

Axane commitment for a sustainable, reliable, and field proven power source through partnership with laboratories and design efforts

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IHTEC 2018 Characterization and quantification of MEA degradation processes

Summary

- 1- Axane activities and partnership presentation
 => Complementary expertises
 => Strategy of choice for studying MEA's degradation
- 2- First degradations phenomena experienced with former configuration
 => Local pinholing of membrane identification/localization
 => Local performance and degradation measurements
- **3- New configuration and new challenges**
- **4- Conclusions**

Axane activities and partnership's targets

Highest Durability to date



NRJ Container # 21 reached 14.100 operating hours without failure System was stopped at the end of the contractual operation period



- Commercial scaled global offer (FC systems, H₂ logistic, services and maintenance)
- From 0.1 to xx kW
- Off-grid, bad-grid (telecom antenna), events (sport, meetings), forklifts, specific applications without ambient air, automotive
- Several hundred systems deployed since 2001, mainly in France and Europe
- System's availability (2012) > 99,4%
- >14 000 h of operation without maintenance
- No MEA failure with standard configuration (~100000h of cumulated operation on whole park)

Axane ⇔ Laboratories

Study MEAs degradation mechanisms and develop mitigation strategies for Axane

<u>system</u>

- Analyse of system working data
- Develop in-situ/ex-situ diagnosis and characterisation tools
- > Optimisation of MEA components and operating conditions
- Promoting study of real-operated MEAs (aged in system) => specific degradation

<u>phenomena</u>

20/04/2018



Axane activities and partnership's targets

AIR LIQUIDE

resh Pt Co/C

0.6

E vs RHE / V

Pt-Co/C (t = 522 h)

Co/C (t = 1007 h)

0.8

1.0

1.2



- Stack and system design
- Manufacture stack and system
- > Optimize operating conditions

Electrocatalist characterizations

Electrochemistry (model electrodes)

Microscopy (SEM, TEM, EDS)



- Ce Demla
- > Fluids transport studies
- Local MEA behaviour
- Internal currents



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- Membrane characterizations
- > Chemistry (µIR, µRaman, ATG/MS)



- Physical chemistry (DMA, DETA)
- Macro. techniques (relaxometry, thermography)

| 75µm | 208µm | 305µm |
|-------|-------|-------|
| 125µm | | |
| 89µm | | |
| 125µm | 162µm | |



j | μA cm

30

20

10

в

0.2

0.4



Axane activities and partnership's targets



MEA degradations are related to materials (membrane, catalyst layers, MPL/GDL) but also to hardware design, operating conditions and fluid management (air/H₂ stoichiometry, pressure...)

Each set of parameters may lead to specific phenomena

→ all these aspects have to be considered

20/04/2018





Two 55-cells stack shut down after failure of air humidifier after 1500h

- => System fault due to H₂ leak
- => Every cell tested to localised leak (pressure drop measurement)





Infrared Thermography



Temperature \rightarrow defect size

 $\Delta T = T_{max} - T_{min}$

The flaws are classified as a function of the generated heat recorded by the camera



Localisation and quantification of membrane defects



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Responsible for stack shut-down



Cracks with lentgh ranging from 0.5 to 1.2 mm promoting unsafe level of H₂ leak



Critical defects appeared more frequently near air outlet (H₂ inlet)

Cathode flooding? May be promoted by humidifier failure (=> insufficient air flow)

Larger pinholes look like mechanical tears
 mechanical constraints

Degradation specific to cell design

De Moor, G., Bas, C., Charvin, N., Moukheiber, E., Niepceron, F., Breilly, N., André, J., Rossinot, E., Claude, E., Albérola, N. D., and Flandin, L., 2012, "Understanding Membrane Failure in PEMFC: Comparison of Diagnostic Tools at Different Observation Scales," Fuel Cells, pp. n/a-n/a







Pressure drop and thermal camera results are consistent

> Infrared thermography is much more sensitive (IR able to disqualify « leak proof » MEAs with local membrane weaknesses)

De Moor, G., Bas, C., Charvin, N., Moukheiber, E., Niepceron, F., Breilly, N., André, J., Rossinot, E., Claude, E., Albérola, N. D., and Flandin, L., 2012, "Understanding Membrane Failure in PEMFC: Comparison of Diagnostic Tools at Different Observation Scales," Fuel Cells, pp. n/a-n/a



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 Segments with a lower current density at nominal operating point.
 Oxygen starvation, flooding.

Heterogeneous channel length linked to gas port positionning (same cell side) !







Carbon corrosion at cathode inlet (start & stop)

- > Segments between inlets and outlets are also more degraded
- > Assumption: dry H₂ bypass => anode purges lead to strong drying => dry/wet cycling, may be related to PEM defects

Degradation phenomena still related to the hardware design

A. Lamibrac, G. Maranzana, O. Lottin, J.Dillet, J. Mainka, S. Didierjean, A. Thomas, C. Moyne, Experimental characterization of internal currents during the start-up of a Proton Exchange Membrane Fuel Cell, Journal of Power Sources, Vol. 196, N°22, pp. 9451-9458, 2011.

Air Liquide, world leader in gases for industry, health and the environment

A closer look at air outlet / H2 inlet area





air bubble

Bubble mapping device applied to localize pinholes through MEAs





A closer look at air outlet / H2 inlet area





First degradation phenomena experienced - Conclusions

Fast and specifically localised membrane pinholing

Local study of pinholes and correlation with MEA performance suggested :

- local mechanical constraints and chemical degradation due to local drying (Air outlet/ H₂ inlet)
- dry/wet cycles on anode side (between inlets and outlets)
- local flooding (outlet section of air channels)

Ways of improvement:

- => tougher membrane material (mechanical reinforcement, chemical stabilizers...)
- => better air and H₂ management :
- Threshold air flow to ensure water removal,
- when possible, hydrogen humidification thanks to recirculation,





J. Kim et al. JPS 365 (2017) pp.419-429

axane

New MEA and improved system management

- Large increase of membrane lifetime
- Low performance degradation probably related to catalyst layer degradation
- Catalyst layers undergo heterogeneous ageing too¹
- New target: 7 performances and Y CL aging in various operating condition \Rightarrow **3** stack efficiency \Rightarrow **4** H₂ consumption



¹Dubau L., Durst J., Maillard F., Chatenet M., André J., Rossinot E., 2011, "Heterogeneities of aging within a PEMFC MEA," Fuel Cells, pp. n/a-n/a



- Multidisciplinary diagnosis and characterisation techniques are useful for a better understanding of MEA degradation
- > They also enabled to identify ways of improvement
- Specific operating conditions and hardware design lead to specific degradation phenomena...which is critical to mitigate MEA degradation



Thank you very much for your kind attention !

