Mechanisms of high potential excursions in fuel cells

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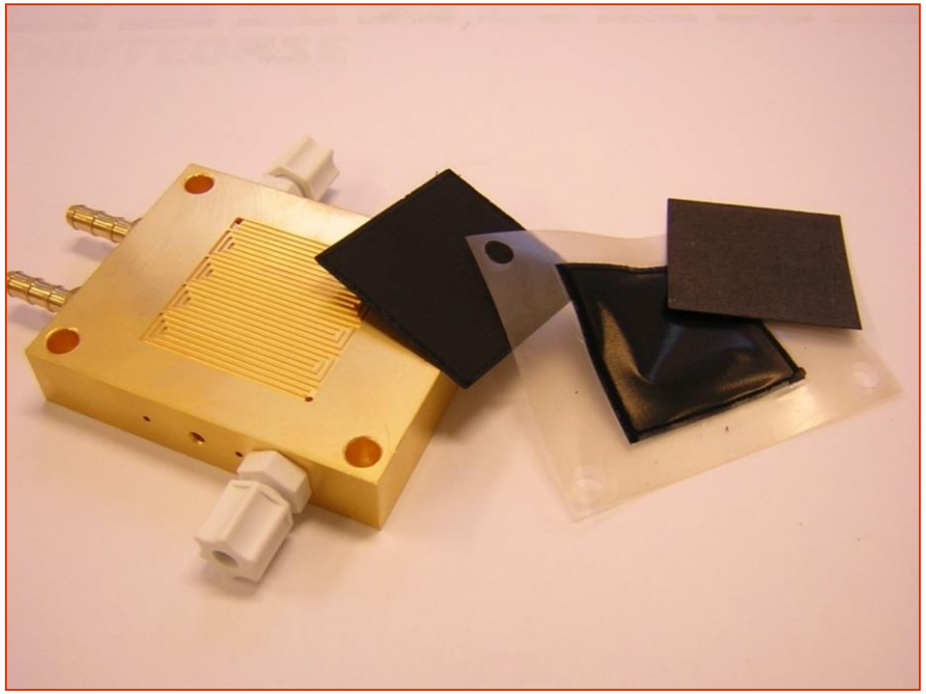


Introduction → Electrochemical Potentials

What is it?

Electrochemical or redox potentials describe equilibria between two species named the oxidant and the reducer, *i.e.*:

$$Ox + ne^- \leftrightarrow Red$$

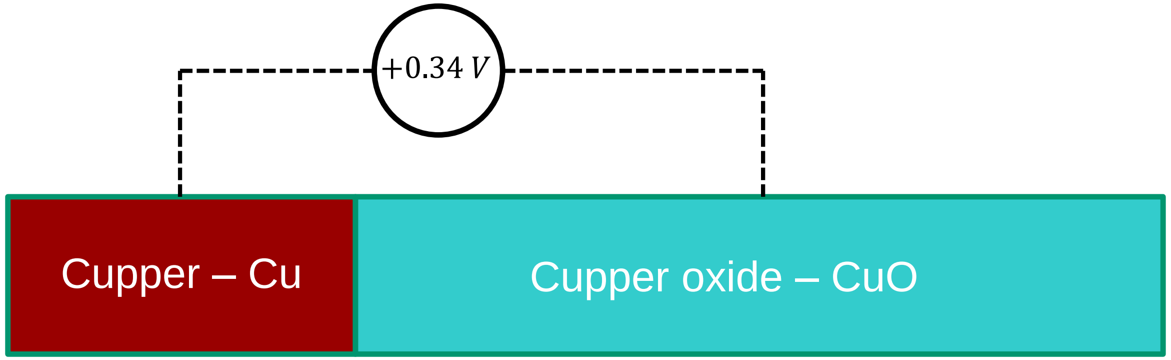
… in standard conditions, this reaction is in equilibrium when the potential is equal to E_0 (or ΔE_0).

For instance…

$$Cu^{2+} + 2e^- \leftrightarrow Cu$$

Cupper is in equilibrium with its oxide when $E_0 = +0.34\,V$.

… which means (1) that there is an actual potential difference ($\Delta E_0 = +0.34\,V$) between cupper and its oxide when they are in equilibrium.



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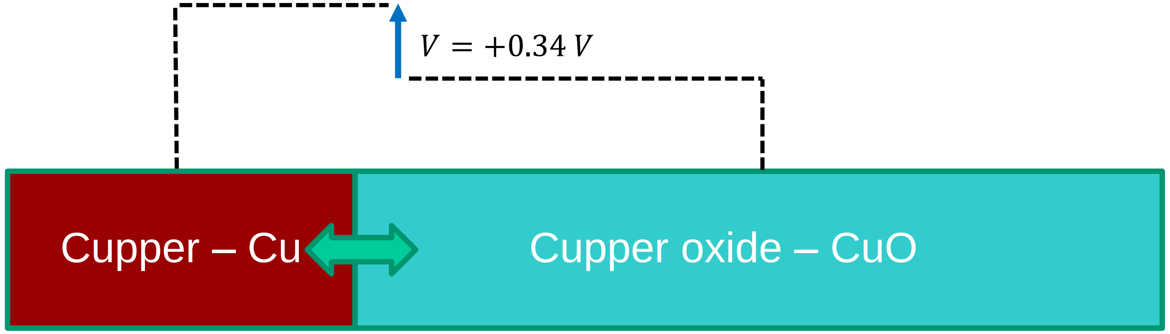
… which means (2) that **above +0.34 V**$^{(*)}$, the reaction will go in the left hand side direction and eventually, cupper will be fully oxidized into Cu^{2+}.



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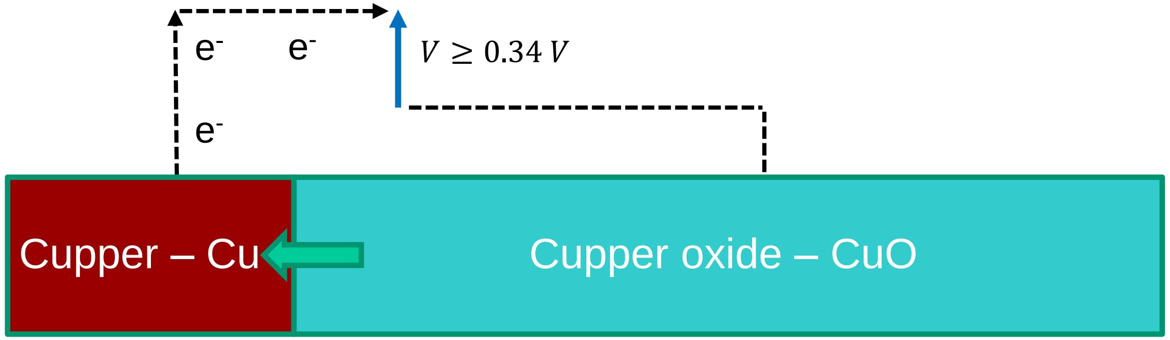
… which means (2) that **above +0.34 V**$^{(*)}$, the reaction will go in the left hand side direction and eventually, cupper will be fully oxidized into Cu^{2+}, and this simply because electrons are attracted by high potentials! $^{(**)}$



(*) To this point, the way potential is applied does no matter.

(**) In practice, the electrons flux is significant only if the charge balance is maintained.

Proton Exchange Membrane Fuel Cells (PEMFC)

- Hydrogen oxidation at the anode

$$H_2 \rightarrow 2H^+ + 2e^-$$

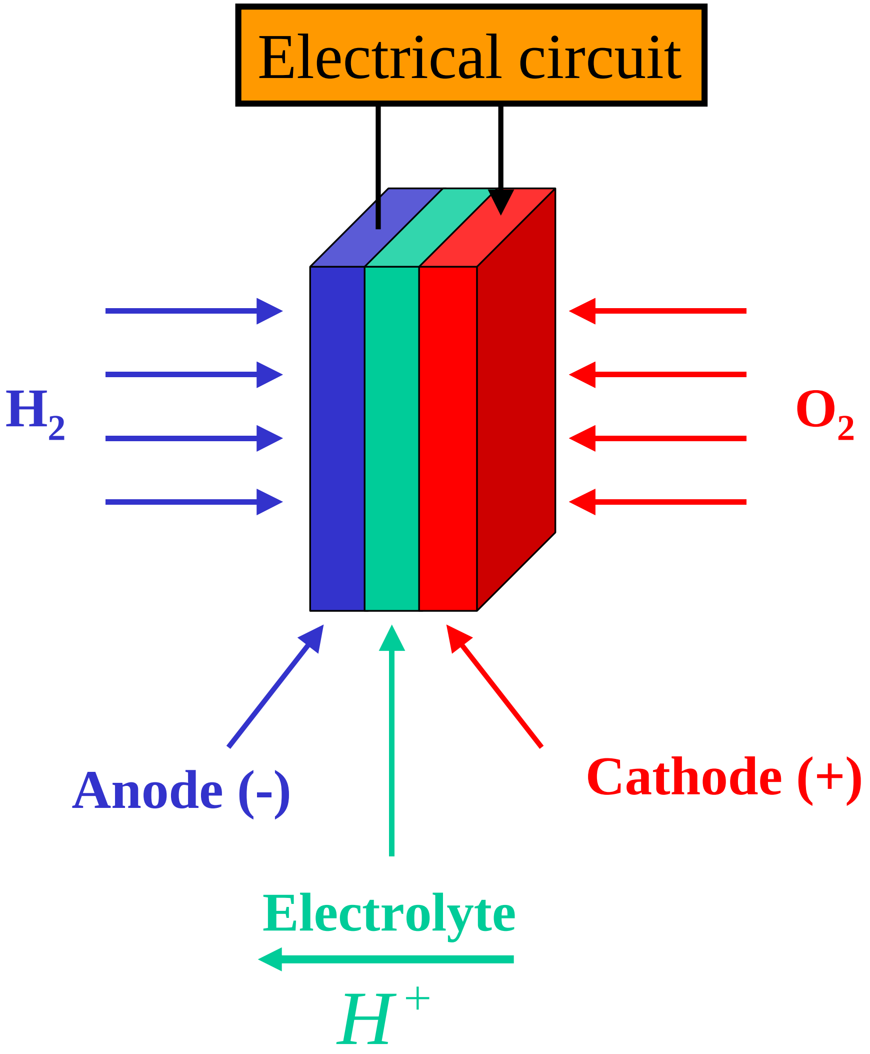
- Oxygen reduction at the cathode

$$^1/_2\,O_2 + 2H^+ + \ 2e^- \rightarrow H_2O$$

- Globally

$$H_2 + {}^1/_2\,O_2 \rightarrow H_2O$$

$$\boxed{E_0 = \frac{-\Delta G_0}{k_{ox}F} = 1.23\ V}$$



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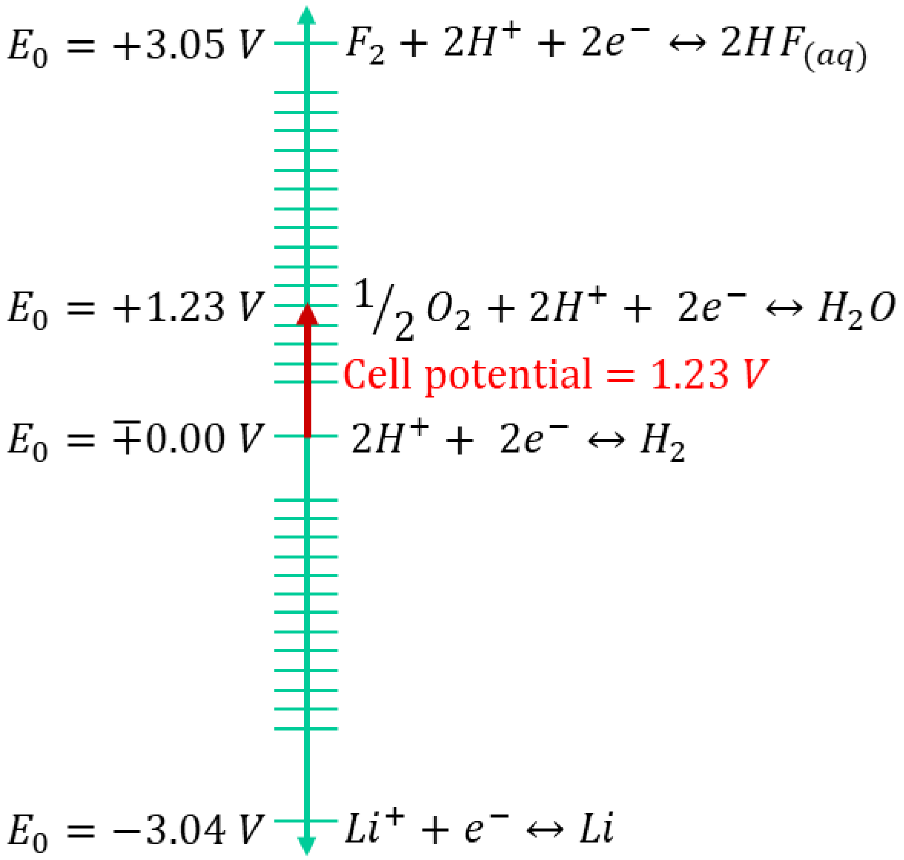

$E_0 = +3.05\ V$ — $F_2 + 2H^+ + 2e^- \leftrightarrow 2HF_{(aq)}$

$E_0 = +1.23\ V$ — $^1/_2\, O_2 + 2H^+ + \ 2e^- \leftrightarrow H_2O$

Cell potential $= 1.23\ V$

$E_0 = \mp 0.00\ V$ — $2H^+ + \ 2e^- \leftrightarrow H_2$

$E_0 = -3.04\ V$ — $Li^+ + e^- \leftrightarrow Li$

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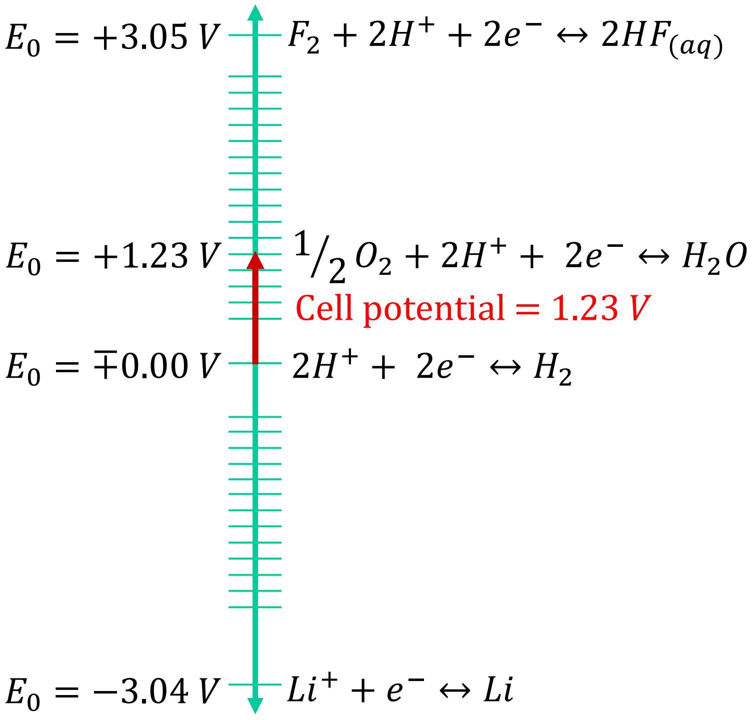

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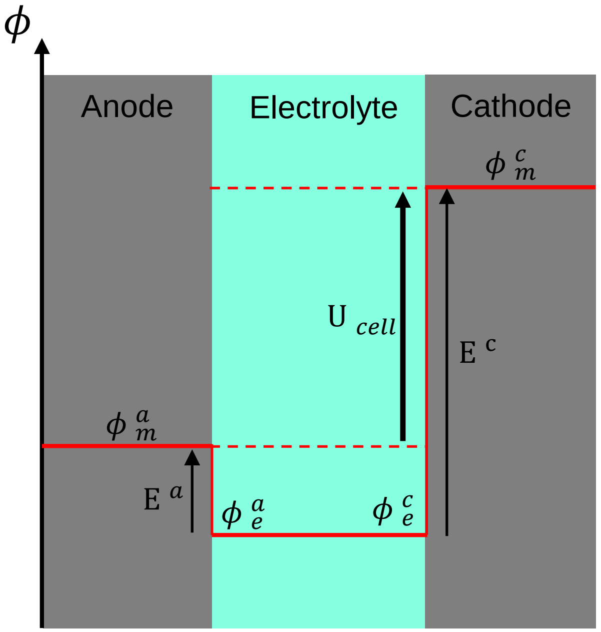
Some comments…

- The fuel cell voltage is quite low, even in ideal conditions, *compared to* *Li-ion* battery cells.

- However… the cathode potential is quite high *compared to* the oxidation potential of the cathode materials: *Pt* and *carbon*.
The cathode potential is lower in Alkaline Fuel Cell.

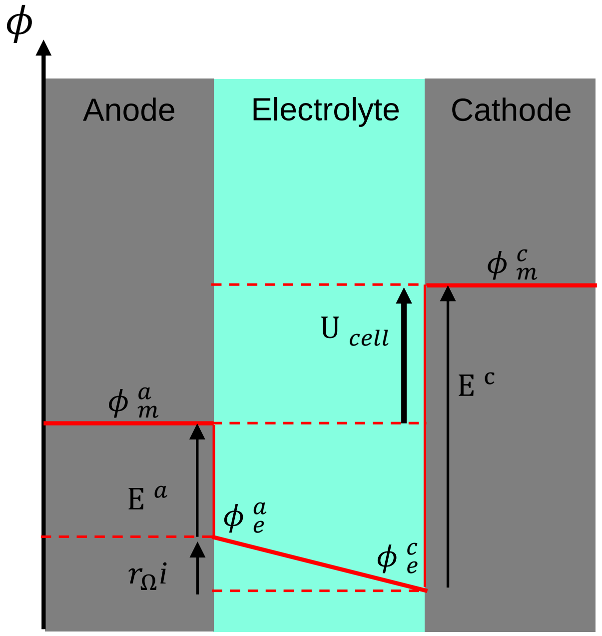
Introduction → Proton Exchange Membrane Fuel Cells (PEMFC)

Potential/voltage diagrams in Proton Exchange Membrane Fuel Cells



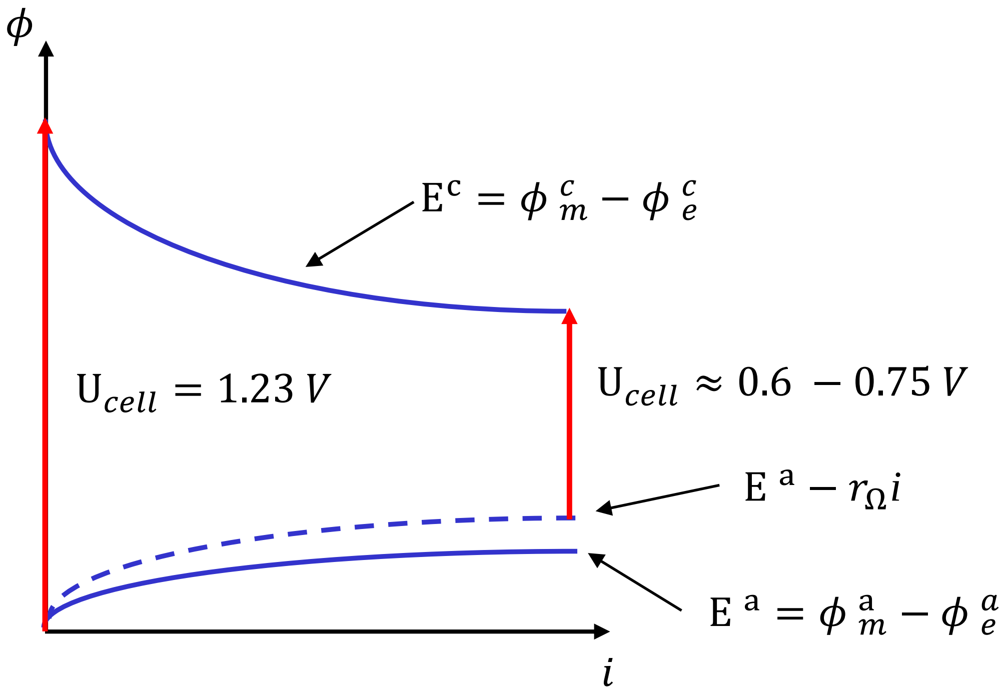
Potentials diagram in open circuit

i = 0 A/cm²



Potentials diagram in operation

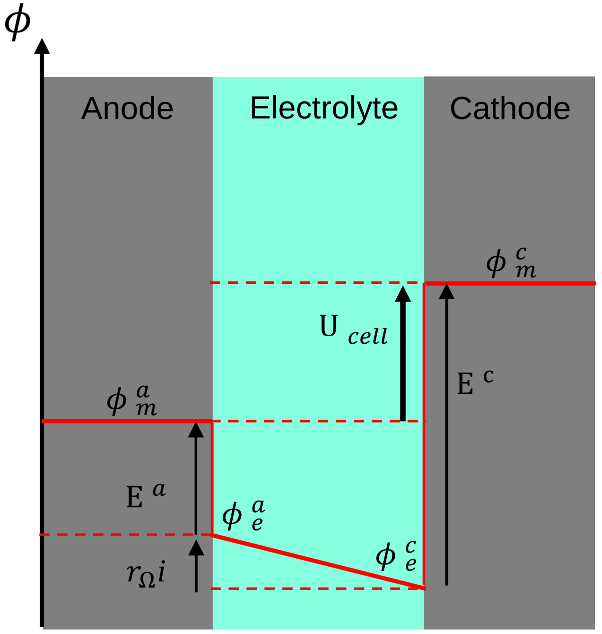
i > 0 A/cm²



$$U_{cell} = \phi_m^c - \phi_m^a$$

Potential/voltage diagrams in Proton Exchange Membrane Fuel Cells

→ Slide 22
→ Slide 25



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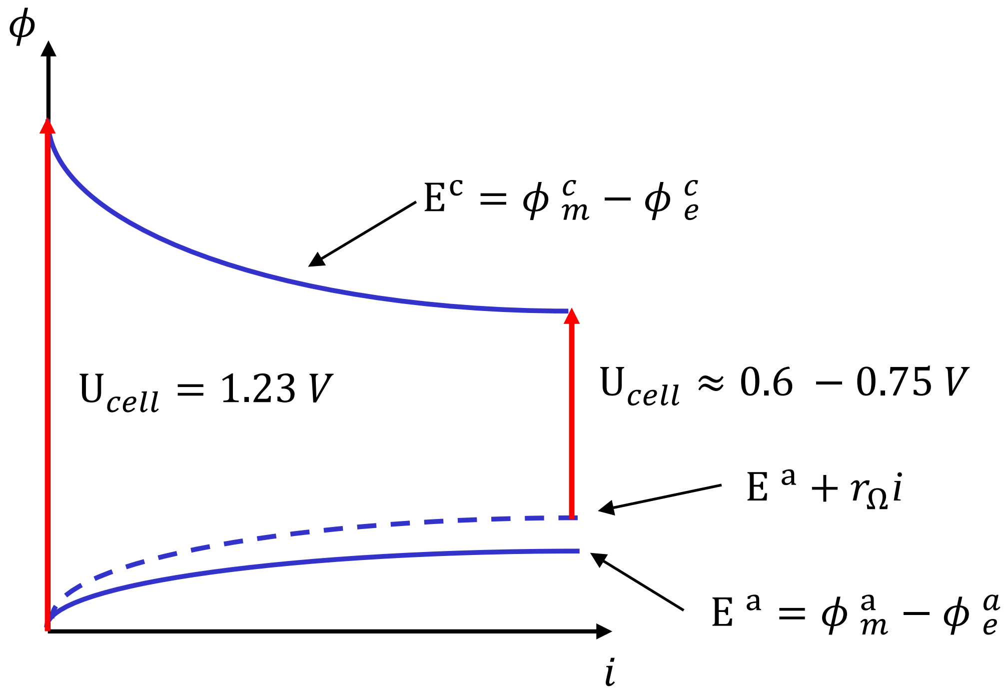


$U_{cell} = \phi_m^c - \phi_m^a$ → This is what we measure at the cell terminals

$$U_{cell} = E^c + \phi_e^c - [E^a + \phi_e^a] = E^c - E^a + \phi_e^c - \phi_e^a = E^c - E^a - r_\Omega i$$

$^1/_2 O_2 + 2H^+ + 2e^- \leftrightarrow H_2O$

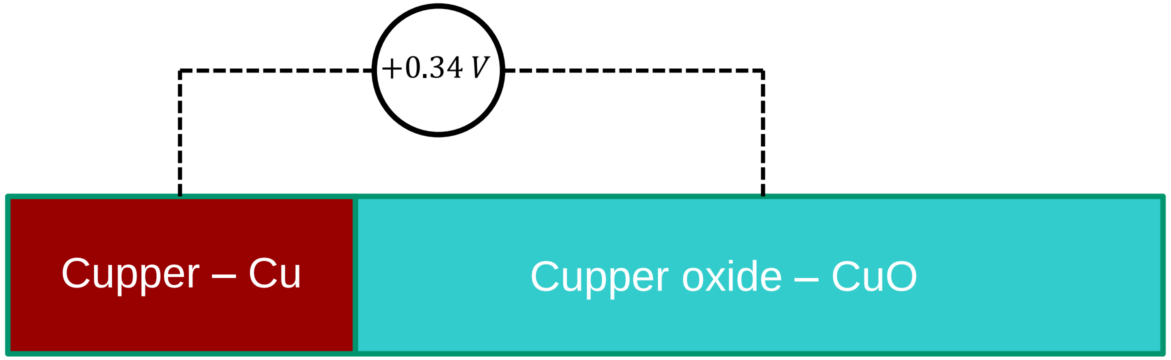
$2H^+ + 2e^- \leftrightarrow H_2$

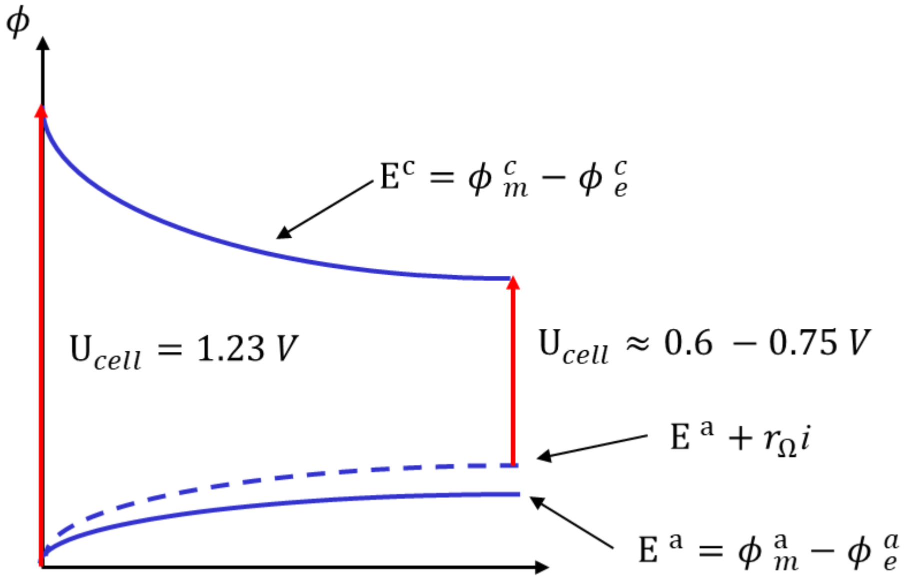
Losses in the electrolyte

Introduction → Summary to this point

1. Electrochemical or redox potentials are *real* potential differences!

$$Cu^{2+} + 2e^{-} \leftrightarrow Cu$$

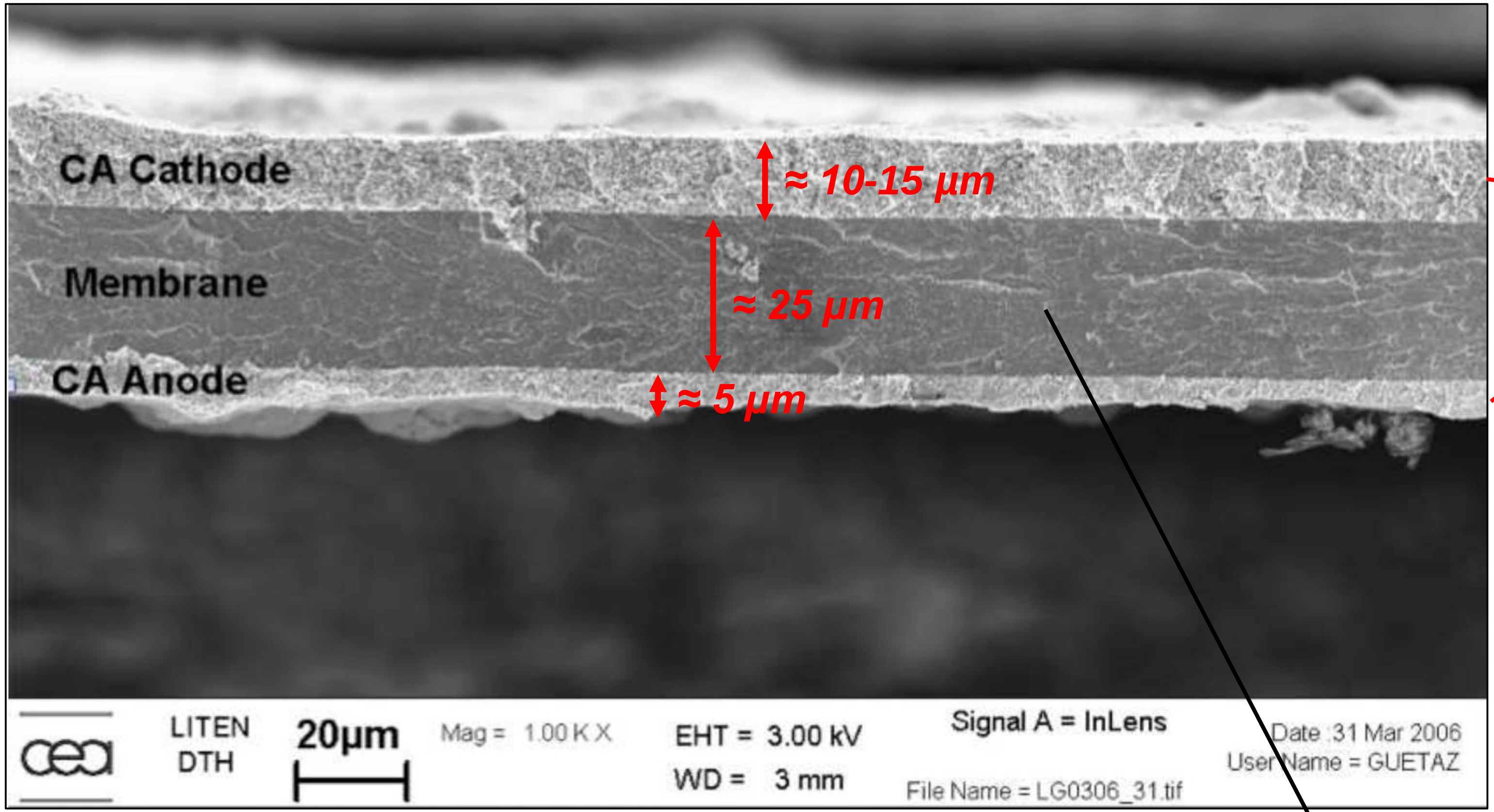




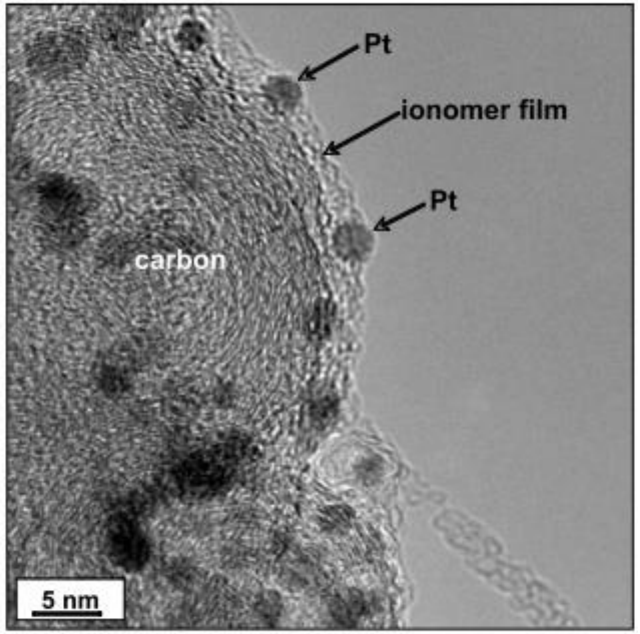
2. The redox potential of *oxygen reduction* corresponds to the *cathode potential* in fuel cells, while the redox potential of *hydrogen oxidation* corresponds to the *anode potential*…
   … but values from taken from thermodynamic tables can be used only *in equilibrium*, without any electrical current. When the cell is under operation, the situation becomes more complex! …Tafel law, Butler-Volmer equation.

3. Materials are *sensitive* to *electrical potentials* and tend to *oxidize* under the effect of *high potentials*.

The electrodes, what are they made of?



B. Vion-Dury, *PhD Thesis, Univ. Grenoble, p.18*



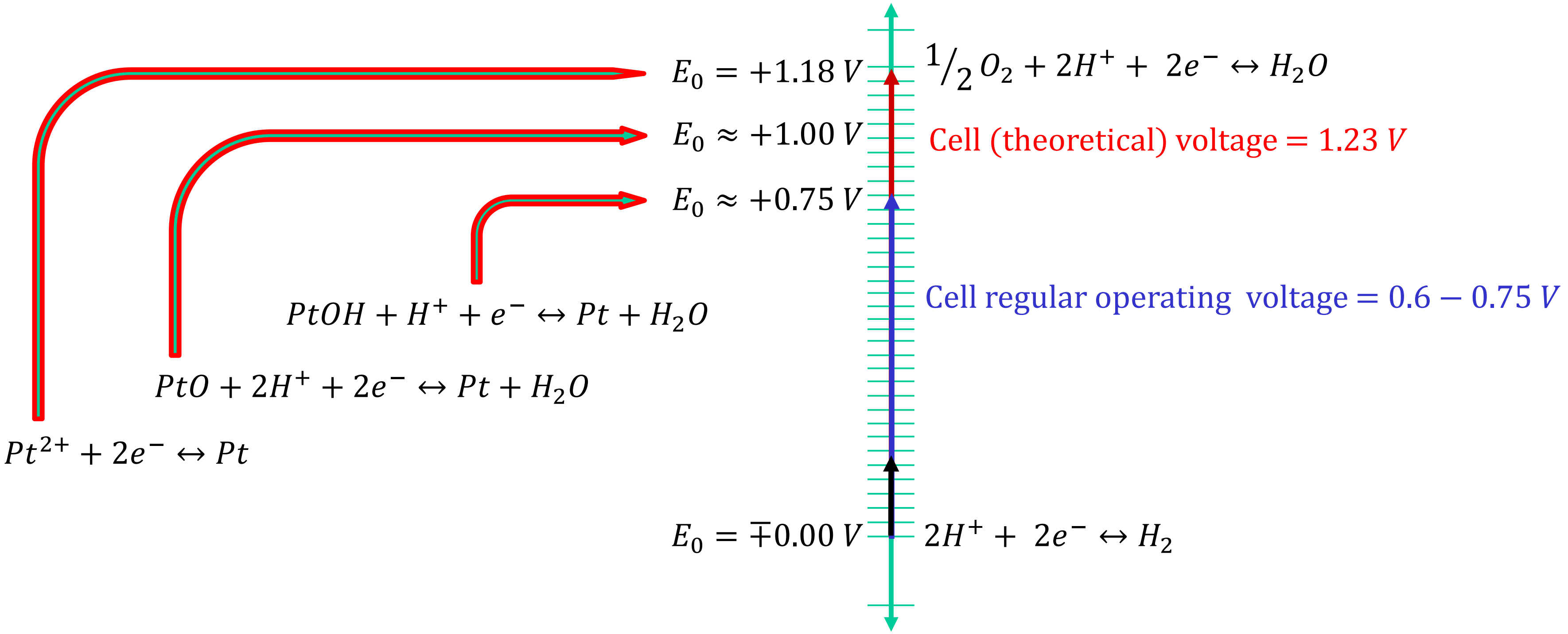
K. More et al., *ECS Transactions, 3 (1) 717-733 (2006)*

Materials are rarely ordered so well in fuel cell electrodes !
… but Pt and carbon, and ionomer, are always there.

Membranes are out of the scope of this presentation but their degradation is a strong issue in PEMFC.

Materials degradation in PEMFC → platinum oxidation

Platinum oxidation potentials



$E_0 = +1.18\ V$ — $^{1}/_{2}\,O_2 + 2H^+ + 2e^- \leftrightarrow H_2O$

$E_0 \approx +1.00\ V$

$E_0 \approx +0.75\ V$

$E_0 = \mp 0.00\ V$ — $2H^+ + 2e^- \leftrightarrow H_2$

Cell (theoretical) voltage = 1.23 V

Cell regular operating voltage = 0.6 – 0.75 V

$PtOH + H^+ + e^- \leftrightarrow Pt + H_2O$

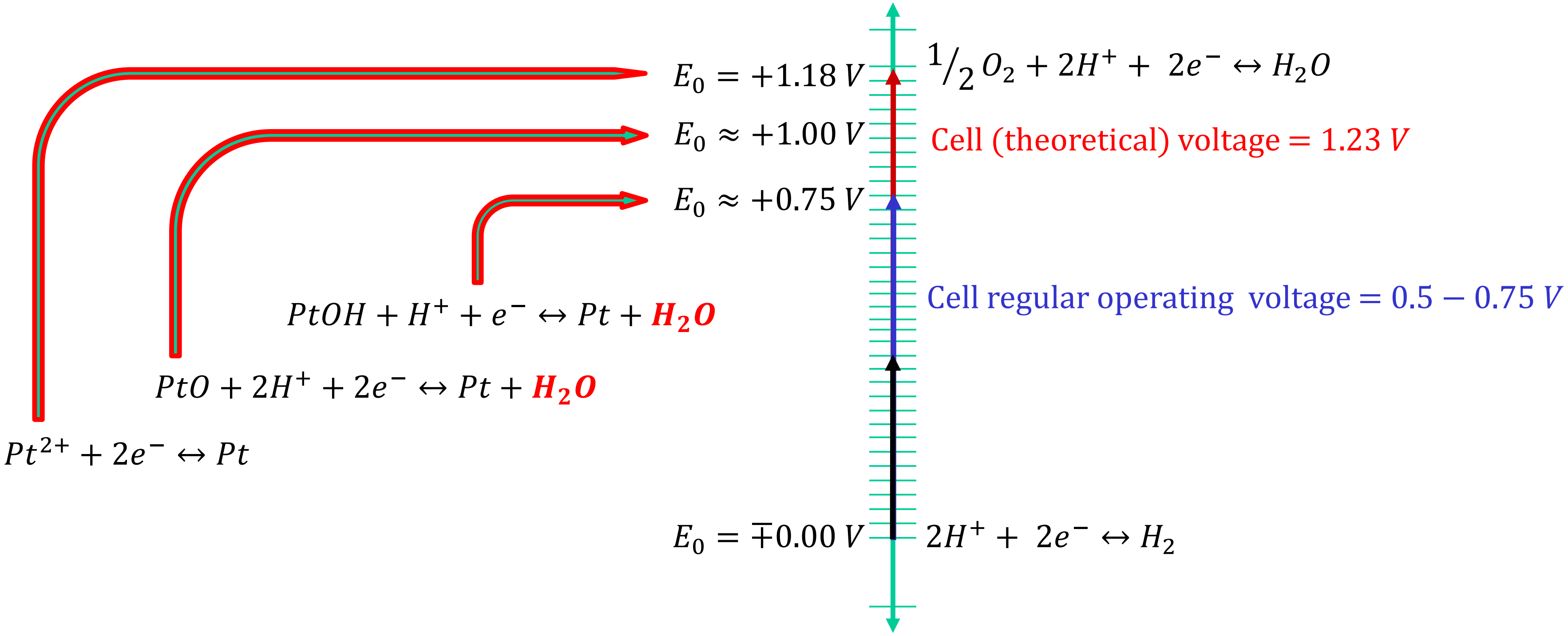
$PtO + 2H^+ + 2e^- \leftrightarrow Pt + H_2O$

$Pt^{2+} + 2e^- \leftrightarrow Pt$

- Platinum oxidation occurs in several steps: PtOH, PtO, and eventually Pt^{2+}.
- … from 0.75 V to 1.18 V… *i.e.* at *potentials lower than the cathode potential*!
- → this explains why stable FC operation is *not possible above 0.75-0.8 V*: PtOH and PtO are not good catalysts!
- → this also explain why the Open Circuit Voltage never goes above 1.0 V… instead of 1.23 V according to thermodynamics.
- !Pt oxidation is mostly reversible! … *except* for $\mathrm{Pt}^{2+} + 2\mathrm{e}^- \leftrightarrow \mathrm{Pt}$ (which should never occur?).

Materials degradation in PEMFC → platinum oxidation

Platinum oxidation potentials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$\,^1/_2\, O_2 + 2H^+ + 2e^- \leftrightarrow H_2O$

$E_0 \approx +1.00\ V$

Cell (theoretical) voltage = $1.23\ V$

$E_0 \approx +0.75\ V$

$PtOH + H^+ + e^- \leftrightarrow Pt + \boldsymbol{H_2O}$

Cell regular operating voltage = $0.5 - 0.75\ V$

$PtO + 2H^+ + 2e^- \leftrightarrow Pt + \boldsymbol{H_2O}$

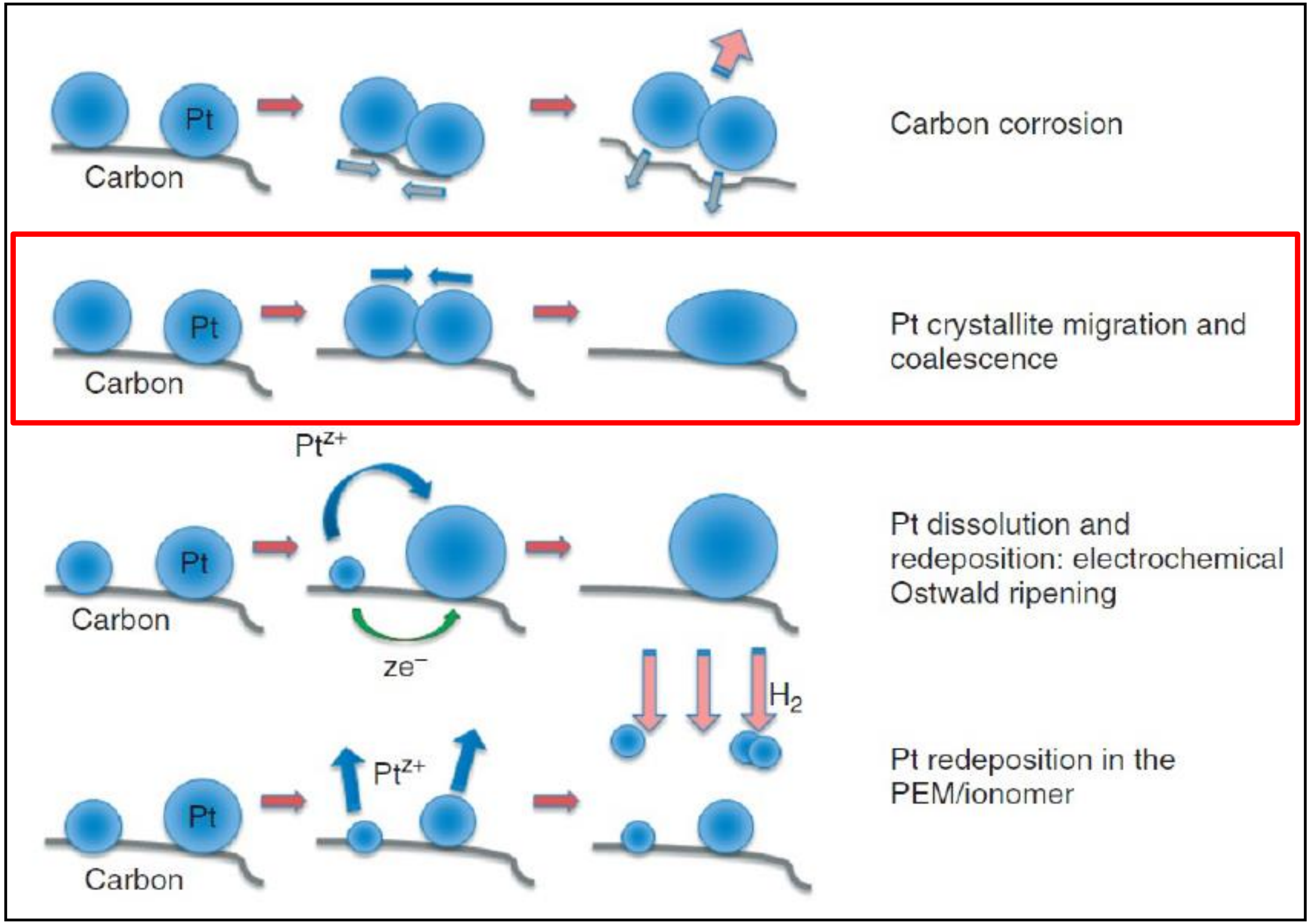
$Pt^{2+} + 2e^- \leftrightarrow Pt$

$E_0 = \mp 0.00\ V$

$2H^+ + 2e^- \leftrightarrow H_2$

- Platinum oxidation occurs in several steps: PtOH, PtO, and eventually Pt^{2+}.
- … from 0.75 V to 1.18 V.
- !Pt oxidation is mostly reversible! … except for $\mathrm{Pt}^{2+} + 2\mathrm{e}^- \leftrightarrow \mathrm{Pt}$ (which should never occur?).
- Long term degradation depends on the number *oxidation/reduction cycles* → most *usual* catalyst degradation mechanism.
- Degradation increases with the amount of *water* in the cell → one of the various issues linked to water management in FC.

Platinum degradation → a matter of electrochemistry… .



L. Dubau et al., *Wiley Interdisciplinary Reviews : Energy and Environment, 3 (6), (2014)*

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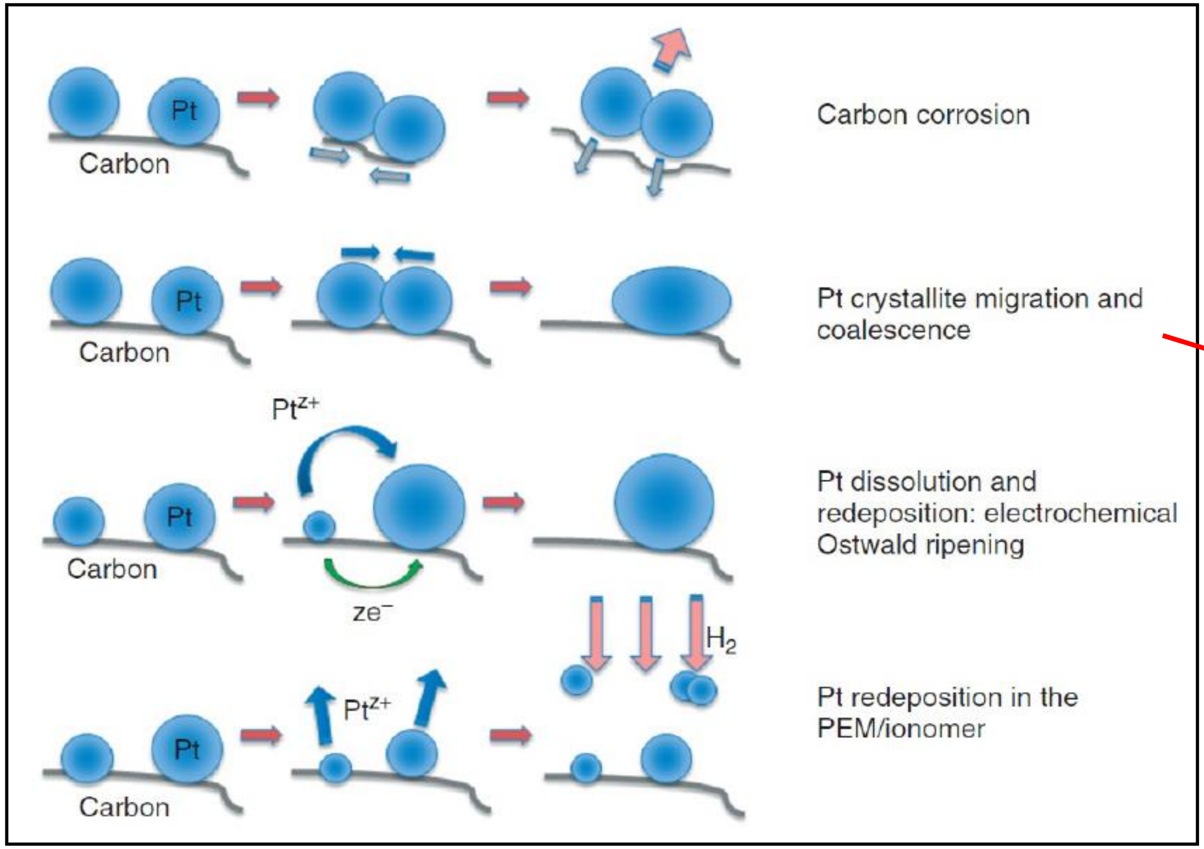

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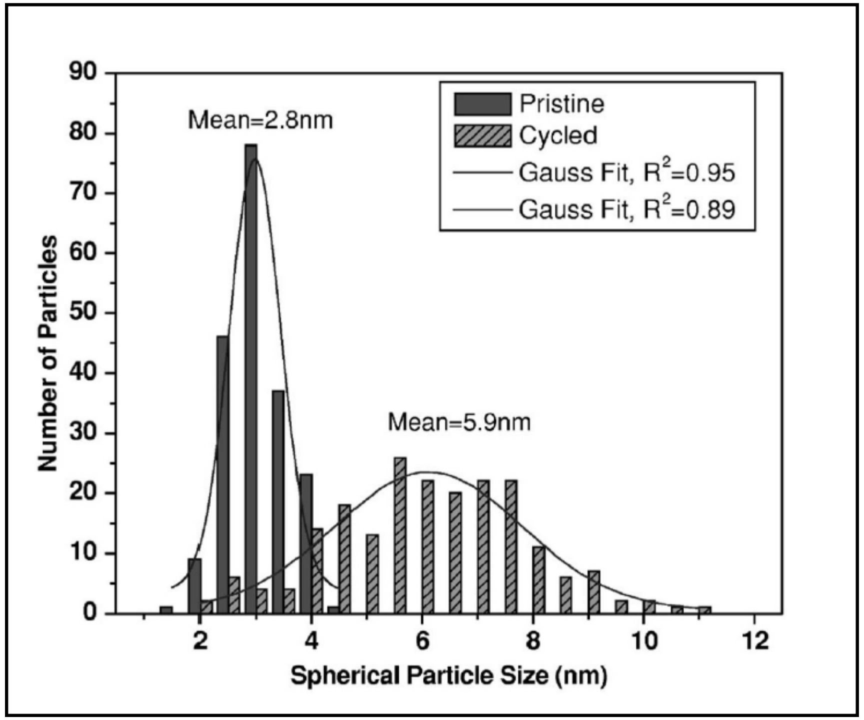


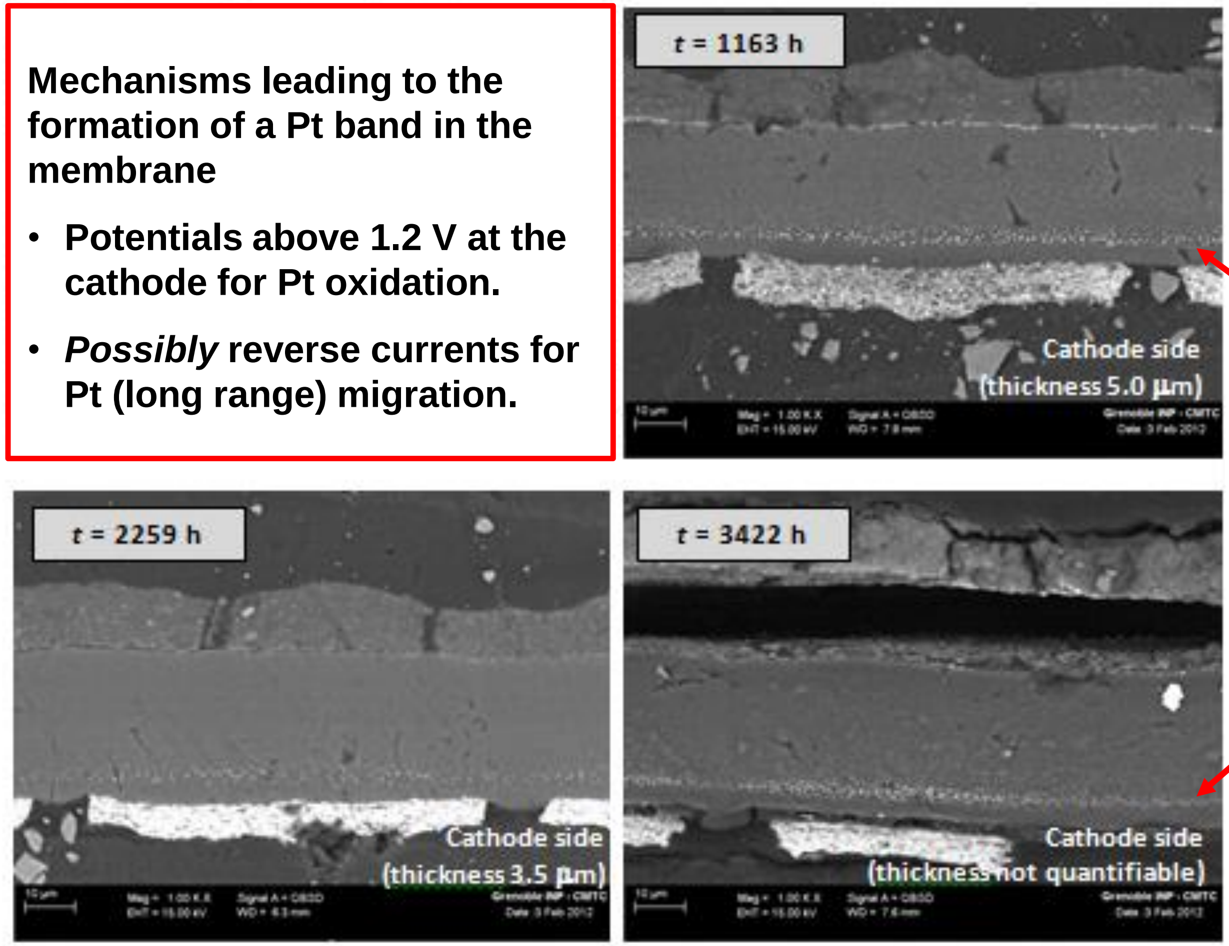
E. Guilminot et al., *J. Electrochem. Soc., 154 (1), (2007)*

Materials degradation in PEMFC

The famous Pt band → only in very special occasions… at least in theory.

Mechanisms leading to the formation of a Pt band in the membrane

- **Potentials above 1.2 V at the cathode for Pt oxidation.**
- ***Possibly* reverse currents for Pt (long range) migration.**

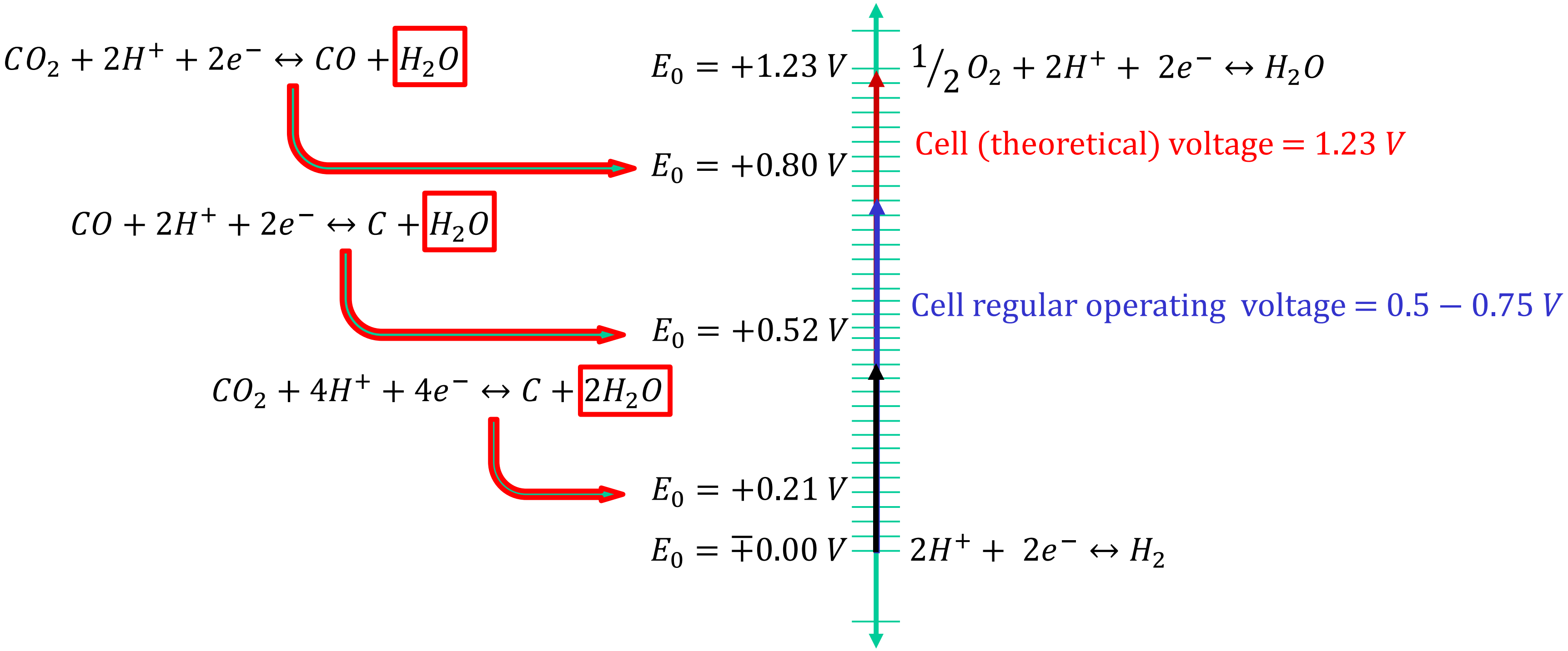


Pt^{2+} migrates and deposits in the membrane

L. Dubau et al., *Wiley Interdisciplinary Reviews : Energy and Environment, 3 (6), (2014)*

♦ !Pt oxidation is mostly reversible! … except for $\mathrm{Pt}^{2+} + 2\mathrm{e}^{-} \leftrightarrow \mathrm{Pt}$ (which should never occur?)

Carbon oxidation



$CO_2 + 2H^+ + 2e^- \leftrightarrow CO + \boxed{H_2O}$

$CO + 2H^+ + 2e^- \leftrightarrow C + \boxed{H_2O}$

$CO_2 + 4H^+ + 4e^- \leftrightarrow C + \boxed{2H_2O}$

$E_0 = +1.23\ V$ $\quad {}^1/_2\, O_2 + 2H^+ + 2e^- \leftrightarrow H_2O$

Cell (theoretical) voltage $= 1.23\ V$

$E_0 = +0.80\ V$

Cell regular operating voltage $= 0.5 - 0.75\ V$

$E_0 = +0.52\ V$

$E_0 = +0.21\ V$

$E_0 = \mp 0.00\ V$ $\quad 2H^+ + 2e^- \leftrightarrow H_2$

- Carbon oxidation potentials are *much lower* than the regular cathode potential range.
- However, *the rate of carbon oxidation* remains *very low* below one Volt.
- … thus, significant carbon oxidation should not occur in fuel cells (but it does sometimes).
- Like in the oxidation of Pt, *H_2O is a key species*.
- … and *Pt reduction* can lead to *carbon oxidation* → most *usual* carbon degradation mechanism in regular operating conditions.

Materials (cathode) degradation → summary to this point

1. Fuel cells operate between *0.6 and 0.75 V*, and one can assume that the *cathode potential* is *only slightly higher* than these values in regular *operating conditions*.

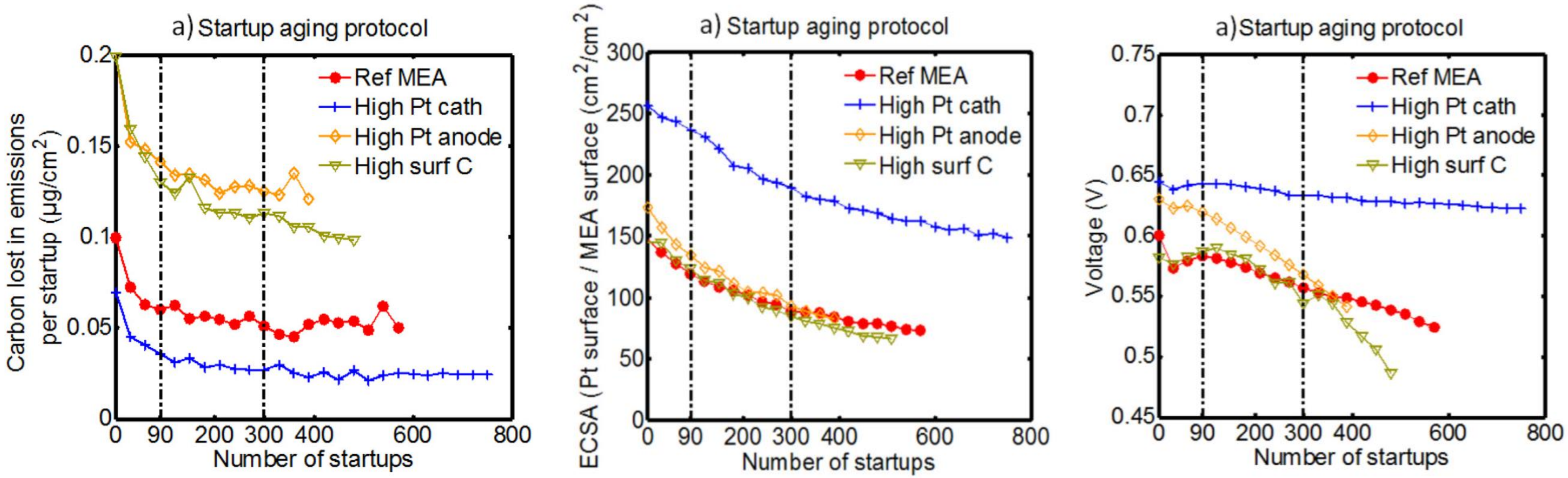
2. Fuel cells voltage higher than 0.75 V *in operation* or 1.0 V in open circuit (OCV) are -*in theory and practice*- *not possible* because of *Pt oxidation* → thus *limiting* the FC efficiency to 0.75/1.23 = 0.61.

3. *Pt* and *carbon* degradation mechanisms at cathode potentials *lower* or *equal* to *1.0 V* are *well understood* and *observed*: *slow carbon corrosion* and *Pt migration* and *coalescence* under the effect of *potential cycling*.

4. … but we also have evidence of *strong degradations*, *i.e strong carbon corrosion* and *Pt full oxidation* under the effect *high potentials*… that are currently explained *only* by *hydrogen starvation*.

Materials degradation in PEMFC → impact of hydrogen starvation

Pt oxidation into Pt^{2+} and significant carbon oxidation can occur!



From ***J. Dillet et al.,*** *J. Power Sources, 250, (2014)*

- Aging protocol consisting in repeated FC startups, by injecting H_2 in the anode compartment
- Independently of the MEA materials used, we observed:
  - significant CO_2 emissions,
  - a significant loss of active surface,
  - a decrease of FC voltage (measured at 0.67 A/cm²).

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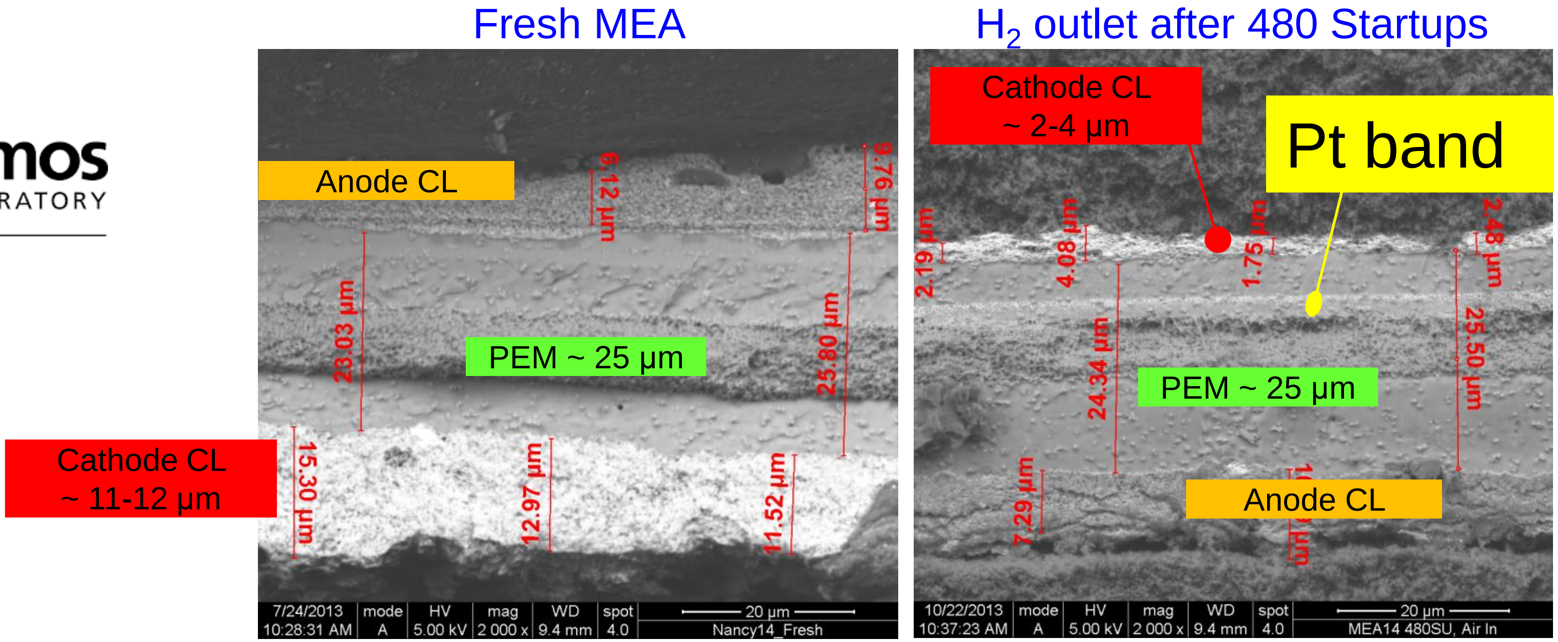

Only a few hours!

*Pictures from **D. Spernjak et al.,** see Komini Babu et al. Applied Energy, 2019 – Aging tests from the Université de Lorraine*

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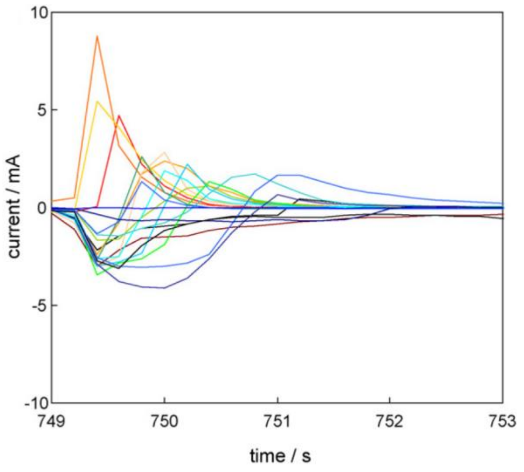
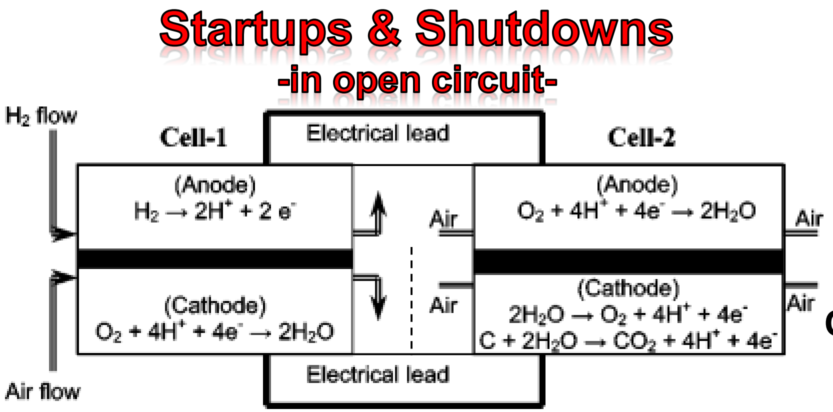
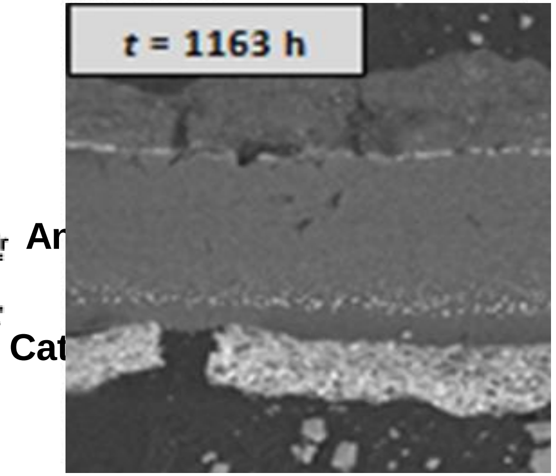
Hydrogen starvation → well known during FC start-up and shut-down



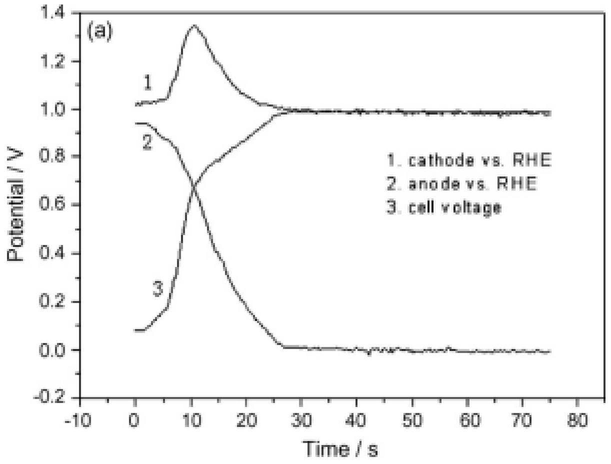
***Siroma et al.**, Journal of Power Sources 172 (2007) 155–162*



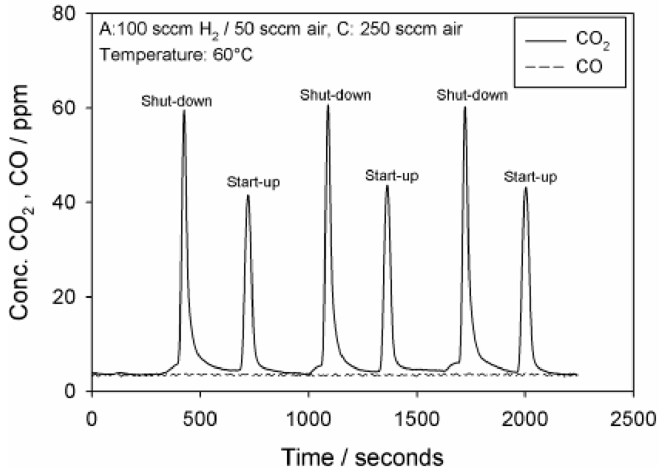
***Reiser et al.**, Electrochemical and Solid-State Letters, 8 (6) A273-A276 (2005)*



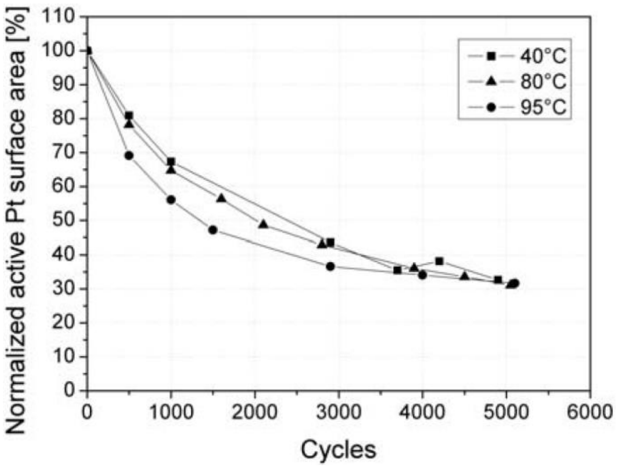
***Dubau et al.**, WIRES, 3 (2014)*



***Shen et al.**, Journal of Power Sources 189 (2009) 1114–1119*

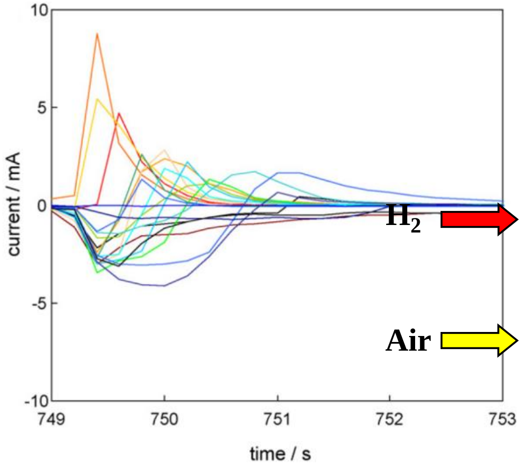


***Ofstad et al.**, ECS Transactions, 16 (2) 1301-1311 (2008)*



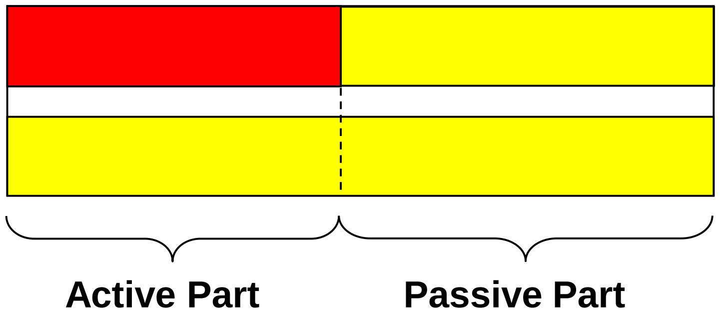
***Linse et al.**, ECS Transactions, 25 (1) 1849-1859 (2009)*

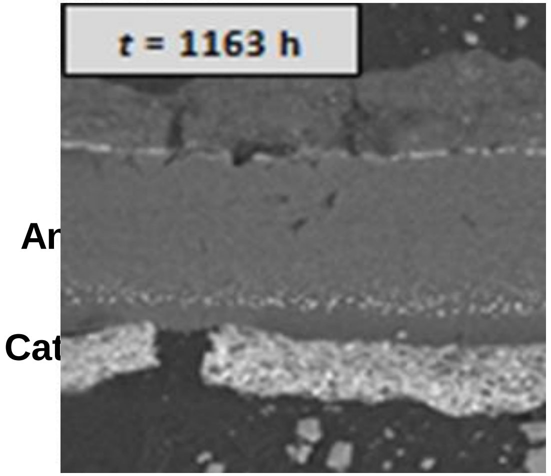
Hydrogen starvation → well known during FC start-up and shut-down



Siroma et al., *Journal of Power Sources 172 (2007) 155–162*

Startups & Shutdowns
-in open circuit-





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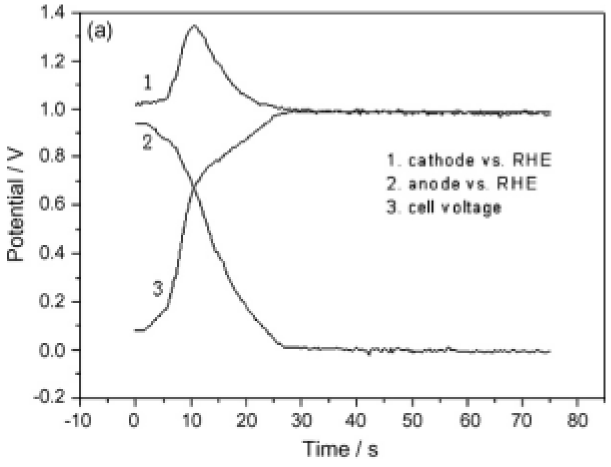

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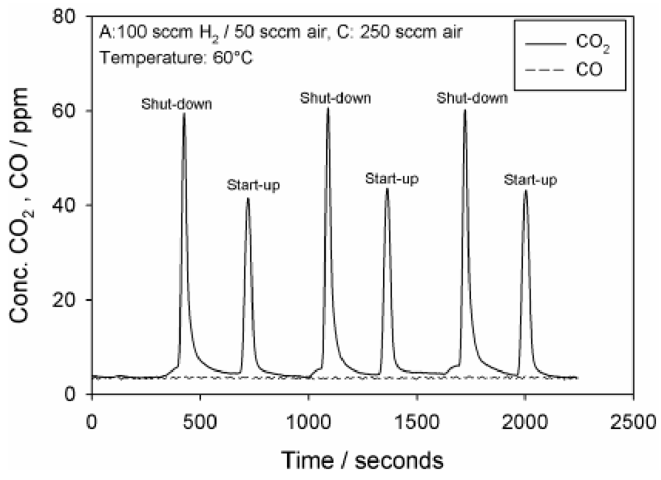

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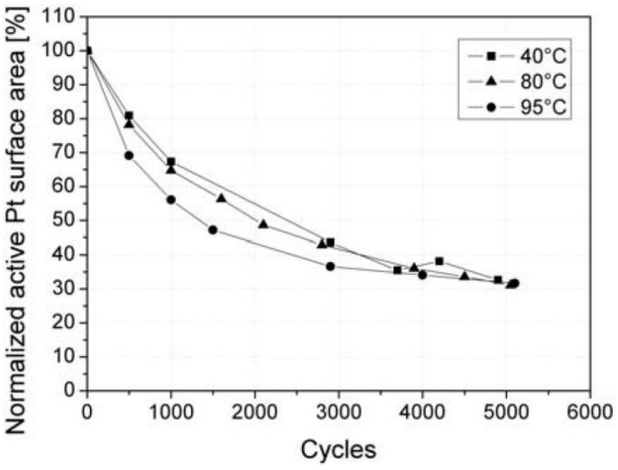
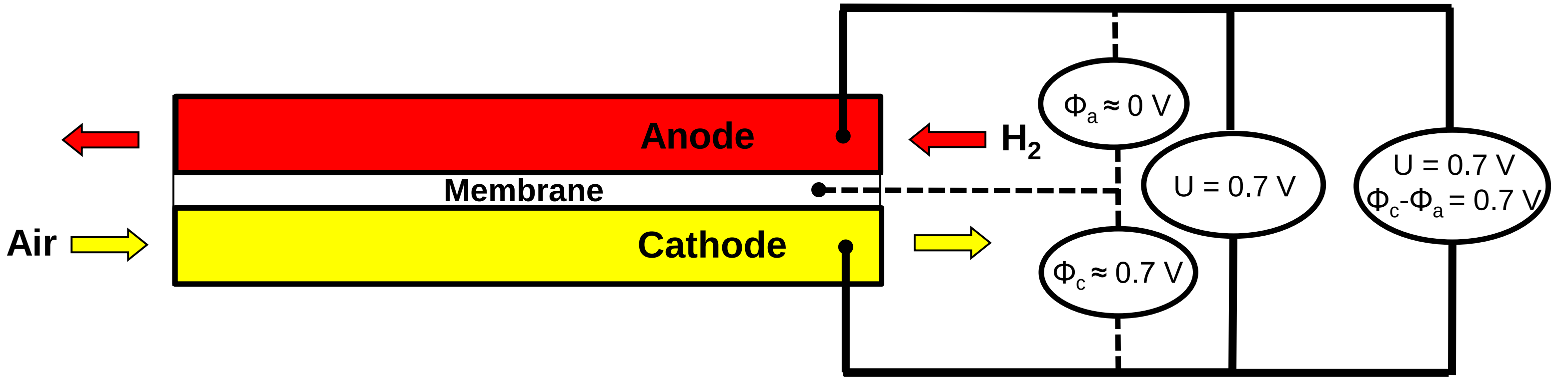


Linse et al., *ECS Transactions, 25 (1) 1849-1859 (2009)*

Hydrogen starvation → possibly a very common issue

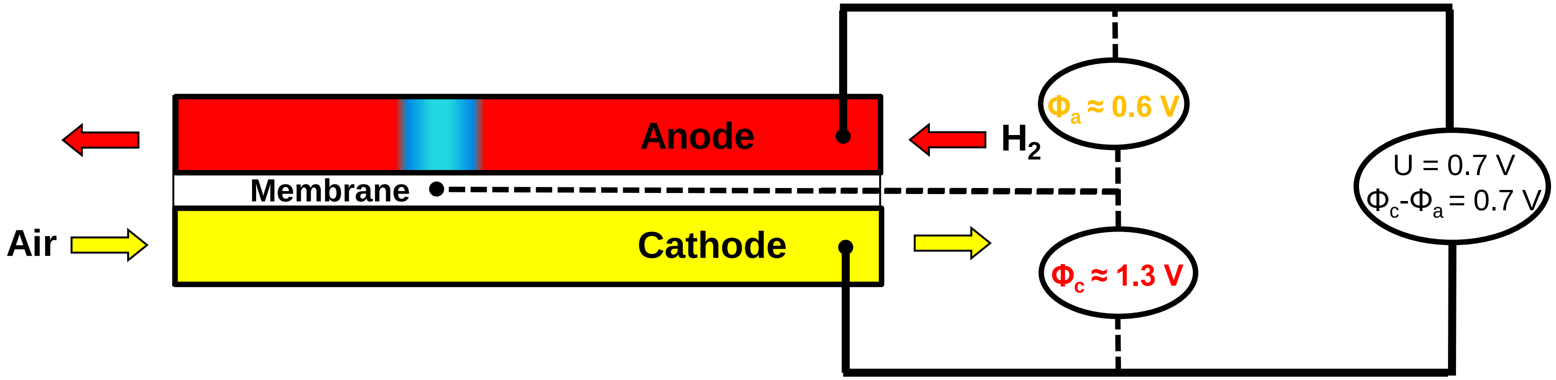
Regular operation



- Hydrogen keeps the anode potential low.
- The anode local potential rises if it is not properly fed with hydrogen.
- → possible high potential excursion at the cathode… because the anode and cathode potential are linked by the equation: $U_{cell} = E^{c} - E^{a} - r_{\Omega} i \approx E^{c} - E^{a}$ (we can neglect the ohmic losses for the sake of simplicity).

Hydrogen starvation → possibly a very common issue

Anode local flooding



- Hydrogen keeps the anode potential low.
- The anode local potential rises if it is not properly fed with hydrogen.
- → possibly high potential excursion at the cathode… because the anode and cathode potential are linked by the equation: $\mathrm{U}_{cell} = \mathrm{E}^{\mathrm{c}} - \mathrm{E}^{\mathrm{a}} - r_{\Omega} i \approx \mathrm{E}^{\mathrm{c}} - \mathrm{E}^{\mathrm{a}}$ (we can neglect the ohmic losses for the sake of simplicity).

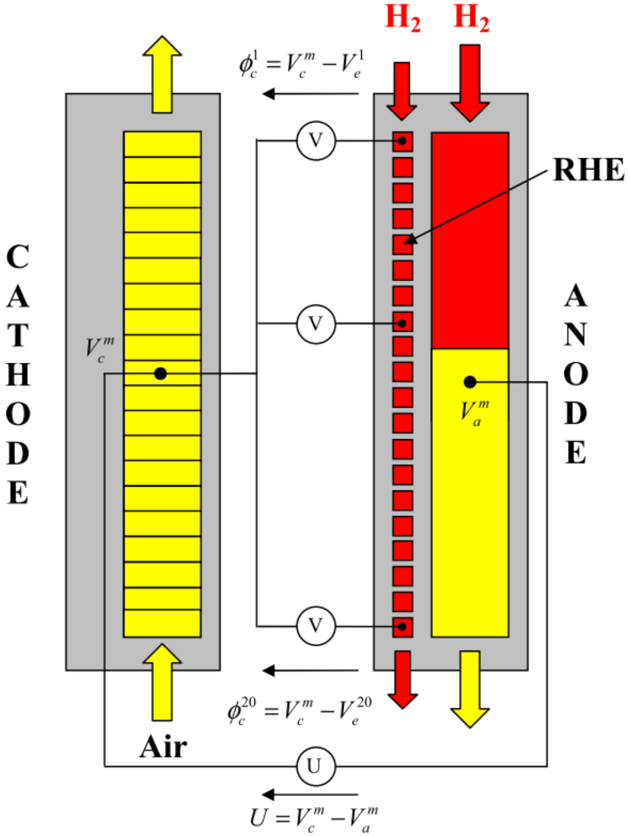
Hydrogen starvation → measurement of electrodes potentials

Measurement of

CO_2 emissions

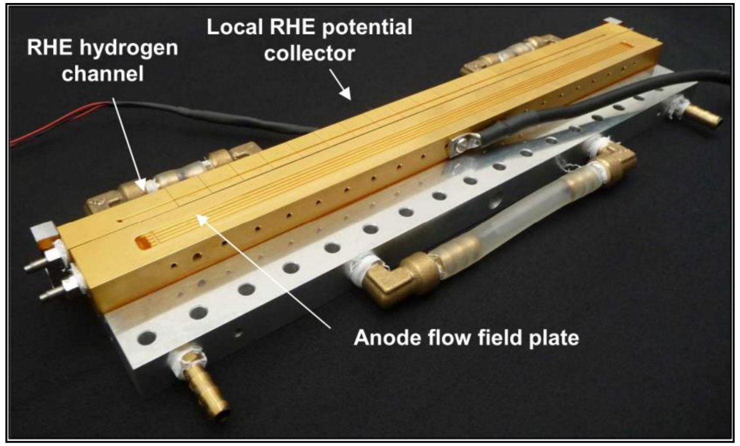
Infrared CO_2 analyzer

600 series by California Analytical Instruments, Inc



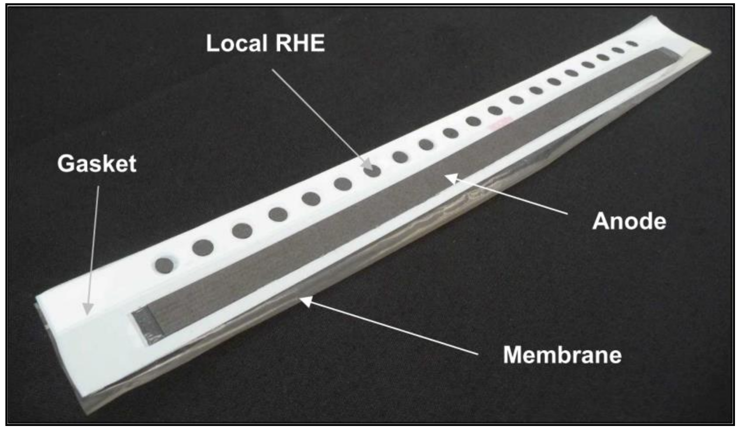
Segmented Cathode Current Collector (20 segments)

- ➢ Local current densities
- ➢ Local Cyclic Voltammetry
- ➢ *Local EIS*



Reference Hydrogen Electrodes

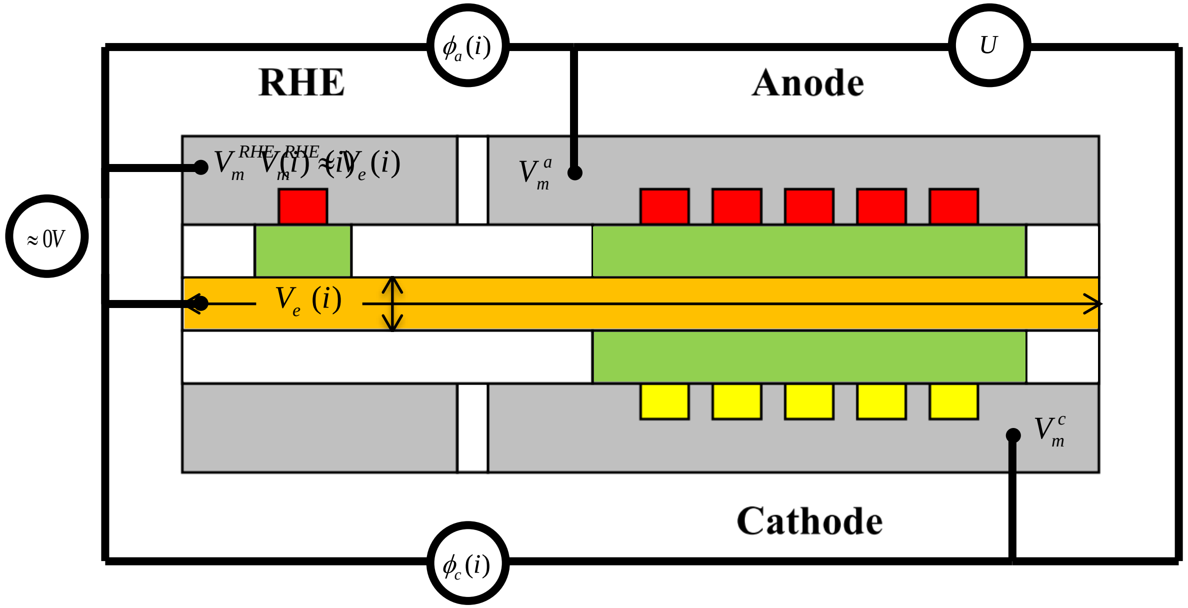
- ➢ Local Anode & Cathode Potentials



Hydrogen starvation → measurement of electrodes potentials

Local potential measurements → segmented cell with reference electrodes [1]

- **The "electrode potential" E^c or E^a are actually potential differences** between the *electron conductive phase* (carbon/catalyst) and the *ion conductive phase* (ionomer/membrane).
- → One must be able to measure the potential in the membrane…
- … and the best way to do that is to use additional electrodes on the membrane!



🟩 **Gas Diffusion Electrode (GDE)**

▨ **Flow field plate**

☐ **Gasket**

🟧 **Membrane**

(RHE): $\phi_{RHE}(i) = V_m^{RHE}(i) - V_e(i) \approx 0V$

Local anode potential: $\phi_a(i) = V_m^a - V_m^{RHE}(i)$

FC Voltage: U= $V_m^c - V_m^a$

Local cathode potential: $\phi_c(i) = U + \phi_a(i)$

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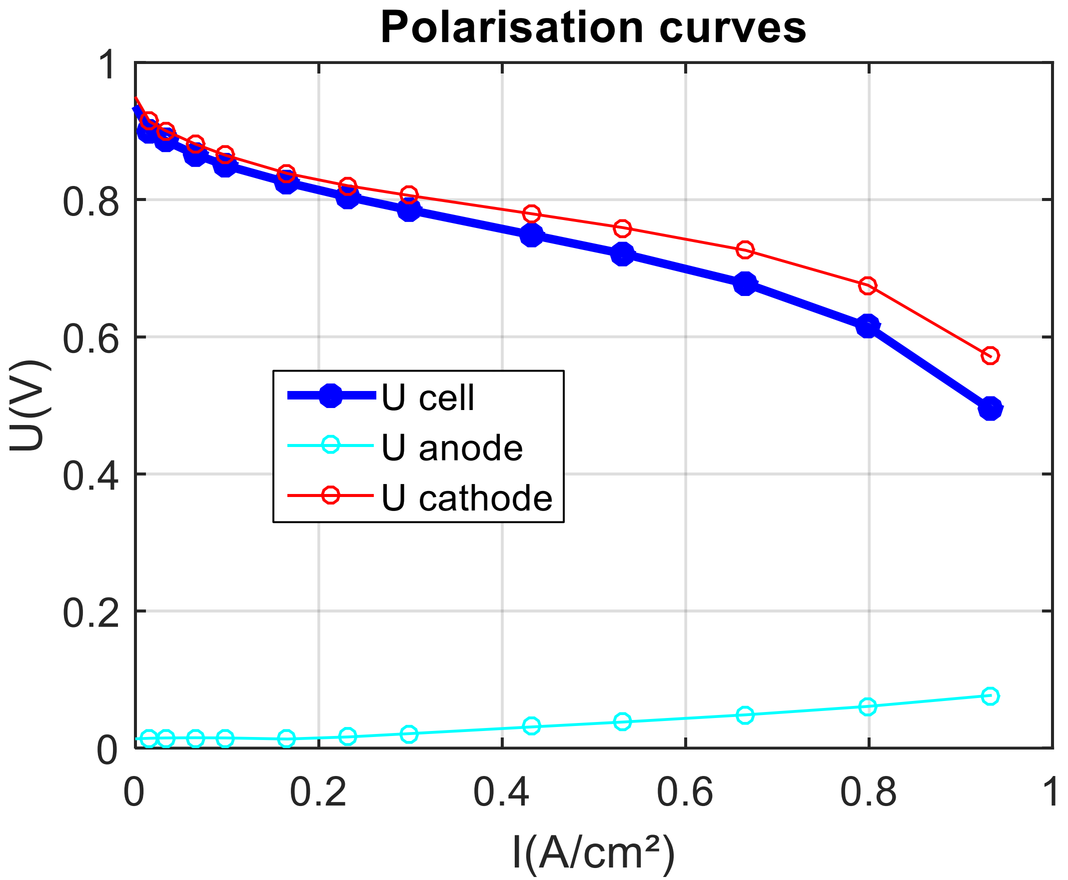
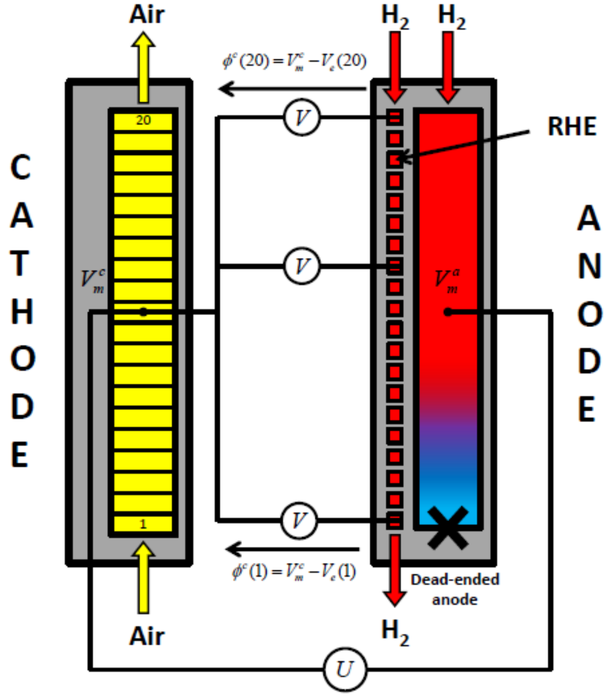
[1] ***Abbou et al.***, *J. Power Sources, 340 (2017)*

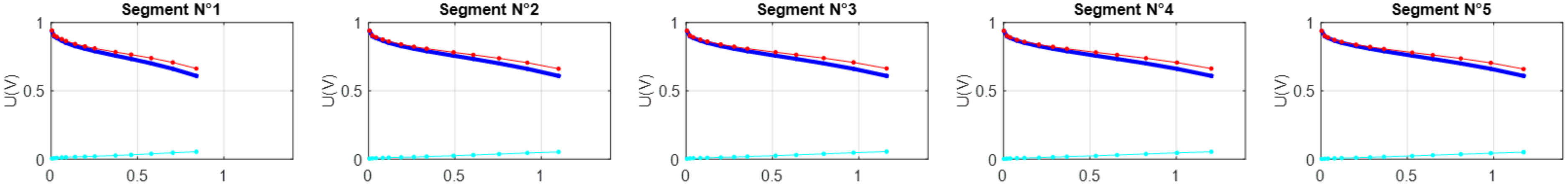
Hydrogen starvation → measurement of electrodes potentials

Local potential measurements in regular operating conditions

→ Slide 9

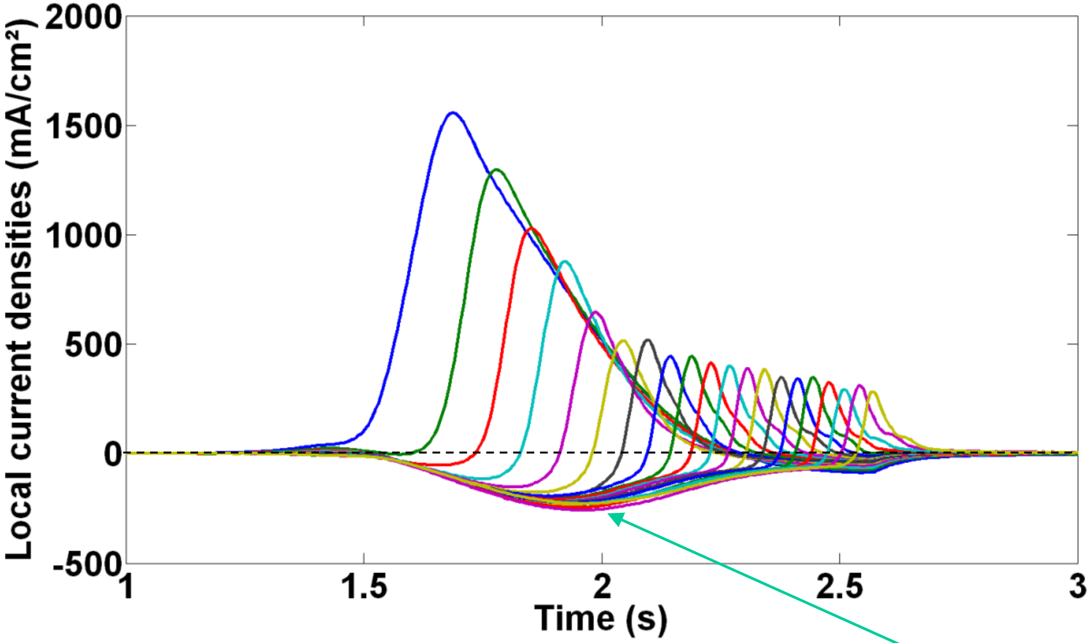






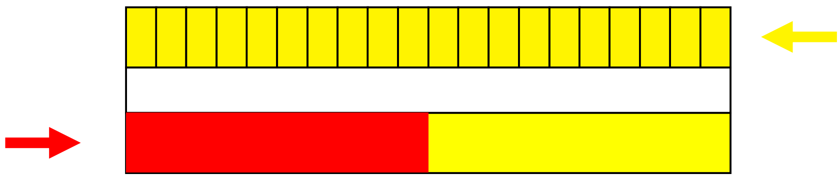
Hydrogen starvation → well known during FC start-up and shut-down

Start-up by Injection of 10 slph Hydrogen in Air



Reverse currents

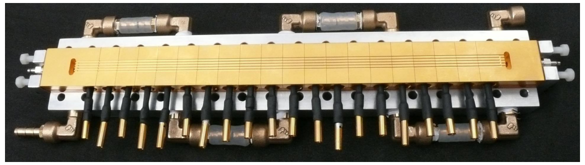
Cathode



Air In

H_2 In

Anode



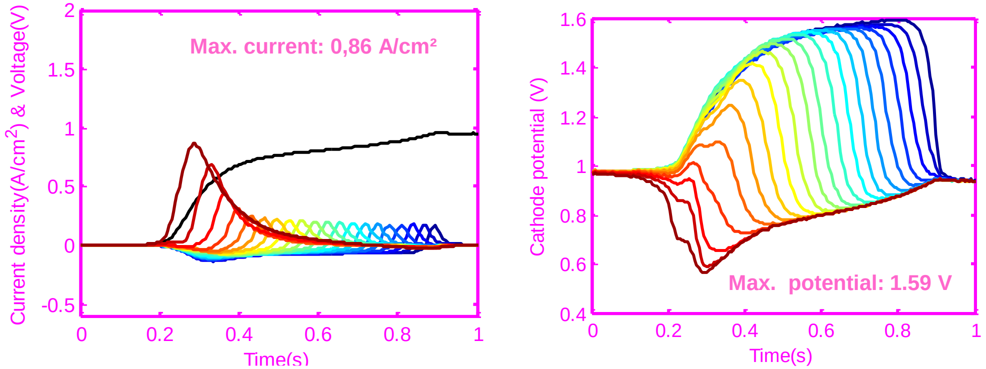
More about internal currents

Hydrogen starvation → well known during FC start-up and shut-down

Reference MEA by *IonPower* + 2 alternatives : high specific surface carbon, and highly graphitized carbon at the cathode

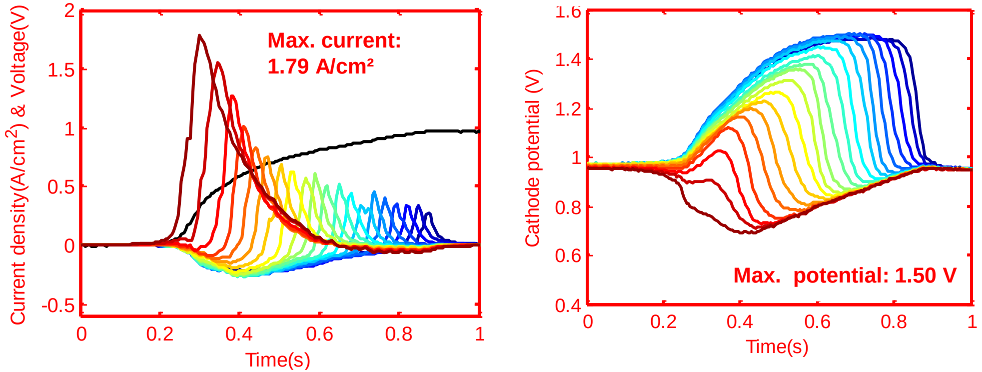
Highly graphitized carbon

Cathode: 0.2 mg/cm² Pt loading
Anode: 0.1 mg /cm² Pt loading
Cathode Carbon support: **110 m²/g**
Anode Carbon support: Vulcan XC72 240 m²/g
GDL: 210 µm SGL24 BC



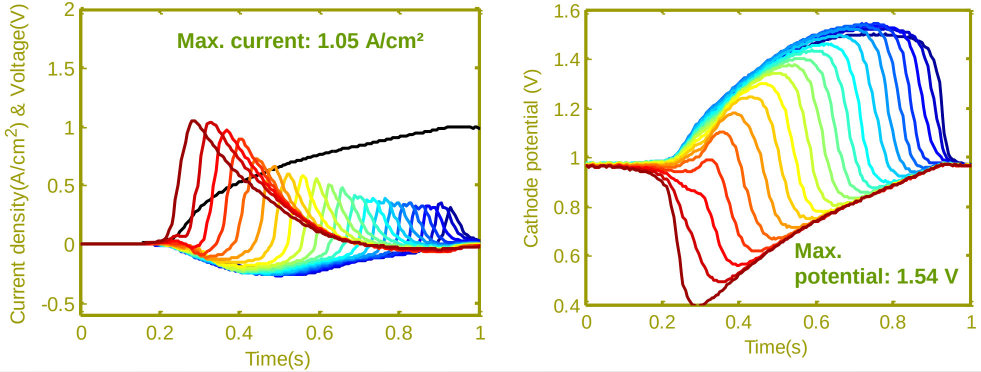
Reference MEA

Cathode: **0.2 mg/cm²** Pt loading
Anode: **0.1 mg/cm²** Pt loading
Cathode Carbon support: Vulcan XC72 **240 m²/g**
Anode Carbon support: Vulcan XC72 240 m²/g
GDL: 210 µm SGL24 BC



Higher specific surface carbon

Cathode: 0.2 mg/cm² Pt loading
Anode: 0.1 mg /cm² Pt loading
Cathode Carbon support: **800 m²/g**
Anode Carbon support: Vulcan XC72 240 m²/g
GDL: 210 µm SGL24 BC

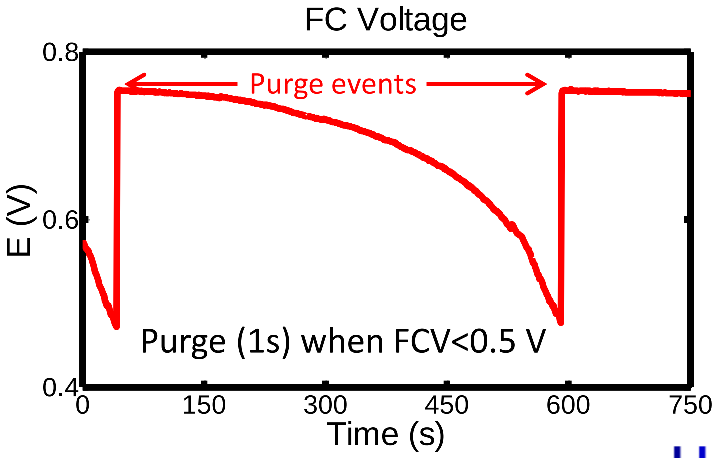


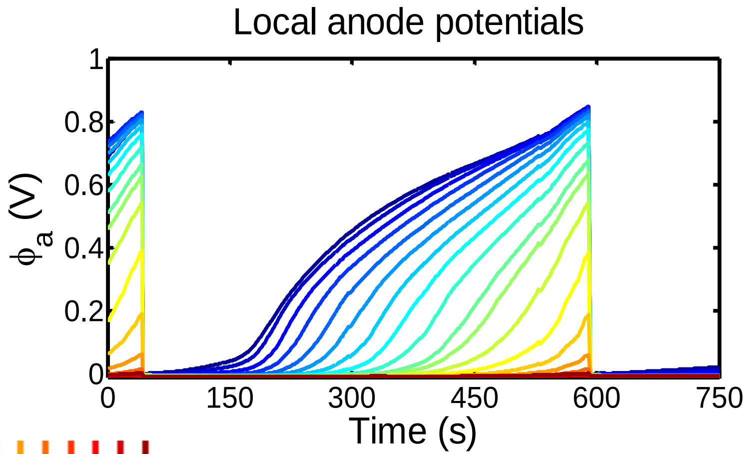
***O. Lottin** et al., ECS Transactions, 69 (2015).*

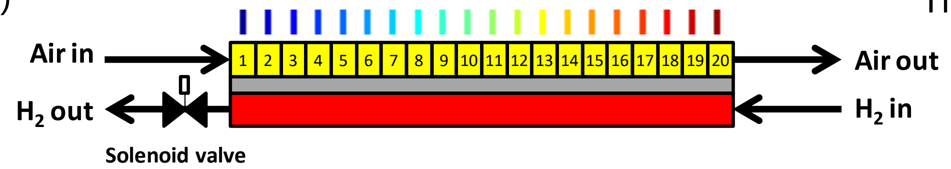
Hydrogen starvation → indeed a common issue ?

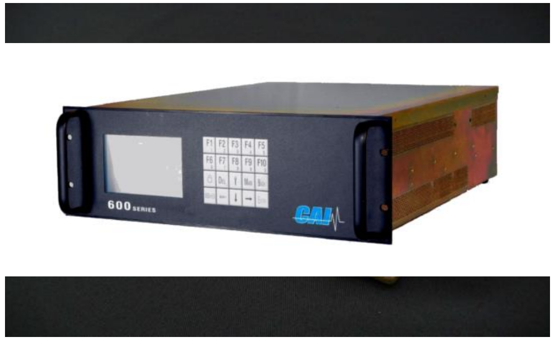
Dead-ended anode with long duration between purges

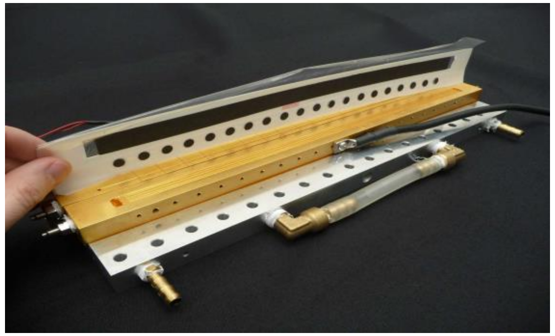
Dead-end/purge sequence – FC average current density set to 0.5 A/cm²









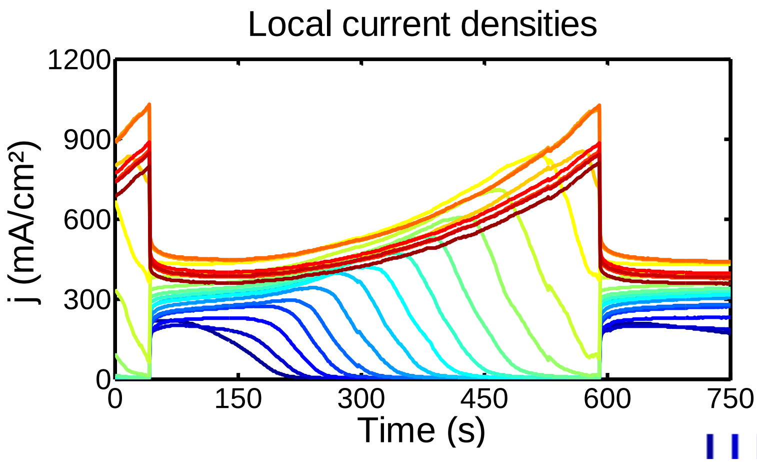


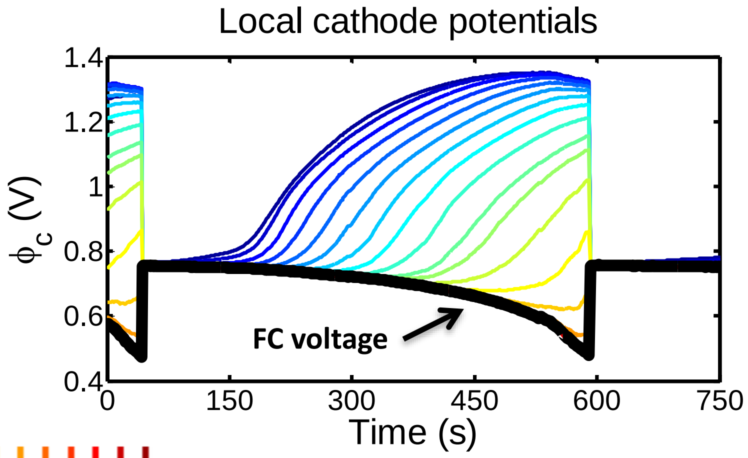
MEA + Reference electrodes

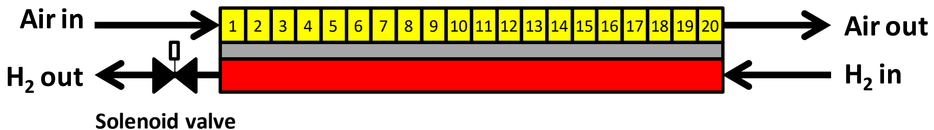
Hydrogen starvation → indeed a common issue ?

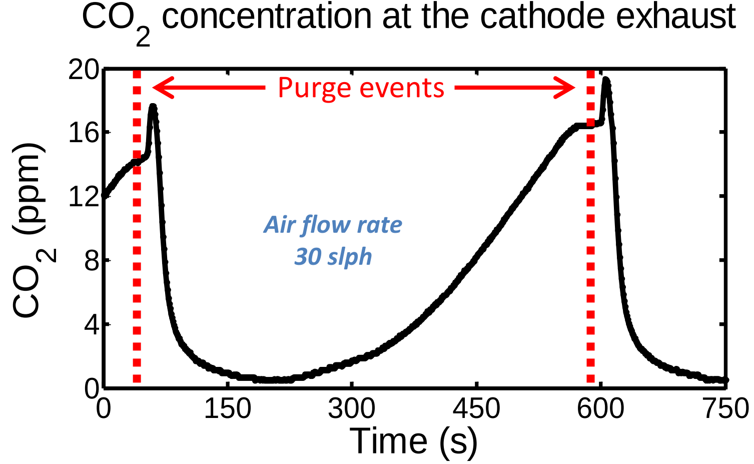
Dead-ended anode with long duration between purges

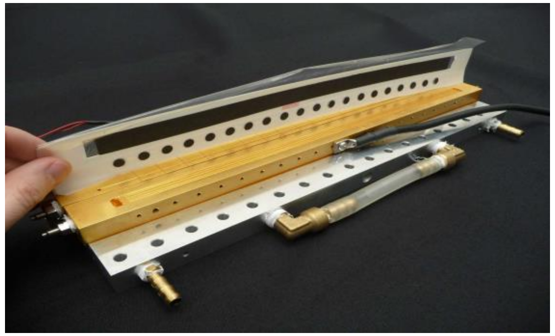
Dead-end/purge sequence – FC average current density set to 0.5 A/cm²









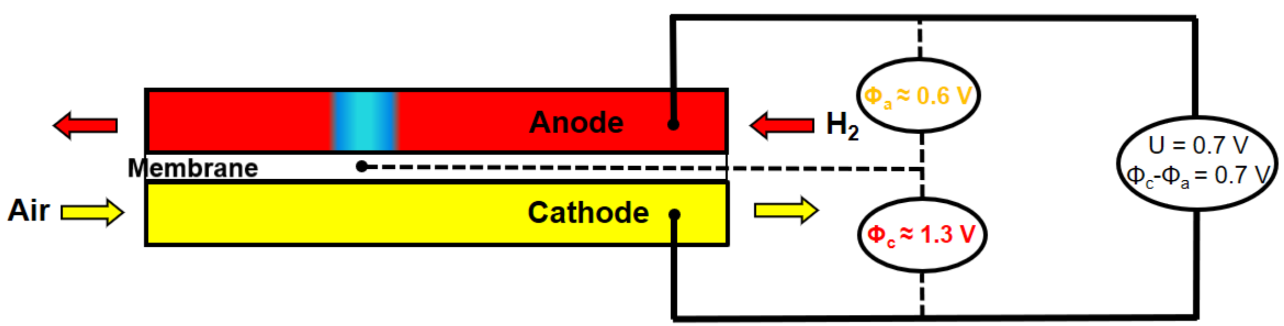


MEA + Reference electrodes

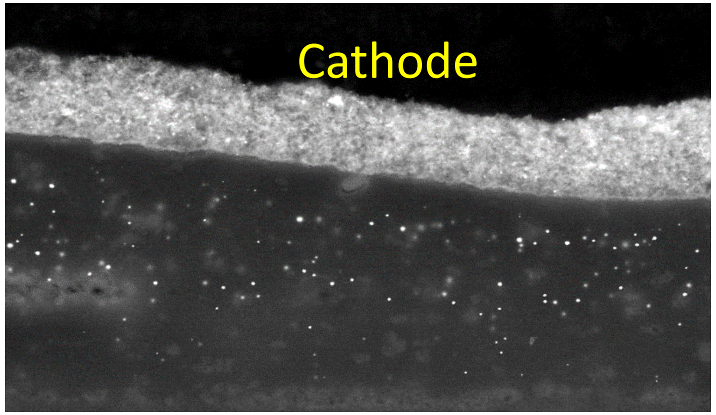
Fuel Cell aging and degradation → to keep in mind

- The low durability of PEMFC is still one of the main limitation to their widespread commercialization.
- Electrode materials degradation is quite well understood in regular operating conditions but there are other cases: *fuel starvation* with *high potential excursion* → leads to much stronger degradations!
- The point that needs to be clarified → is *fuel starvation* a *specific* (start-up, shut-down…) or *common* phenomenon?

Anode local flooding



This is a scenario, not a result!

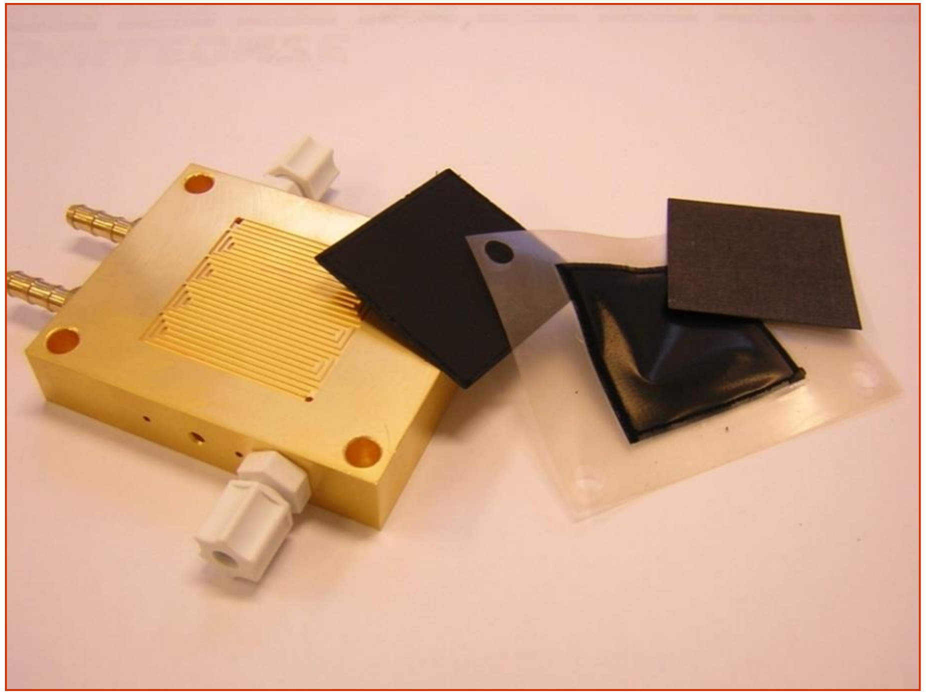


… but there is evidence of Pt in the membrane after FC "regular" operation (constant current).

Picture from L. Dubau - LEPMI

Mechanisms of high potential excursions in fuel cells

Thank you for your attention!

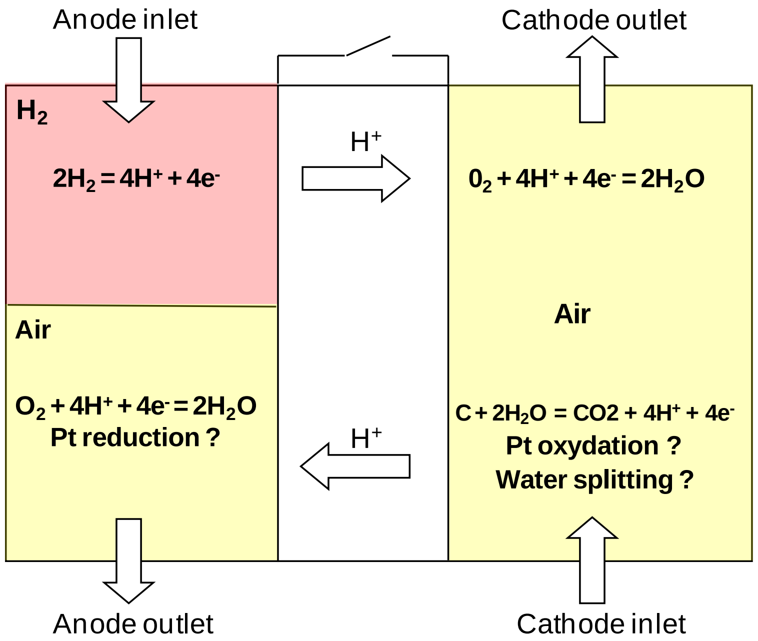


Laboratoire Energies & Mécanique Théorique et Appliquée

Unité Mixte de Recherche *Université de Lorraine, CNRS*

Experimental set-up → segmented cell with reference electrodes

Segmented cell → internal (forward and reverse) currents



Anode inlet

Cathode outlet

H_2

$2H_2 = 4H^+ + 4e^-$

H^+

$0_2 + 4H^+ + 4e^- = 2H_2O$

Air

Air

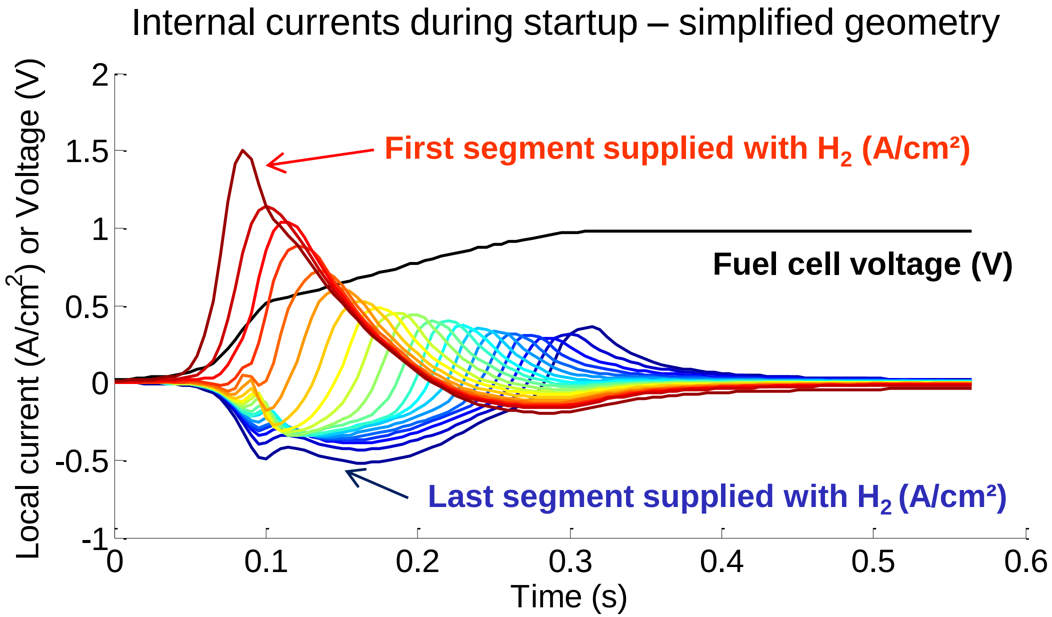
$O_2 + 4H^+ + 4e^- = 2H_2O$
Pt reduction ?

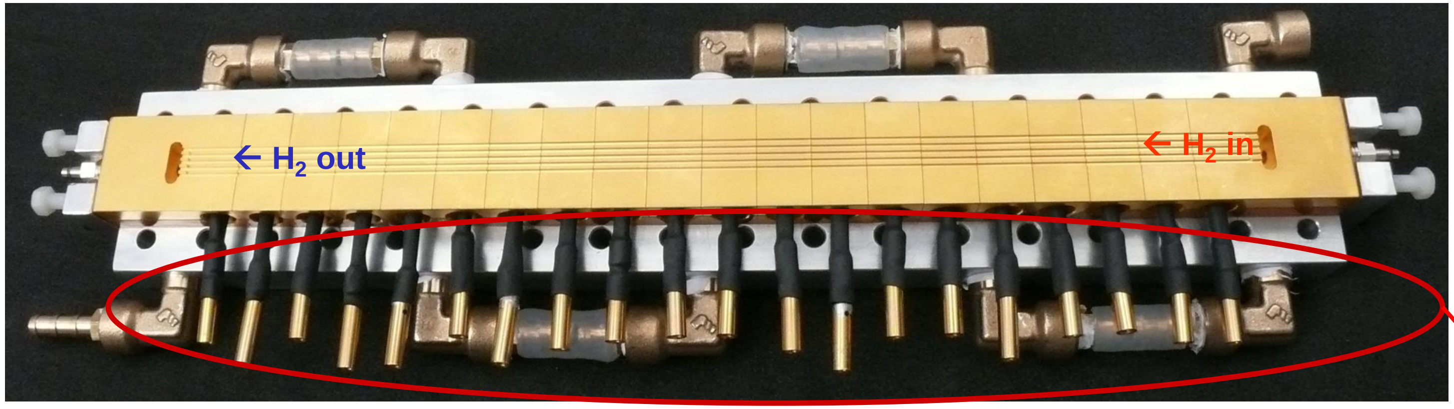
H^+

$C + 2H_2O = CO2 + 4H^+ + 4e^-$
Pt oxydation ?
Water splitting ?

Anode outlet

Cathode inlet





J. ***Dillet*** *et al., J. Power Sources, 2014*

Terminals used to collect and then measure the local current densities but ***the potential of whole segmented plate remains homogeneous***.

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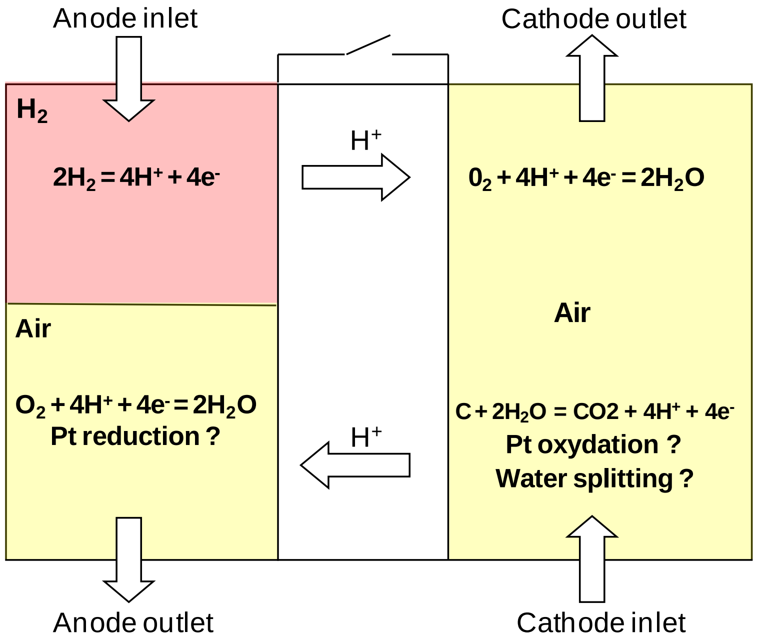
Experimental set-up → segmented cell with reference electrodes

Reverse currents → *1:* with any flow field geometry



Anode inlet

Cathode outlet

H_2

$2H_2 = 4H^+ + 4e^-$

H^+

$O_2 + 4H^+ + 4e^- = 2H_2O$

Air

Air

$O_2 + 4H^+ + 4e^- = 2H_2O$

Pt reduction ?

H^+

$C + 2H_2O = CO2 + 4H^+ + 4e^-$

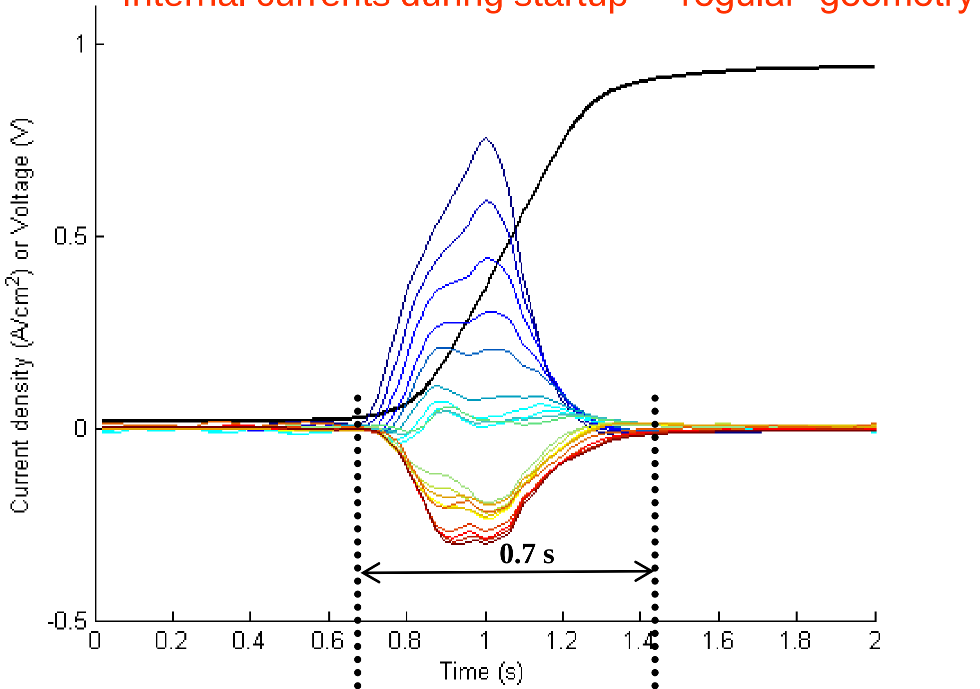
Pt oxydation ?

Water splitting ?

Anode outlet

Cathode inlet

Internal currents during startup – “regular” geometry



See also **Z. Siroma** et al., J. Power Sources, 2007

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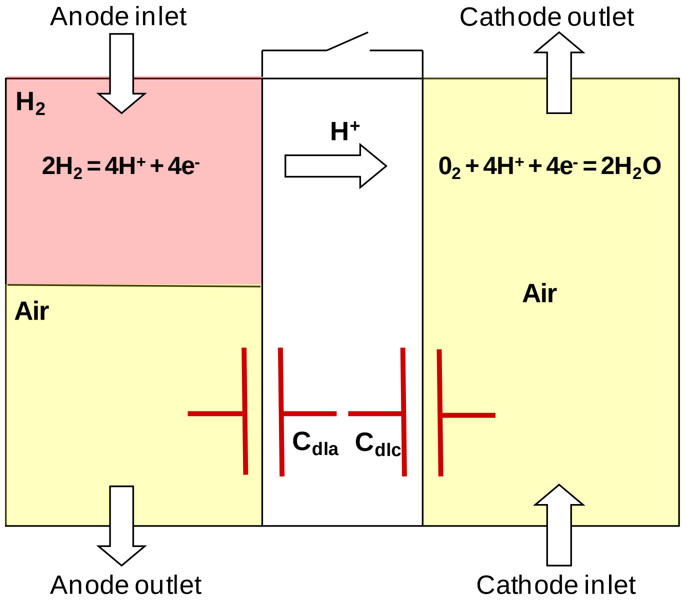
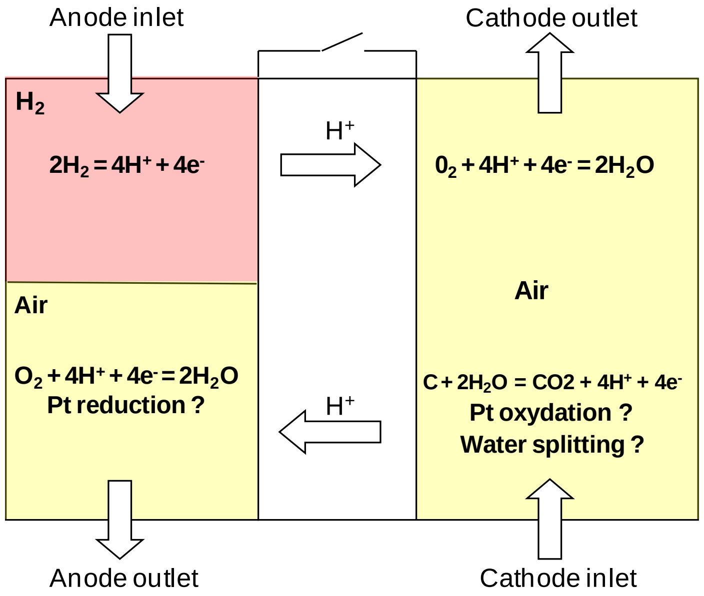
Experimental set-up → segmented cell with reference electrodes

Reverse currents → 2: capacitive + faradic contributions





Important features about the reverse currents

- Capacitive contribution → charge/discharge double layer capacities
- Faradic contribution → reduction reactions in the anode compartment and oxidation reactions in the cathode compartment
- The extent of these various faradic contributions is not well known

***A. Lamibrac** et al., J. Power Sources, 2011.*
***J. Dillet** et al., J. Power Sources, 2014.*

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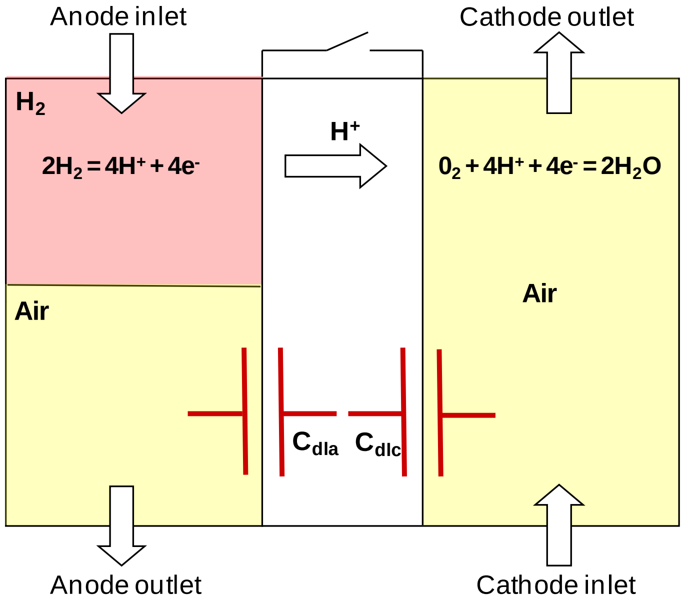
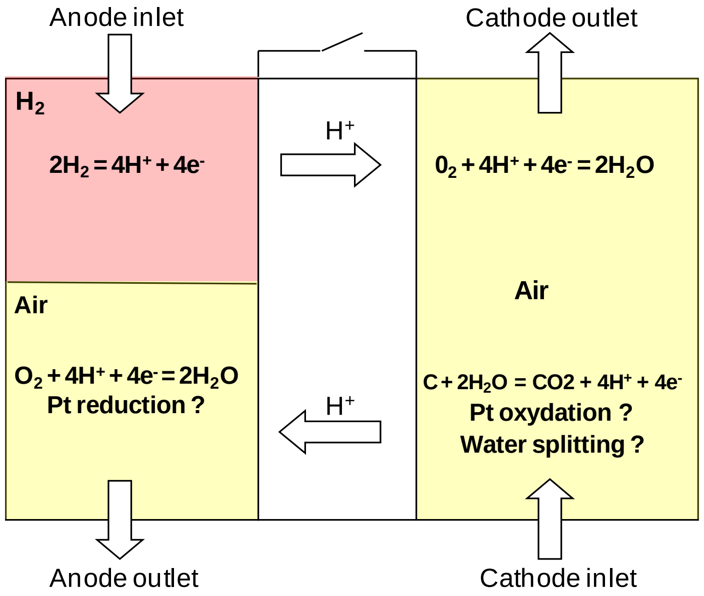
Experimental set-up → segmented cell with reference electrodes

Reverse currents → 3: a matter of time…





Important features about the reverse currents

- Their intensity increases with the gas injection velocity…
- … but the total charge passing between the active and passive parts decreases.
- → the main factor governing carbon corrosion is the common residence time of air and H_2 in the anode compartment.

A. Lamibrac *et al., J. Power Sources, 2011.*
J. Dillet *et al., J. Power Sources, 2014.*

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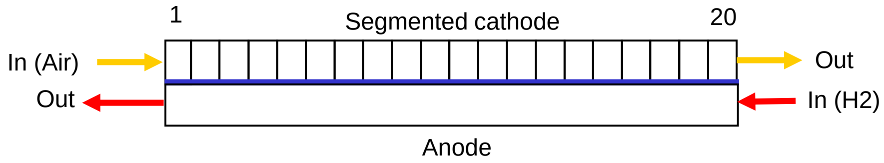
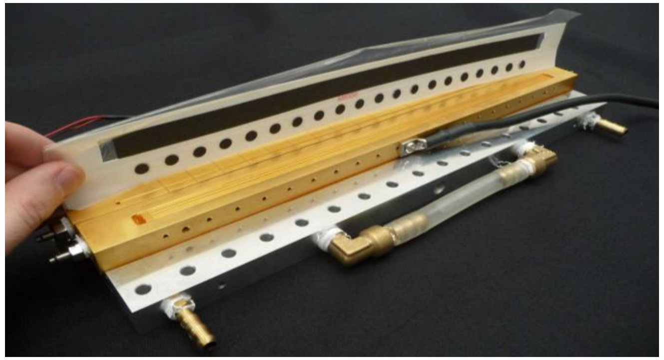
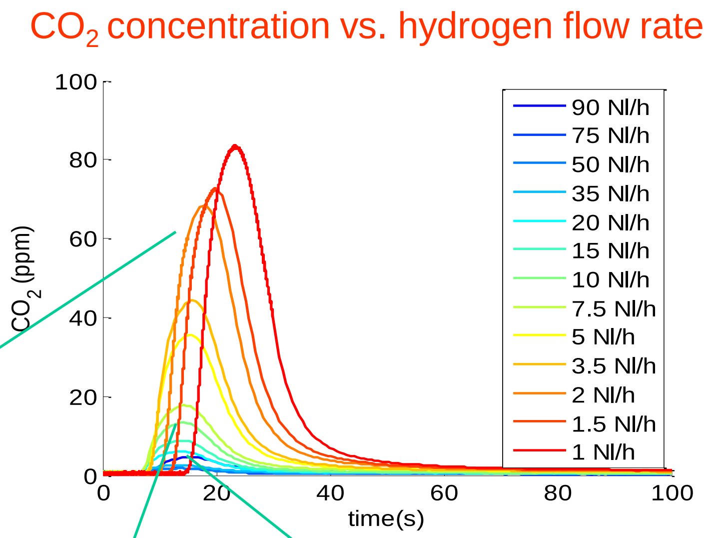
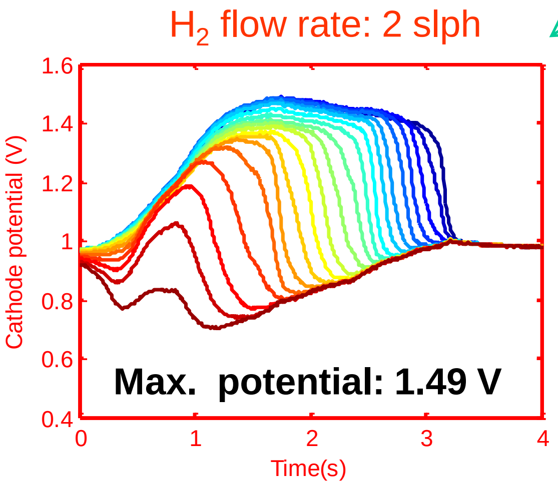
Experimental set-up → segmented cell with reference electrodes

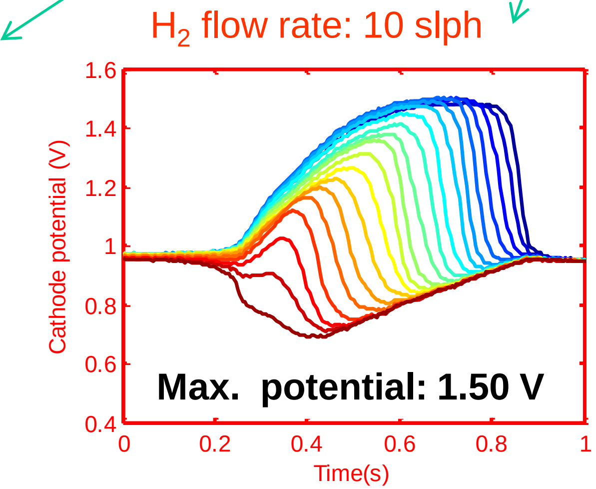
Reverse currents → 3: a matter of time…

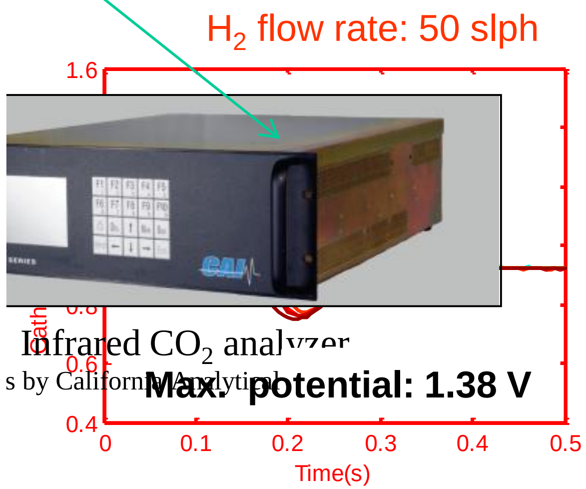












Infrared CO$_2$ analyzer
s by California Analytical

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The electrodes… a quite complex component

What are they here for?

- To **catalyze** the half-reactions
- To **transport electrons**
- To **transport ions**
- To let the **reactants and products flow** to the active sites (catalyst)

→ At least 3 different materials or 3 phases

- **Platinum** to catalyze the reactions and transport electrons
- **Some ionomer** (electrolyte material) to transport ions
- **Gas** (and sometimes **liquid**) reactants/products

→ Since Pt is (too much) expensive

- **3 solid materials**: carbon, platinum, and the ionomer
- … forming a **porous structure** to let the reactants/products flow toward the CL

Getty Images/iStockphoto

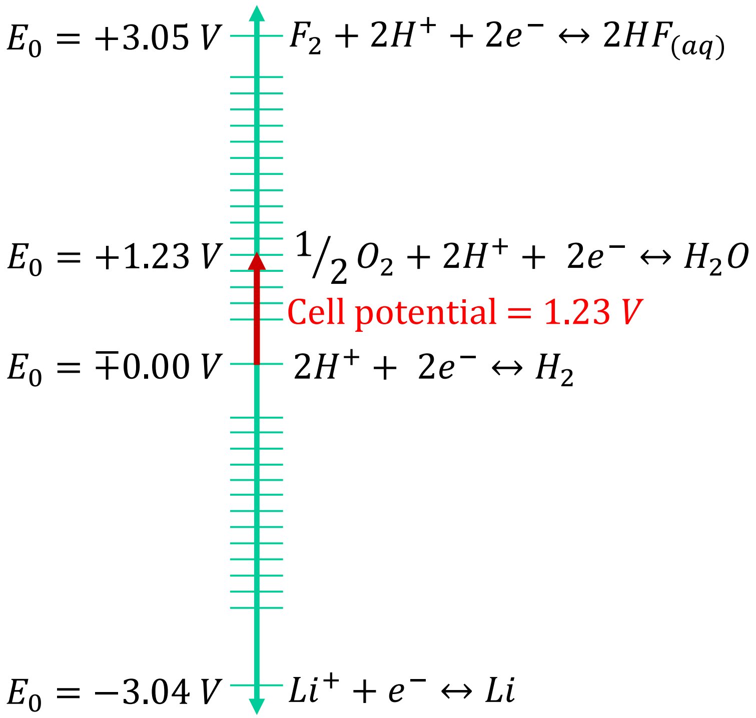
→ Mass transport is the main *non-electrochemical* issue limiting fuel cell performance

→ Mass transport is not (or less) an issue in battery electrodes… because products and reactants stay where they are

→ The three phases description is actually… not (fully) true

Introduction → Proton Exchange Membrane Fuel Cells (PEMFC)

Proton Exchange Membrane Fuel Cells (PEMFC) *vs.* Alkaline Fuel Cells (AFC)



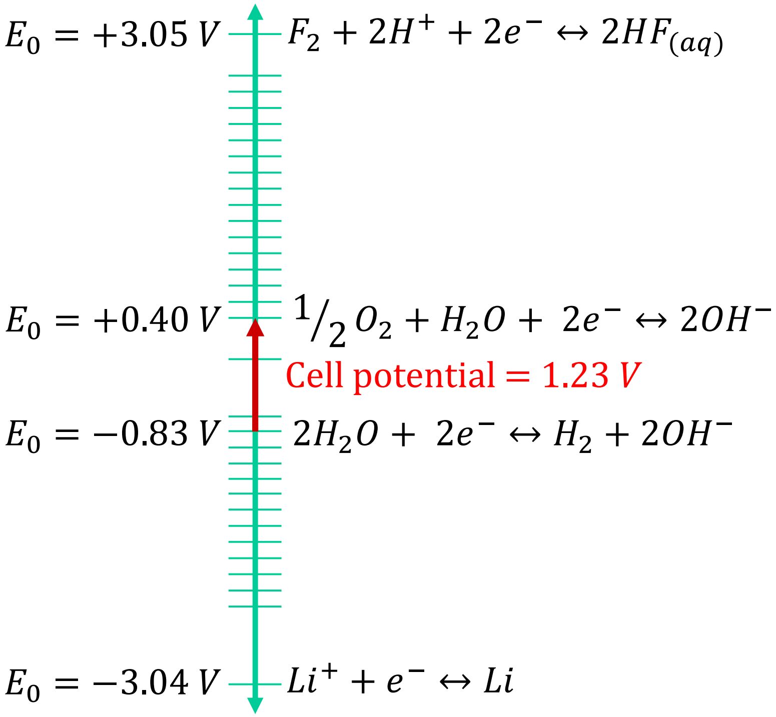
$E_0 = +3.05\ V$ — $F_2 + 2H^+ + 2e^- \leftrightarrow 2HF_{(aq)}$

$E_0 = +1.23\ V$ — ${}^1/_2\, O_2 + 2H^+ + 2e^- \leftrightarrow H_2O$

$\text{Cell potential} = 1.23\ V$

$E_0 = \mp 0.00\ V$ — $2H^+ + 2e^- \leftrightarrow H_2$

$E_0 = -3.04\ V$ — $Li^+ + e^- \leftrightarrow Li$



$E_0 = +3.05\ V$ — $F_2 + 2H^+ + 2e^- \leftrightarrow 2HF_{(aq)}$

$E_0 = +0.40\ V$ — ${}^1/_2\, O_2 + H_2O + 2e^- \leftrightarrow 2OH^-$

$\text{Cell potential} = 1.23\ V$

$E_0 = -0.83\ V$ — $2H_2O + 2e^- \leftrightarrow H_2 + 2OH^-$

$E_0 = -3.04\ V$ — $Li^+ + e^- \leftrightarrow Li$

- ♦ Higher anode and cathode potentials
  - → expensive materials (lower efficiency at low current)
- ♦ Acid electrolyte (H^+)
  - → very efficient ionomer membranes
  - → can be fed with air instead of pure O_2

- ♦ Low anode and cathode potentials
  - → cheap materials (better efficiency)
- ♦ Alkaline electrolyte (OH^-)
  - → no stable anionic membrane
  - → sensitive to CO_2 ($CO_2 + 2OH^- \rightarrow CO_3^{2-} + H_2O$)