# FURTHER-FC

**F**urther **U**nderstanding **R**elated to **T**ransport limitations at **H**igh current density towards future **E**lect**R**odes for **F**uel **C**ells

Co-funded by the European Union

# Discussion on MEA performance limitations

## Final Workshop

# OUTLINE

General considerations

CCM losses

Proton transport resistance thanks to EIS measurements

What else can we extract from EIS?

Oxygen transport resitance thanks to LCA measurments

Decoupling electrokinetic from transport limitations

Conclusion

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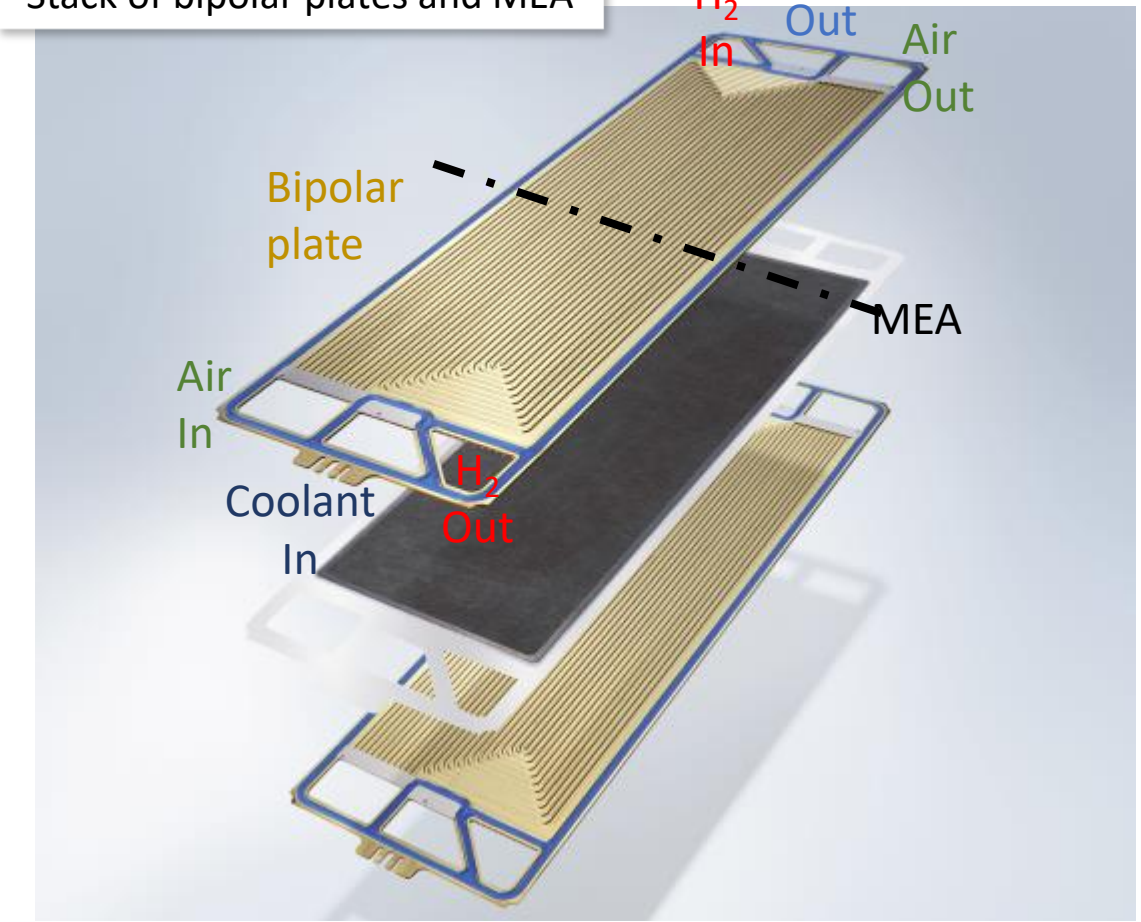
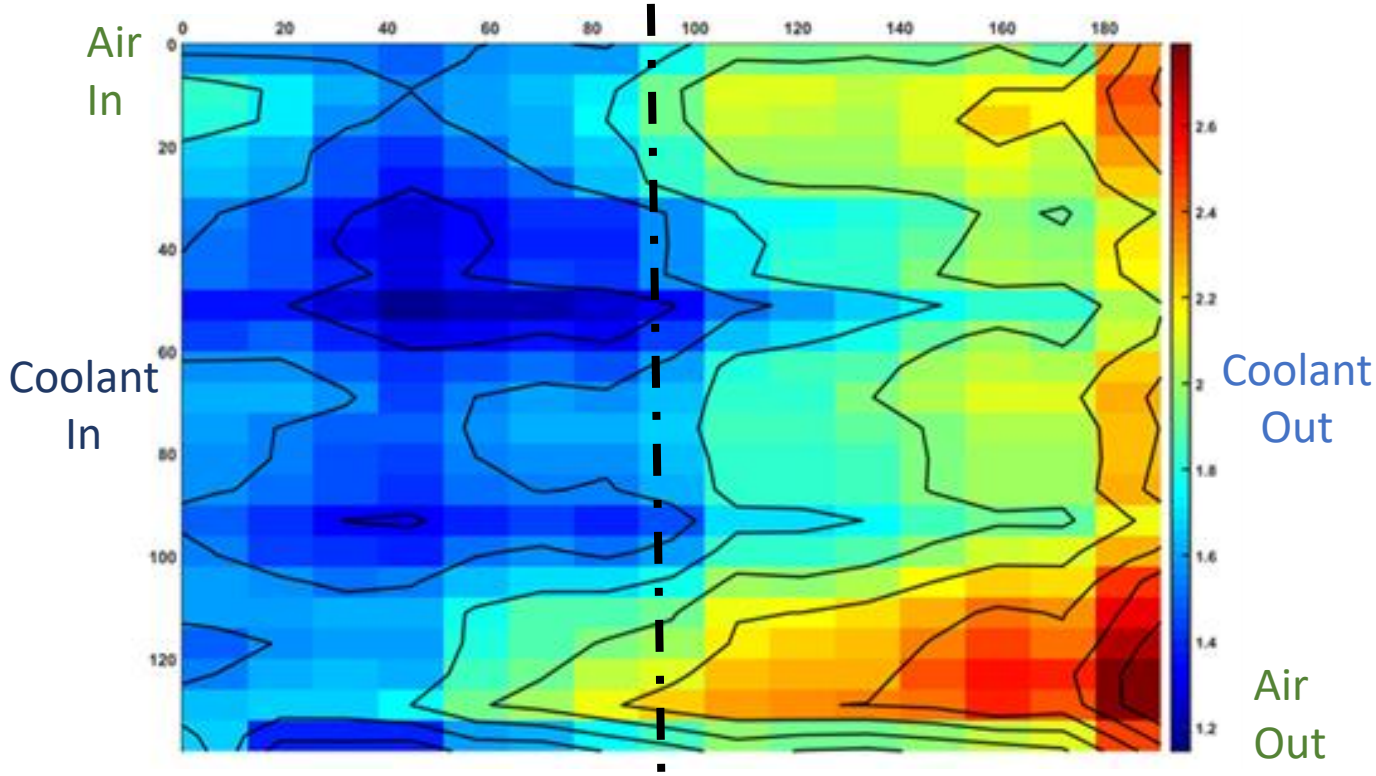
# Heterogeneities at the cell level

Stack of bipolar plates and MEA



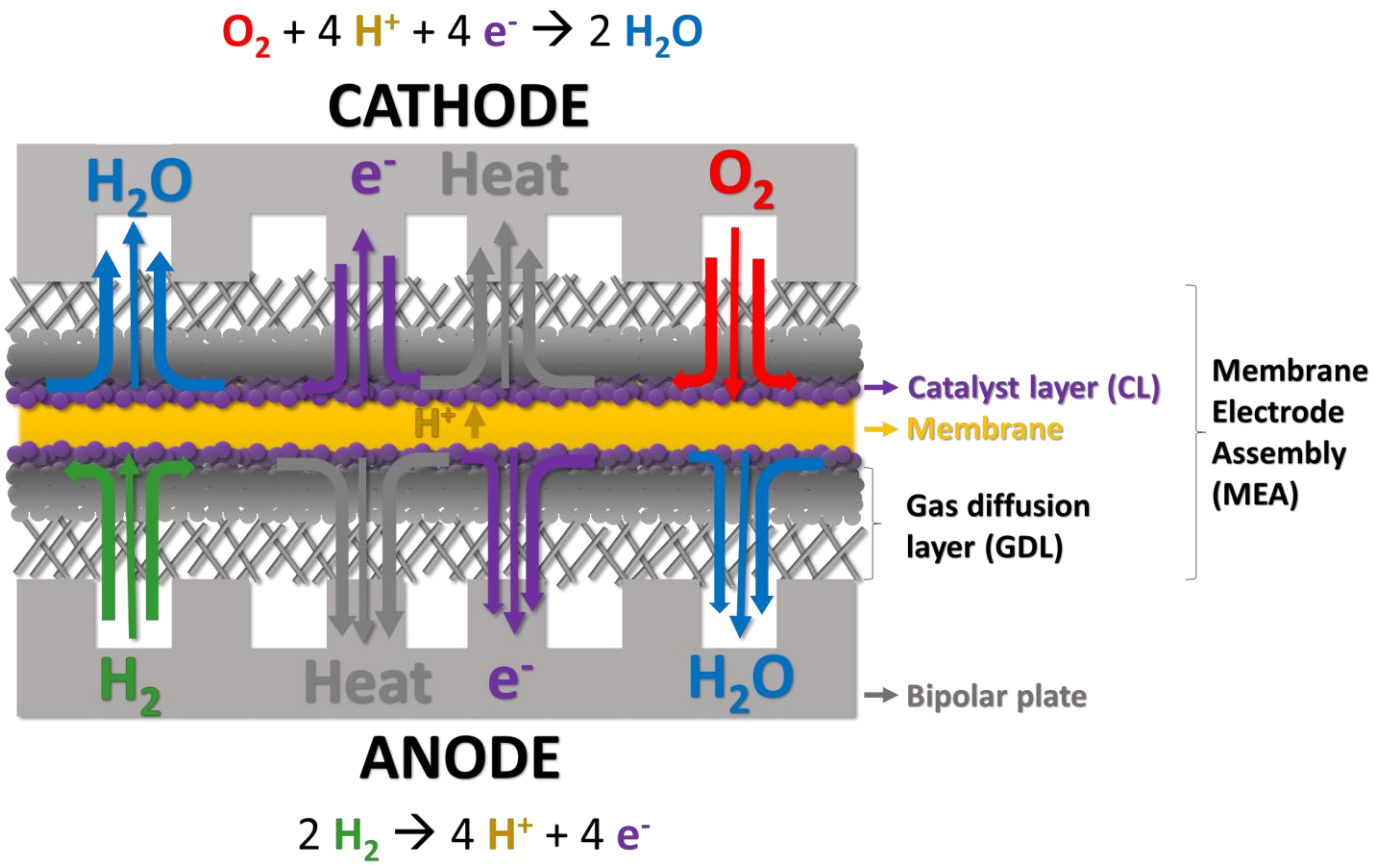

Current density distribution




**Heterogenous operation from inlet to outlet** even in well designed stack (homogeneous compression) because of evolution of concentration of reactants and product and temperature

# Heterogeneities at the cell level

3D schematic of bipolar plate and MEA cross-section in the stack active area




**Homogeous operation between each land/channel repeat unit** in a well designed stack (homogeneous distribution of products and evacuation of water in all the channel & homogeneous compression)

**Heterogeneous operation at the land/channel scale** because of GDL transport limitations

Heat transport limitation in GDL

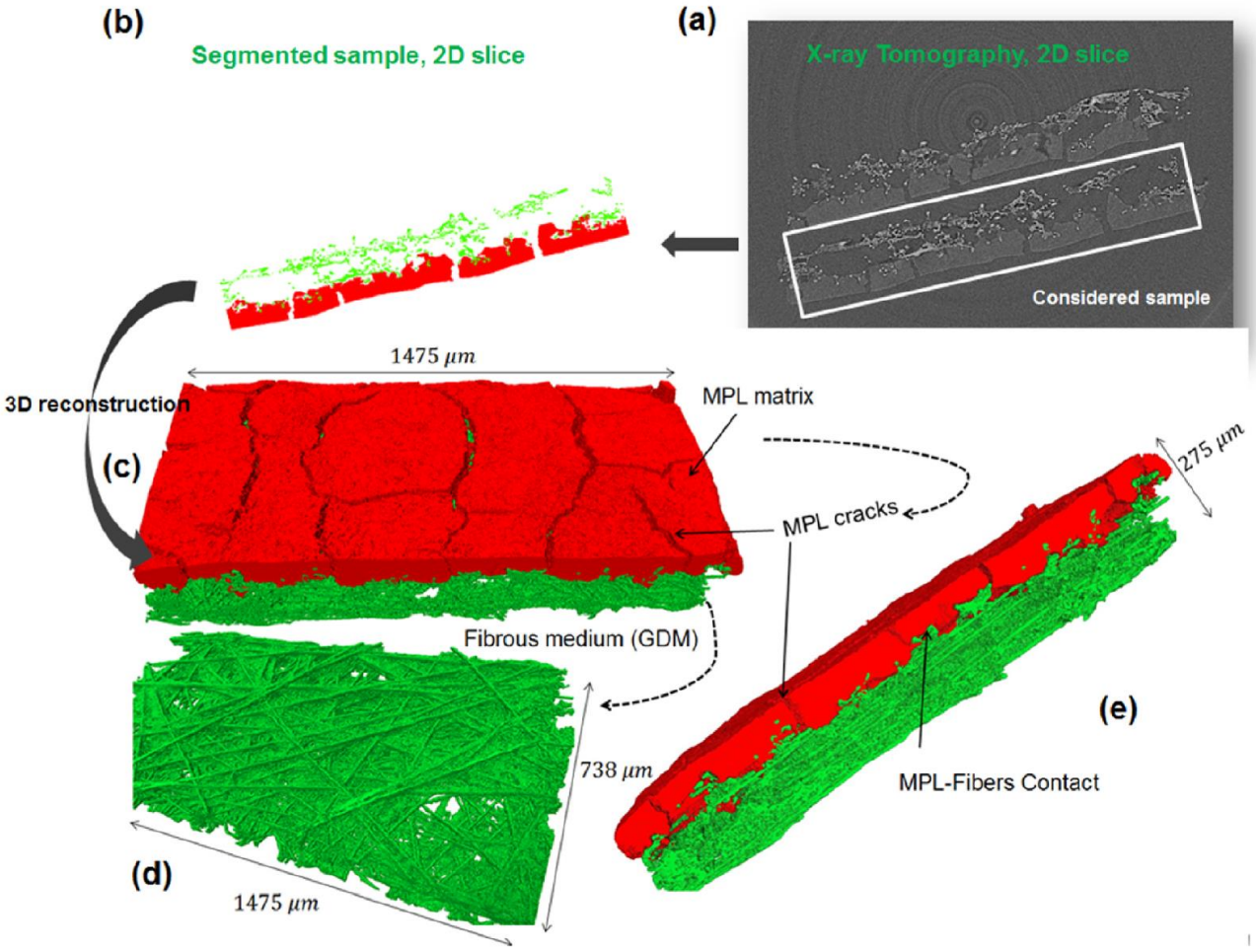
→ T gradient between membrane and bipolar plate

Several °C at high current density

→ Lower RH in the CCM than in the BP

# GDL transport properties

## A multiscale approach combining two imagery techniques




## From GDL structure to transport properties

MPL structure from FIB-SEM
+ GDM structure from X-Ray CT
→ Computation of $O_2$ effective diffusion tensor (MPL)
→ Through plane effective diffusion coefficient
→ Integration in the 2D cell model : through-plane + along channel (land/channel averaged)

- Knudsen diffusion is important in the MPL matrix.
- The MPL has a quite significant impact on the GDM-MPL diffusion property compared to the GDM alone.
- The MPL-GDM overlap region has a quite noticeable impact on the diffusion resistance of the assembly.
- MPL cracks have a relatively weak impact on the GDM-MPL assembly diffusive resistance, with a reduction by about 10%.

Demanding and must me done for each GDL

Next step: include the two phase flow

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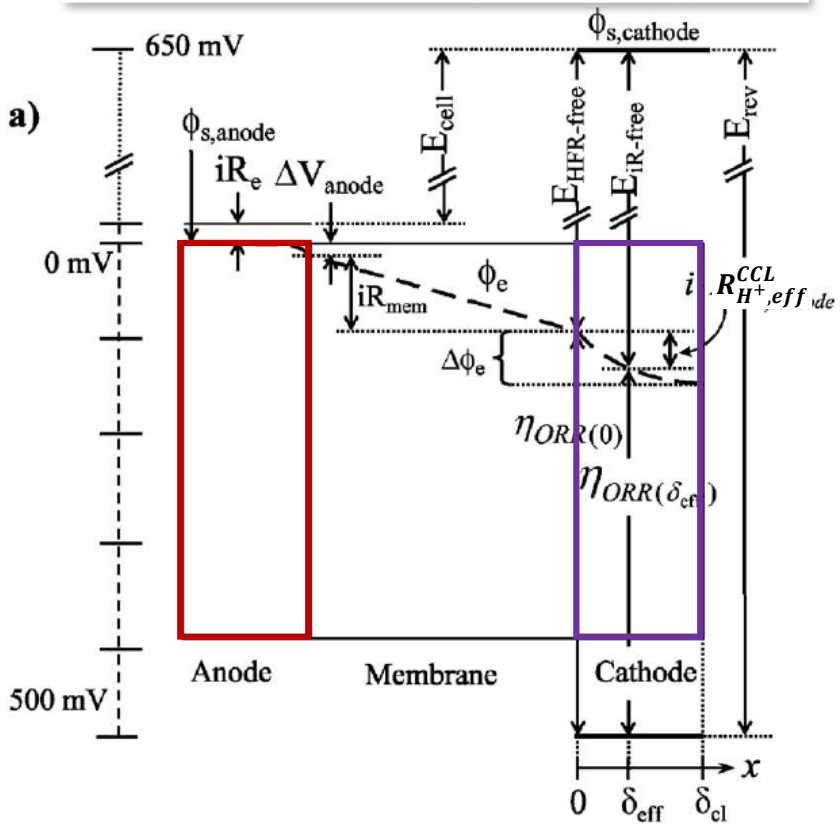
# CCM performance limitations

Schematic of the voltage losses across the CCM




Neyerlin *et al.* **2007** *J. Electrochem. Soc.* **154** B279

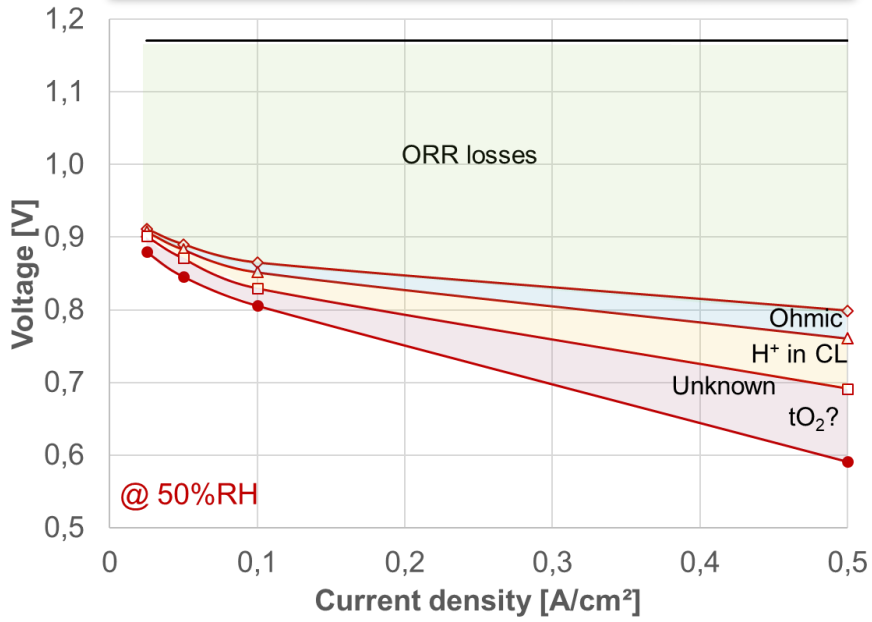
$$U_{cell} = U_{rev} - R_{Ohm} \cdot I - \eta_{HOR} - R^{ACL}_{H^+,eff} \cdot I - \eta_{ORR} - R^{CCL}_{H^+,eff} \cdot I - \eta_{transport(gas)}$$

ACL: Anode Catalyst Layer
CCL: Cathode Catalyst Layer

Neglect losses at the anode (kinetic and transport)

$$U_{cell} = U_{rev} - \eta_{ORR} - R_{Ohm} \cdot I - R^{CCL}_{H^+,eff} \cdot I - \eta_{transport(gas)}$$

Polarisation curve loss breakdown

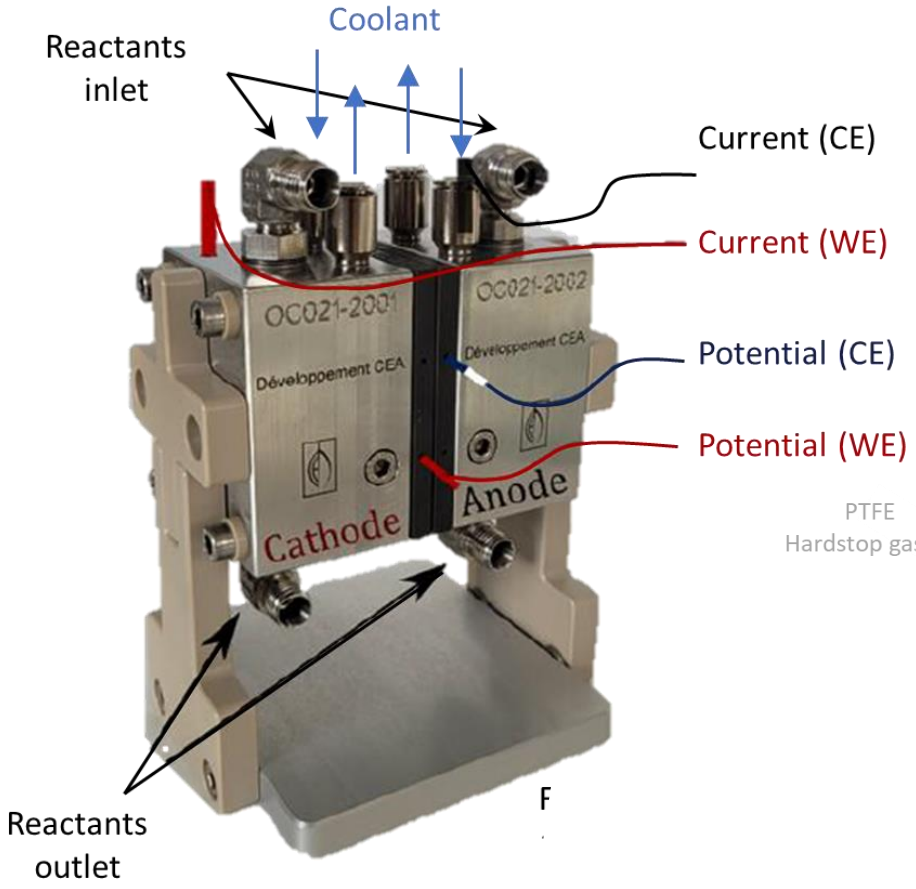


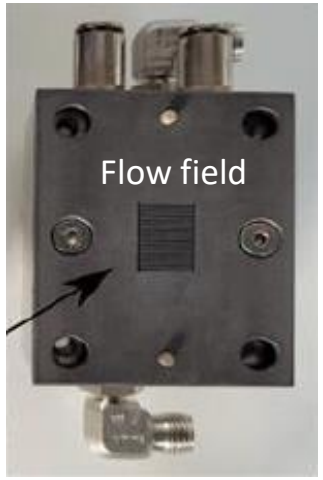

**Hyp. :** Decoupling between electrokinetic and transport losses

# How to quantify the losses?

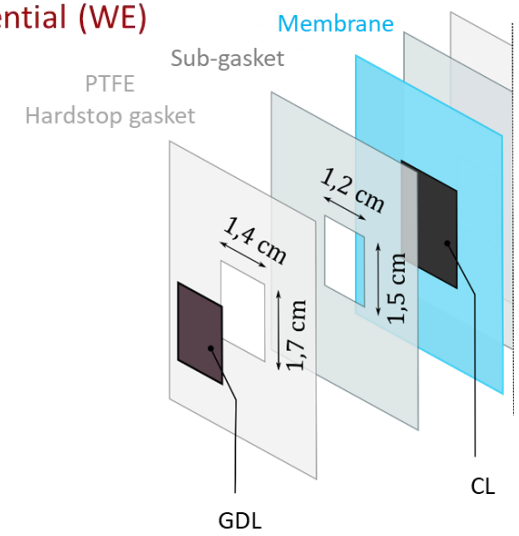
## Best possible control of conditions across the active area

Differential cell




MEA Assembly




- **High reactant stoichiometry** ( > 30 @ 3A/cm²)
  → Minized inlet to outlet « heterogeneity »
- **Small land & channel** (250 µm)
  → reduced land/channel « heterogeneity »

→ **In-plane operation** as **homogeneous** as possible

- Gas velocity in channel similar to that in stack

→ **Representative** of part of the active area **of a stack**

Control of T, RH, $PH_2$, $PO_2$, Ptot

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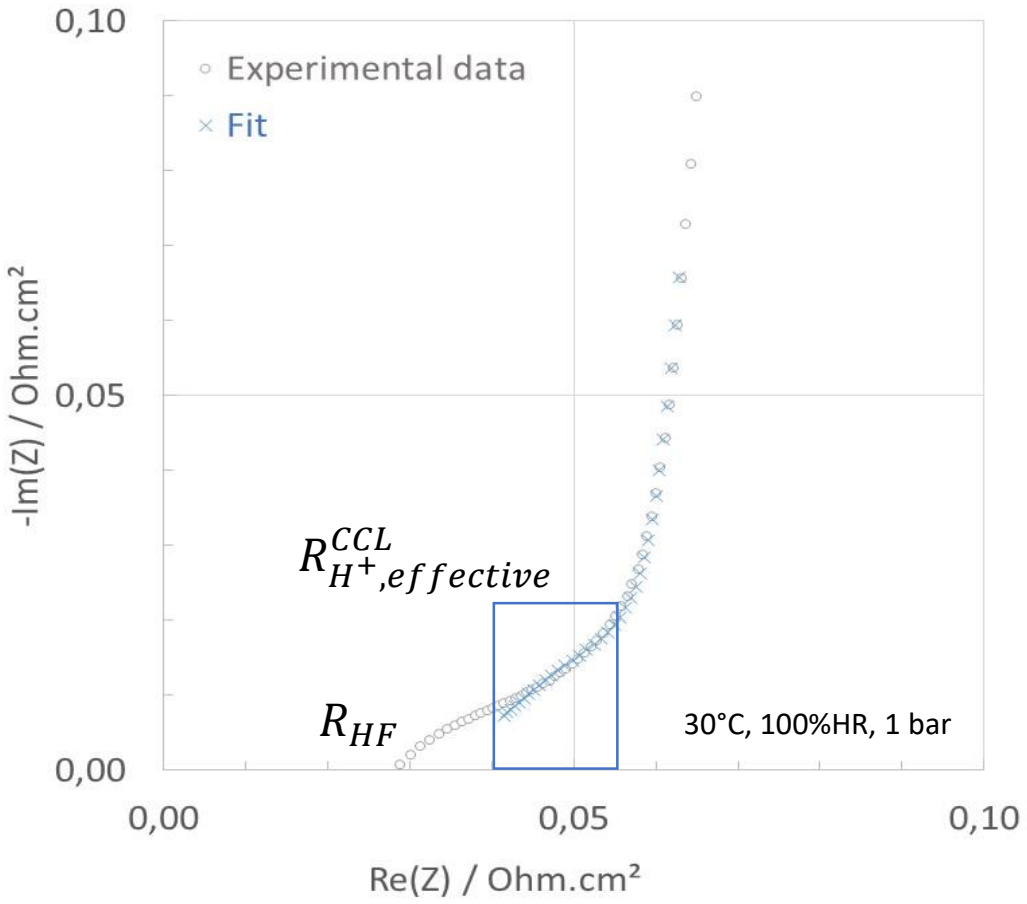
# Proton transport limitations

## How to properly quantify them?

EIS under $H_2/N_2$ _ No ORR




**Ideal case:** $R_{HF} \sim R_{Ohm} \sim R^{membrane}_{H^+} + R^{GDL}_{e^-}$ $\quad 5 < R^{GDL}_{e^-} < 10\ mOhm.cm^2$

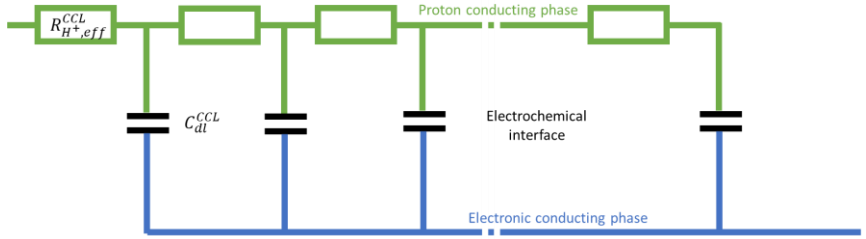
**Hyp.:** $R^{CCL}_{e^-} \ll R^{CCL}_{H^+,eff}$

**If not:** $R_{HF} \sim R_{Ohm} \sim R^{membrane}_{H^+} + R^{GDL}_{e^-} + \frac{R^{CCL}_{e^-} \cdot R^{CCL}_{H^+,eff}}{R^{CCL}_{e^-} + R^{CCL}_{H^+,eff}}$

Proper quantification of $R_{Ohm}$ needs to fit the whole impedance $Z_{H_2/N_2}$

Transmission line model blocking electrode

$R^{CCL}_{e^-}$ is neglected

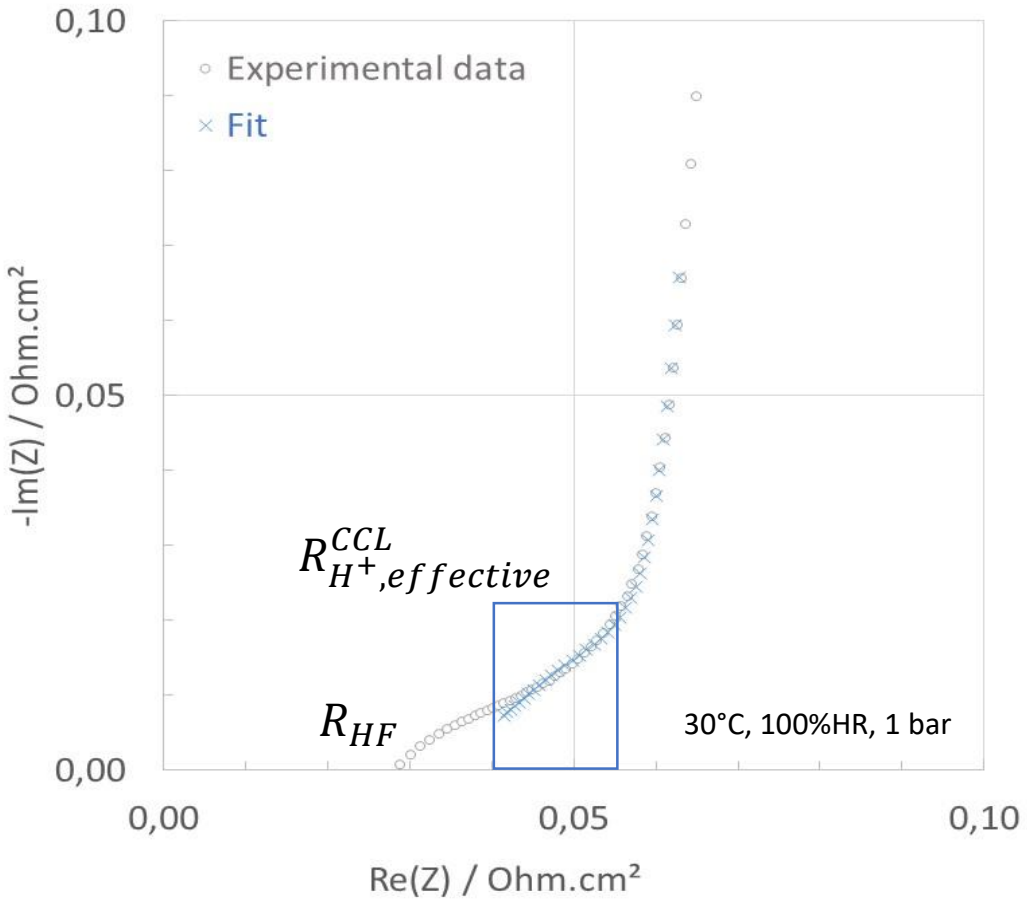



$$Z_{H_2/N_2} = R_{Ohm} + R^{CCL}_{H^+,eff} \frac{coth\left(\sqrt{i \cdot \omega \cdot R^{CCL}_{H^+,eff} \cdot C^{CCL}_{dl}}\right)}{\sqrt{i \cdot \omega \cdot R^{CCL}_{H^+,eff} \cdot C^{CCL}_{dl}}}$$

# Proton transport limitations without ORR

## How to properly quantify them?

EIS under $H_2/N_2$ _ No ORR




$$Z_{H_2/N_2} = R_{Ohm} + R^{CCL}_{H^+,eff} \frac{coth\left(\sqrt{i \cdot \omega \cdot R^{CCL}_{H^+,eff} \cdot C^{CCL}_{dl}}\right)}{\sqrt{i \cdot \omega \cdot R^{CCL}_{H^+,eff} \cdot C^{CCL}_{dl}}}$$

**Hyp.**

- $R^{CCL}_{e^-}$ is neglected
- Ideal capacitor
- No Faradaic reaction : blocking Working Electrode (WE)

**Limits**

- Faradaic reaction : HOR of $H_2$ crossing membrane from CE
  → Limit the minimum frequency for a proper fit (~few 10 Hz)
- Inductance of the connections
- Impedance of the potentiostat
  → Limit the maximum frequency for the fit (~few kHz)
- Non ideal capacitive behaviour
  → Use Constant Phase Element (CPE) instead of C

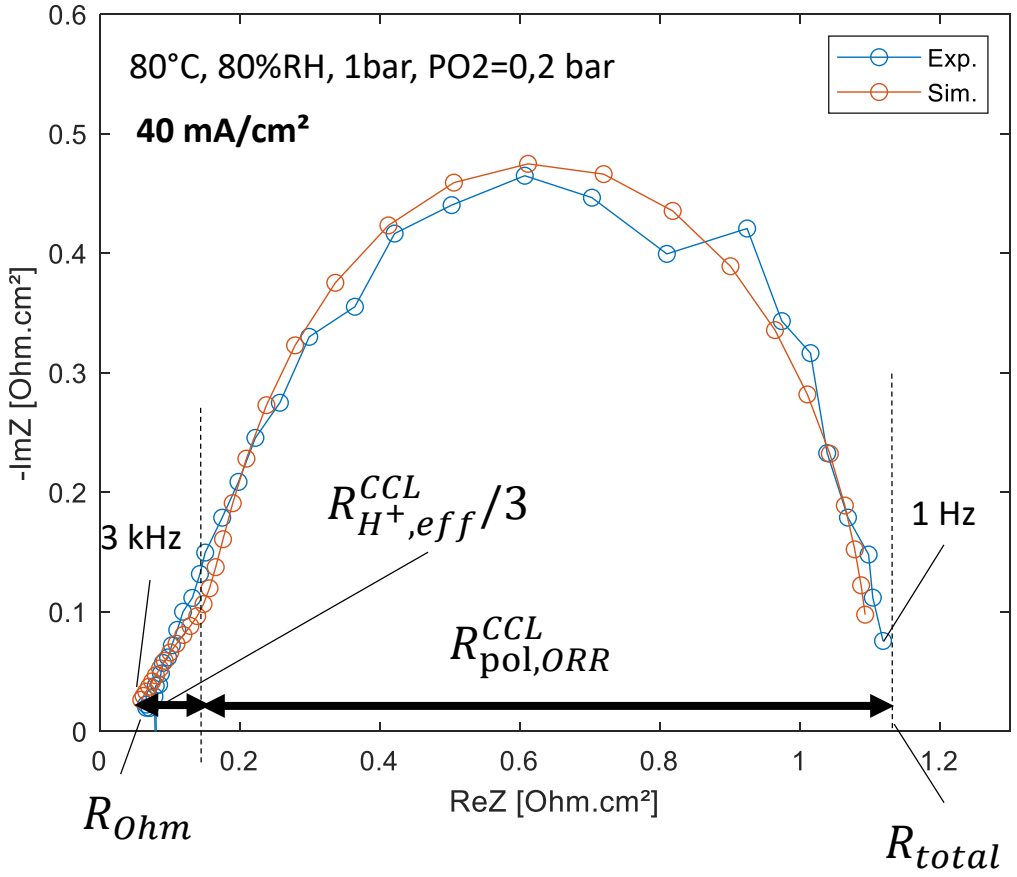
Siroma *et al.* **2015** *Electrochimica Acto* **160** 313

**More accurate with larger resistance, e.g. smaller geometrical active surface**

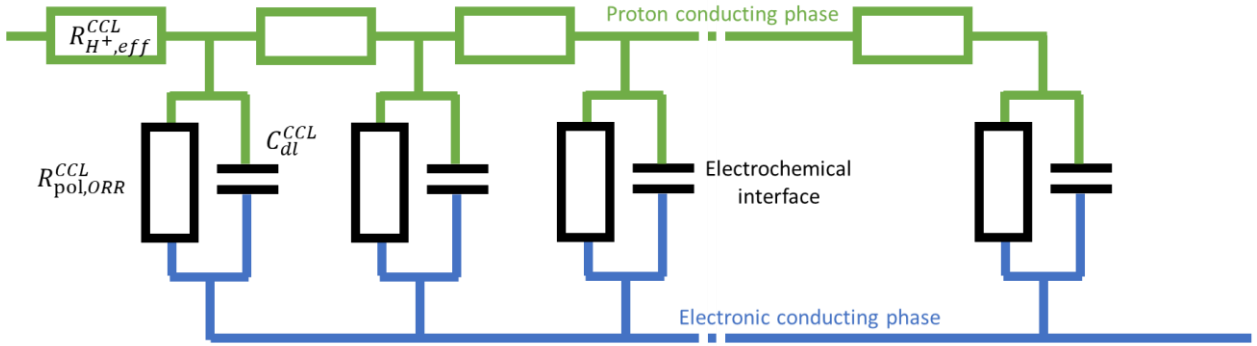
# Proton transport limitations with ORR

## Low current density

EIS under $H_2/O_2$ _ORR




Proper quantification needs to fit the whole impedance $Z_{H_2/O_2}$




$$Z_{H_2/O_2} = R_{H^+,eff}^{CCL} \cdot \frac{\coth\left(\sqrt{i \cdot \omega \cdot R_{H^+,eff}^{CCL} \cdot C_{dl}^{CCL} + \frac{R_{\mathrm{pol},ORR}^{CCL}}{R_{H^+,eff}^{CCL}}}\right)}{\sqrt{i \cdot \omega \cdot R_{H^+,eff}^{CCL} \cdot C_{dl}^{CCL} + \frac{R_{\mathrm{pol},ORR}^{CCL}}{R_{H^+,eff}^{CCL}}}}$$

# Proton transport limitations with ORR

## Low current density

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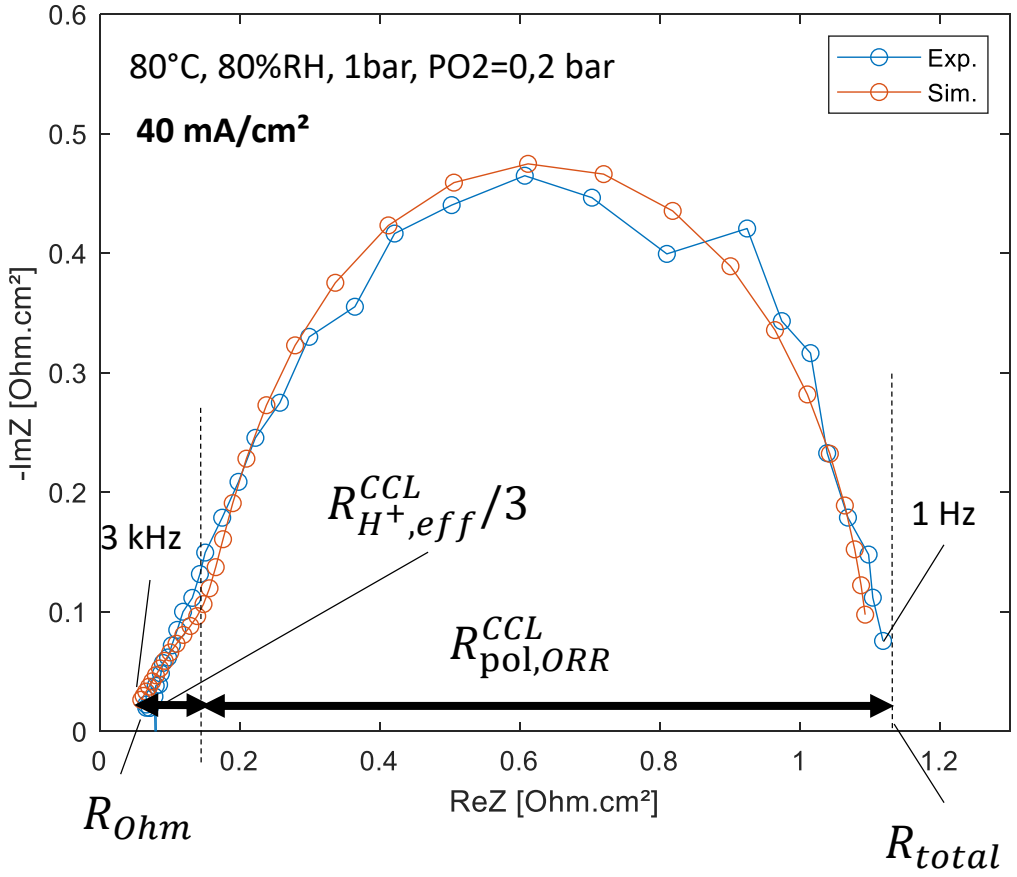


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**Hyp.**

- No gas transport limitations at all
- Homogeneous operation across the CL

**Limits**

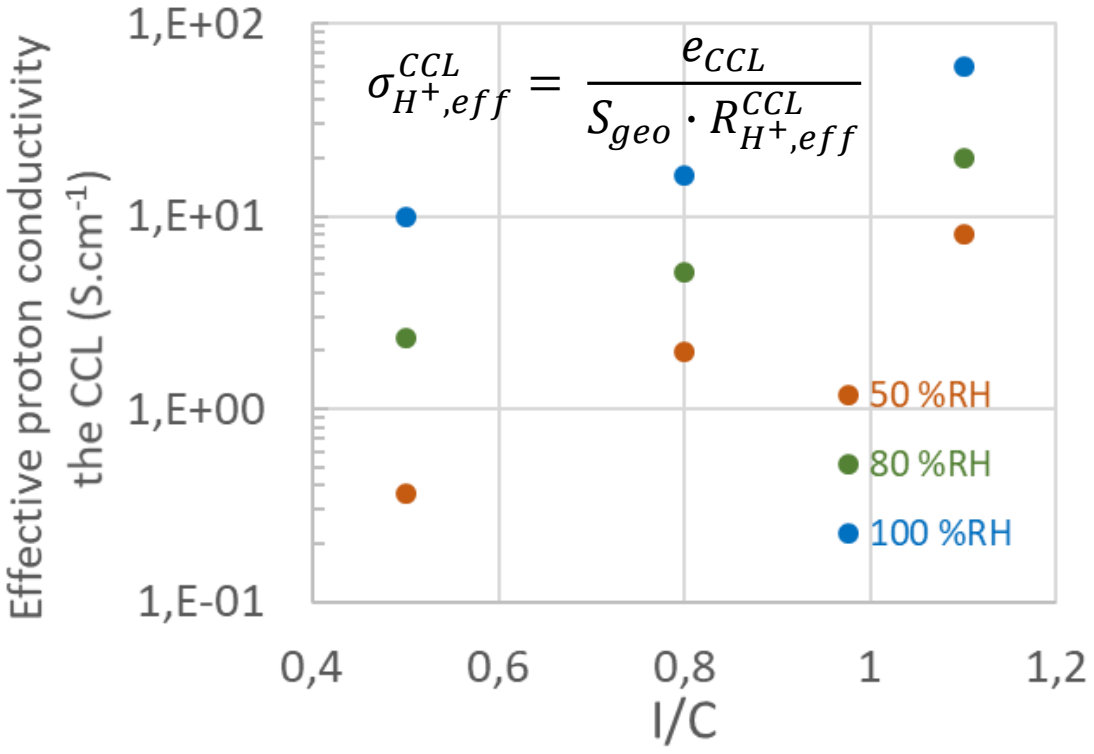
- No reliable analytical model when transport limitations can not be neglected
  - → Fitting at low current density (<0.1 – 0.2 A/cm²)
- Inductance of the connections
- Impedance of the potentiostat
  - → Limit the maximum frequency for the fit (~few kHz)

# Proton transport limitations

## What does FURTHER-FC teach us?

$\sigma_{H^+,eff}^{CCL}$ for different I/C

*Reference I/C = 0.8*




**$\sigma_{H^+,eff}^{CCL}$ increases as I/C and RH increase**

$e_{ccL} = 6.5 \cdot 10^{-4}\ cm$, thickness of CCL

**In–plane H⁺ conductivity of ultra-thin film measured ex-situ**

$$\sigma_{H^+,in-plane}^{D2020\ thin\ film,\ 7\ nm@\ 80°C,80\%RH} = \sigma_{H^+}^{thin\ film} = 1\ mS.cm^{-1}$$

**Theoritical effective CCL H⁺ conductivity from ultra-thin film**

$$\sigma_{H^+,eff}^{CCL\ theo\ @\ 80°C,80\%RH} = \sigma_{H^+}^{thin\ film} \frac{\emptyset_v^{D2020}}{\tau} < 0.215\ mS.cm^{-1}$$

$\emptyset_v^{D2020} = 0.215$, volume fraction of D2020 in CCL

$\tau = 1$, minimum tortuosity of the ionomer network

**Experimental effective CCL H⁺ conductivity**

$$2 < \sigma_{H^+,eff}^{CCL\ exp.@\ 80°C,80\%RH} < 20\ mS.cm^{-1} \gg \sigma_{H^+,eff}^{CCL\ theo\ @\ 80°C,80\%RH}$$

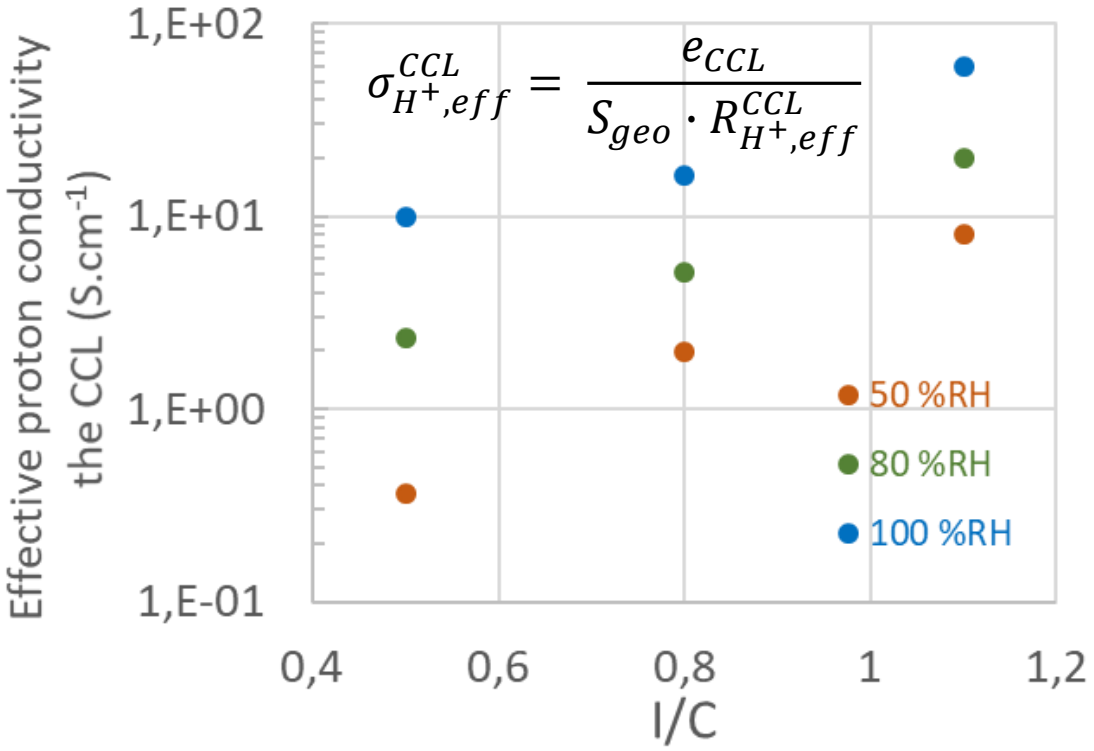
**Values for ultra-thin ionomer conductive films may not be representative**

# Proton transport limitations

## What does FURTHER-FC teach us?

$\sigma_{H^+,eff}^{CCL}$ for different I/C

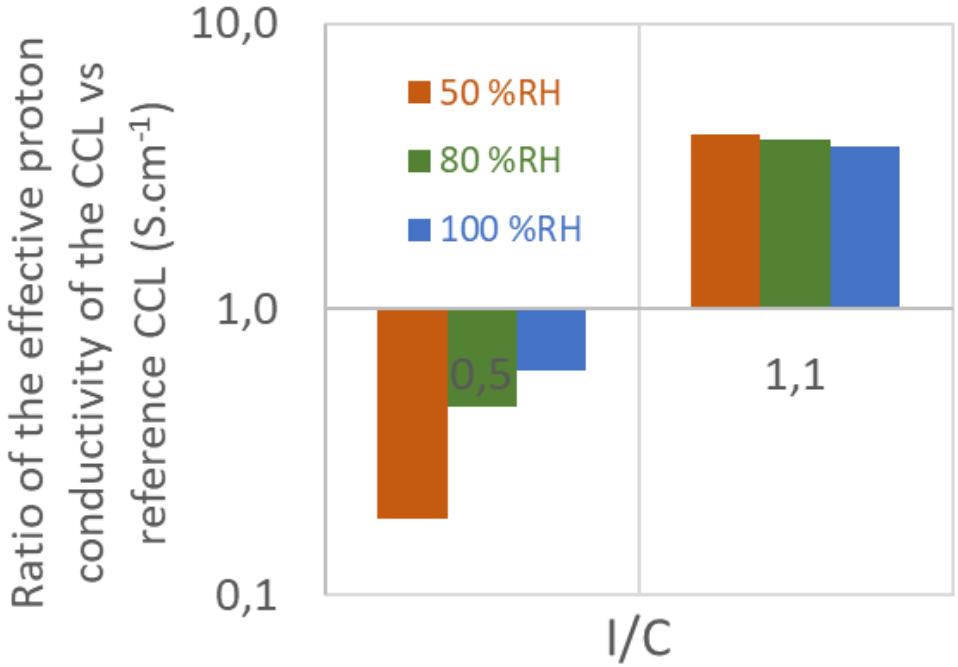
*Reference I/C = 0.8*




**$\sigma_{H^+,eff}^{CCL}$ increases as I/C and RH increase**

Ratio of $\sigma_{H^+,eff}^{CCL}$ for different I/C

*Reference I/C = 0.8*




$R_{H^+,eff}^{CCL}$ ratio depends on RH for low I/C

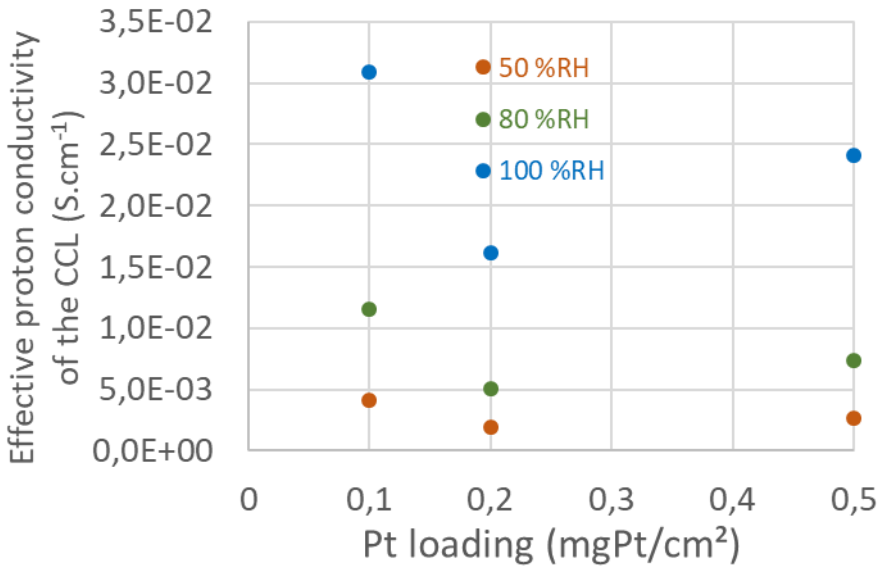
→ **Water « independently » of ionomer plays a role in $H^+$ transport**

$e_{ccL} = 6.5 \cdot 10^{-4}\ cm$, thickness of CCL

# Proton transport limitations

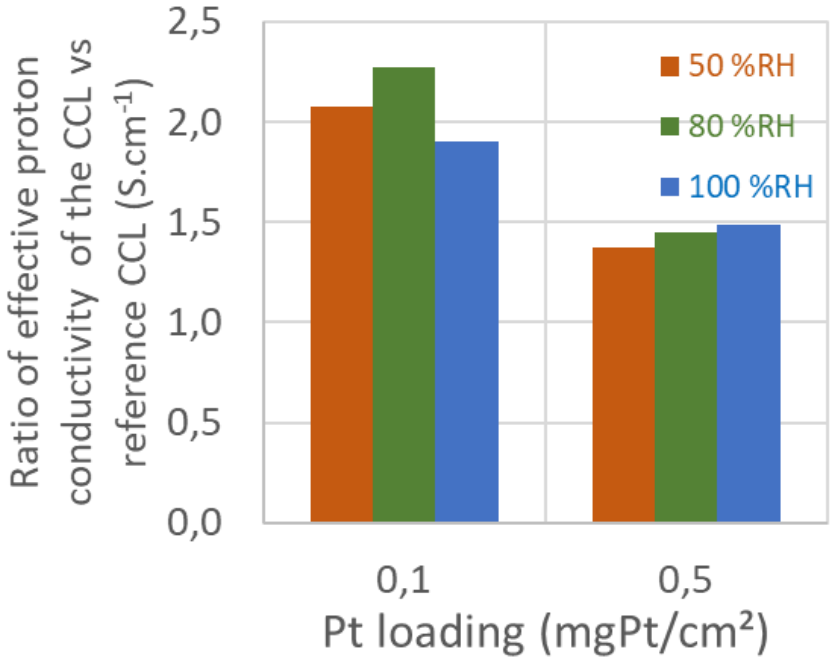
## What does FURTHER-FC teach us?

$\sigma_{H^+,eff}^{CCL}$ as a function of CCL thickness for different RH




Ratio of $\sigma_{H^+,eff}^{CCL}$ for different Pt loading

*Reference 0.2 $mg_{Pt}/cm^2_{geo}$*




- $\sigma_{H^+,eff}^{CCL}$ ratio does not depend RH

→ Similar overall behaviour for all RH

- $\sigma_{H^+,eff}^{CCL}$ ratio does depend on Pt loading/CCL thickness and ≠ 1

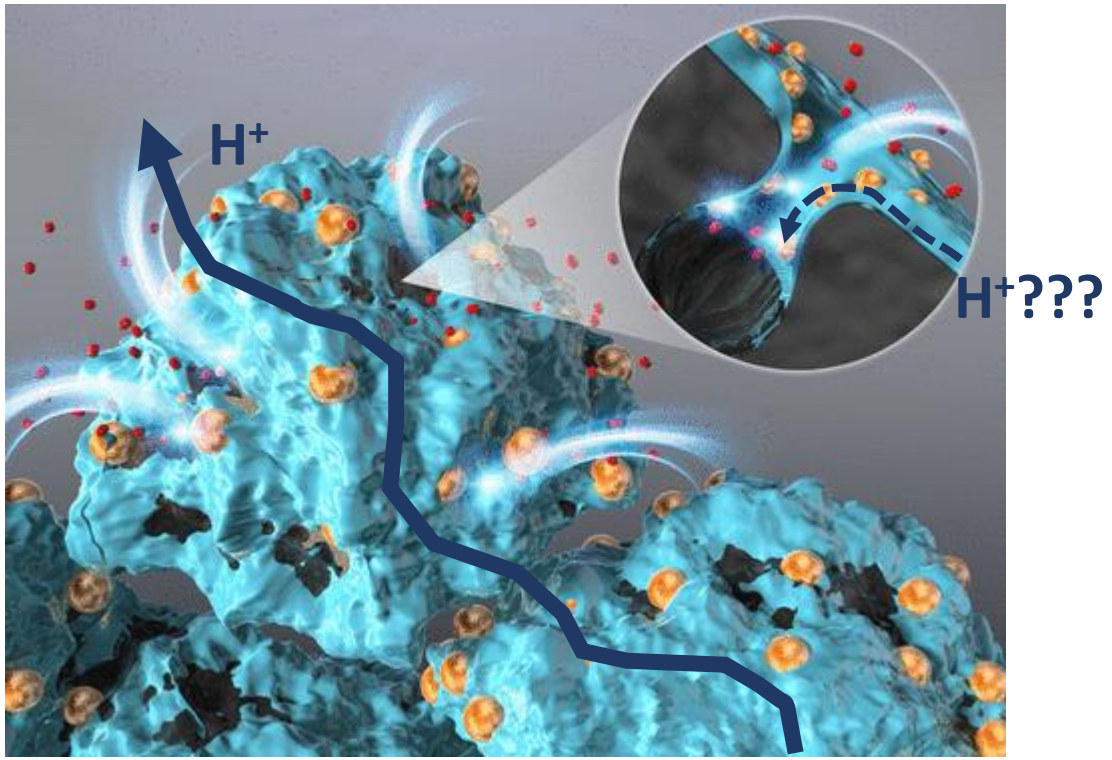
→ Difference in ionomer distribution through CCL?

# Proton transport limitations

## Does it give relevant information to correlate with losses?

Scheme of Pt/C catalyst layer

*Artist's impression*




Kobayashi *et al.* **2021** *ACS Appl. Energy Mater.*
*Univ. Yamanashi*

$R^{CCL}_{H^+,eff}$ proton transport across CCL

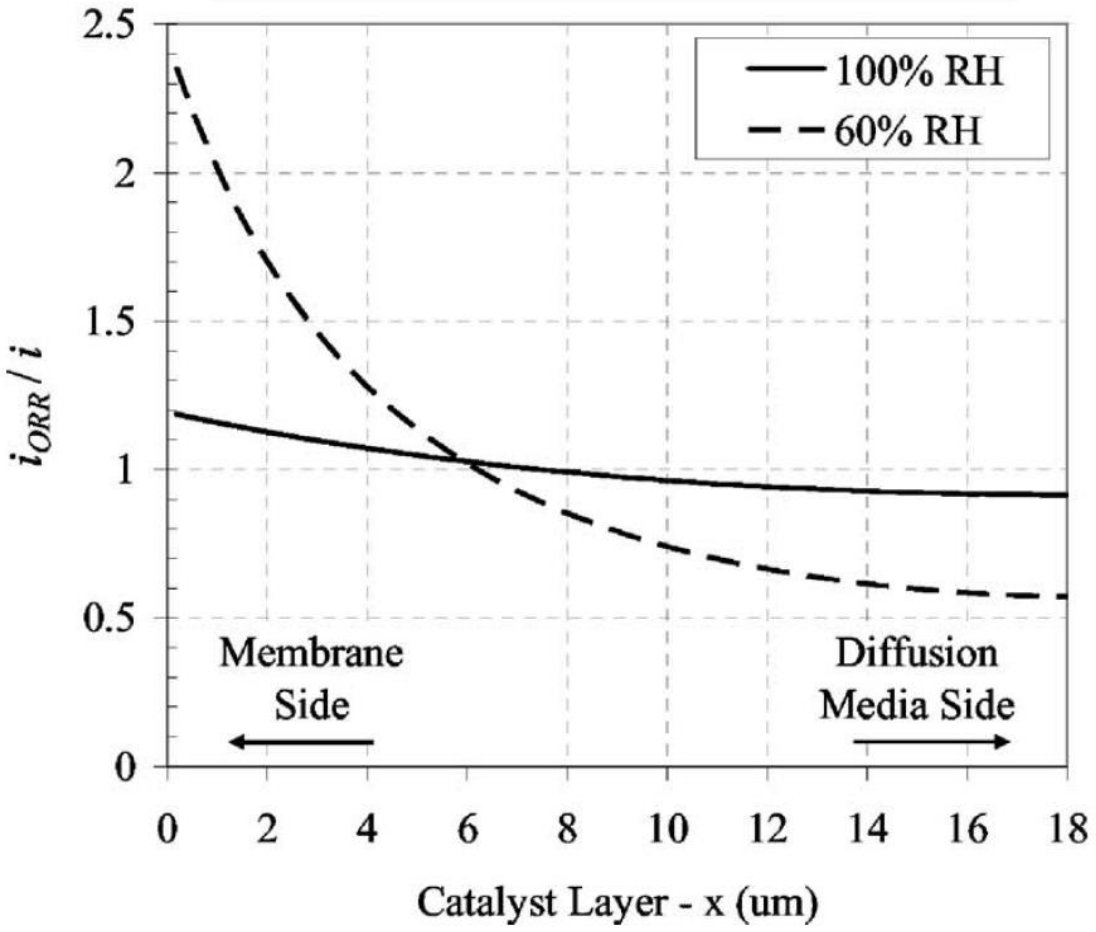
Does $R^{CCL}_{H^+,eff}$ tackles $H^+$ transport limitations at the nm scale e.g. inside the nanopores of the C???

→ **It is currently of paramount importance**

# Proton transport limitations

## What does it correspond to?

ORR current density distribution across CCL




Neyerlin *et al.* **2007** *J. Electrochem. Soc.* **154** B279

**Limits**

- $R_{H^+,eff}^{CCL}$ implies heterogeneous operation across CCL
  → Not the same reaction rate on all catalytic sites

- $R_{H^+,eff}^{CCL}$ losses due to $H^+$ transport across CCL
  → No information on its impact on effective use of catalyst or to kinetic losses due to heterogeneous operation

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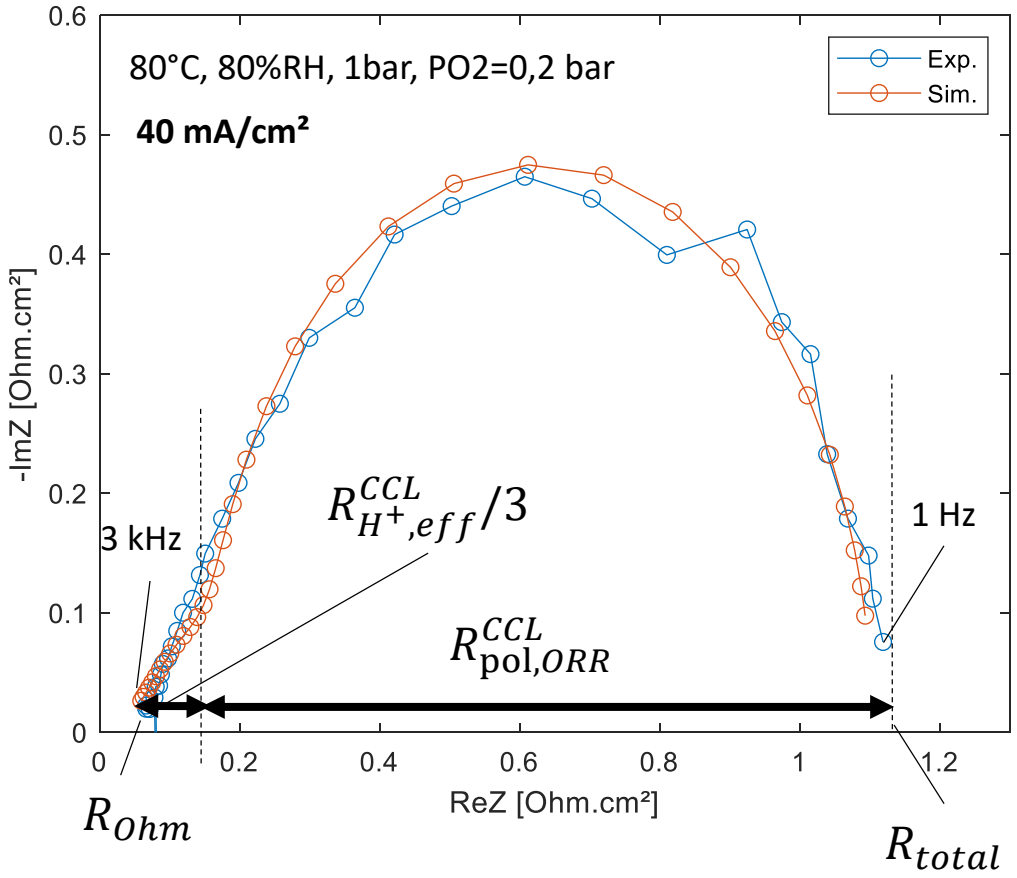
# Electrochemcal properties from EIS

## Low current density

EIS under $H_2/O_2$ _ORR




$$Z_{H_2/O_2} = R_{H^+,eff}^{CCL} \cdot \frac{\coth\left(\sqrt{i \cdot \omega \cdot R_{H^+,eff}^{CCL} \cdot C_{dl}^{CCL} + \frac{R_{\mathrm{pol},ORR}^{CCL}}{R_{H^+,eff}^{CCL}}}\right)}{\sqrt{i \cdot \omega \cdot R_{H^+,eff}^{CCL} \cdot C_{dl}^{CCL} + \frac{R_{\mathrm{pol},ORR}^{CCL}}{R_{H^+,eff}^{CCL}}}}$$

**Limits**

- $R_{\mathrm{pol},ORR}^{CCL}$ does not give you any information on the catalyst activity for ORR or the overall activity of your CCL

$$U_{cell} = U_{rev} - \eta_{ORR} - R_{Ohm} \cdot I - R_{H^+,eff}^{CCL} \cdot I - \eta_{transport(gas)}$$

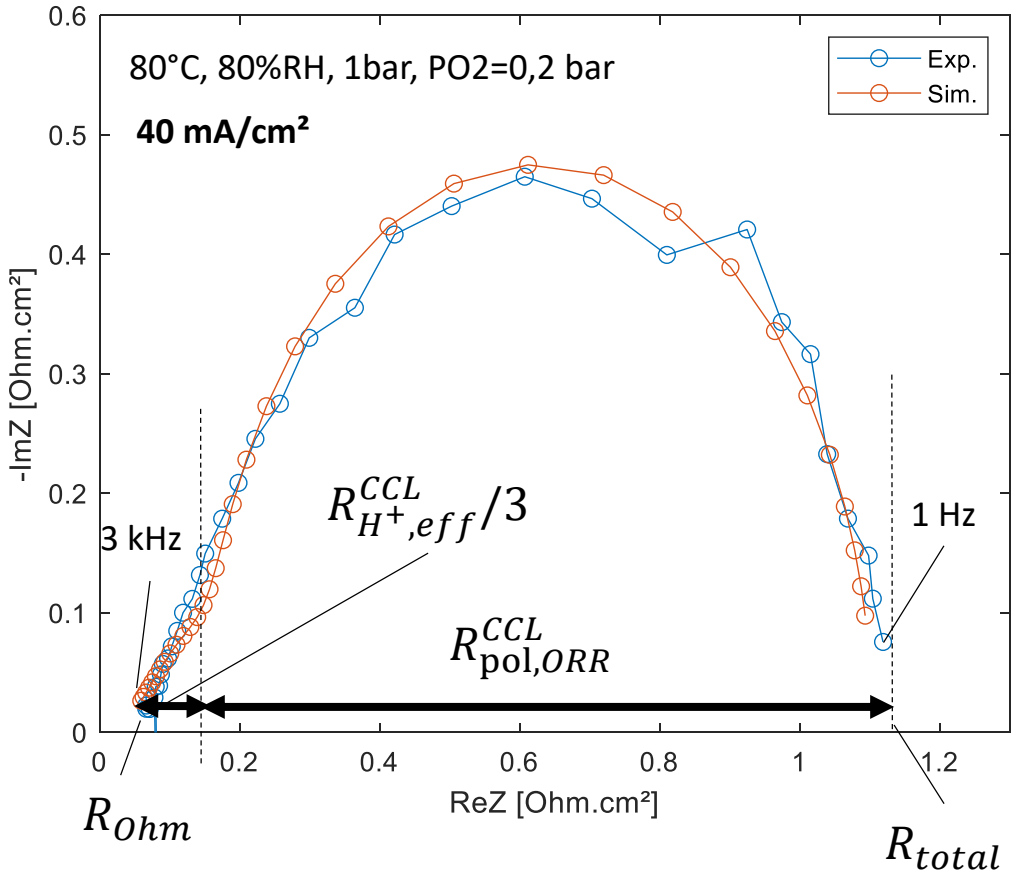
If no transport limitations and correction from Ohmic drop and $H^+$ transport limitation in CCL

$$U_{corr} = U_{cell} + R_{Ohm} \cdot I + R_{H^+,eff}^{CCL} \cdot I = U_{rev} - \eta_{ORR}$$

# Electrochemcal properties from EIS

## Low current density

EIS under $H_2/O_2$ _ORR




$$R_{\mathrm{pol},ORR}^{CCL} = -\left(\frac{d\eta_{ORR}}{dI}\right)_{\omega\to 0}$$

Simple Butler-Volmer:

$$I = I_0\left(e^{\frac{-\alpha_C F\,\eta_{ORR}}{R\,T}} - e^{\frac{(1-\alpha_C) F\,\eta_{ORR}}{R\,T}}\right)$$

Tafel approximation: $|\eta_{ORR}| > \frac{3\,R\,T}{\alpha_C F}$

$$I = I_0\, e^{\frac{-\alpha_C F\,\eta_{ORR}}{R\,T}}$$

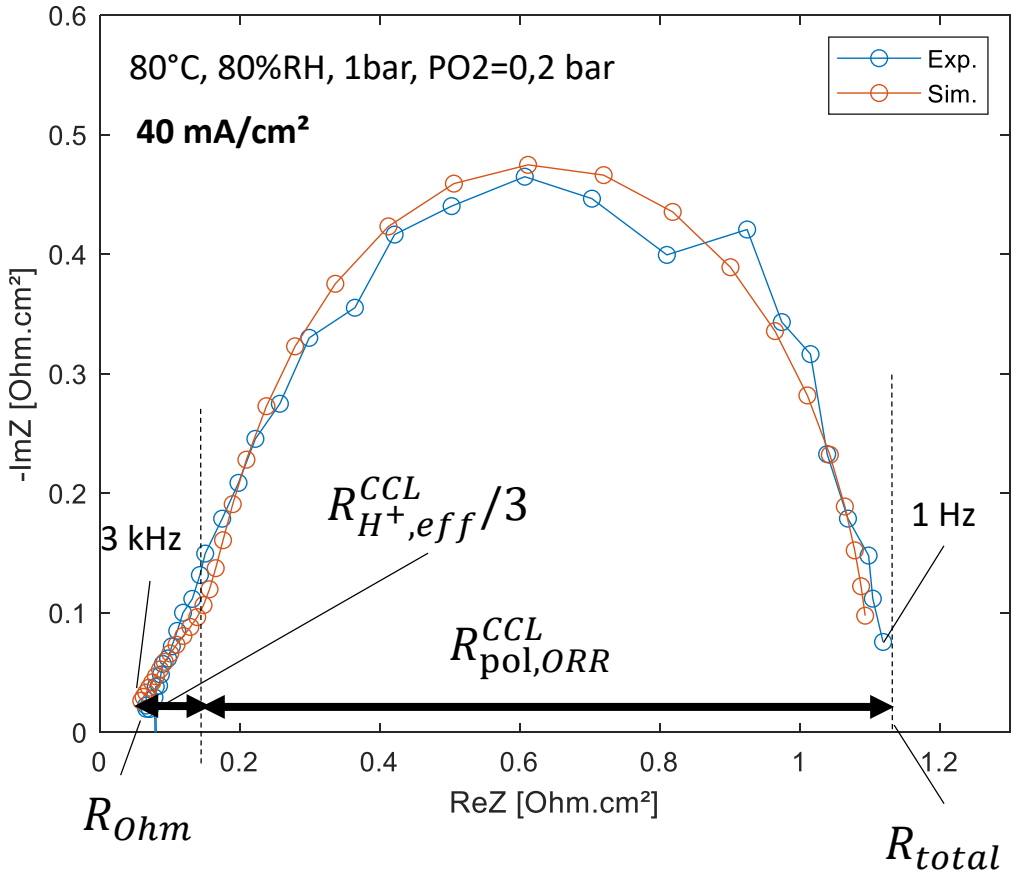
$$\eta_{ORR} = -b\,\ln\frac{I}{I_0} \qquad b = \frac{R\,T}{\alpha_C\,F}\text{ : Tafel slope}$$

$$R_{\mathrm{pol},ORR}^{CCL} = \frac{b}{I}$$ If no transport limitations

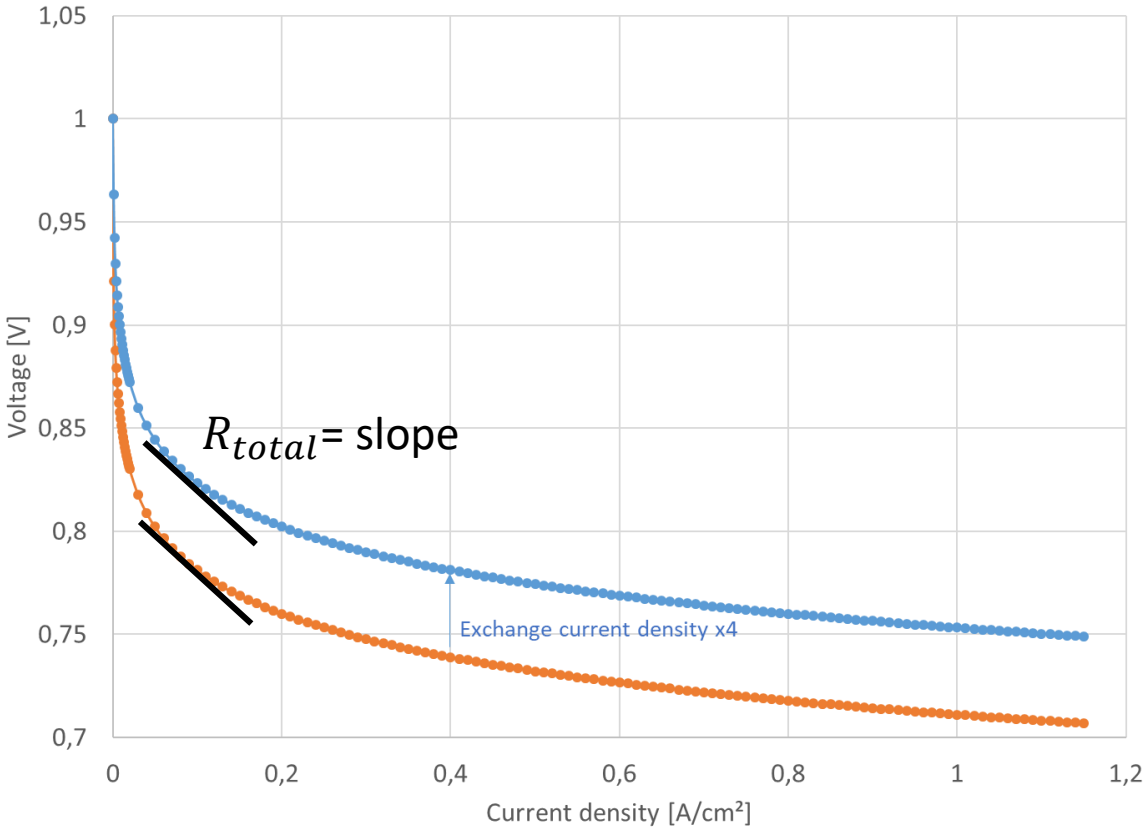
# Electrochemcal properties from EIS

## Low current density

EIS under $H_2/O_2$ _ORR




Simulated I-V curve without transport limitations



# Electrochemcal properties from EIS

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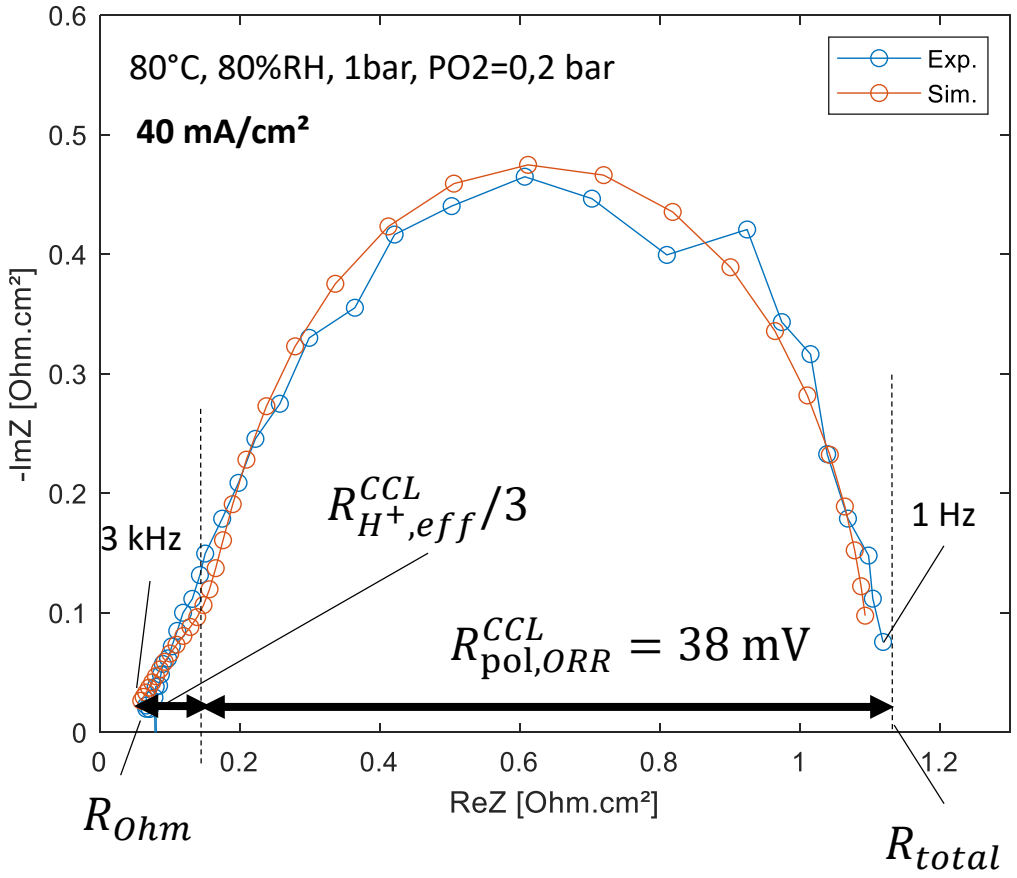
EIS under $H_2/O_2$ _ORR




$$R_{\mathrm{pol},ORR}^{CCL} = -\left(\frac{d\eta_{ORR}}{dI}\right)_{\omega \to 0} \qquad b = \frac{R\,T}{\alpha_C\,F} : \text{Tafel slope}$$

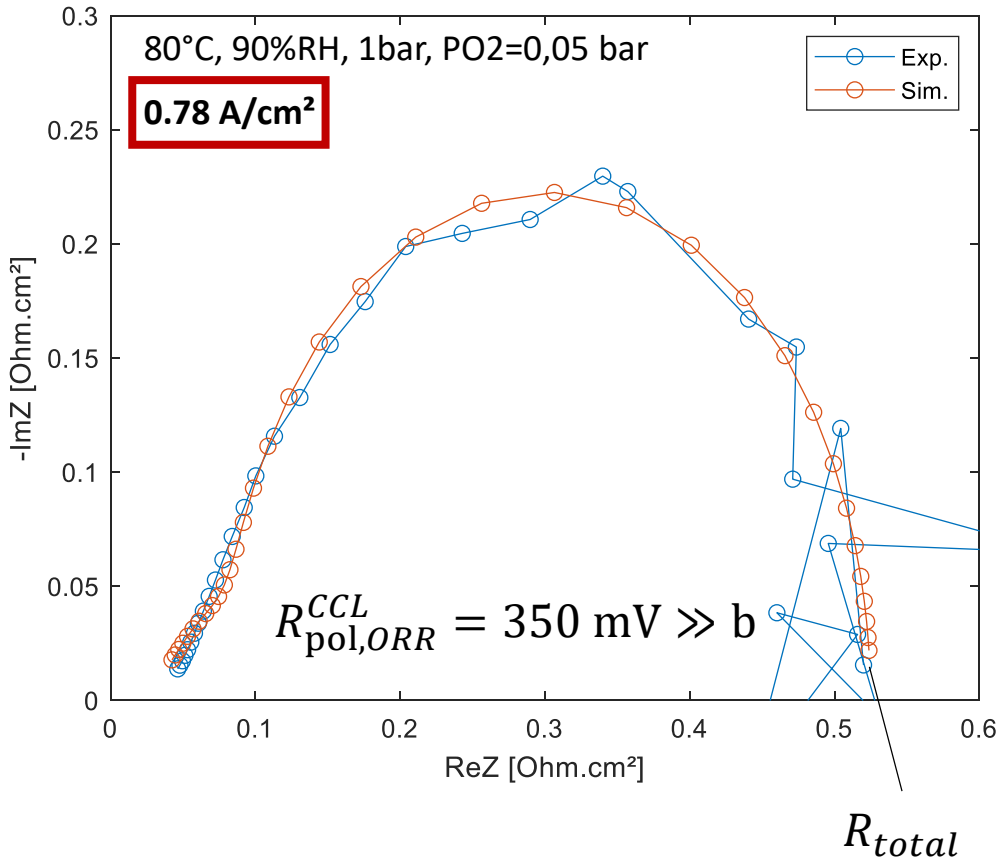
$R_{\mathrm{pol},ORR}^{CCL} = \frac{b}{I}$ **If transport limitations have negligible impact**

If $R_{\mathrm{pol},ORR}^{CCL} \cdot j > b$ **transport limitations most probably not negligible**

# Transport limitations from EIS

## High current density

EIS under $H_2/O_2$ _ORR




But where is the famous second loop???

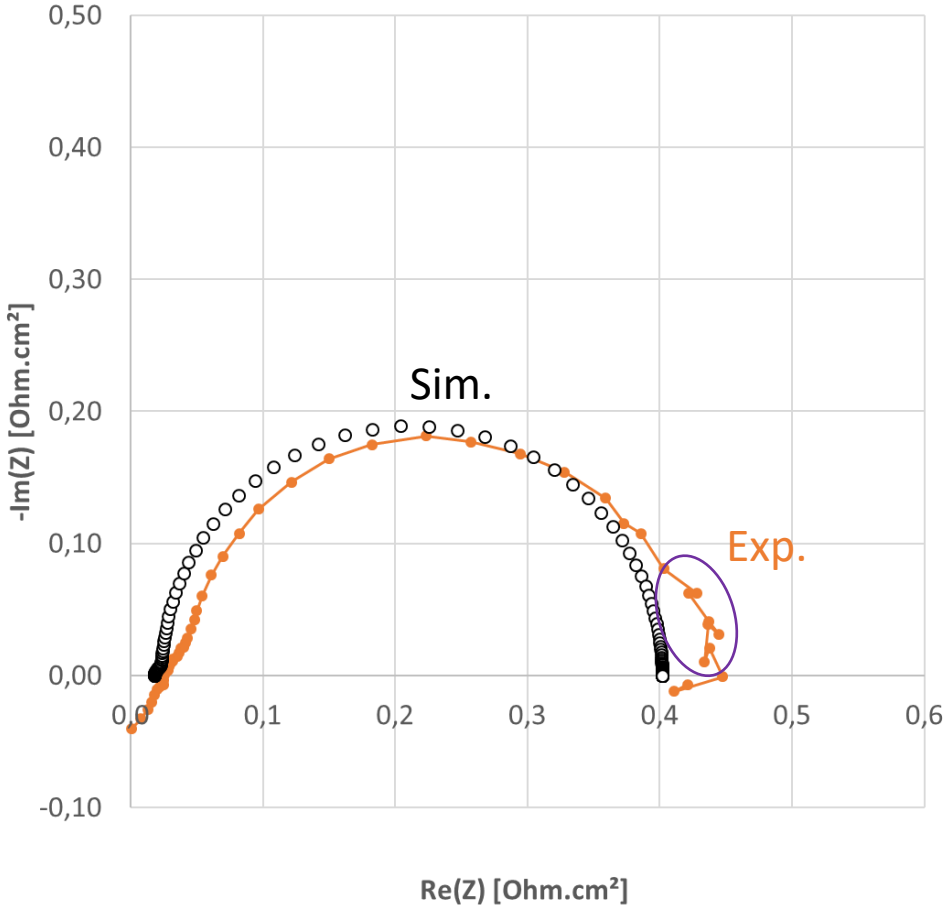
Limits:

- $R^{CCL}_{\mathrm{pol},ORR}$ **includes the coupling between kinetic & transport**

# Transport limitations from EIS

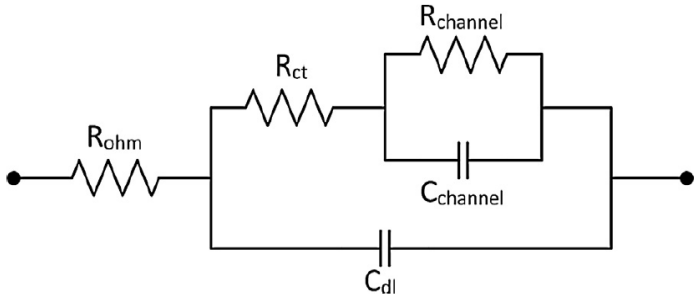
## High current density

EIS under $H_2/O_2$ _ORR




## But where is the famous second loop???

May be due to transport limitations along/within the channel




$$Z = R_{ohm} + \frac{1}{\frac{1}{R_{ct} + \frac{1}{\frac{1}{R_{channel}} + i\omega C_{channel}}} + i\omega C_{dl}}$$

$$R_{channel} = \frac{\gamma R_{ct}}{2St_{O_2} - 1}$$

$$C_{channel} = \frac{2St_{O_2} - 1}{\gamma R_{ct}} \frac{V}{2\overline{Q}}$$

Chanderis *et al.* **2015** *Electrochimica Acto* **180** 581

# OUTLINE

General considerations

CCM losses

Proton transport resistance thanks to EIS measurements

What else can we extract from EIS?

**Oxygen transport resitance thanks to LCA measurments**

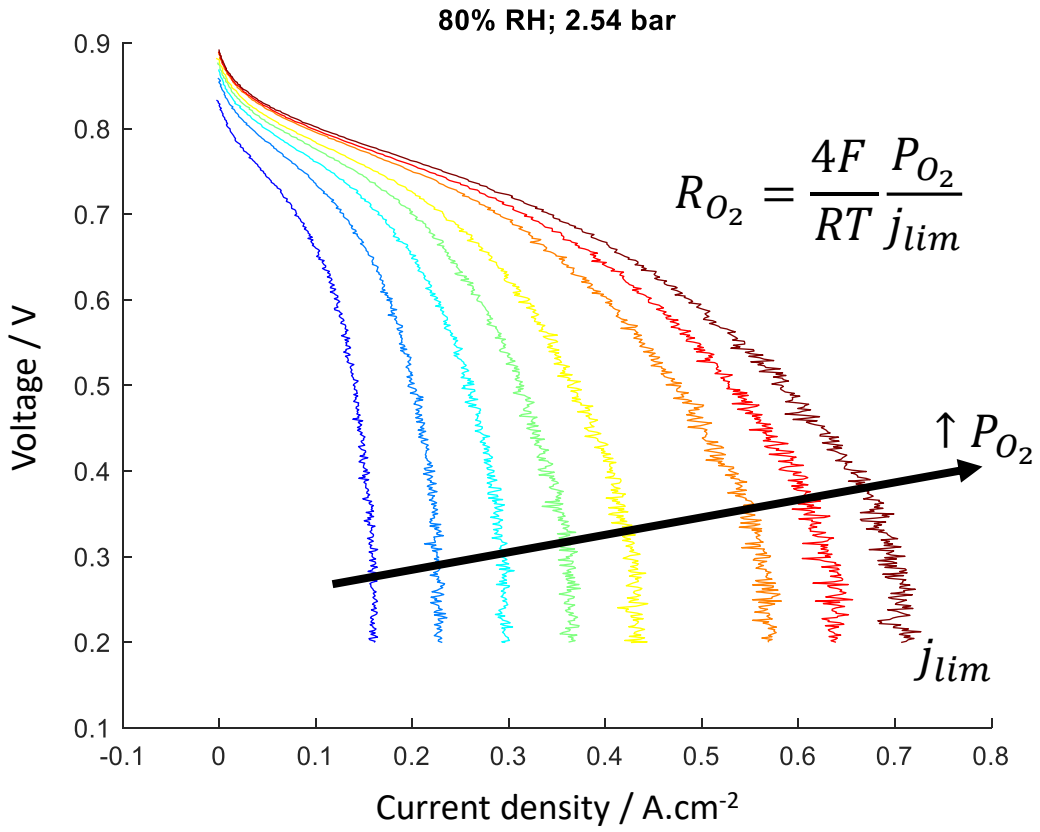
Decoupling electrokinetic from transport limitations

Conclusion

# $O_2$ transport limitations

## Limiting Current Analysis (LCA) measurement principle and analysis

I-V for different $O_2$ concentration



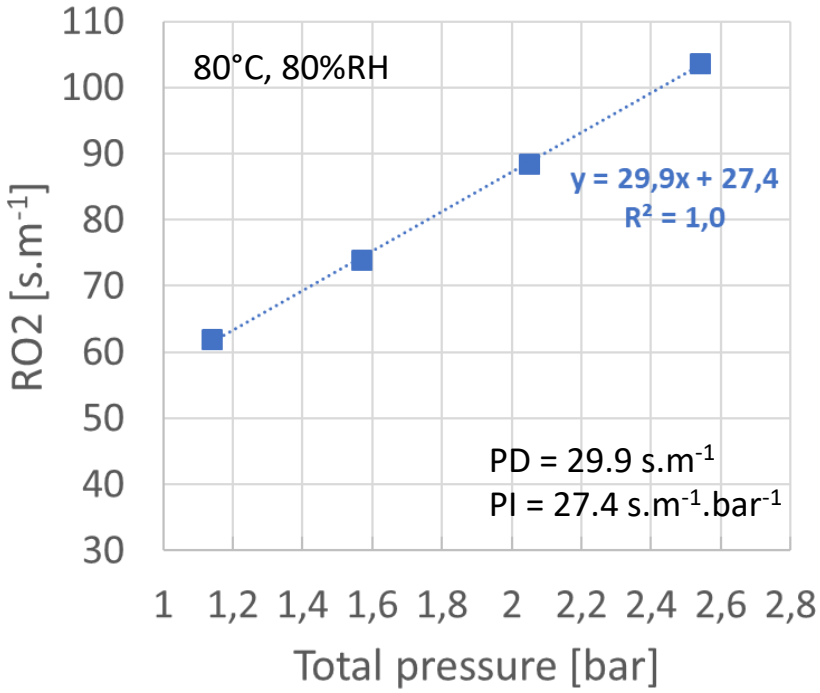

Baker *et al.* **2009** *J. Electrochem. Soc.* **156** B991

**Pressure Dependent** (PD) term:

- Fickian diffusion in largest pores (d > mean free path $O_2$ ~ 100 nm @ 80°C, 1bar )

**Pressure Independent** (PI) term:

- Knudsen diffusion in smallest pores (d < 100 nm)



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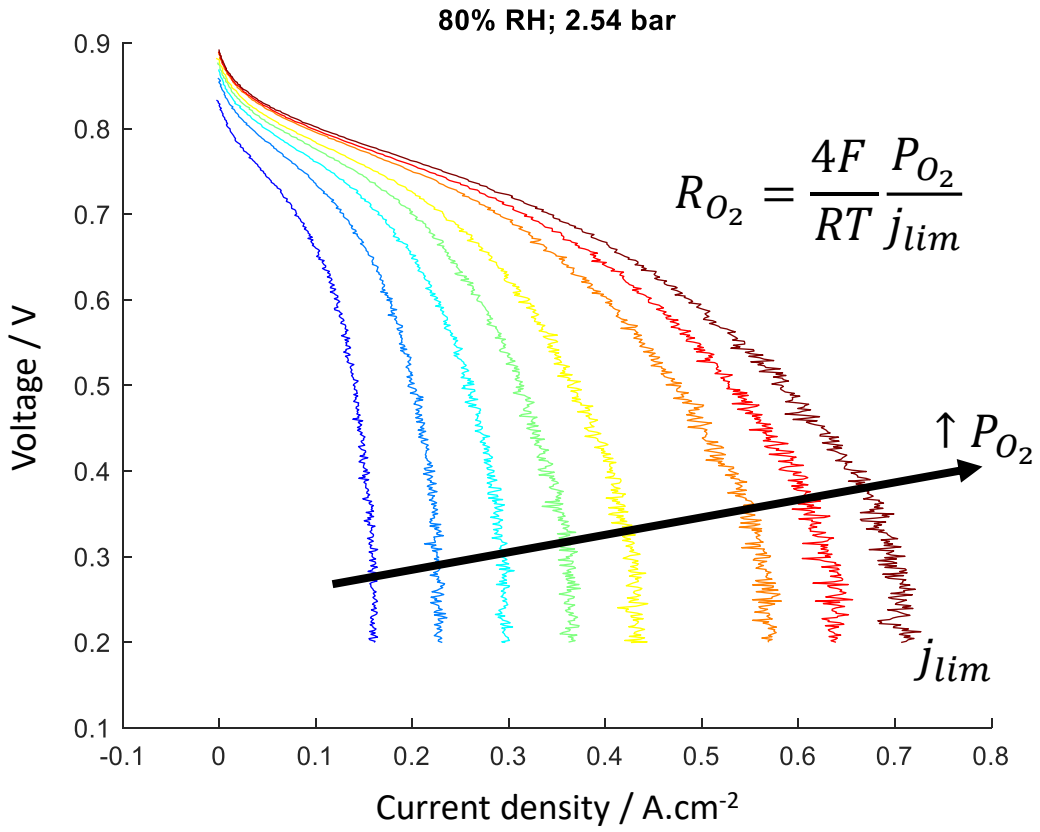


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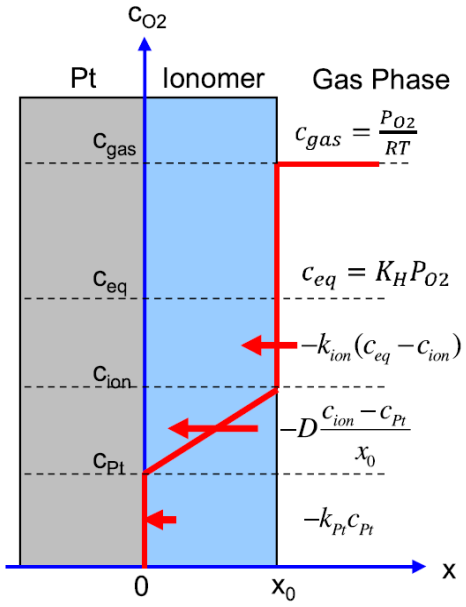
- Diffusion within electrolyte (hydrated ionomer and/or water)
- Transfert through gas/electrolyte interface
- Transfert through electrolyte/Pt interface

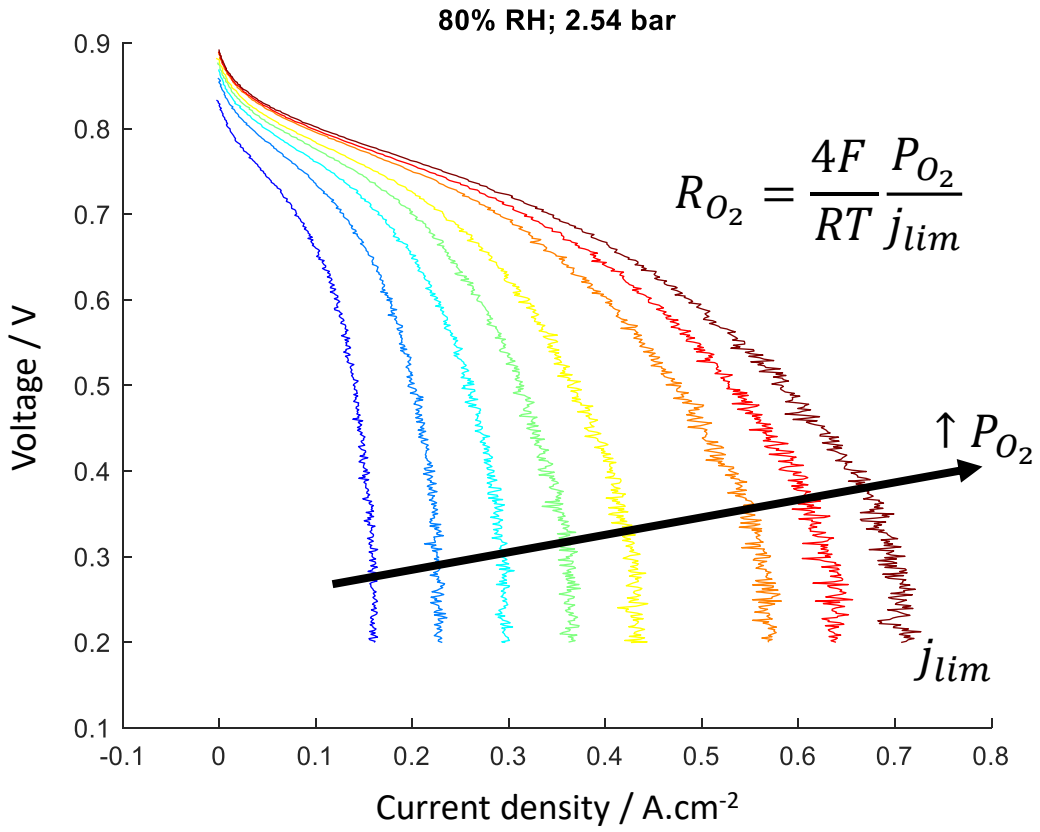



Kudo *et al.* **2016** *Electrochimica Acta*, 682

# $O_2$ transport limitations

## Limiting Current Analysis (LCA) measurement conditions

I-V for different $O_2$ concentration

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- Transfert through electrolyte/Pt interface

**Hyp.**

➢ Current is limited only by $O_2$ transport and not by $H^+$ transport or kinetic

**Caution must be taken in the choice of measurement conditions**

➢ RH must be sufficiently high not to be limited by the $H^+$ transport

➢ RH must not be too high to avoid excessive water condensation

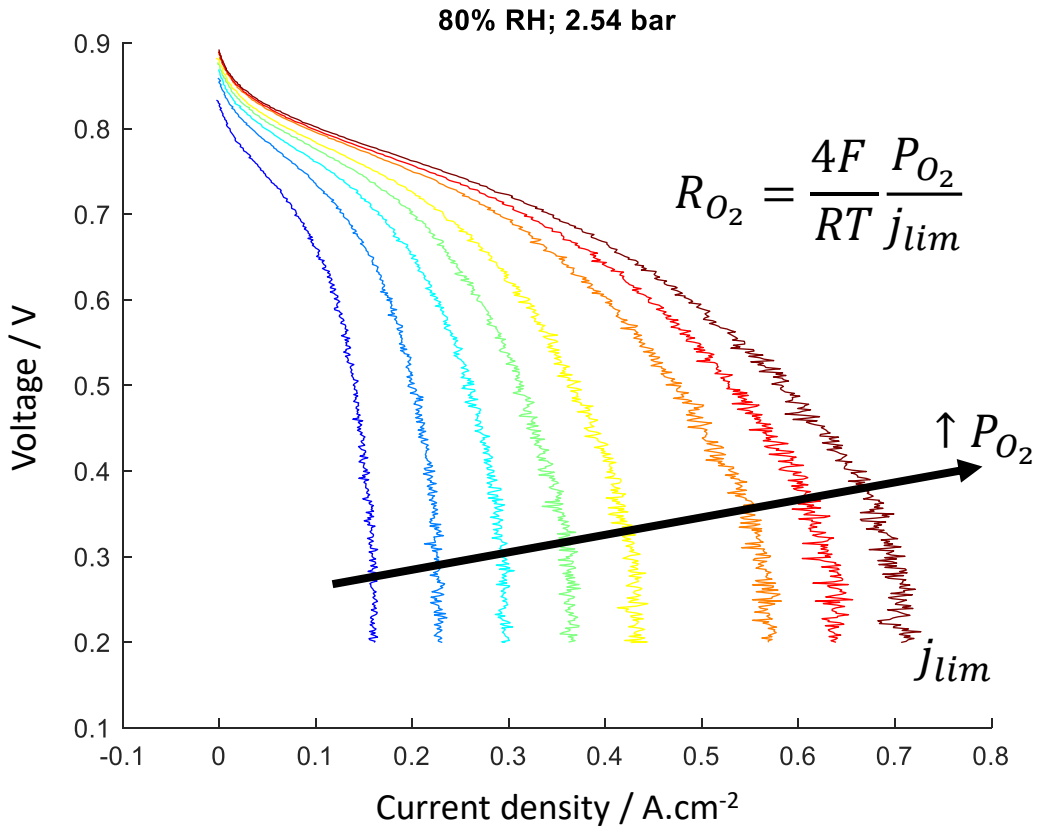
➢ Increased water condensation as current density increases

**FURTHER-FC**

**→ J @ 0.2V 80/80%RH, $x_{O2}$ < 2%**

# $O_2$ transport limitations
## Limiting Current Analysis (LCA) results

I-V for different $O_2$ concentration



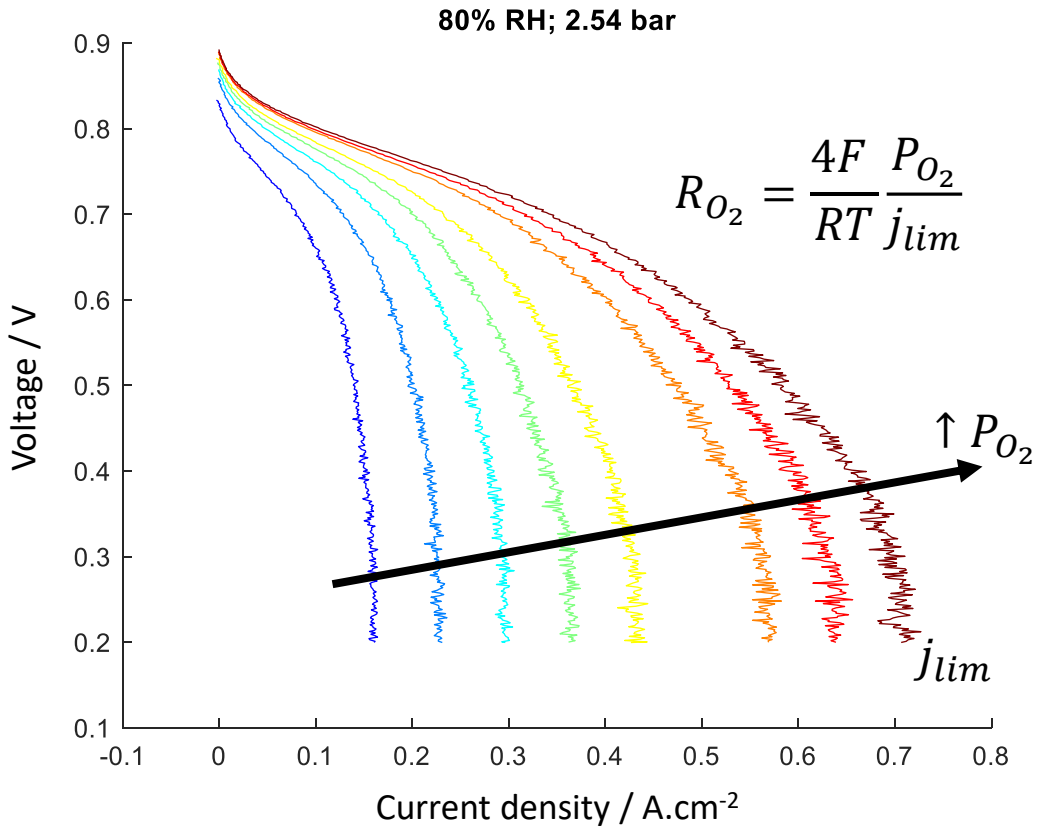

Baker *et al.* **2009** *J. Electrochem. Soc.* **156** B991

**Pressure Dependent** (PD) term:
- not impacted by CCL variation
- only impacted if non-reproducible parts of the GDL was used

**Pressure Independent** (PI) dominated by CCL composition and structure and decreases with:
- ➢ increasing roughness factor (more active sides)
- ➢ decreasing I:C ratio (less ionomer coverage and water)
- ➢ GC as Pt support vs HSAC (no diffusion in nanopores)
- ➢ increasing Pt:C ratio (thinner CCL)
- ➢ HOPI vs D2020, but limited to HSAC support and not visible for GC

# $O_2$ transport limitations
## Limiting Current Analysis (LCA) results

I-V for different $O_2$ concentration

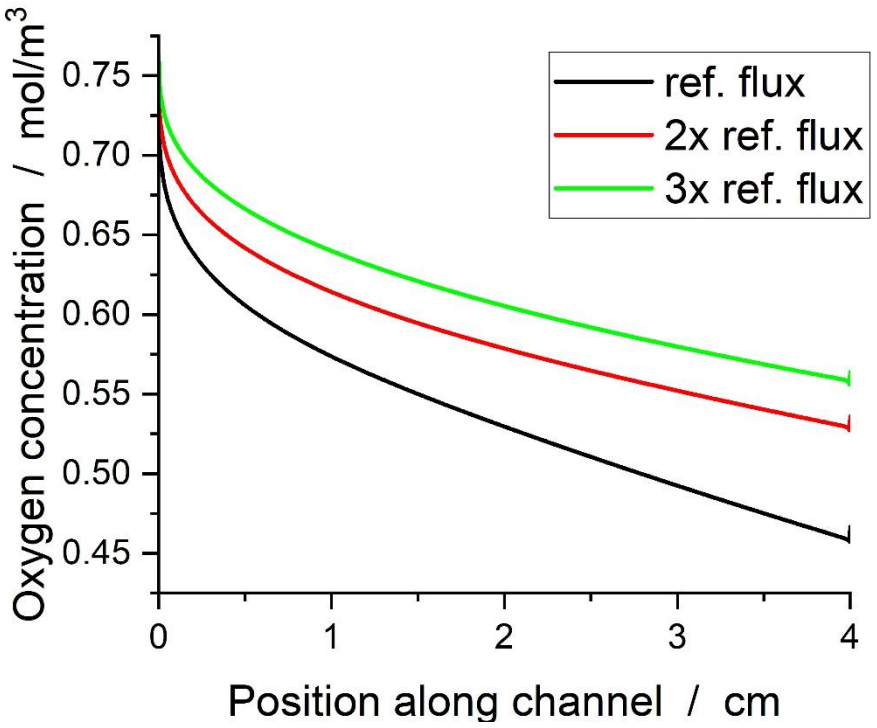



Baker *et al.* **2009** *J. Electrochem. Soc.* **156** B991

**Limits**

- Not possible to dissociate the different contributions in the PI
- Not possible to have reliable information as a function of RH
- $O_2$ through-plane transport limitations in the channel

$O_2$ concentration at the GDL/channel interface along the channel



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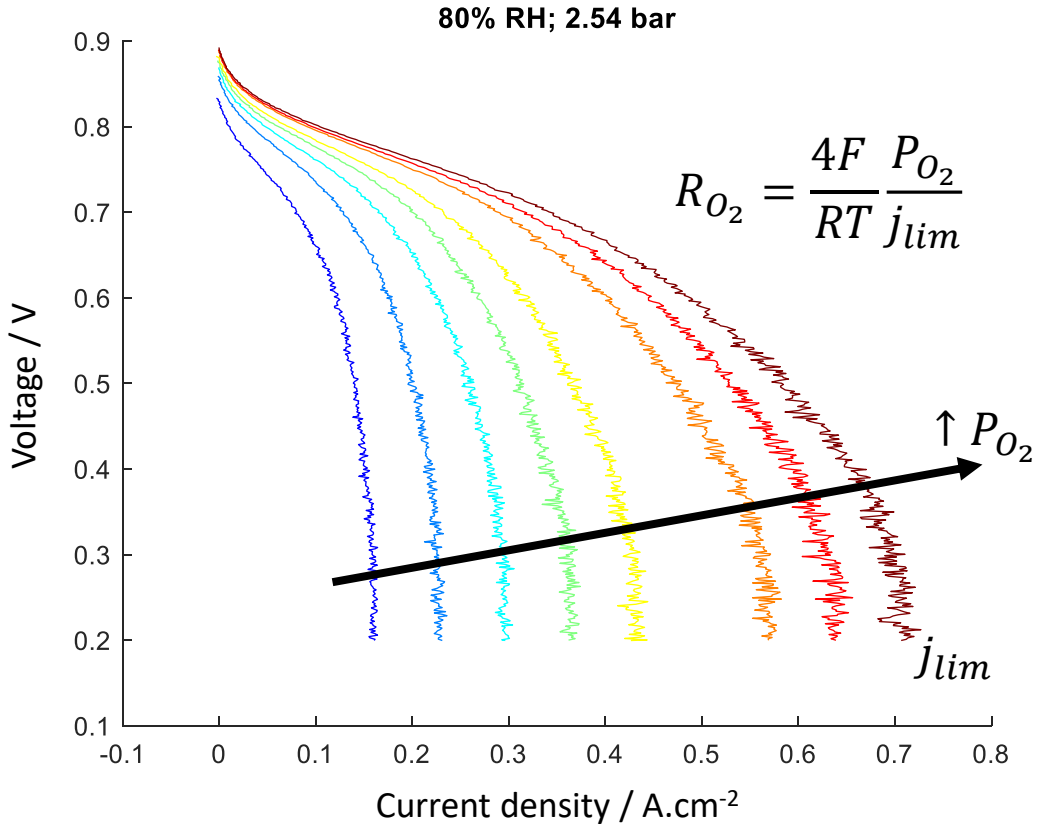
I-V for different $O_2$ concentration

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**Limits**

- Not possible to dissociate the different contributions in the PI
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- $O_2$ through-plane transport limitations in the channel

**FURTHER-FC approach:**

→ Use **numerical models to analyse LCA**

→ Use **LCA to validate the models**

# OUTLINE

General considerations

CCM losses

Proton transport resistance thanks to EIS measurements

What else can we extract from EIS?

Oxygen transport resitance thanks to LCA measurments

**Decoupling electrokinetic from transport limitations**

Conclusion

# Decoupling transport from kinetic

Kinetic often described with Butler-Volmer equation

$$i_r = i_0 \gamma_{CL} \left[ \exp\left(\frac{\alpha n F}{RT}\eta\right) - \exp\left(-\frac{(1-\alpha) n F}{RT}\eta\right) \right]$$

$$i_0 = i_0^{\circ} \left( \prod_{\nu_j > 0} a_j^{\gamma_j} \right)^{1-\alpha} \left( \prod_{\nu_j < 0} a_j^{-\gamma_j} \right)^{\alpha}$$

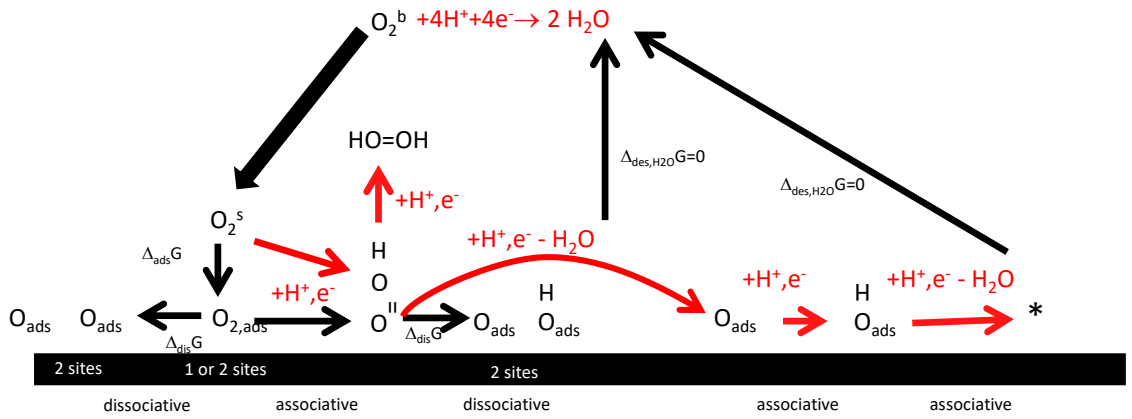
$$i_0^{\circ} = nF\left(k_{ox}^{\circ}\right)^{1-\alpha}\left(k_{red}^{\circ}\right)^{\alpha}$$

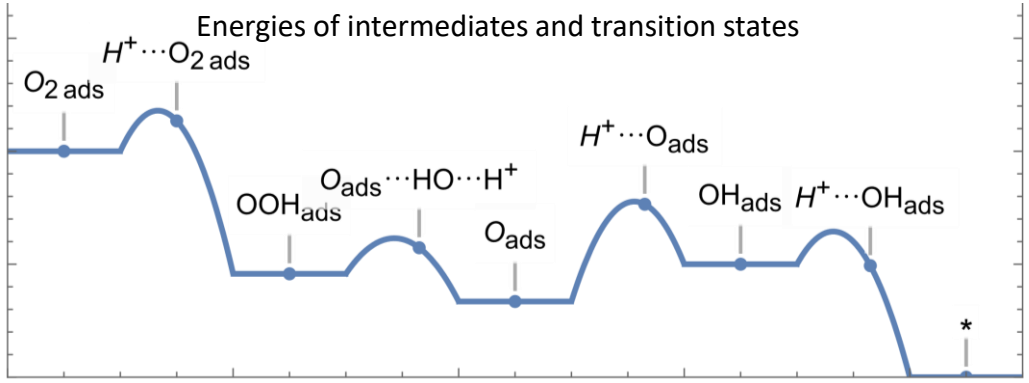
$$\eta = \emptyset_{e^-} - \emptyset_{H^+} - E_{eq}$$

Depends on the local concentration of reactant and product
→ **Depends on $O_2$ transport limitations**

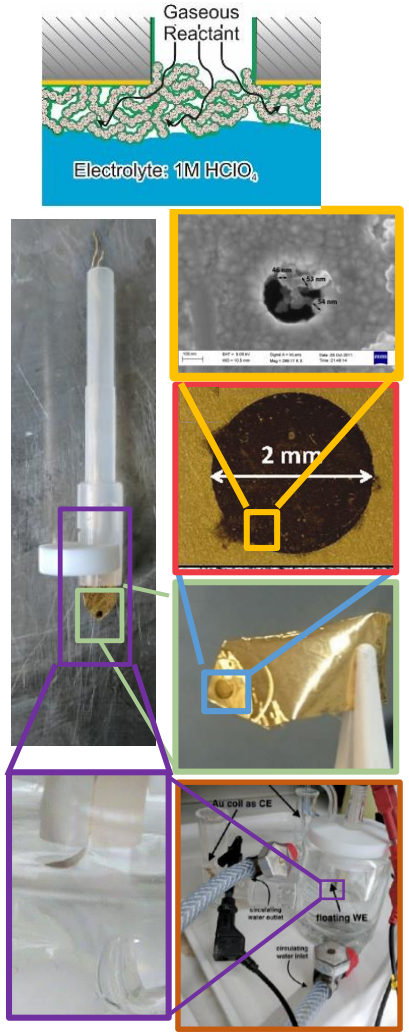
Depends on the local potential within the electrolyte
→ **Depends on $H^+$ transport limitations**

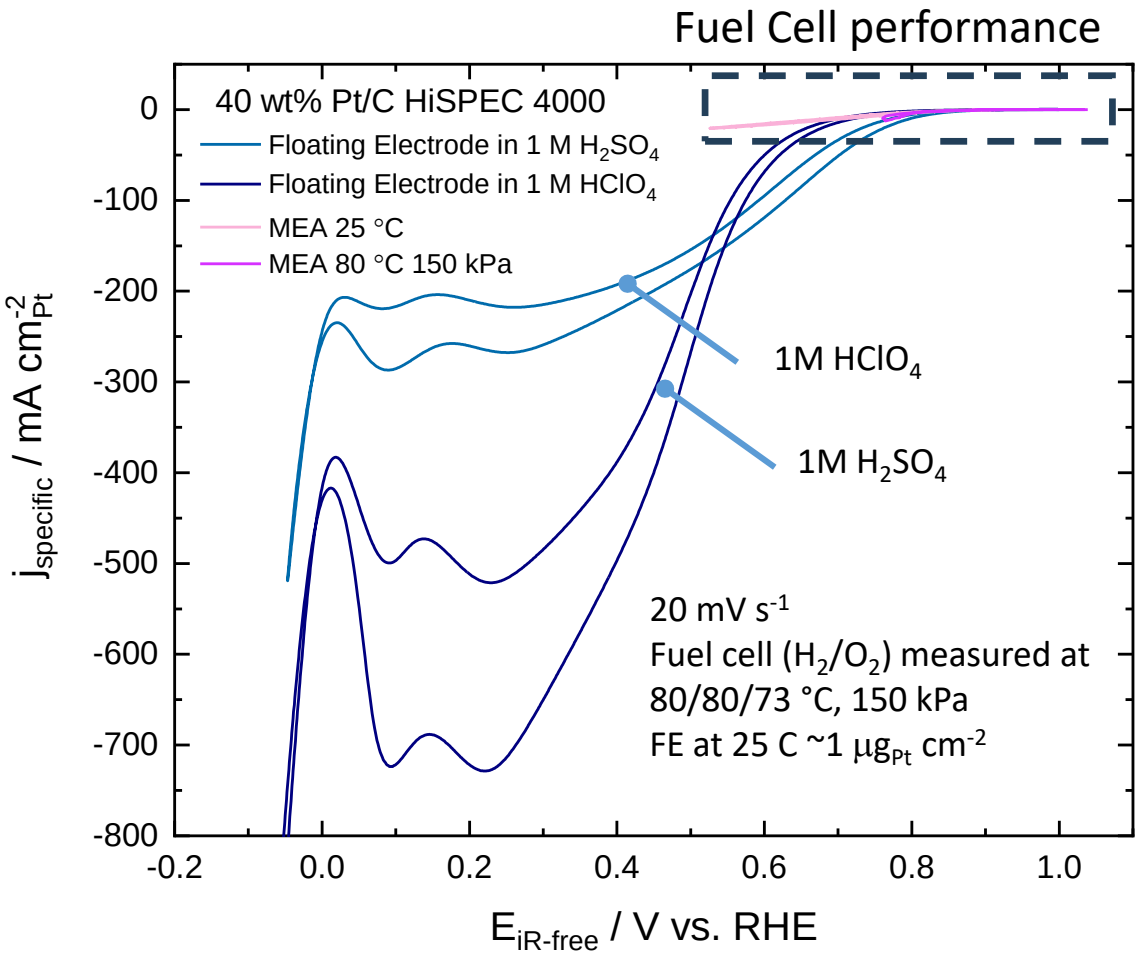
**Extremely difficult to decouple kinetics from transport limitations experimentally**

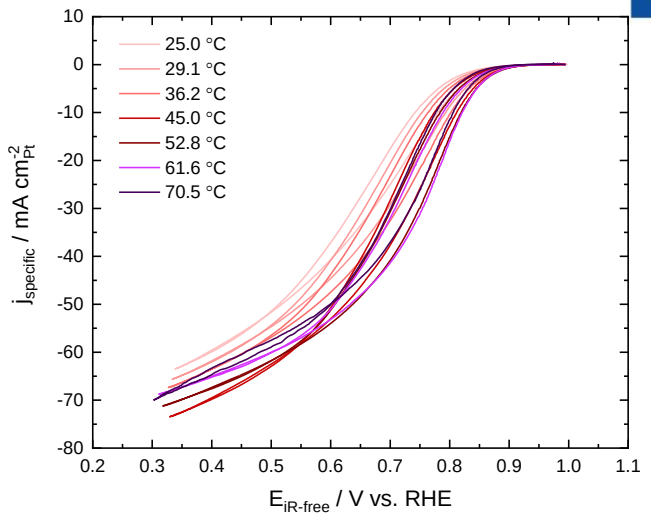


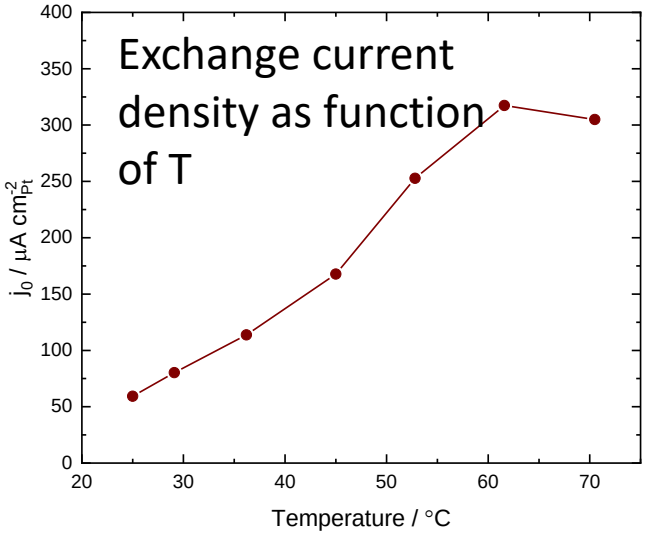



# Decoupling transport from kinetic
## Floating electrode



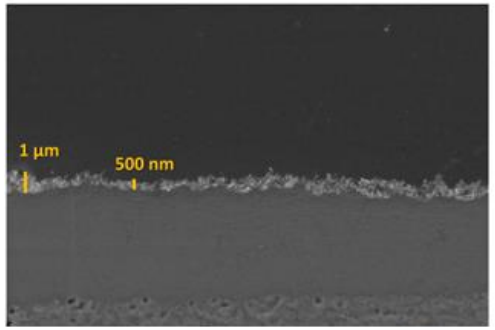

Jackson, S Kucernak et al, . *ACS Catalysis, 2022, 12*(1), 200-211. doi:/10.1021/acscatal.1c03908

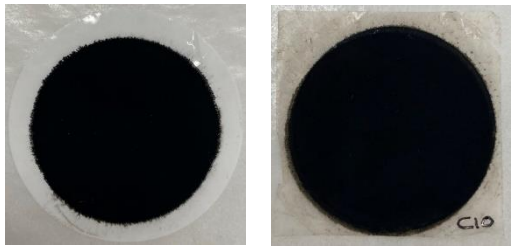
Conclusion: Pt has plenty of activity

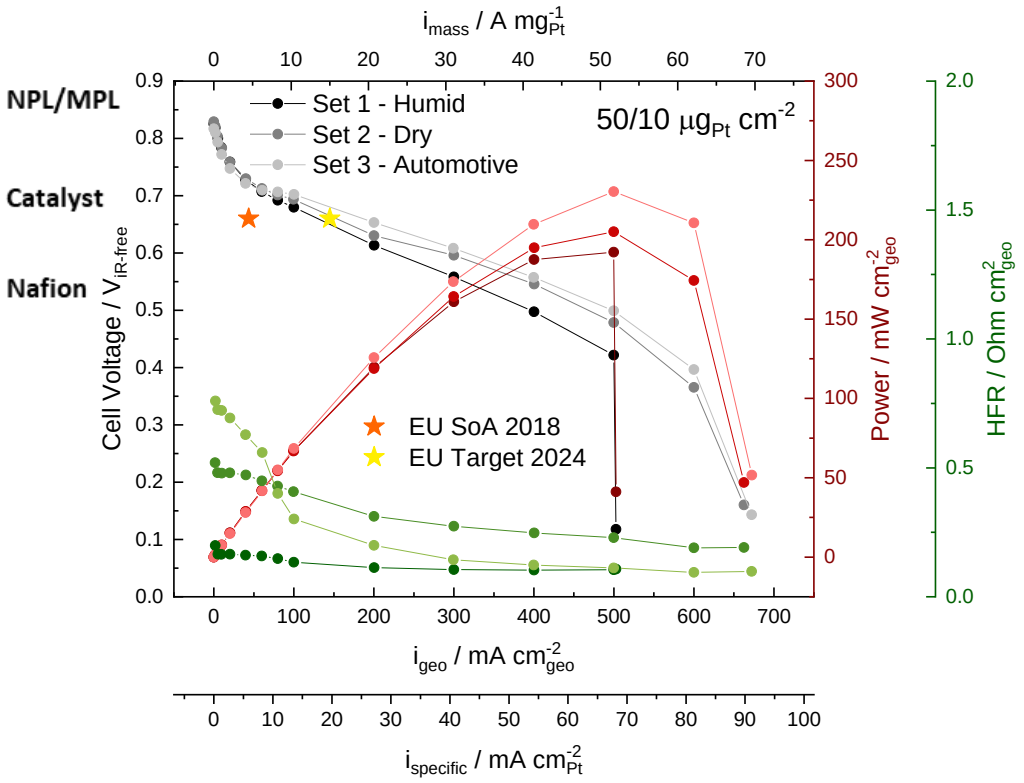
# Decoupling transport from kinetic

## Ultrathin CCL

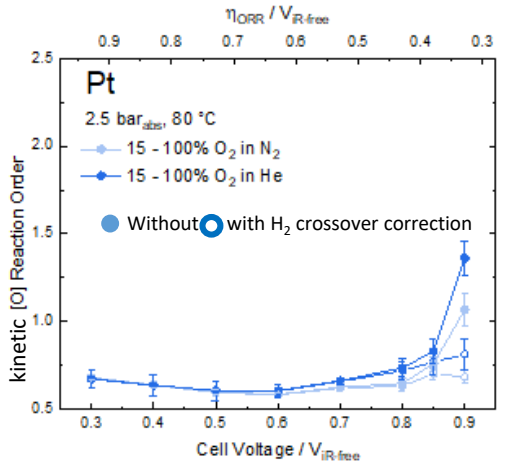
Imperial College London

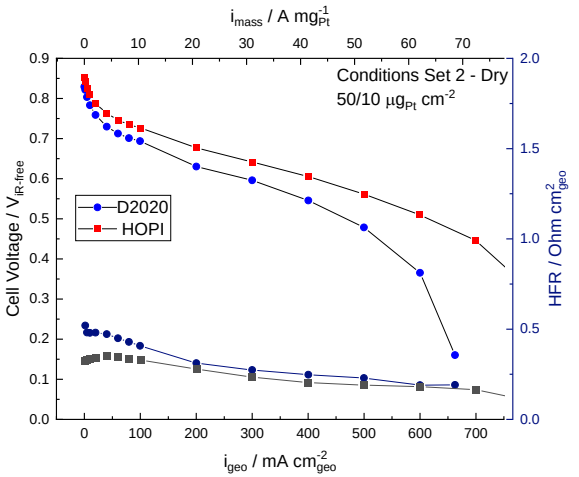


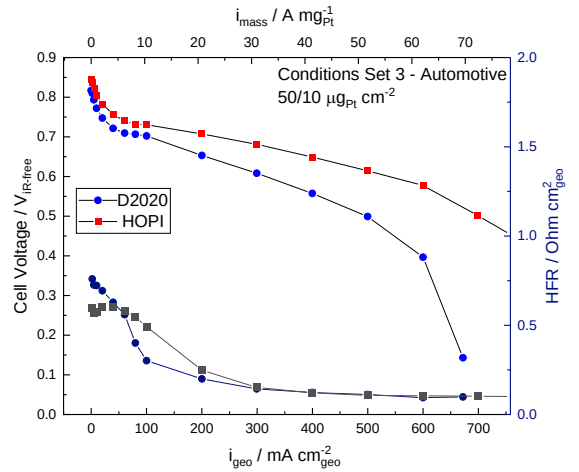






CCMs with loading down to 3 $\mu g_{Pt} cm^{-2}$




In mass activity terms, catalysts can reach required performance goals at low loading

- Kinetic reaction order is between 0.5-0.75 (0.3-0.8V)
- Mass transport at catalyst/ionomer interface not an issue
- For 10 $\mu_{Pt}$ $cm^{-2}$ cathode layer, significant shift in performance when using HOPI across entire current range

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# Conclusions and FURTHER-FC approach

- Transport limitations very difficult to quantify properly experimentally
- Coupling between transport and electrokinetic will always exist
- Simple Butler Volmer model is insufficient to describe electrochemistry of ORR on Pt
- Not possible to quantify with a simple analytical model the impact of transport losses on performance

**FURTHER-FC approach:**

**Do not use this simplifed analytical approach**

→ **Compute the transport properties** from the « real » structure of the components

→ Reduce the number of parameters to be fitted by the model

→ **Use physical model** to directly fit the experimental data

→ Quantify the contribution of the different components on the performance losses

# The TEAM


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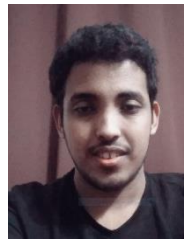
Dr. Isotta Cerri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Dr. Joël Pauchet


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Dr. Thomas Jahnke


Pr. Anthony Kucernak


Dr. Colleen Jackson


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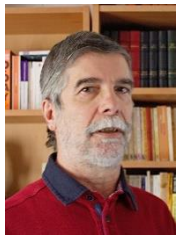
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