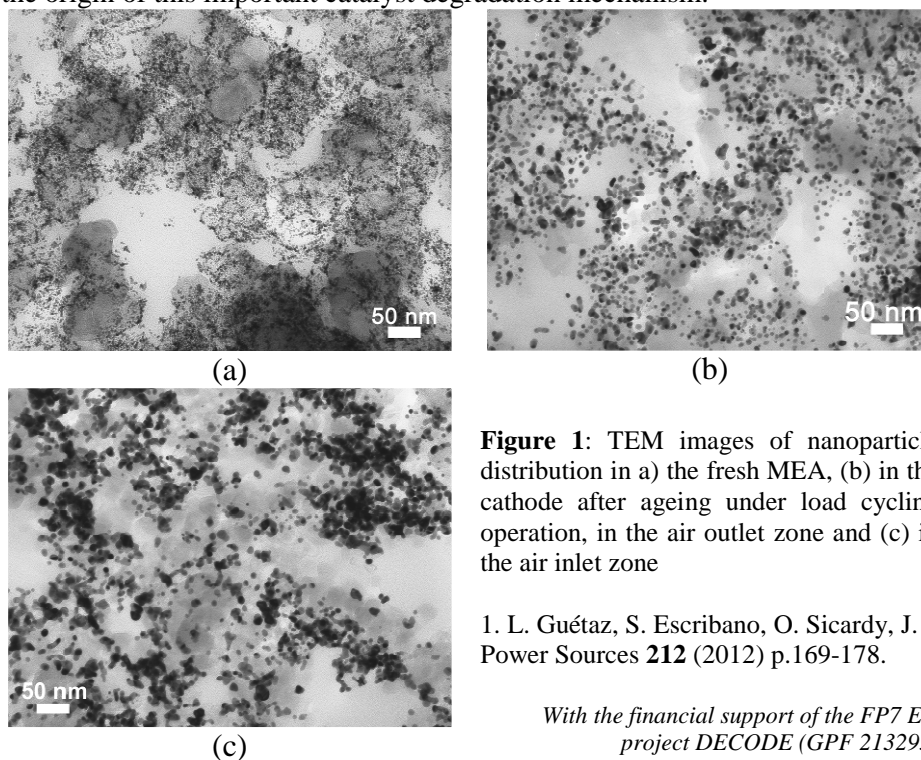
Figure 1: TEM images of nanoparticle distribution in a) the fresh MEA, (b) in the cathode after ageing under load cycling operation, in the air outlet zone and (c) in the air inlet zone

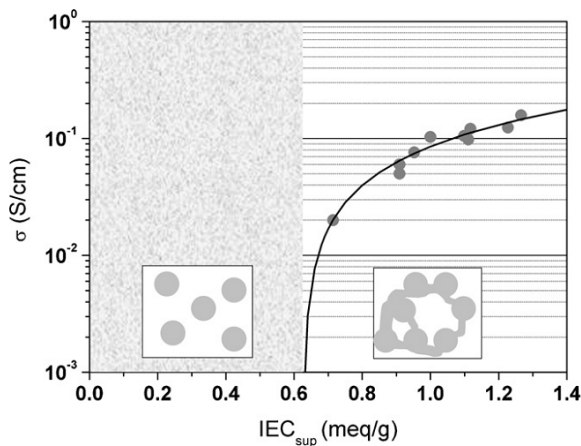1. L. Guétaz, S. Escribano, O. Sicardy, J. Power Sources **212** (2012) p.169-178.

Investigation of ionomer structure through its dependence on ion exchange capacity (IEC)[1]

Eddy Moukheiber, Gilles De Moor, Lionel Flandin, Corine Bas

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Perhaps the most successful systems to date, perfluorosulfonic acid (PFSA) based membranes have been used in portable, stationary, and automotive commercial applications of PEMFC technology. In addition to providing an attractive combination of performance and reliability, these polymers demonstrated high durability.

The ion exchange capacity (IEC) of conventional ion-exchange perfluorinated membranes based on the so-called Long-Side-Chain (LSC) polymers and their Short-Side-Chain (SSC) Aquivion® derivatives [2-4] was determined using a series of experimental techniques. Newly developed and already used experimental analyses were compared in terms of sensitivity to the determination of the ion exchange capacity. The use of complementary techniques allows better determination of IEC with an uncertainty of about 3%. This IEC determination method was then applied to membranes with large chemical differences such as copolymers and reinforced membranes. In addition, based on these analyses, relationship between DMA, WAXS, TGA and conductivity parameters and either IEC or the molar number of tetrafluoroethylene (TFE) per comonomer unit are underlined, leading to a more comprehensive approach of architecture in perfluorosulfonic acid (PFSA) polymer membranes.

Figure 1- Morphological aspect of proton conductivity in PFSA membranes
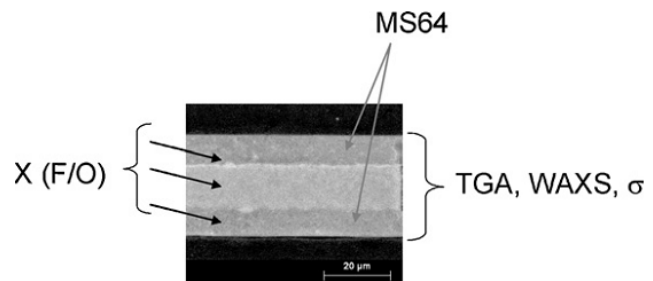
Figure 2- Different techniques used for measuring the IEC of external layers and the whole membrane Nafion XL100

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Detection of MEA's flaws in PEMFC: "in-situ" Relaxometry combined with "ex-situ" Infrared Imagery

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Understanding MEA degradation mechanisms is one of the main issues to improve their durability and thereby meet commercial viability. However, the complex relationships between laboratory approaches and industrial applications are poorly developed and very challenging to establish. Laboratory "in-situ" tests are indeed mainly performed on single cells with well controlled parameters and focused on specific factors. Although this kind of study is intrinsically limited and hardly allows forecasting what happens in real systems. In contrast feedbacks from real life applications generally focus on macroscopic approaches (Cell voltage, polarization curves, IES...), but fruitful correlation among the data collected is difficult. In conclusion there is a strong need in the fuel cell area for fast, sensitive and reproducible ways to investigate polyelectrolyte degradations. In the present work, fast and robust specific macroscopic tools were developed both to characterize and to localize defects, such as pinholes or short-circuits, within the MEA. The identification of defective cells without disassembling a real-life Axane's system was performed in a single step through a passive electrical technique so-called "relaxometry" [1-3]. This powerful passive technique allows a fast characterization of an entire stack (hundreds of MEA in few minutes). It is based on the measurement of electrical properties over time. The principle of the measurement is simple and based on three phases: *a forced charging*, a current or a voltage is applied to the MEA during a given time; *a self discharge*, the assembly is held at open-circuit and freely discharges; *a forced discharge*, to return MEA to an equilibrium state. The principle of the technique is described in the Figurea. The self discharge is the most discriminant step. Indeed, a fast relaxation of the potential is directly related to membrane defects such as electronic shortcuts. The flaws were further tracked inside MEA using Infrared Thermography in order to obtain their precise location. This last technique allows pointing out the exact position of flaws inside the MEA with a very high sensitivity as described in the Figureb. It was possible by calibration to detect pinholes with size close to 75 µm. Consequently, with the numerous data collected (several hundreds of MEA macroscopically analysed), it was shown that every pinhole in the stack could be detected by combining the techniques. The method was also of great help to perform more specific analysis, such as SEM analyses, around flaw areas in order to identify membrane degradation mechanisms after ageing. The set of diagnostic tools will be of great help to design the next Axane's Fuel Cell generation, with a better understanding of the membrane or system failure modes for instance by varying the operating conditions.
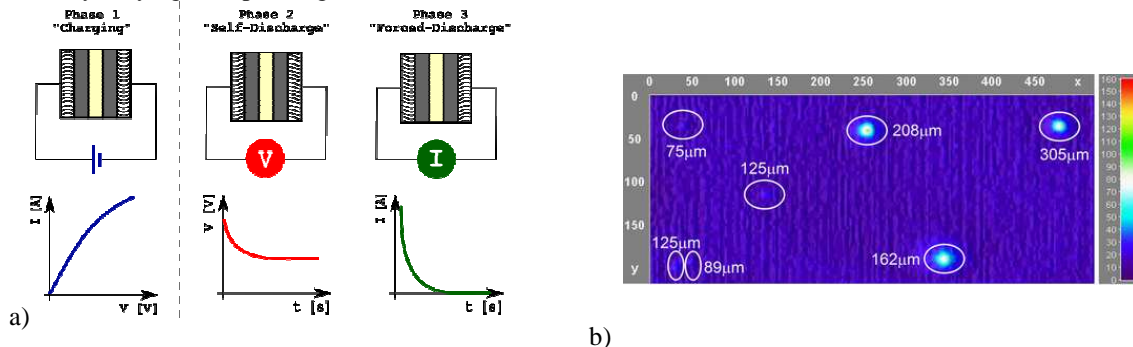
Figure: a) Schematic representation of the relaxometry devices applied to a synchronous measurement of a stack; b) Hole size calibration by IR thermography

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Chemical stabilization of polymer electrolytes used in PEMFC

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Proton exchange membranes (PEM) are key components in fuel cell (FC) systems as they allow proton transport between the two electrodes, without any gas mixing. However, the enhancement of the durability of the PEM lifetime is critical to viability for the FC. The aging of the membrane is due to a chemical / electrochemical and a mechanical degradation. Hydrogen peroxide and its products of decomposition (HO• and HO_2•) generated during the fuel cell operation are considered as one of the important vectors of the membrane degradation[1, 2].

The first works devoted to a strategy of chemical stabilization of membranes appeared only very recently (2003). These works are based on a radical scavengers approach[3, 4]. Following a different strategy, our work intends to develop sacrificial stabilizers able to decompose hydrogen peroxide formed in fuel cell operation. Two stabilizers based on thiourea (THP) and tetrasulfide (TS) groups were synthetized. In order to avoid leaching of the stabilizer induced by membrane swelling in the fuel cell, the stabilizer were grafted onto SiO_2 nanoparticles (figure 1). Nanoparticles dispersion in the membrane is expected to have a strong impact on both conductivity and mechanical properties of the membrane, and was thus thoroughly studied. AFM analyses performed on the cross section of cryo fractured membranes have shown an excellent dispersion of functionalized THP loads whereas the formation of aggregates was observed with functionalized TS. (figure 2a). The ability of stabilizers to limit the membrane degradation was evaluated with a sPAEK membrane, for different aging times in an H_2O_2 environment. The most efficient stabilizer appears to be the one based on THP groups. However, this stabilizer cannot be used because strong acid base interaction with the sulfonic groups of the polymer reduced the proton conductivity of the membrane and as a consequence the performances of the FC. On the other hand, the oxidation of the TS stabilizer leads to the formation of sulfonic groups and consequently to an increase of the ionic conductivity of the membrane (figure 2b).
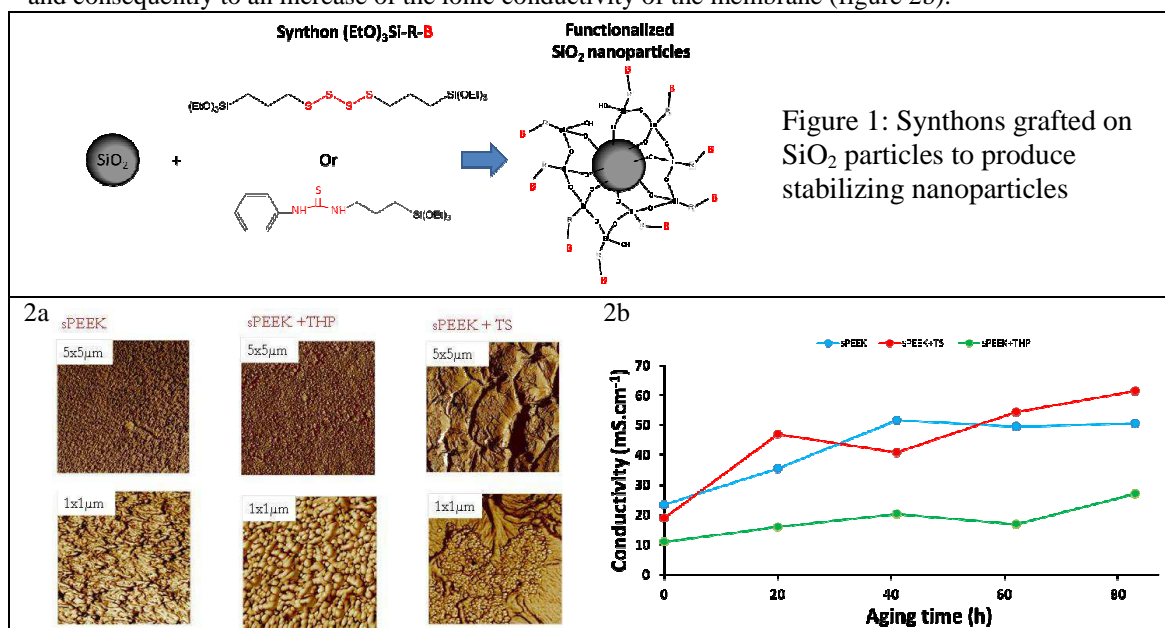

Figure 1: Synthons grafted on SiO_2 particles to produce stabilizing nanoparticles

Figure2: a-AFM image of cryo-fractured membranes (tapping mode/phase contrast) and b-Ionic conductivity of the composite membranes versus aging time in H_2O_2 vapor at 70°C

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Insights into the Degradation Mechanism of Radiation Grafted Fuel Cell Membranes

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Several thousand hours of durability have been attained with radiation grafted membranes in the fuel cell at a temperature of 80°C under constant as well as dynamic load conditions [1-3]. The insights gained so far into the aging phenomena suggest that the chemical degradation mechanism in radiation grafted membranes is fundamentally different from those found in perfluoroalkylulfonic acid (PFSA) membranes. Whereas catastrophic failure is often encountered in PFSA membranes as a result of localized degradation and pinhole formation, properly designed radiation grafted membranes rarely develop pinholes and mechanical failures, even in heavily degraded samples. Therefore, the likelihood for catastrophic failure is much lower.
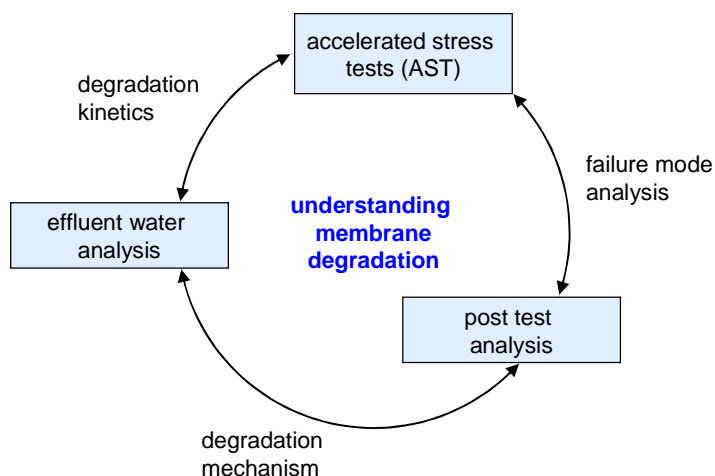
Figure 1: Schematic of the strategy adopted to study the chemical degradation mechanisms in radiation grafted membranes.

In this contribution, we will discuss and evaluate the various techniques that are being explored to probe the aging phenomena in radiation grafted membranes. In contrast to PFSA membranes, the analysis of the fluoride content in the product water is not meaningful and other analytical methods have to be used, such as ion chromatography. Our strategy to further our understanding of the prevalent degradation phenomena encompasses a combination of accelerated stress tests in the single cell, combined with analysis of the effluent water and post-test analysis of the membrane, for instance using FTIR spectroscopy (Figure 1). Moreover, a current status report of the development regarding performance and durability will be given.

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In Situ Characterization of Styrene-based Radiation Grafted Membranes with Nitrile Groups in the Fuel Cell

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To obtain tailor-made and cost-effective membranes for polymer electrolyte fuel cells (PEFCs), we are looking at utilizing the radiation grafting method which is based on versatile processes and low-cost raw materials. In the preparation of styrene-based radiation grafted membranes, functional monomers, styrene for instance, are grafted into pre-irradiated polymer base films followed by sulfonation, by which we introduce proton exchange sites and thus conductivity into the membrane. In the development of PSI membranes, methacrylonitrile (MAN) was used as co-monomer to promote the grafting of α-methylstyrene (AMS), and the obtained membranes displayed a conductivity of around 100 mS/cm and significantly higher durability in the fuel cell compared to pure styrene grafted membranes[1]. Our further work revealed that styrene/MAN co-grafted membranes also exhibited much longer lifetime than pure styrene grafted membranes under a normal operating condition[2]. The effect of nitrile groups on membrane performance and durability is being evaluated in our current work through testing styrene/MAN and styrene/ acrylonitrile (AN) co-grafted membranes (Figure 1) in the single cell, the results of which will be presented.
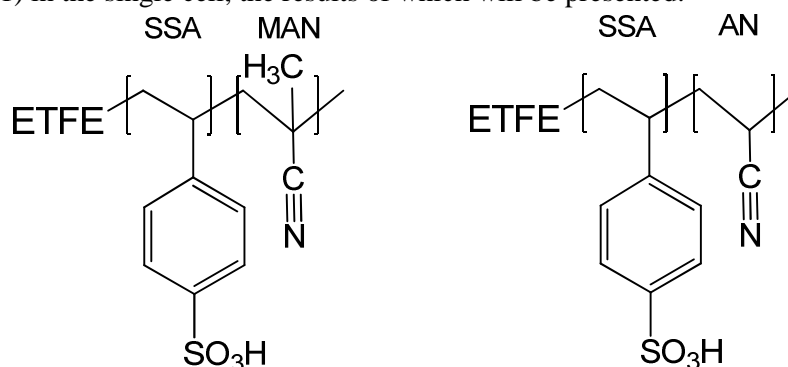
Figure 1: Schematic of the architectures of the membranes used in the study. SSA: styrene sulfonic acid

Open circuit voltage (OCV) hold tests have been widely employed as an accelerated stress test (AST) to promote membrane degradation in PEFCs due to the enhanced gas crossover[3]. The purpose of using accelerated degradation conditions is to achieve a high testing throughput. In our study, the nitrile group containing membranes have been tested under the OCV accelerated condition with online measurement of the high frequency resistance (HFR), and pre-test / post-test determination of the hydrogen permeation to investigate the change in gas barrier property. In this contribution, the effects of the presence of nitrile groups towards mitigating degradation of our membranes found in the OCV hold tests will be reported, and the possible stabilization mechanism will be discussed. Furthermore, the *post mortem* analysis based on Fourier transform infrared spectroscopy (FTIR) is conducted after the OCV hold test in order to investigate the composition change and characterize the degradation of membranes.

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The Essential Work of Fracture (EWF) of PFSA membranes: a way to understand MEA degradation

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In this research, the Essential Work of Fracture of Small Side Chain (SSC), Long Side Chain (LSC) and PTFE reinforced PFSA type films were studied with Cotterell tests [1, 2]. The effect of reinforcement, chain orientation, water and temperature were evaluated. The PTFE reinforced membrane showed a better resistance to crack initiation characterized by a high essential work of fracture w_e and a better resistance to crack propagation characterized by a higher slope βwp. The effect of chain orientation showed that TD-notch specimens displayed the highest resistance to fracture which suggest that the control of crack initiation or propagation could be optimized by pointing the direction of the gas channel perpendicularly to the orientation of the polymer chains, i.e. to rolling process during manufacturing. Moreover, the resistance to crack initiation was found to be dependent on water content of the membrane regardless of the length of side chain while the resistance to crack propagation was found constant for both types of membranes. Finally, the combine effect of water and temperature was found to further reduce the resistance to crack initiation however it strengthens the resistance to crack propagation.

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POSTERS

Influence of support morphology on MEA performance using polyaniline as alternative support material

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In the last decades, there has been a lot of effort towards the development of new catalysts and catalyst preparation techniques, but there has been considerably less interest in the support itself and its morphology in particular [1]. It is well known that the electrodes structure is a key issue for the fuel cell performance, as it is responsible for electron, proton, reactant and product transport. Especially the water content from humidification and product water, the so-called water management, has a huge effect on fuel cell performance and components degradation [2].

One step towards the controlled processing of 3D structured electrodes is the use of polymer fibers and adapted electrode fabrication techniques, as for instance a modified layer-by-layer technique (LbL). The fibers can be synthesized in different lengths and with different diameters. By tuning their morphology over a wide range its influence on the fuel cell performance can be determined.

To this aim, the supports morphology needs to be tuned without changing its chemical characteristics. This can be done either by using the electrical and proton conducting polyaniline (PANI) itself or in its carbonized form. Chemical similar PANI with different morphologies was obtained via a wet chemical approach (figure 1). This PANI has been decorated with Pt nanoparticles using HCOOH reduction at room temperature [3]. The mild reaction conditions ensure that there is no decomposition of the polymer and no change in the morphology.
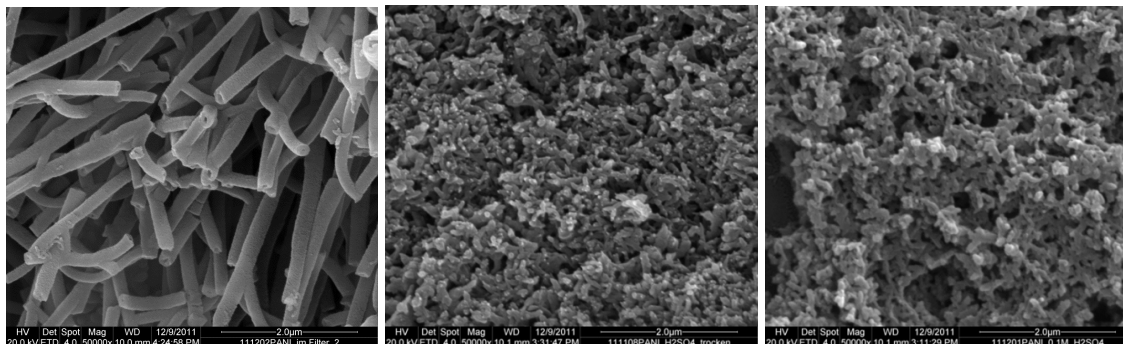
Figure: Differently-shaped PANI. Left: long wires produced in 0.4 M HAc; middle: short wires produced in 1 M H_2SO_4; right: granular material produced in 0.1 M H_2SO_4. Reaction conditions: 0.450 ml aniline dissolved in 50 ml acid rapidly mixed with 1.423 g ammonium peroxydisulphate dissolved in 50 ml acid then vibration-free stored for 24 h.

Membrane-electrode assemblies (MEA) of the different PANI materials have been prepared and will be tested in a home-made testbench as cathode with a standard carbon black supported catalyst as anode. The focus of these investigations will be on their performance at first, but also on their long term stability and durability. For the latter, the effect of the material and the resulting electrode structure will be analyzed.

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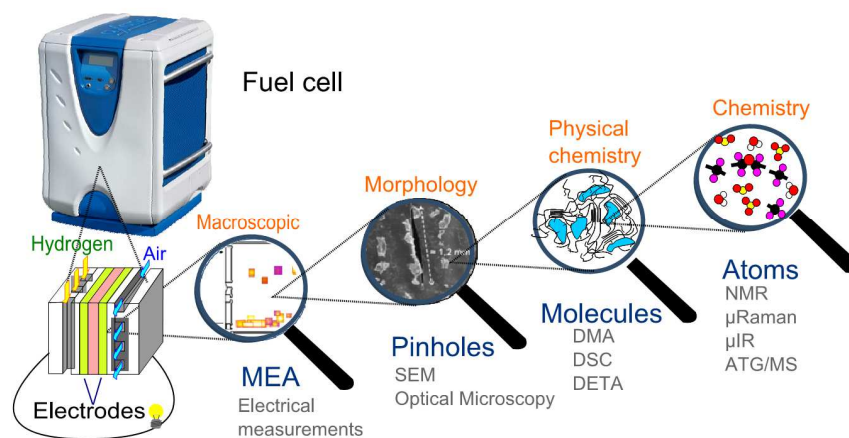
Understanding MEA failure in PEMFC using multi-scale characterizing tools

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Proton Exchange Membrane fuel cells (PEMFC) are expected to be an interesting alternative as power supply in portable, automotive and stationary applications because of their high power density and low environmental impact. Still some barriers have to be overcome before PEMFC become actually a viable market option. In order to access the viability of fuel cell systems, one still needs to increase their performances and durability.

The LMOPS team has worked in this area for almost 10 years exclusively with Axane (Air liquid group) and has developed innovative tools to characterize and understand fuel cell performances over time. As depicted in the figure below, we are currently involved in probing the performances of the systems at many different scales from the entire stack down to the chemical degradation that may occur in service. On the macroscopic scale [1], a unique set of measurements tools has been developed such as relaxation and thermal camera techniques which allowed identifying degraded cells mainly by perforation. Other experimental methods at meso/microscopic scale such as infrared spectroscopy, dynamic mechanical analysis or TGA, reliable in the literature for the characterization of different types of composites, were applied here for the study of MEAs. Some of these techniques are specific to the study of the membrane [2] while others concern the study of the ionomer of the active layer or membrane-electrode interface [3, 4].

Figure: Hierarchical analysis of the performance and durability of fuel cell developed at LMOPS.

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FTIR spectroscopic imaging of aged gas diffusion layers (GDL)

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Regarding fuel cells as an environmental friendly energy converter one of the major unsolved problems beside the costs is an insufficient lifetime. In polymer electrolyte fuel cells humidity management is one of the key points for their performance. The water management is mainly determined by the hydrophobic/hydrophilic properties of the gas diffusion layer (GDL) including the micro porous layer. Thus the degradation of GDL and micro porous layers (MPL) therein are the key point for the understanding of reduced fuel cell lifetimes.

In the past the partially decomposition of the polymers in the GDL and the MPL was observed by X-ray photoelectron spectroscopy measurements on fresh and aged GDL and MPL. The decomposition of the polymers due to fuel cell operation changes the hydrophobic/hydrophilic properties of the GDL, MPL and catalyst layer and consequently also the water balance in the cell.

Disadvantage of these XPS investigations are that the measurements were performed under ultrahigh vacuum conditions and the X-ray radiation induces also a decomposition of the polymers, therefore the FTIR spectroscopy as a non-destructive investigation method for the polymers and their distribution is very interesting. The imaging of the gas diffusion components shows a significant inhomogeneity for both, fresh and aged, GDLs.
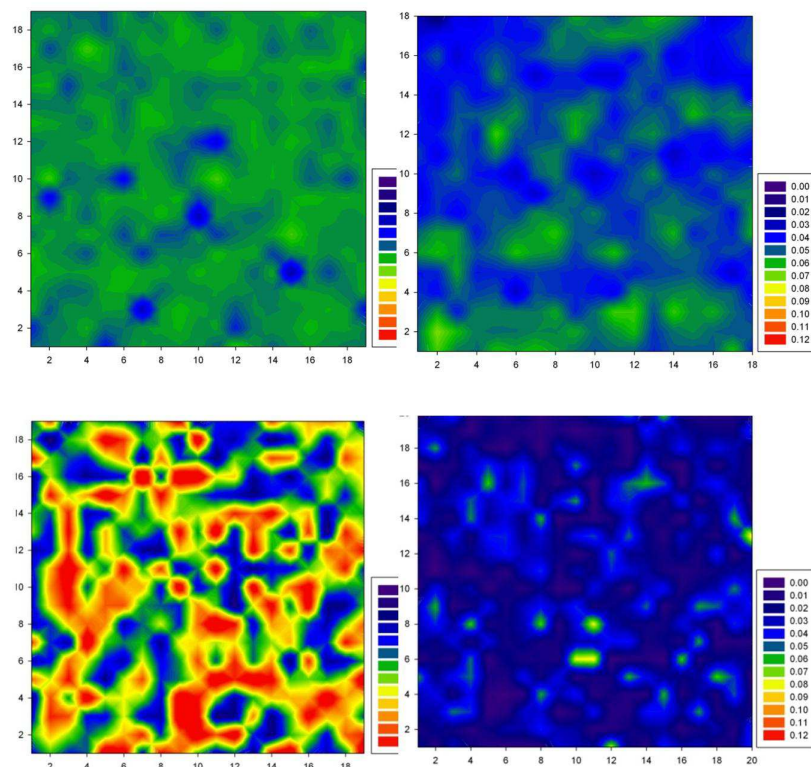
Fig. 1: Intensity of the C-F vibration measured of MPL (on top) and the paper side (bottom) of a commercial GDL. The fresh component is on the left side, the aged on the right side. The investigated area is 50x50 mm2, measured 20x20 points.

Chemical imaging of proton exchange fuel cell components using vibrational spectroscopy

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The performance and durability of proton exchange fuel cells (PEFC) is strongly affected by numerous degradation processes that occur under fuel cell operation conditions. It is therefore desirable to obtain detailed information of the corresponding ageing mechanisms in order to avoid them by improving the design of the individual PEFC components. Beside standard, yet complex *ex-situ* experiments such as XPS, chemical imaging using vibrational spectroscopic methods is a powerful approach for physical characterization of PEFC components and degradation.

This poster demonstrates the applicability of Fourier transform infrared microscopy (FTIR) and Raman microscopy to investigate membrane-electrode-assemblies (MEA) and their components, e.g. the gas diffusion layer (GDL) and micro porous layer (MPL). For the study of the catalyst layer other methods that provide direct information on elemental material composition are more suitable. The FTIR measurements were performed on single points as well as by scanning the sample and also by imaging the surface. Raman measurements were also performed on single points as well as by scanning the sample surface. The different imaging techniques allow to characterize the sample in different local resolutions.

The data presented in this poster comprise fresh MEAs only that can be used as reference samples to study specific degradation mechanism.
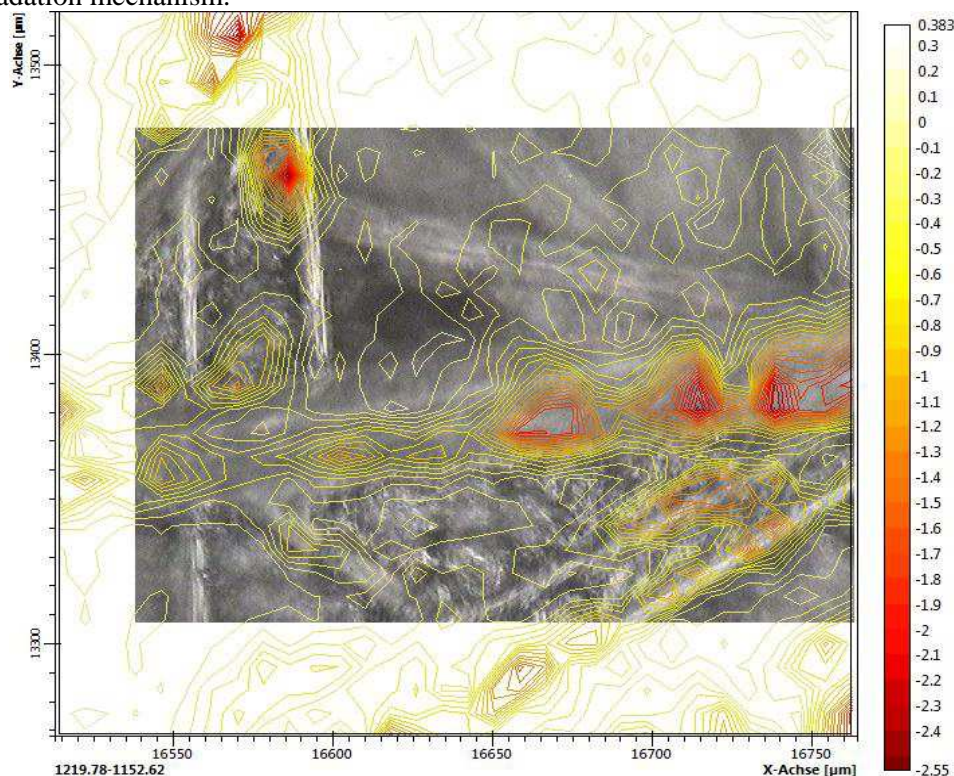
Fig. 1:   chemical image of PTFE in a GDL overlaid with a conventional VIS microscopy image. The measured area of 260 x 260 mm2 corresponds to 64 x 64 pixels

FTIR imaging of aged GDLs

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The performance loss induced by the degradation of fuel cells is one of main problems beside the costs for introduction of fuel cells for mass market applications. The water management is a crucial parameter for the performance of fuel cells. The hydrophobic/hydrophilic character of the fuel cell components especially it of the gas diffusion layer (GDL) has a strong effect on the water management. Therefore, the alteration of GDL due to the aging is an important degradation process.

FTIR spectroscopy allows to investigate the surface composition of the fuel cell components. Using the peak area of the C-F vibration signal the relative concentration of the hydrophobic agents (PTFE) on the surface of the GDL and MPL (micro porous layer) can be determined.

With exception of the sample, which was operated for 1200 h, a decrease in the PTFE concentration can be observed.
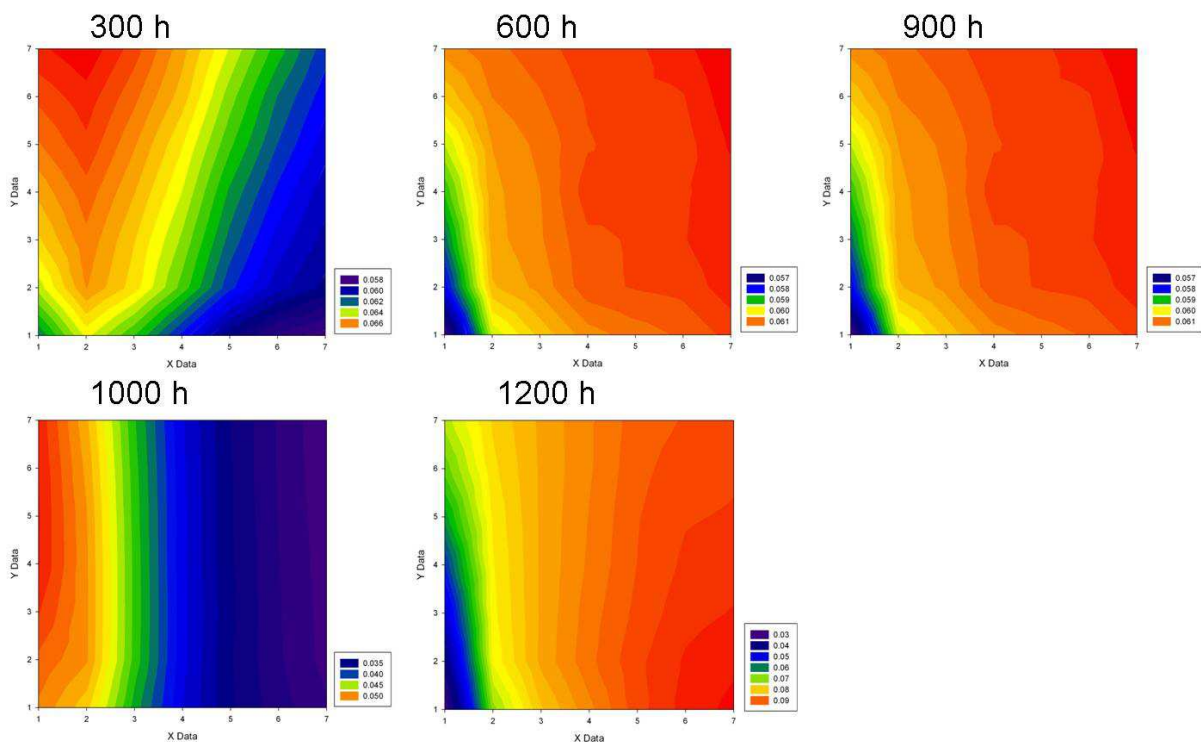
Fig. 1: FTIR-ATR image with an area of 150µm x 150 µm (7x7 points) of aged GDL (300 h, 600 h, 900 h, 1000 h and 1200h).

Pt-Co nanocatalyst degradation after fuel cell operation studied by EELS elemental mapping

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Pt-Co alloy nanoparticles are one of the most promising fuel cell catalysts for cathode, due to their higher catalytic activity than Pt nanoparticles. However as cobalt is not stable in the fuel cell environment, their activity decreases over the time. Therefore in order to get a better understanding of the degradation mechanism of the Pt-Co nanoparticles chemical changes in the near surface regions of nanoparticles should be monitored. In this context, an analytical technique with a high spatial resolution is needed. The electron energy loss spectroscopy (EELS) combined with the most advanced aberration corrected scanning transmission electron microscopy (AC-STEM) is a powerful technique for this application.

In this work we have combined EELS done using GIF Quantum and AC-STEM (a FEI TITAN Ultimate operating at 200kV) to investigate the degradation of Pt_3Co nanoparticles after fuel cell operation under cycling mode. On EELS spectra we can distinguish the characteristic signals for the Pt N_3, O-K and Co-$L_{2,3}$ edges at 518 eV, 532 eV and 780 eV respectively. The matrix of spectra was denoised using the standard principal component analysis and chemical maps are constructed using a power-law background subtraction. These maps (Figure 1) highlight clearly a Pt enriched shell surrounding the PtCo core. Two mechanisms are suggested for the formation of this layer: i) the dissolution of the Co from the surface or ii) the Ostwald ripening mechanism that leads to the re-deposition of Pt^{Z+} ions on the largest particles.
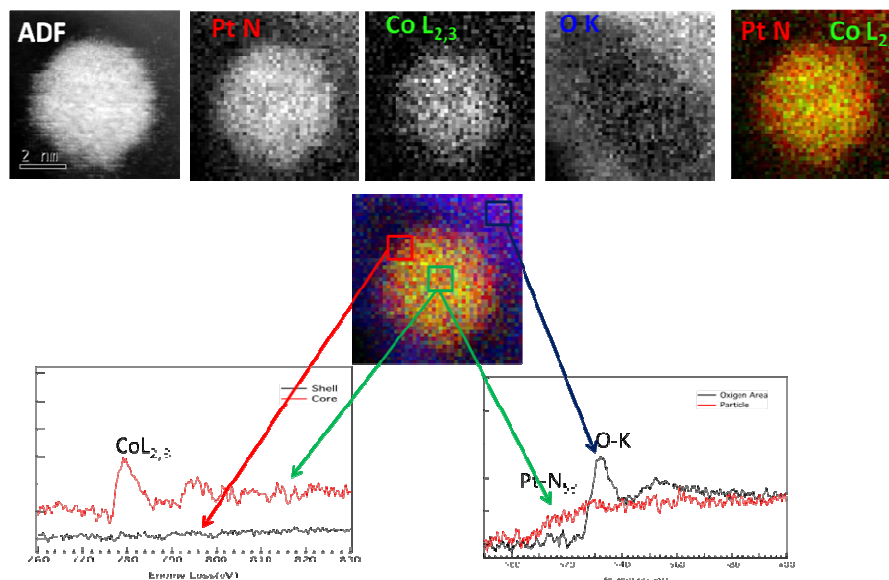
Figure 1: ADF/STEM image of a Pt-Co nanoparticles after fuel cell operation and EELS elemental maps of Pt N_3, Co-$L_{2,3}$ and , O-K.. The color map composition highlights clearly a Pt enriched shell surrounding the PtCo core. EELS spectra acquired using the spectrum imaging mode and extracted from different locations of the aged PtCo nanoparticle supported on carbon black show the difference local composition.

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Thank you for your participation!