MEA; MANUFACTURING PROCESSES & QUALITY TECHNIQUES

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MEA: THE DIFFERENT STEPS OF MANUFACTURING

Catalyst + ionomer ← Material technology choice

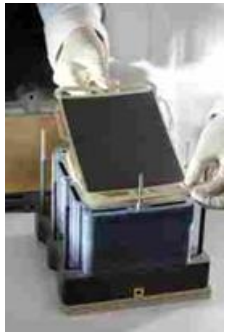
Ink formulation

AL printing

Process

CCM + rim + GDL

GDE + membrane +rim ← Technology choice



7 layers assembly ← design

Process

stack

Impact on:
- performance
- durability
- cost

FIRST: 7 LAYERS ASSEMBLY

Alignement of the MEA (GDL + CCM + rim) is a requirement.

1 defected MEA (misaligned, issue on the rim) causes **failure on the whole stack**:
- Leakage, gas by pass, not homogenous compression

Check tolerance of MEA alignment Vs Stack tolerance chain.

At lab scale : not an issue, what about high speed manufacturing ?

2D control
on 50 points

N° AME : V31b (Anode) Date: 14/09/17

DESIGN : Inhouse | PROJET : MATISSE

Fournisseur Renfort : | Matériau : | Epaisseur :

Fournisseur GDL : LITEN | Chargée : | non | N° lot :

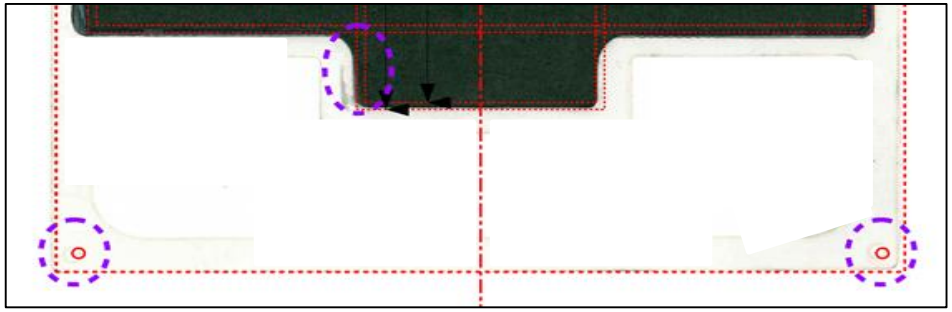
Fournisseur Membrane : | Type : | N° lot :

Référentiel trou Bas Droit | oui | Ecarts Eventuels

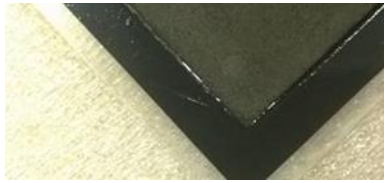
Contrôle Tridim

Repère	Point	Nominal	Mesure	Ecart	Repère	Point	Nominal	Mesure	Ecart
Détrompeurs	1	-48,00	-48,14	-0,14	D	26	42,03	41,92	-0,11
	2	1,50	1,53	0,03		27	3,50	3,60	0,10
	3	2,20	2,31	0,11		28	-3,50	-3,48	0,02
	4	-144,50	-144,49	0,01		29	-44,25	-44,19	0,06
	5	244,50	244,54	0,04		30	10,00	10,11	0,11
	6	2,20	2,34	0,14	E	31	25,33	25,25	-0,08
	7	244,50	244,59	0,09		32	3,50	3,58	0,08
	8	2,20	2,36	0,16		33	-51,75	-51,82	-0,07
	9	2,20	2,34	0,14		34	-92,75	-92,64	0,11
	10	-144,50	-144,46	0,04	F	35	42,03	41,88	-0,15
	11	0,00	0,02	0,02		36	3,50	3,69	0,19
A	12	241,00	240,94	-0,06		37	-100,25	-100,43	-0,18
	13	202,47	202,51	0,04		38	-141,00	-140,83	0,17
	14	-100,25	-100,41	-0,16		39	10,00	9,93	-0,07
	15	-141,00	-140,90	0,10	EXT	40	152,50	152,73	0,23
	16	10,00	10,04	0,04		41	4,00	4,04	0,04
B	17	241,00	240,98	-0,02		42	-148,50	-148,69	-0,19
	18	219,17	219,26	0,09		43	252,50	252,50	0,00
	19	-51,75	-51,95	-0,20		44	248,50	248,52	0,02
	20	-92,75	-92,60	0,15		45	-4,00	-3,98	0,02
C	21	241,00	240,95	-0,05	GDL	46	6,25	6,28	0,03 ✓
	22	202,47	202,58	0,11		47	6,25	6,52	0,27 ✓
	23	-3,50	-3,59	-0,09		48	52,28	51,88	-0,40 ↩
	24	-44,25	-44,19	0,06		49	52,28	52,46	0,18 ✓
	25	10,00	10,24	0,24	Commentaires:				
BON POUR LIVRAISON			OUI	NON					

misaligned GDL



wrinkle



Air pocket under subgasket

Dust on the subgasket

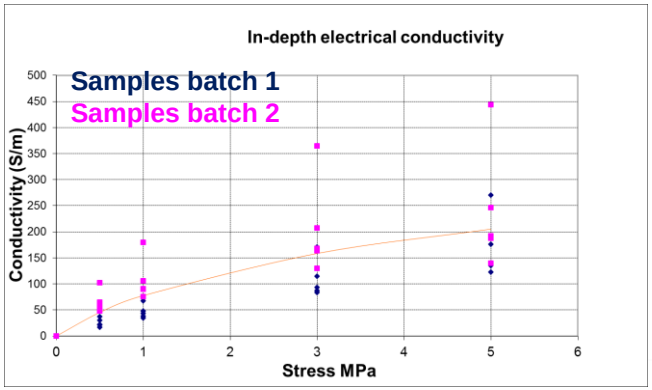
GDL: A KEY ELEMENT

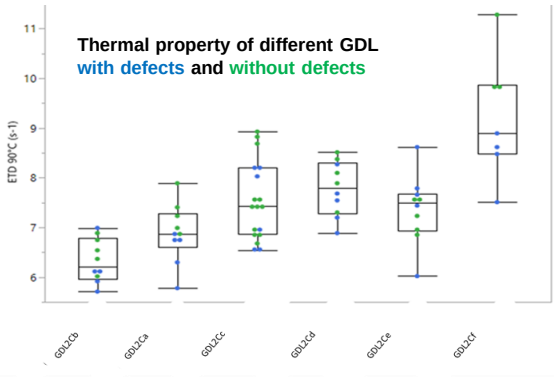
Understanding the impact of GDL properties on stack performance is still to be improved : **Defect definition**

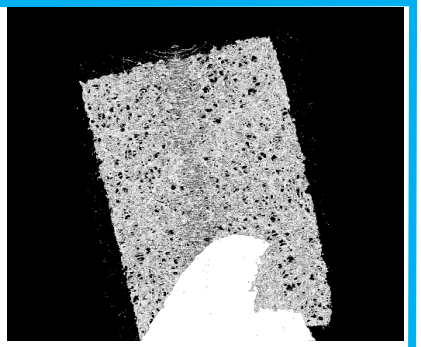
Manufacturing
- Process/parameters
- Requirements
- Selection (criteria)

1. **Better understand the link between (local) properties of GDL to their performance/durability**
2. **Propose improvements**

Off-line characterization

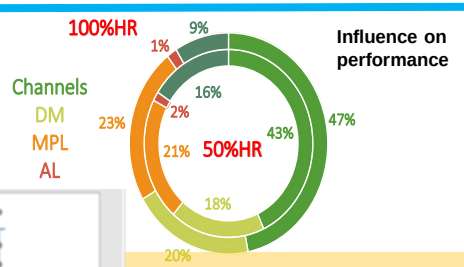




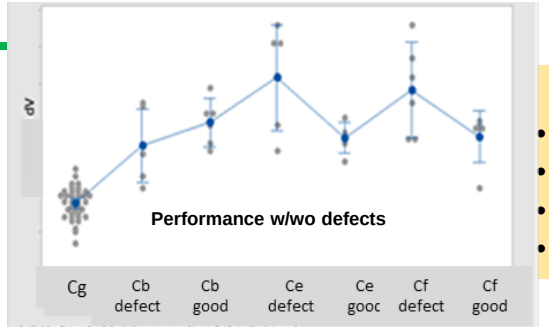


3D image of a commercial GDL

Assembling
- Process/parameters
- Requirements



Testing
- Performance
- Durability
- Requirements
- Acceptance (criteria)



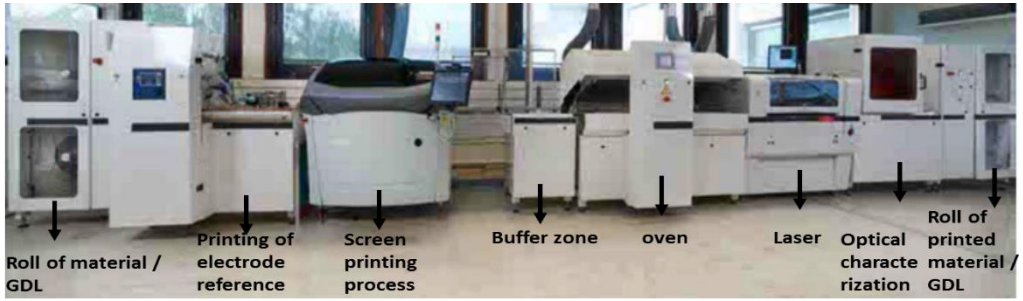
On-line characterization

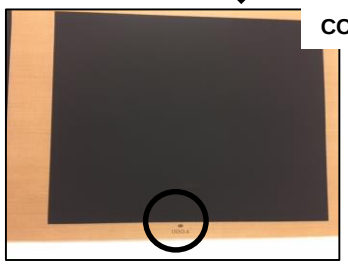
1. **What is a defect?**
2. **Define selection/acceptance criteria as a trade-off quality/price:**
   - **General ones?**
   - **Specific ones depending on the MEA, BPP, OC…**
3. **On-going DIGIMAN project as an example**

ACTIVE LAYER: IN PLANE

Electrode characterization

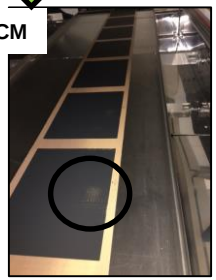
Pilot line of electrode manufacturing in line detection

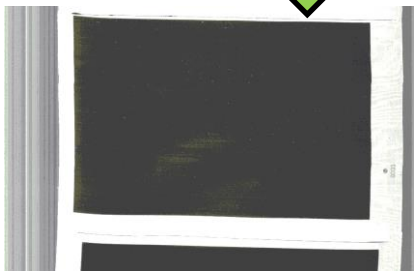




numbering

CCM

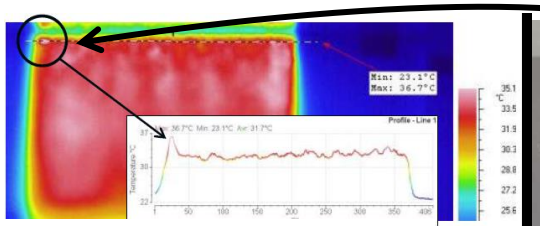




Macro defect:
optical check thanks to contrast AL Vs support

Defects detection on GDE with Reactive Impinging Flow Excitation (RIF)

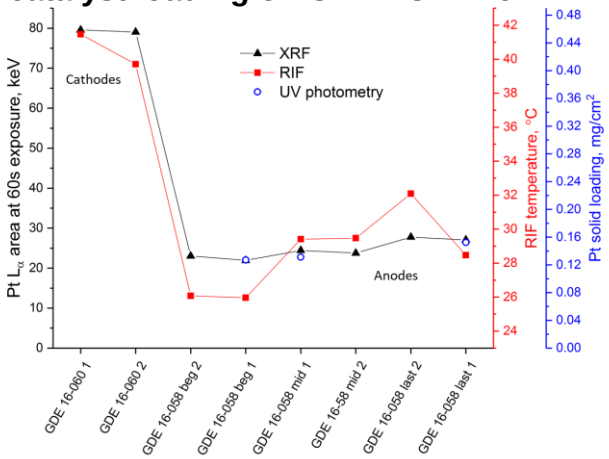
NREL NATIONAL RENEWABLE ENERGY LABORATORY



Hot spot → thicker smudge

Cool spot → thinner coating

Link between defects detection and **catalyst loading on GDE : On line**



adaptation of the ink for each :

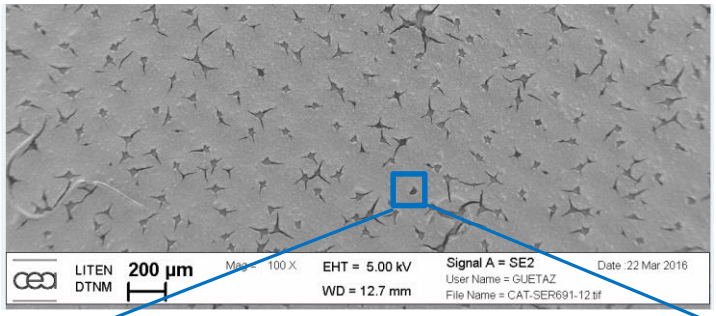
- Process
- Material technology (ionomer, catalyst)
- Electrode design

ACTIVE LAYER: IMPACT OF THE PROCESS

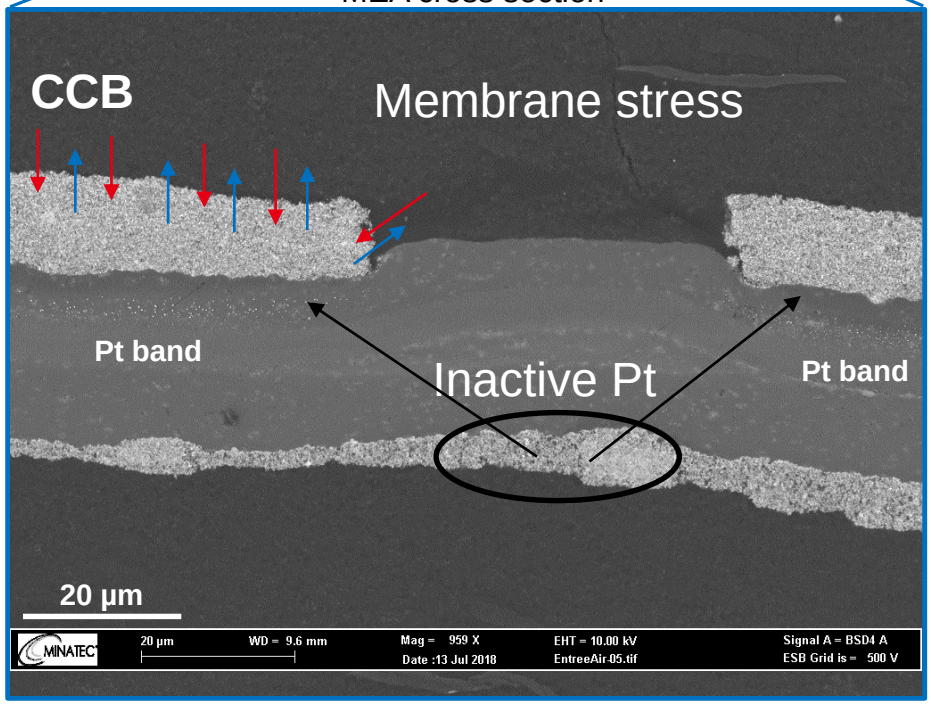
Holes and cracks in the AL impact the membrane integrity;

Cracks from : MPL and/or process

AL front view



MEA cross section



Active Layer inhomogeneity

- no AL → membrane penetration

No Pt band in the membrane:

→ validates the sample preparation

→ Might cause issue on durability

→ Impact on BoL performance ?

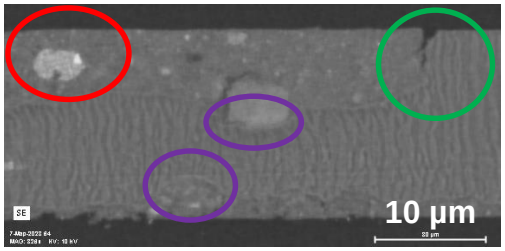
Not or only few

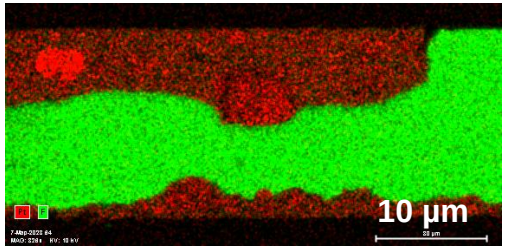
(reinforced membrane)

SEM observation after : 150 hrs – 10,000 km in HyKangoo

ACTIVE LAYER: THROUGH PLANE

CCM cross section : SEM characterization





Pt in red
F in green

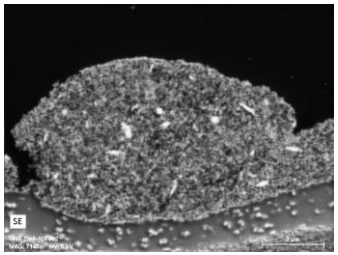
- Aggregates without polymer → inactive Pt, loss of ECSA
- Membrane penetration in AL crack → membrane weak point ?
- AL aggregates penetration in AL → thinner membrane, weak point ?

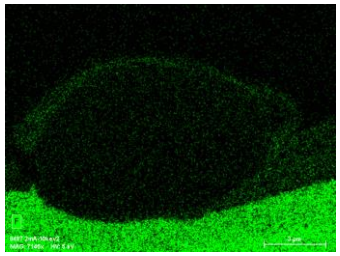
Catalyst aggregates :
- no ionomer,
- no catalyst ripening
- Inactive Pt

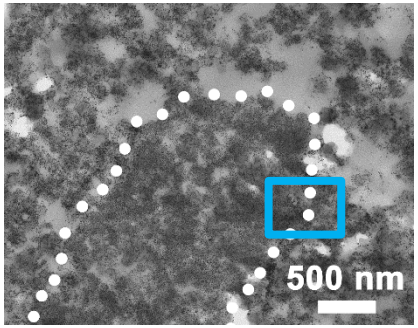
→ impact on Pt loading
→ Impact on ageing

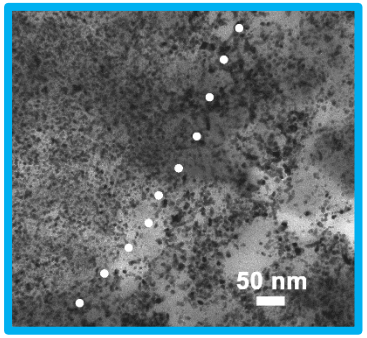
- Deformation of the membrane
- Short circuit / hole

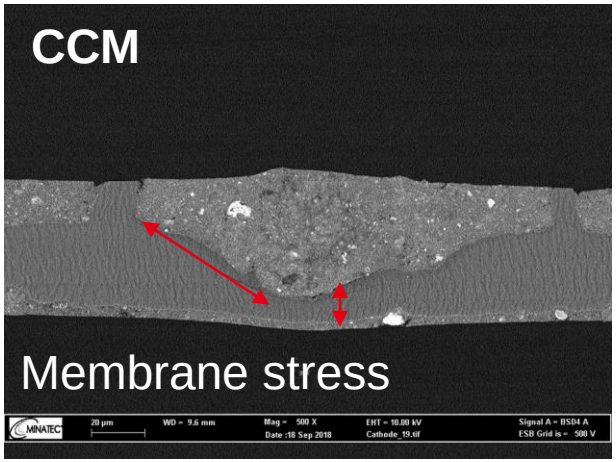
→ Impact on ageing
→ failure











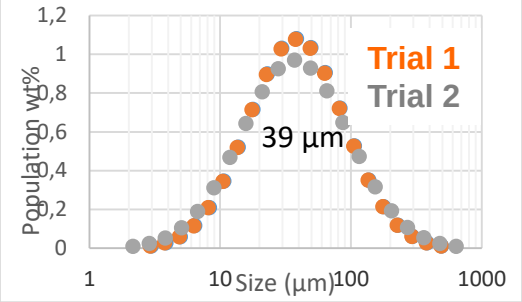
CATALYST AND INK PREPARATION

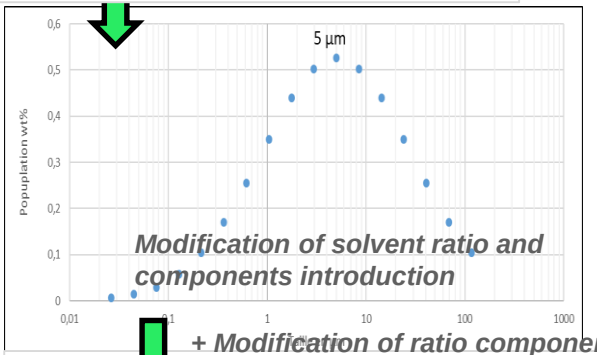
The ink preparation process affecting:
- the numbers of aggregates and the viscosity

To be tuned for each material technology (ionomer, catalyst) and process

Initial ink

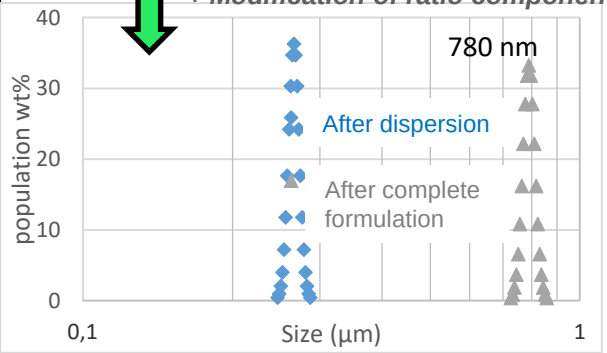
Impact of solvent and ratio components





+ Modification of ratio components

Optimized ink

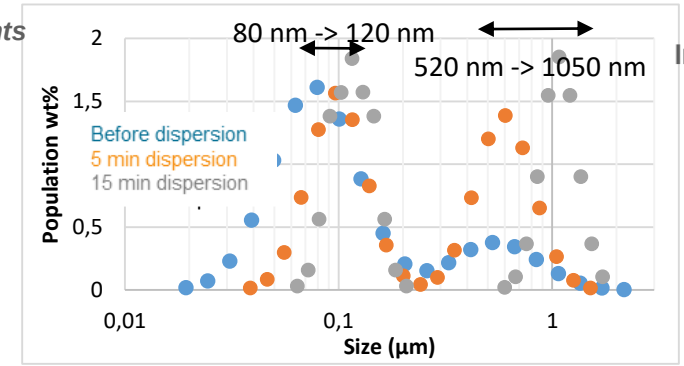


Electroacoustic + zeta probe :

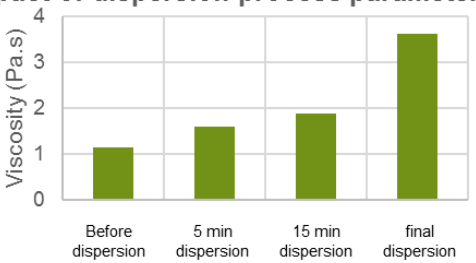
→ chasing the granulometry of the ink

→ Ink validation before AL printing

Impact of dispersion process parameter



Impact of dispersion process parameter

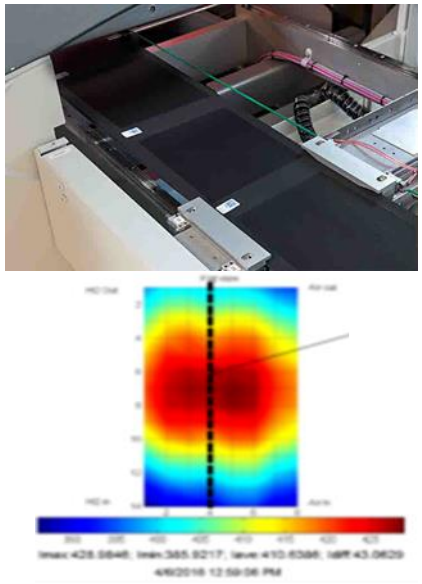


OPERANDO CHARACTERIZATION OF MEA : SEGMENTED BOARD

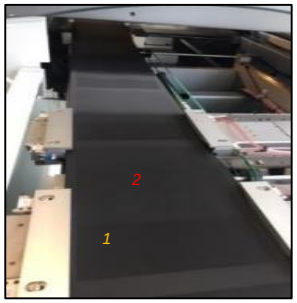
Current density distribution mapping CDDM
→ MEA homogeneity
→ impact of RH, stoichio, fuel composition

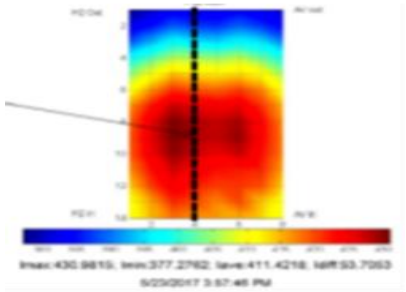
A tool to study the impact of one « defected » MEA on the stack and « defect » propagation

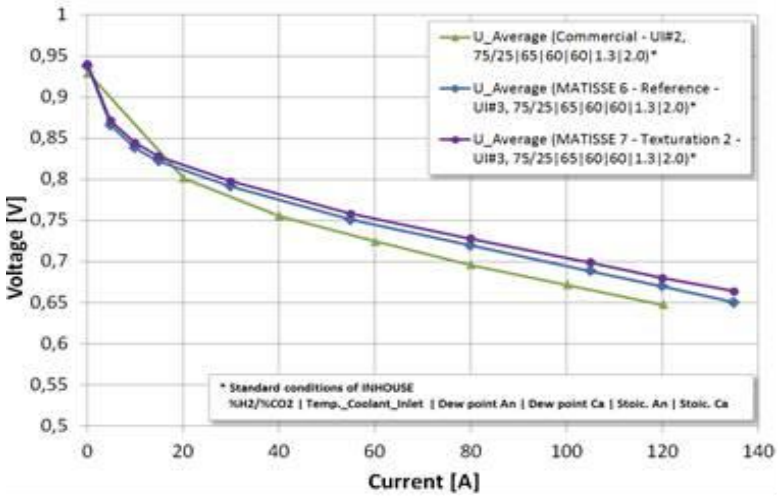
Uniformed electrode



Textured electrode





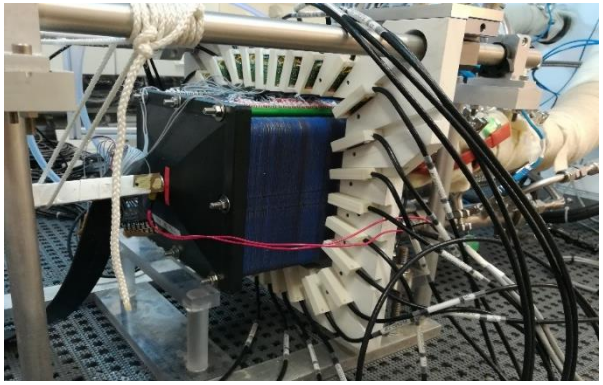


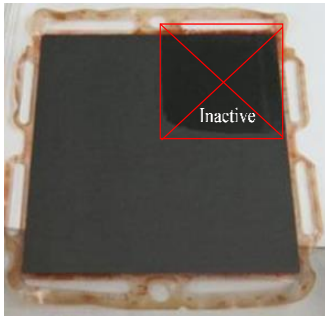
Different AL designs give different CDDM

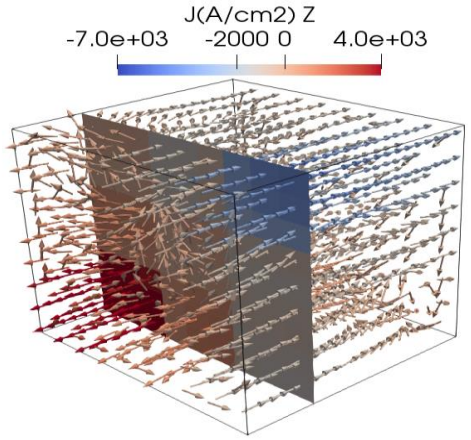
OPERANDO CHARACTERIZATION OF MEA : MAGNETIC FIELD AND ELECTROCHEMICAL IMPEDANCE

Instrumented stack integrating a « defected MEA »

Lyes IFREK, Thesis, 2017

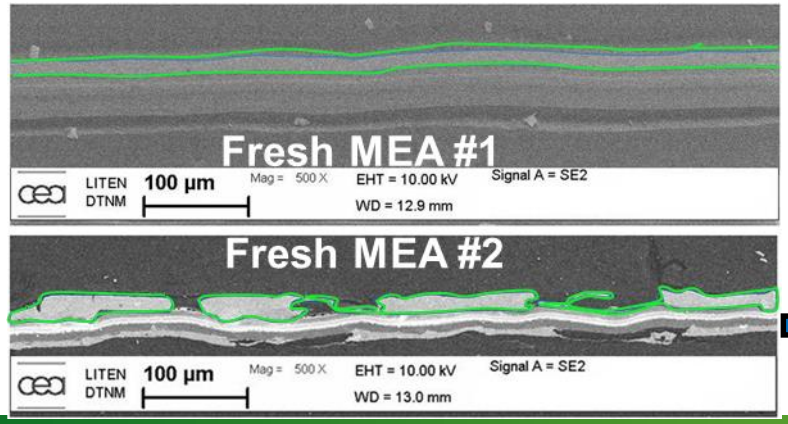


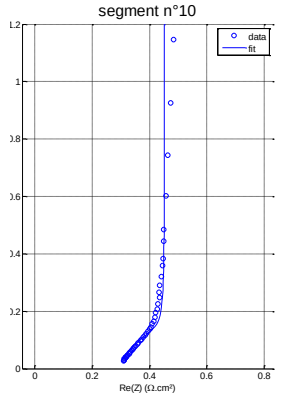


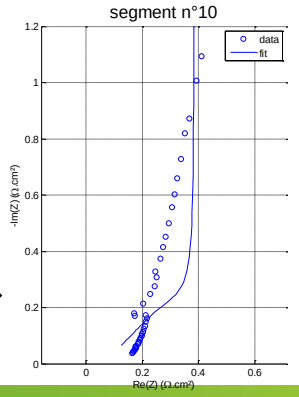


Evolution of EIS between : homogeneous AL and voluntary defected AL

Thomas Gaumont, Thesis, 2017

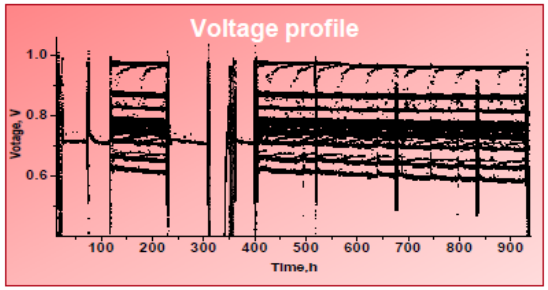


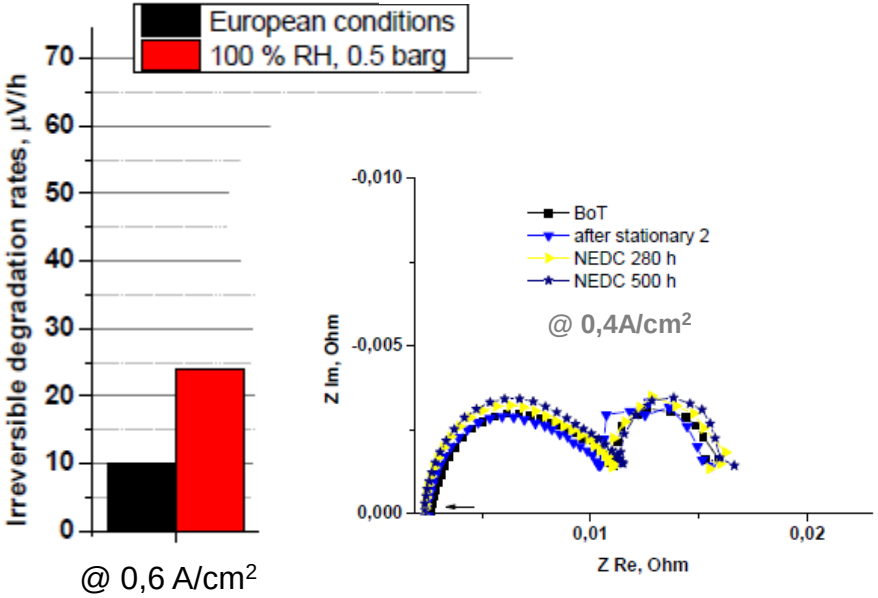




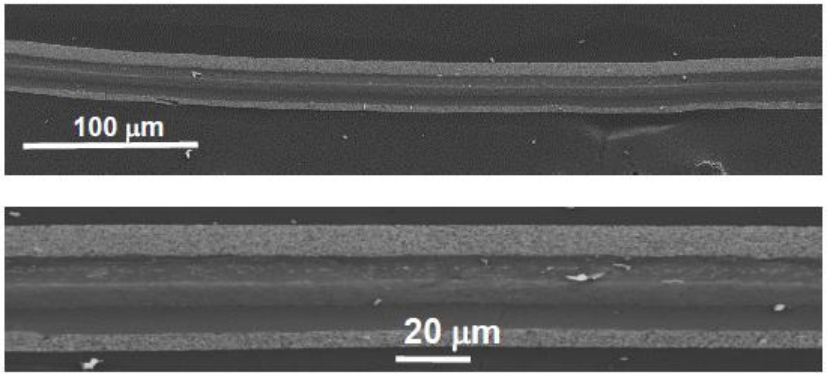
DURABILITY TEST WITH HIGH QUALITY ACTIVE LAYER

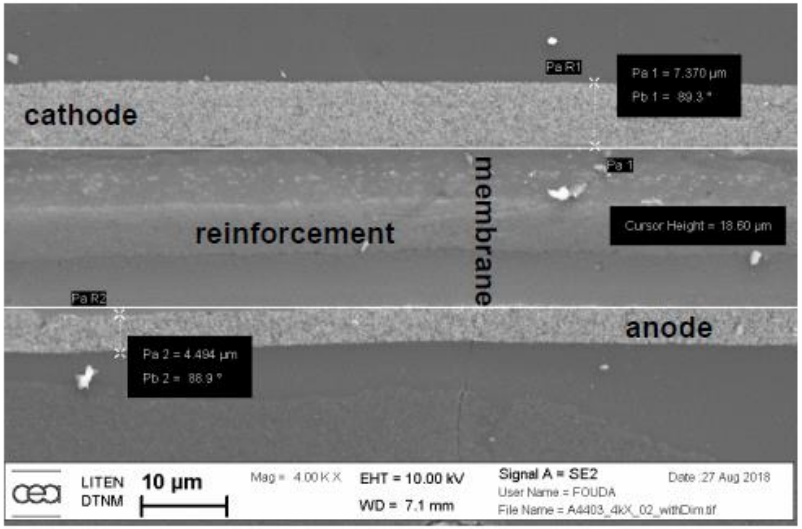
CEA CCM:
- cathode loading 0,35 mgPt/cm^2
- Anode loading 0,1 mgPt/cm^2





SEM on MEA cross section
after 900 hrs (stationary + Fc-DLC)





CONCLUSION

- What is a defect ?

→ Need to establish defect specification and ranking

- Failure of the stack
- Decrease of BoL performance
- Decrease of durability → decrease of EoL performance Vs failure
- Next generation material

→ Understanding the link :

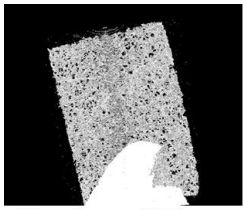
1\ material and component specification → stack performance
2\ material and component specification → stack durability

→ Material and component validation :

- Definition of the main parameters to be tracked
- Definition of the quality assurance (on line Vs off line)
- 100 % areal checking Vs selected validation points

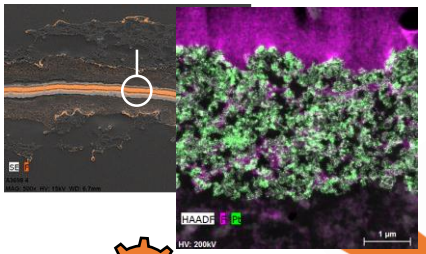
- Available tools for defect detection ?

Component structure



MEA Integration

Ink processing & manufacturing



Inferface optimisation

Ex situ and single component characterisation

In situ and integrated component characterisation

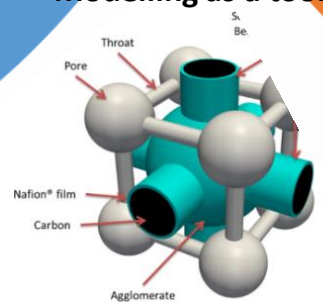
Cell performance

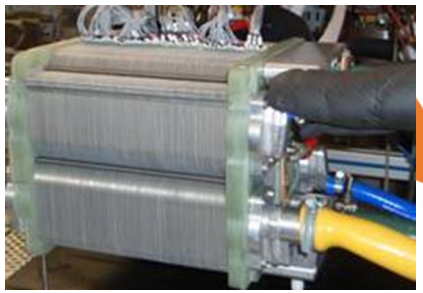
Defect < Component specification < over quality

Impact on each physical parameter

Inspection of the main parameter quality management policy

Modelling as a tool





Full stack characterisation

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MERCI POUR VOTRE ATTENTION

THANKS FOR YOUR ATTENTION

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