HETEROGENEITIES AT THE CELL LEVEL BY MODELING AND EXPERIMENTS

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Fraunhofer Institute for Solar Energy Systems ISE

First Workshop on Multiscale Modeling for PEMFCs

Grenoble, June 12-13, 2014

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AGENDA

- Fuel Cell Systems Department at Fraunhofer ISE
- Motivation
- Spatially Resolved Analysis
  - The Multi-Channel Characterization System (MCCS)
  - The Segmented Cell
- Local Measurement of Current, Voltage, EIS and HFR
- Special Cases
  - Local Perturbation
  - Hydrogen Evolution on the Cathode
- Conclusion / Outlook

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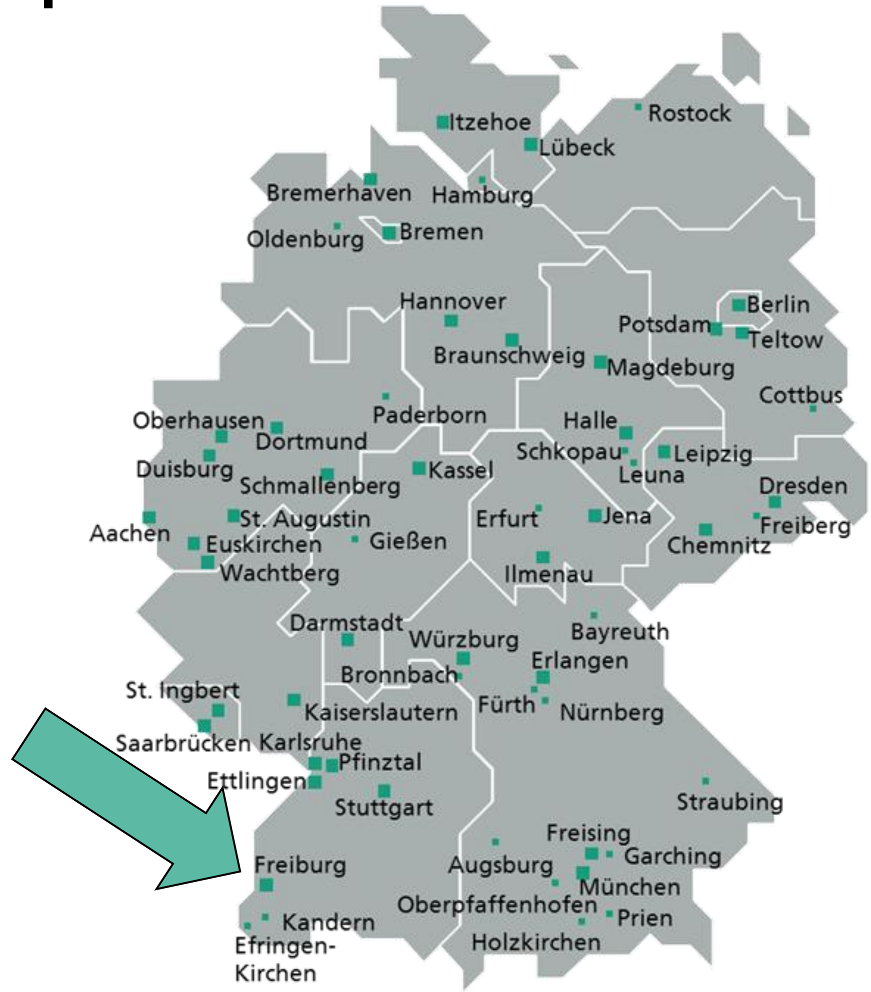
The Fraunhofer-Gesellschaft is the largest organization for applied research in Europe.



- 22,000 employees
- 66 institutes & research units
- Budget 2 billion €
- > 70% with contract research
- growing international activities

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Our department Fuel Cell Systems is assisting industry for over 20 years now.

- Budget 2.2 Mio. € (2013)
- 7 engineers, 5 scientists, 2 PhD students, 1 Technician, ca. 15 students
- Markets:
  - automotive fuel cells
  - back-up power
  - portable generators & micro fuel cells

References

3M
BALLARD®
BASF The Chemical Company
CEKA CEKAtec AG
DAIMLER
EADS
elringklinger
EISENHUTH
DLR
DMT pe
Freudenberg
FuelCon Tomorrow's energy. We make it work.
FEINMETALL
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PM PROTON MOTOR Proton Power Systems plc Group
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Serenergy® The Power of Simplicity
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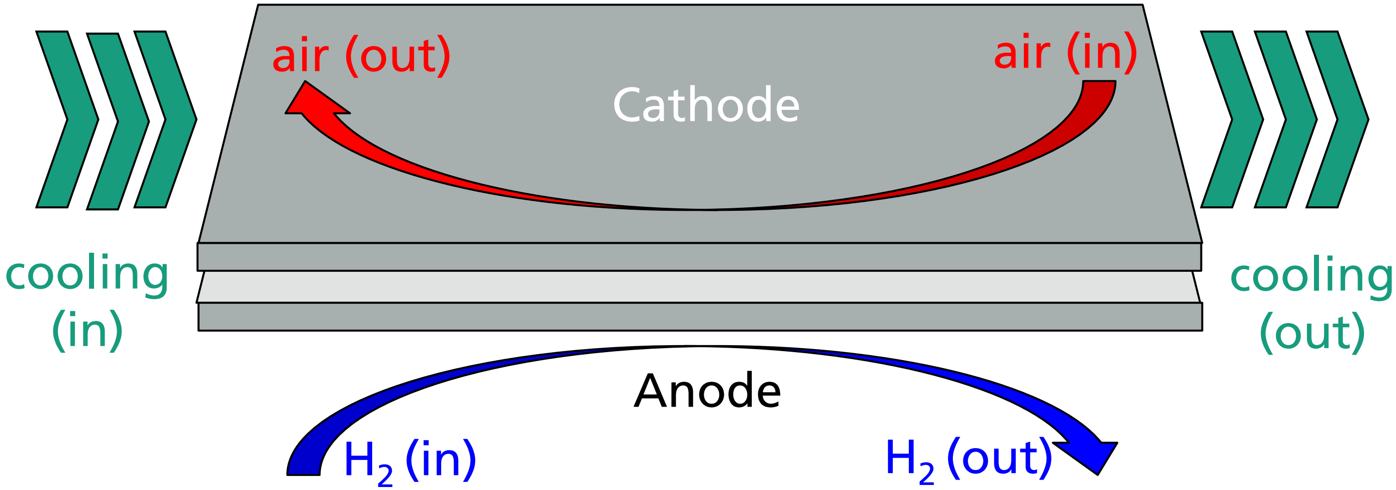
Technological breakthroughs need a new understanding of processes with high time and spatial resolution.

- Fuel cell electrical vehicle with long range
- Performance specifications and cost targets are hard to reach
  - Power/cell:
    > 1 W/cm^2 @ 675mV
  - Total operation time:
    > 5000 h
  - Freeze start capability:
    @ -20 °C
  - Cost per kW:
    30-40 €

Test drive with Daimler F-Cell of Fraunhofer ISE.

Different coupled processes within a fuel cell lead to inhomogeneities over the active cell area.



- Declining concentration of oxygen and hydrogen along the channel
- Rising humidity and temperature of gases along the channel
- Rising temperature of cooling liquid from inlet to outlet
- ➢ To optimize cell design, materials, and operation strategy these inhomogeneities must be understood

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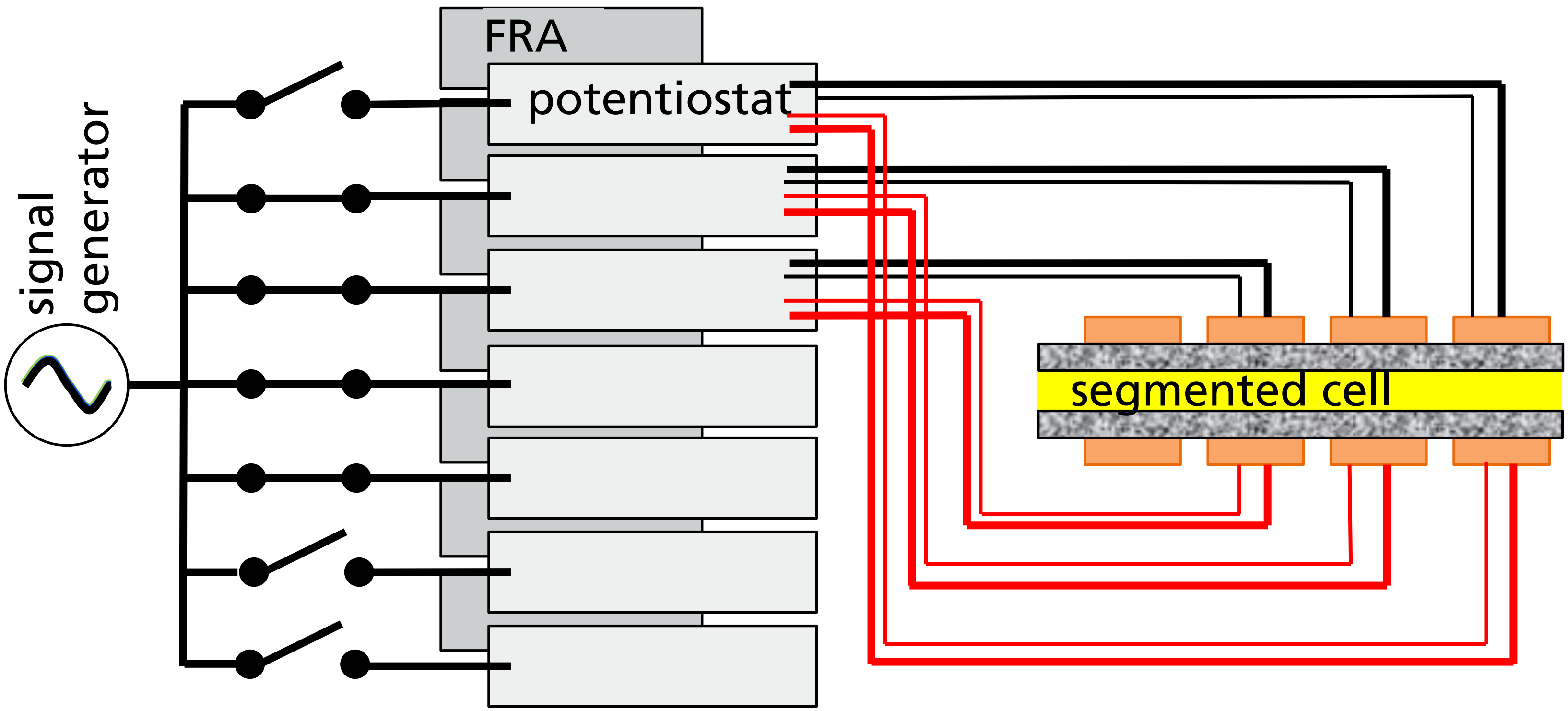
Schematic set-up of the MCCS with the segmented fuel cell

- Every segment is loaded by its own potentiostat (synchronized)
- Every potentiostat communicates with its own FRA



68-channel-characterization system for spatially resolved analysis of electrochemical energy converters

- Potentiostats:
  50 x (+/- 5 A; +/- 5 V) &
  18 x (+/- 30 A / +/- 5 V)
  together with 50 +18 FRAs
- Frequencies: 0.1 Hz to 10 kHz
- current / voltage mapping
  (up to 790 A)
- el.-chem. impedance spectroscopy
- chronovoltammetry, chronoamperometry

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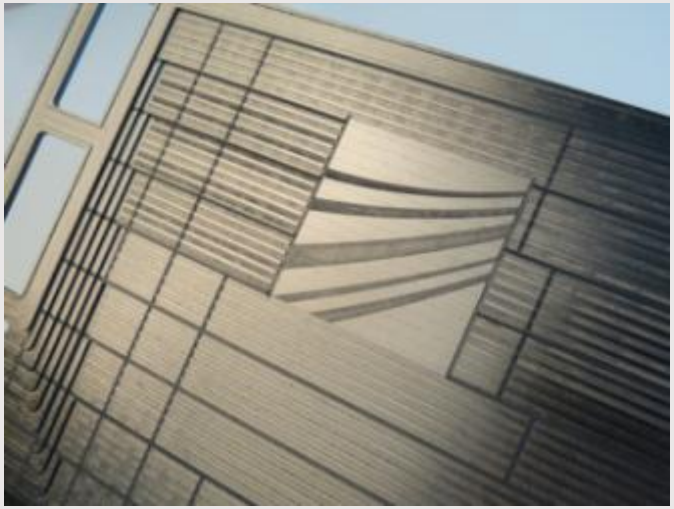
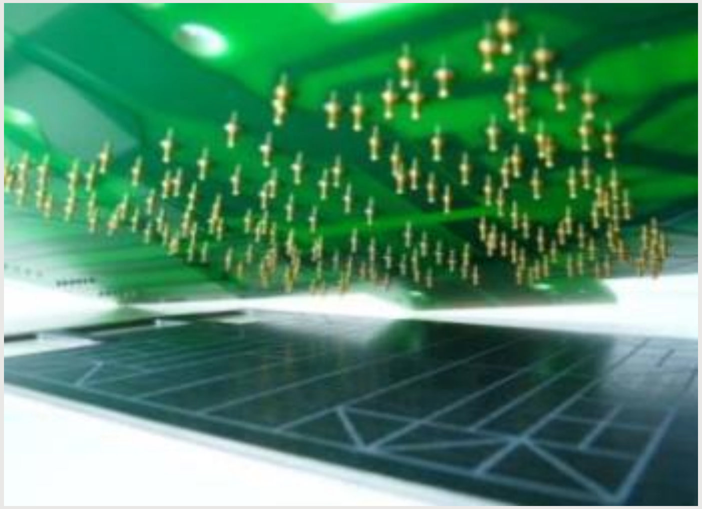
For spatially resolved cell characterization we segment the bipolar plate.

- Electrical isolation of the bipolar plate
- Electrical isolation of GDL and electrode possible
- Up to 68 segments



segmentation of cells



contact pins

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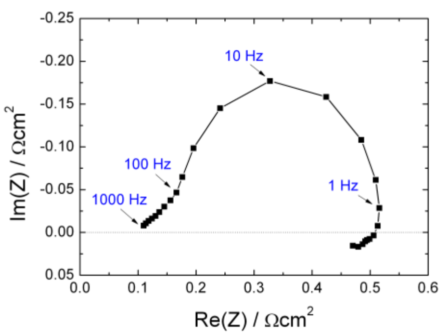
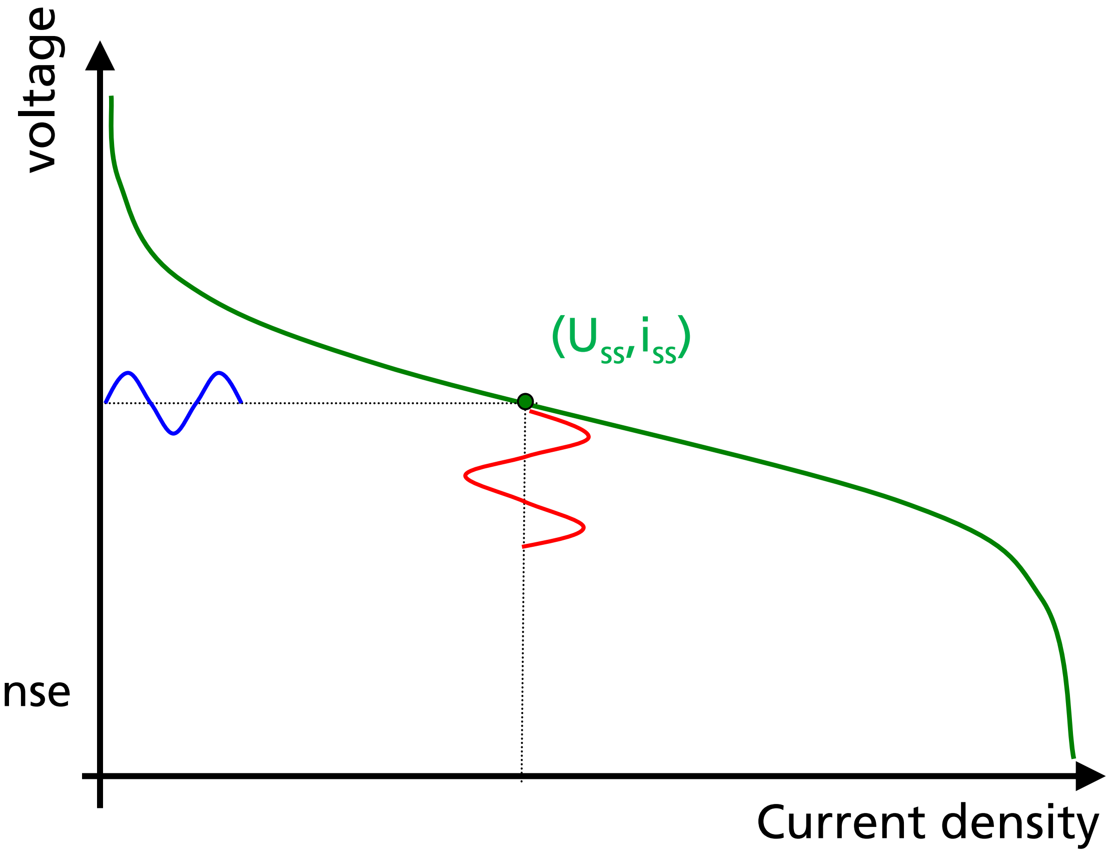
Electrochemical impedance spectroscopy (EIS) is measured at a specified steady state point.



- Steady-state point (U_{ss}, i_{ss})
- Superimposed by small perturbation $U(t) = U_{ss} + U_{AC} \sin(2\pi\, f\, t)$
- Frequency dependent response $i(t) = i_{ss} + i_{AC} \sin(2\pi\, f\, t + \varphi)$



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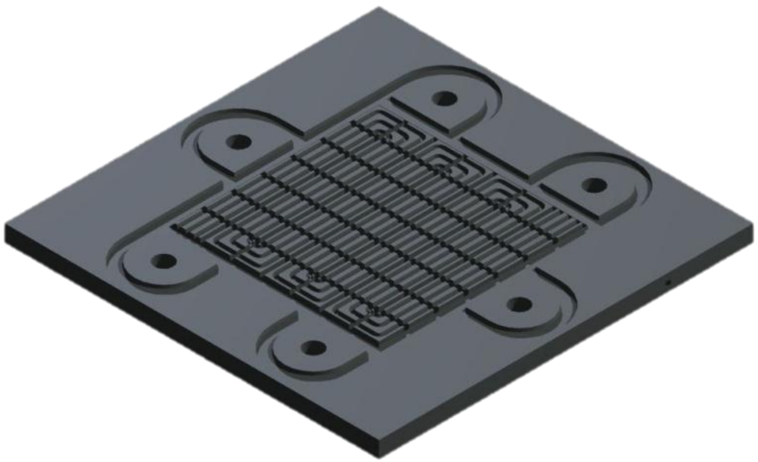
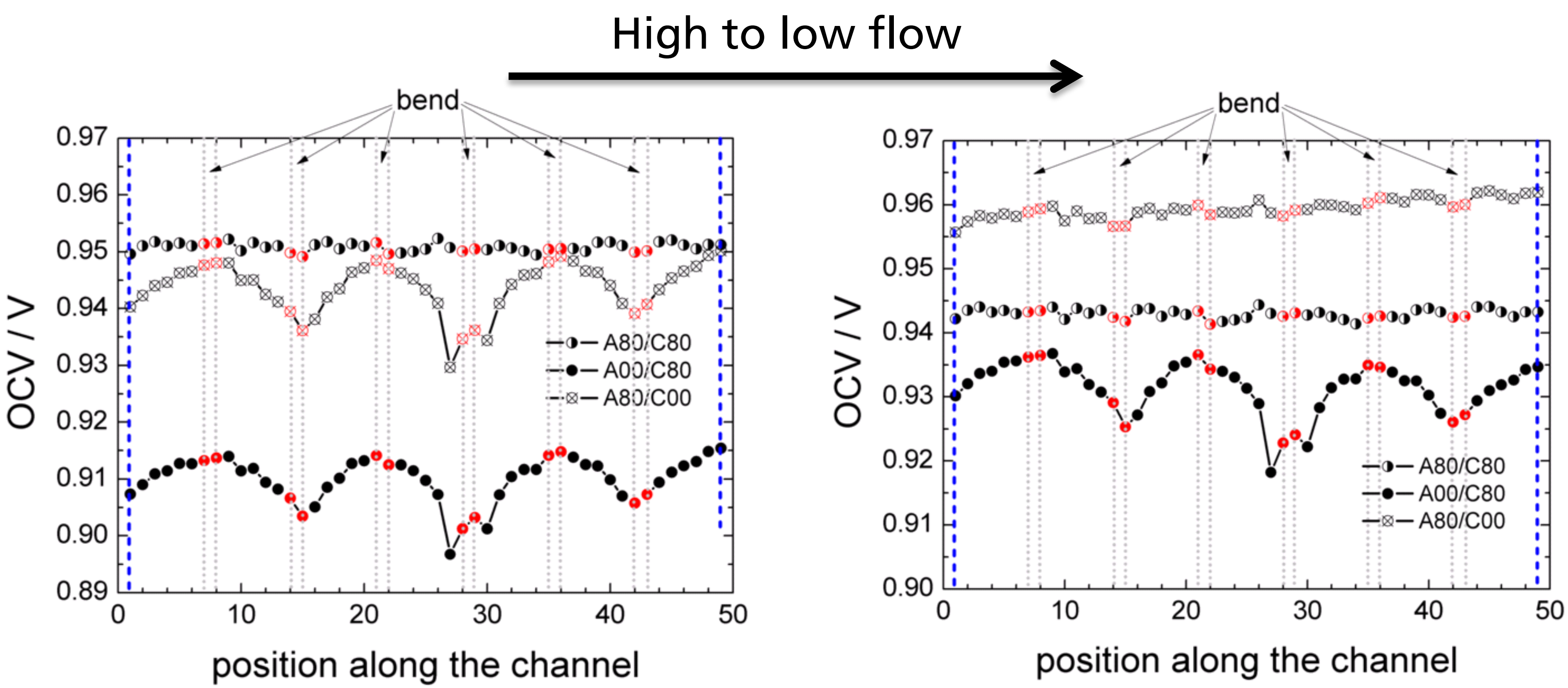
Spatially resolved characterization of a specific single cell



- Active area of 49 cm^2 (7 x 7 square matrix)
- Insulation between the segments achieved with epoxy
- 3 channel serpentine flow field
- Gore membrane with a SIGRACET GDL 25 BC

The hydration level of the cell can be monitored via its spatially resolved OCV

humidity and flow rate affect hydration of the membrane



Gerteisen et al. Int. J. Hydrogen Energy, 37 (2012) 7736-7744

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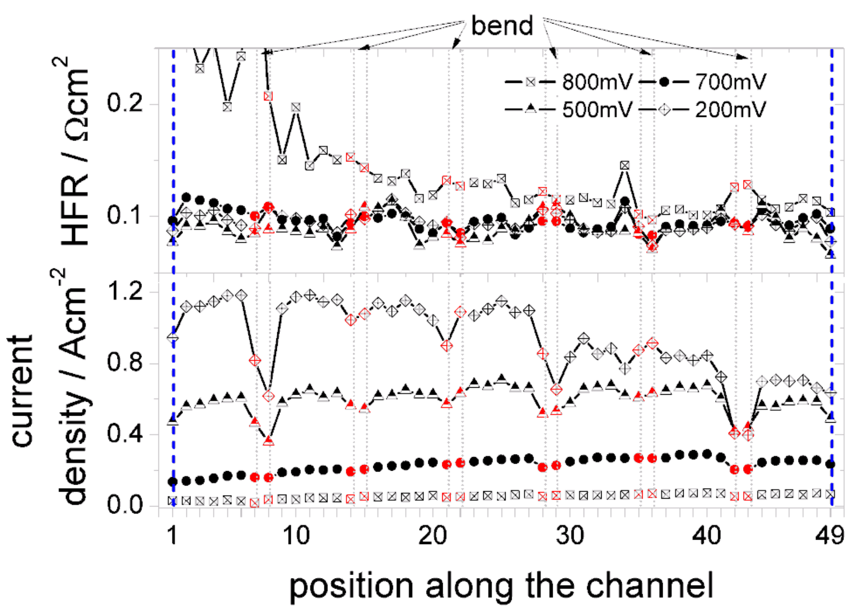
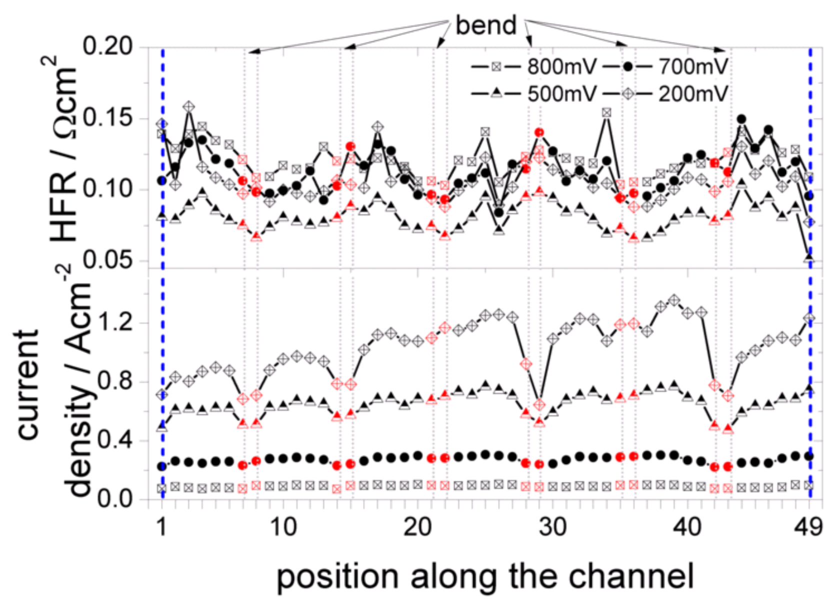
The hydration level of the cell can be monitored via its spatially resolved HFR and current density

flow configuration and its effect on hydration

Co-flow



Counter-flow



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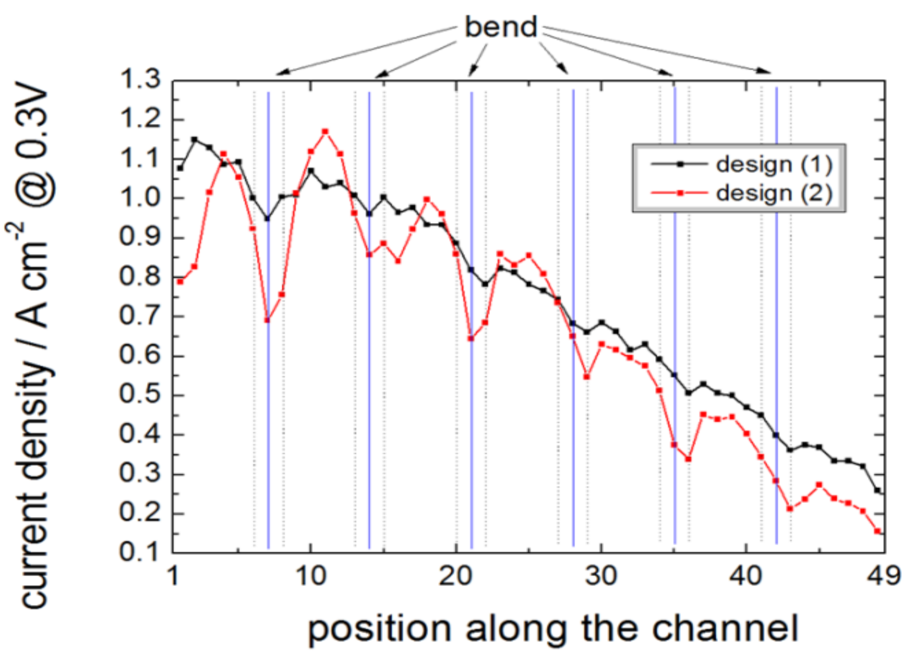
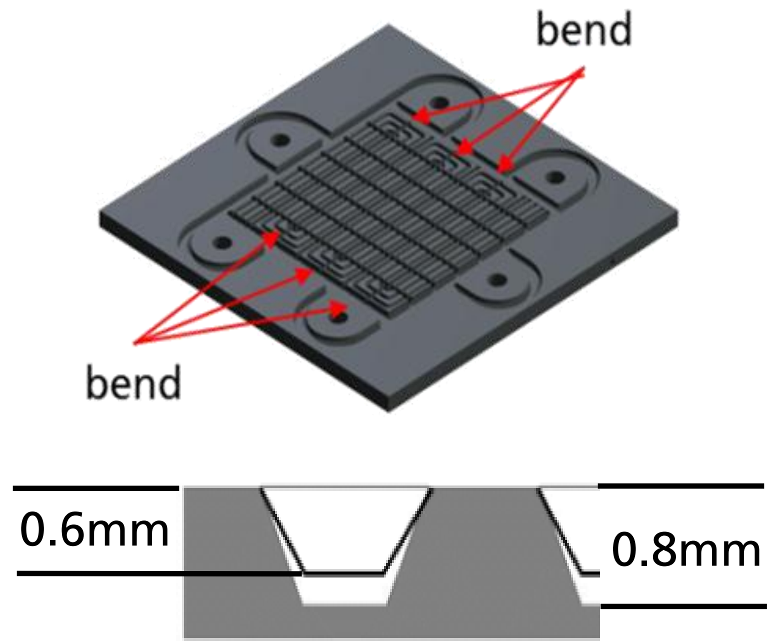
Spatially resolved characterization
development of cell design and operation strategy

- Current density is limited in the regions of the air channel bends
- Performance is improved by a flow field redesign





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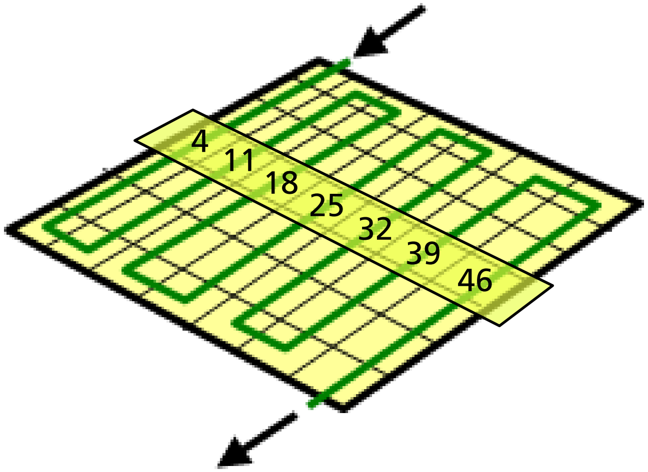
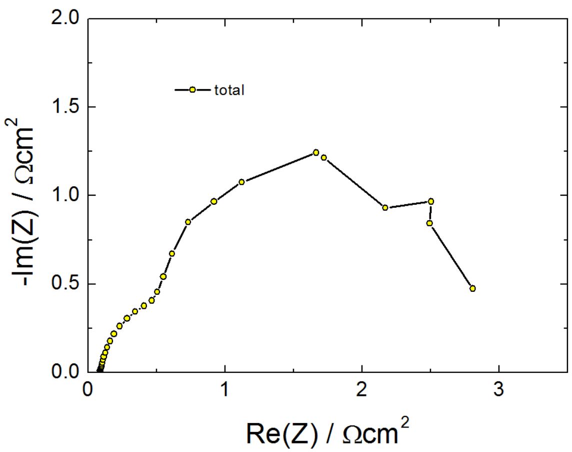
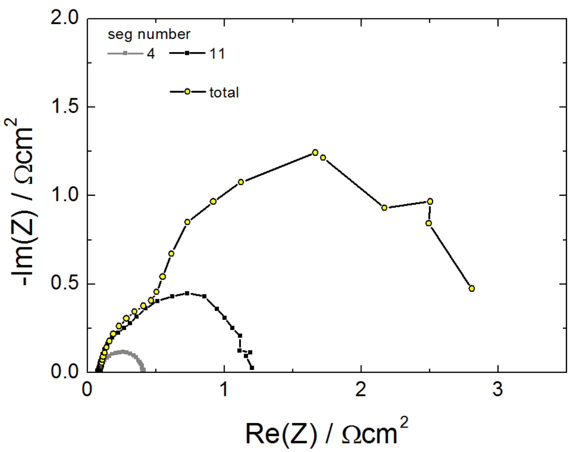
Local Analysis of the electrochemical impedance spectra

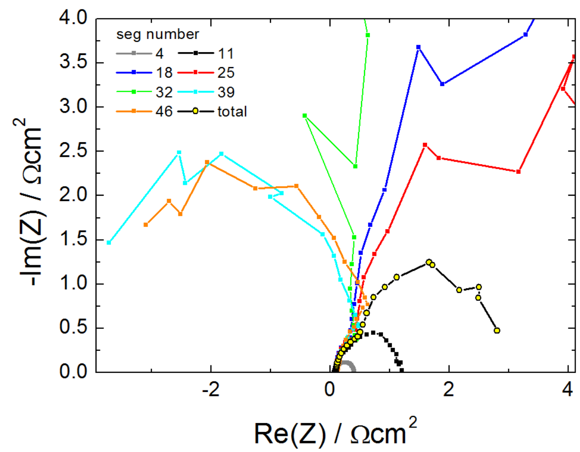
Operating conditions

- Anode: H_2 with λ_{H2}>>1
- Cathode: air with λ_{O2}<2 @ U<0.6V
- Co-flow mode
- Atmospheric pressure









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First explanation was given by Schneider et al.[1]

1. I.A. Schneider, S. A. Freunberger, D. Kramer, A. Wokaun & G.G. Scherer, J. Electrochem. Soc. 2007 154(4): B383-B388
2. T. Jacobsen, P. V. Hendriksen, S. Koch, Electrochimica Acta 2008, 53(25): 7500–7508
3. A. A. Kulikovsky, *J. Electrochem. Soc. 2012 159(7): F294-F300*

Basic idea:

d.c. current per segment: $i_{seg} = i_0\, c \exp(\eta/b)$

a.c. current response: $\Delta i_{seg} = i_0 \exp(\eta/b)\,(\Delta c + c\,\Delta\eta/b) - C_{DL}\, d_t\Delta\eta$

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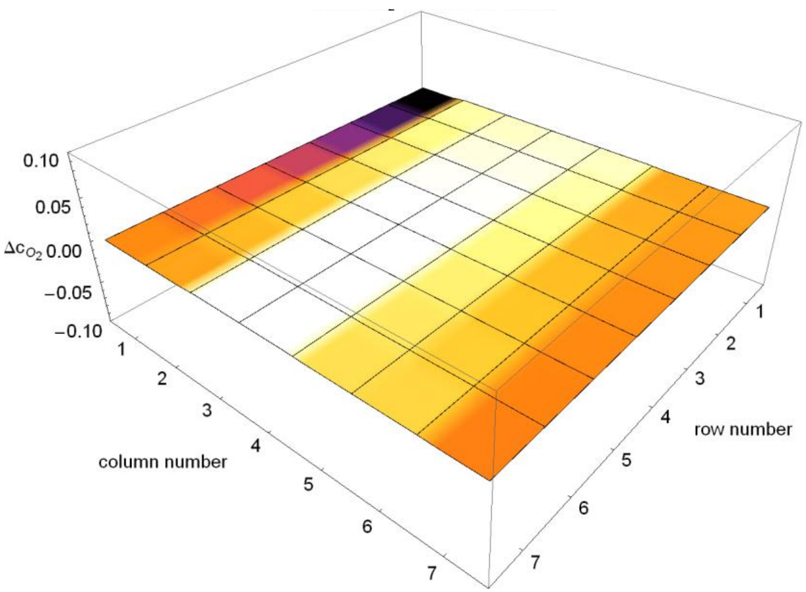
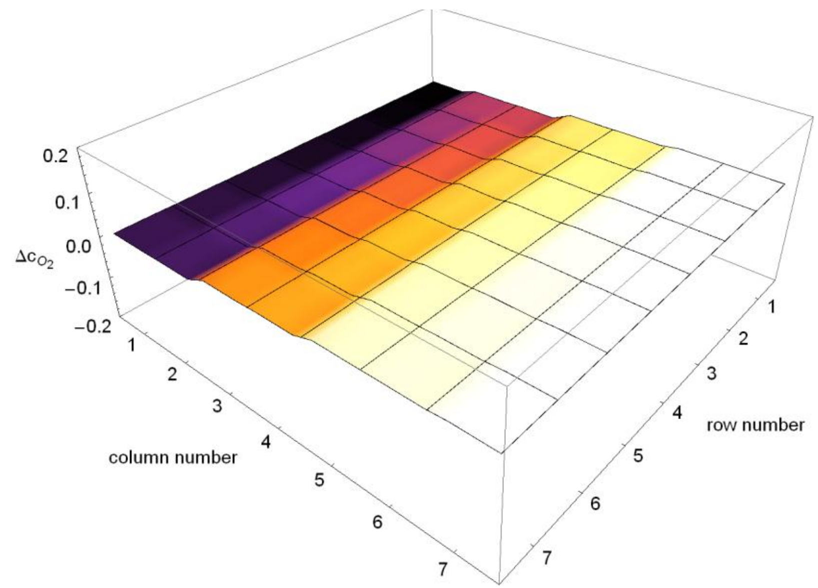
normed O_2 conc oscillation in channel

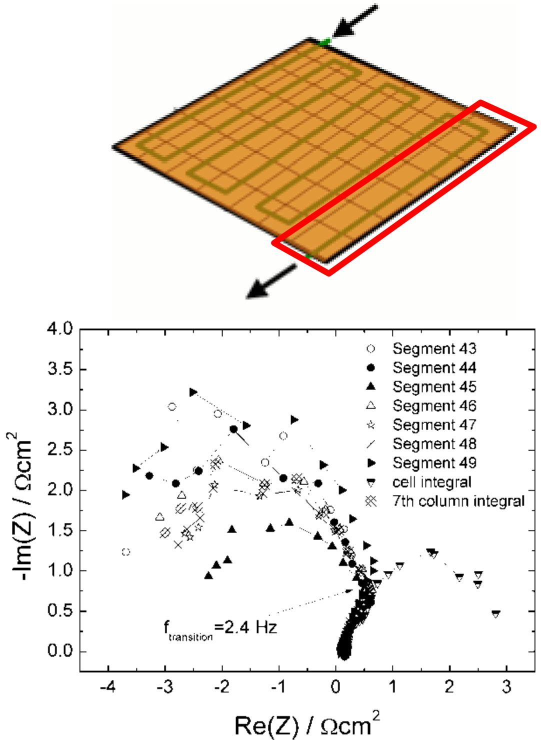


normed O_2 conc oscillation in CL



Measured impedance characteristic by local perturbation

- MCCS has the capability to perturb specific segments
- Perturbation of the **whole** cell (all segments)



Zamel et al. Fuel Cells, 13 (2013) 910-916

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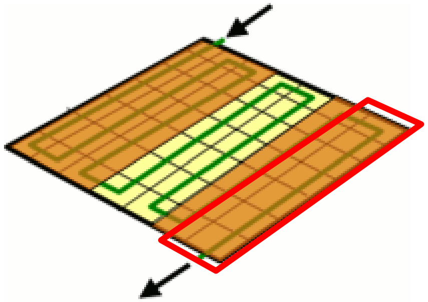
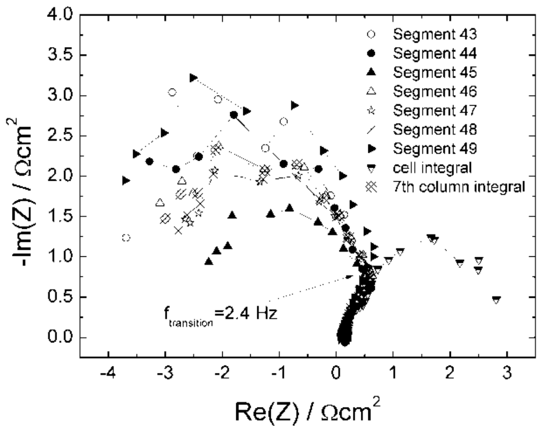
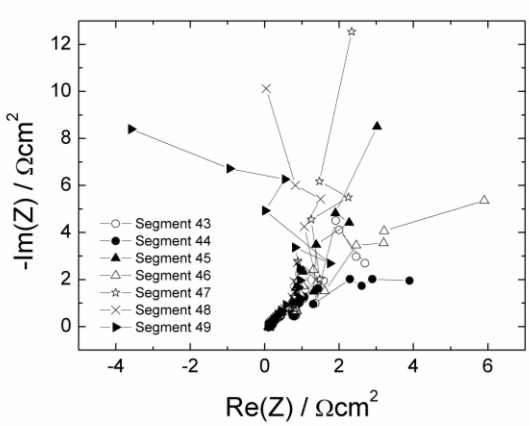
Measured impedance characteristic by local perturbation

- MCCS has the capability to perturb specific segments
- Perturbation of **col #1,2,3,6,7**

All segments of
col#7 are flipped







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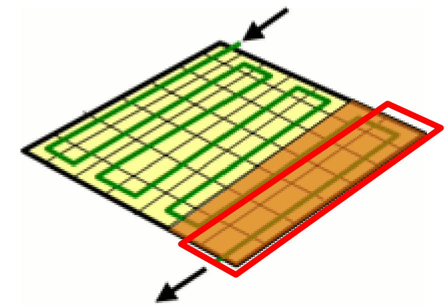
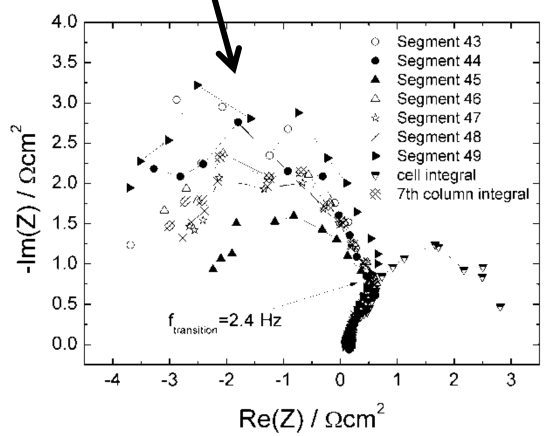
Measured impedance characteristic by local perturbation

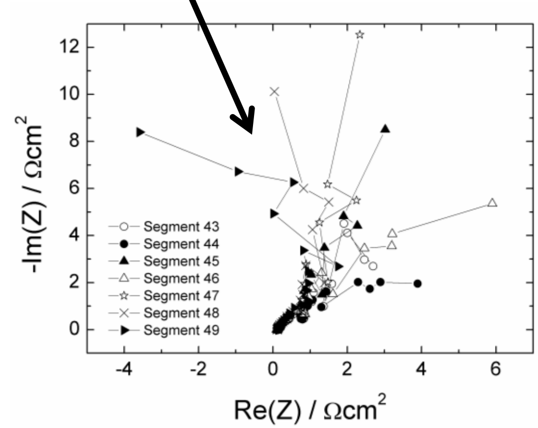
- MCCS has the capability to perturb specific segments
- Perturbation of **col #6,7**

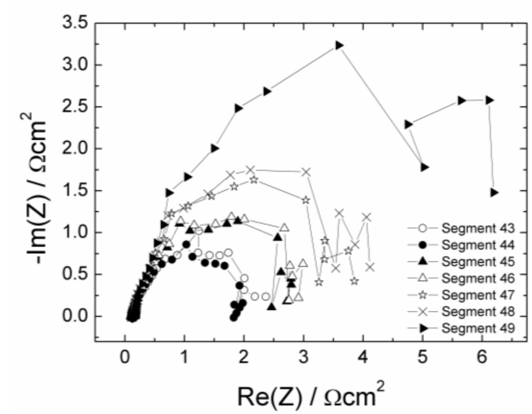


All segments of col#7 are flipped



Some segments of col#7 are flipped





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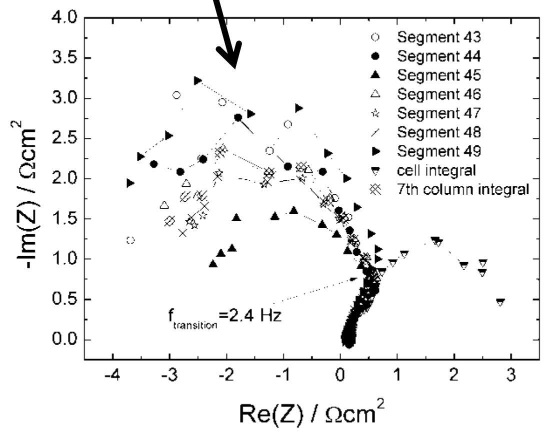
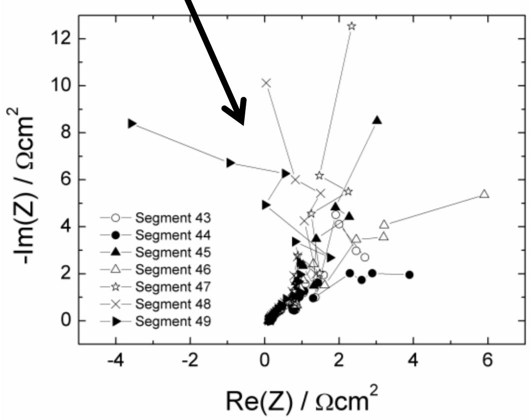
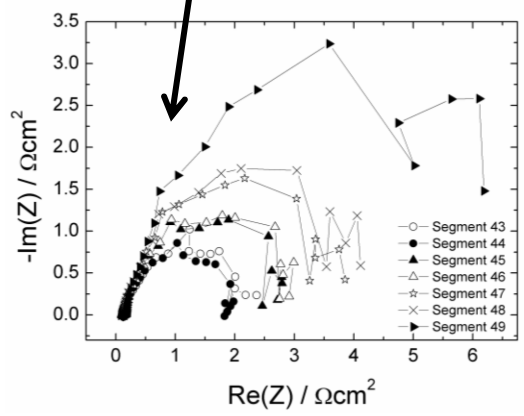


Some segments of col#7 are flipped



No segment of col#7 is flipped



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EIS-Simulation by a simplified 2+1D model

GDL

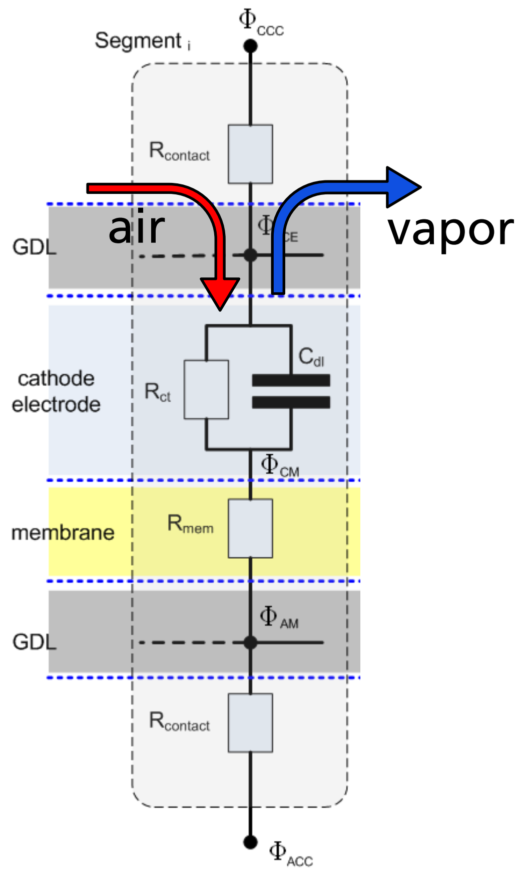
- Fick's diffusion of air and water vapor in GDL

CCL

- Tafel law describes charge transfer resistance
- Double layer charging

ACL

- Anode polarisation neglected



GDL

- Phase change of water in GDL
- Fickian diffusion approach for saturation

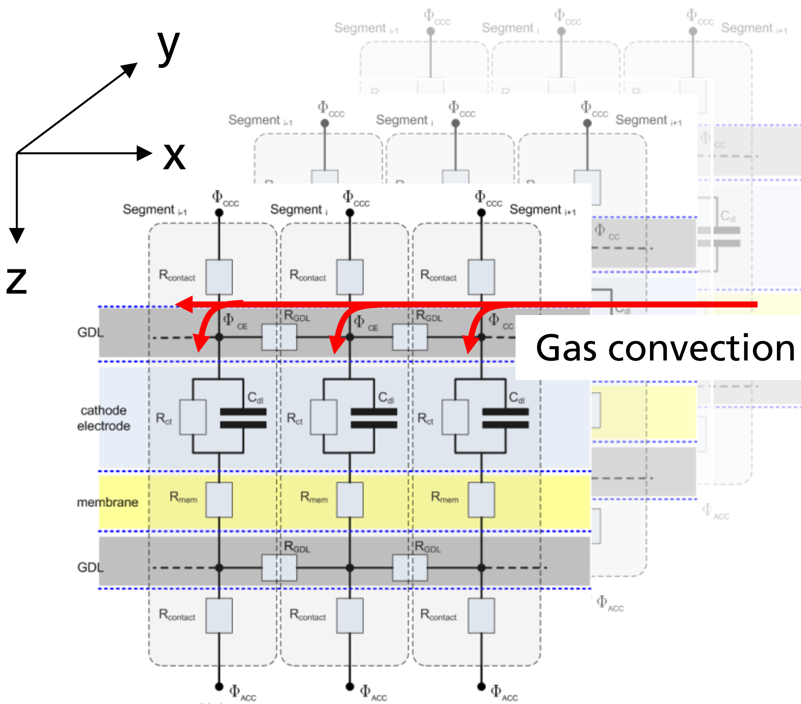
membrane

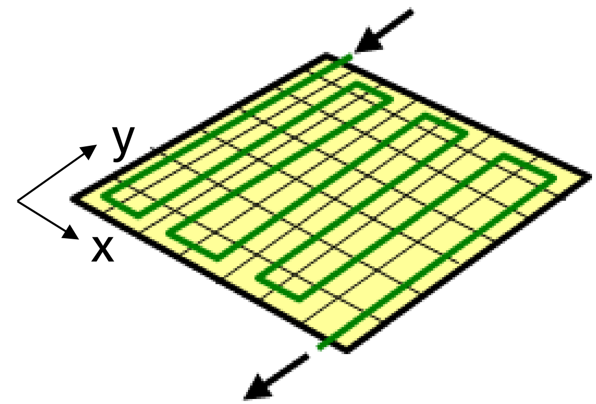
- Ohmic loss in membrane as a function of the water content

interfaces

- Contact resistance on cathode and anode

EIS-Simulation by a simplified 2+1D model





Fuel cell segments are coupled to nearest neighbors (x-y-direction) by

- Gas convection of air and vapor according to flow field pattern
- Cross current in GDL
- In-plane permeation of liquid water

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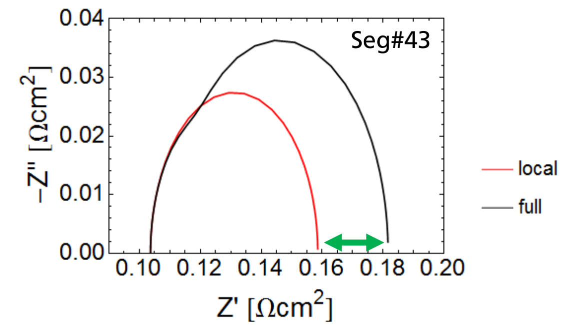
Stoichiometric impact on the local EIS

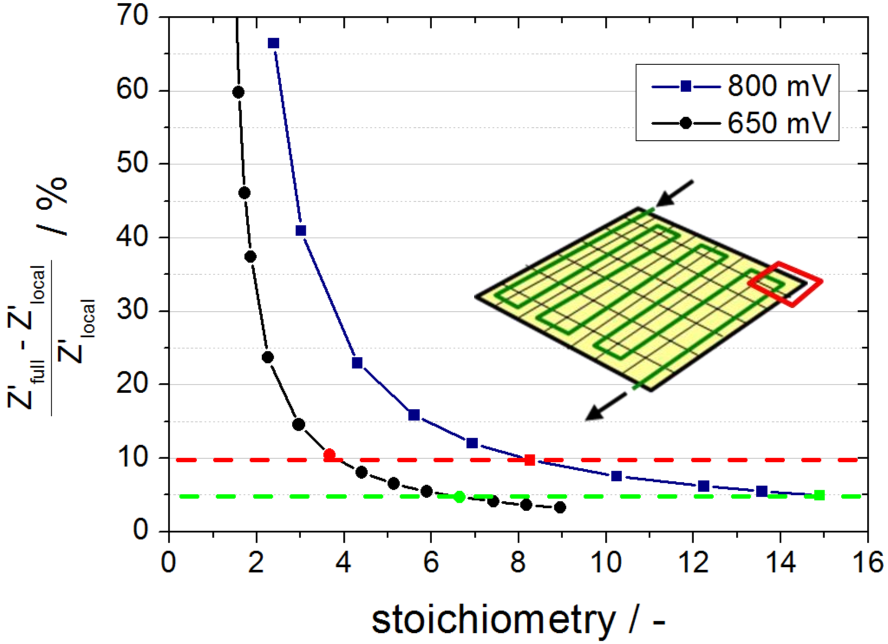
Comparison of full perturbation vs local perturbation @ low frequency (10mHz) shows…

- large deviance at $\lambda_{air} < 3$
- reduced artifact at high λ_{air}
  - error<5% @ $\lambda_{air} > 7$ @ 650mV
  - error<5% @ $\lambda_{air} > 15$ @ 800mV

→ High stoichiometry needed for analysing mass transport processes in the porous transport layers

→ Otherwise this effect has to be taken into account by modeling





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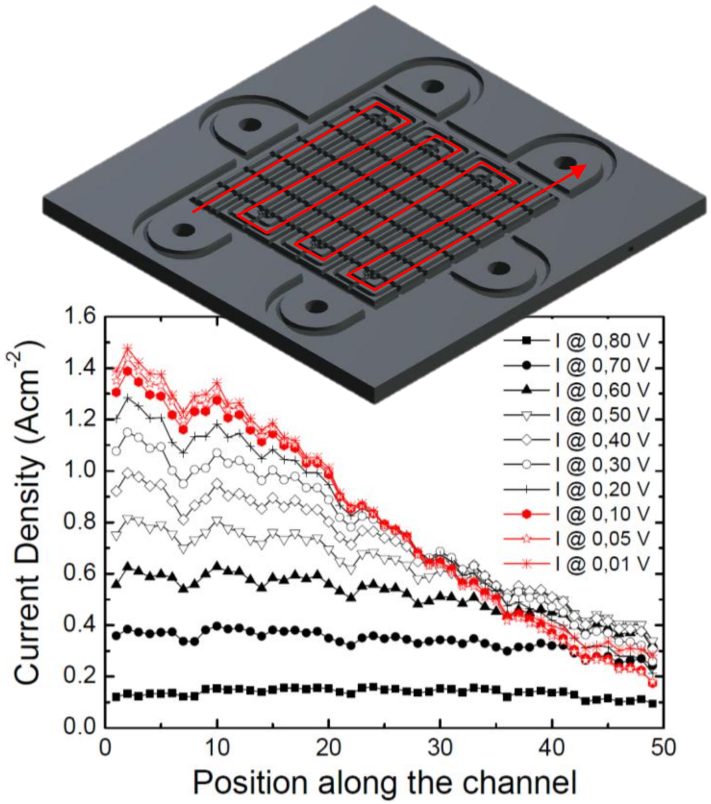
A bistable behavior in the performance of the cell is measured under the steady state condition

An increase in current is measured despite depletion of oxygen $\lambda_{o2} < 1$

- Spatially resolved polarization curves with steady-state operation



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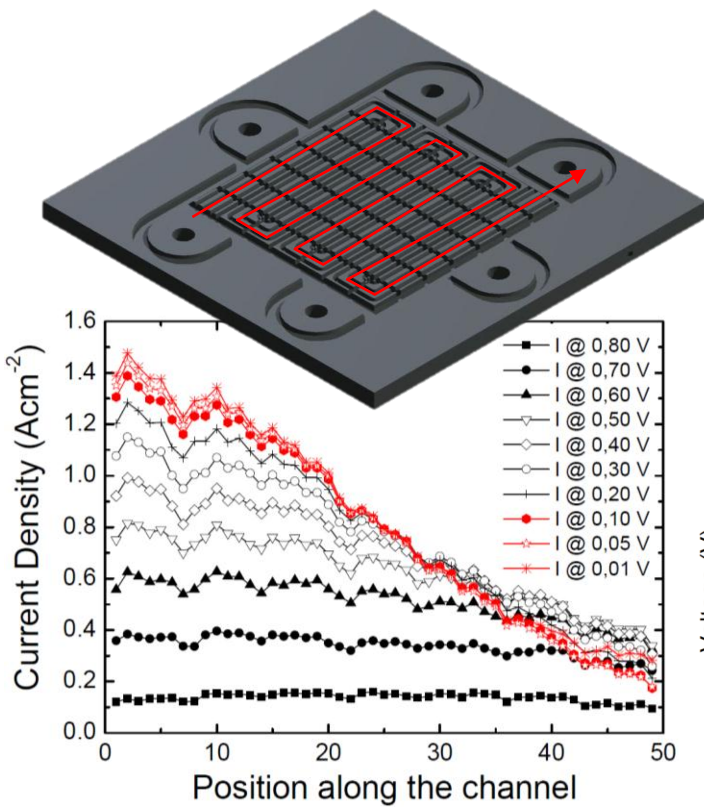

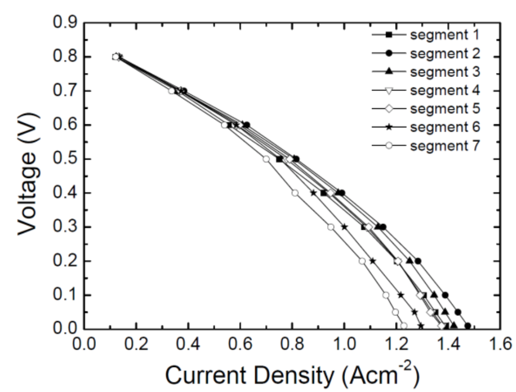

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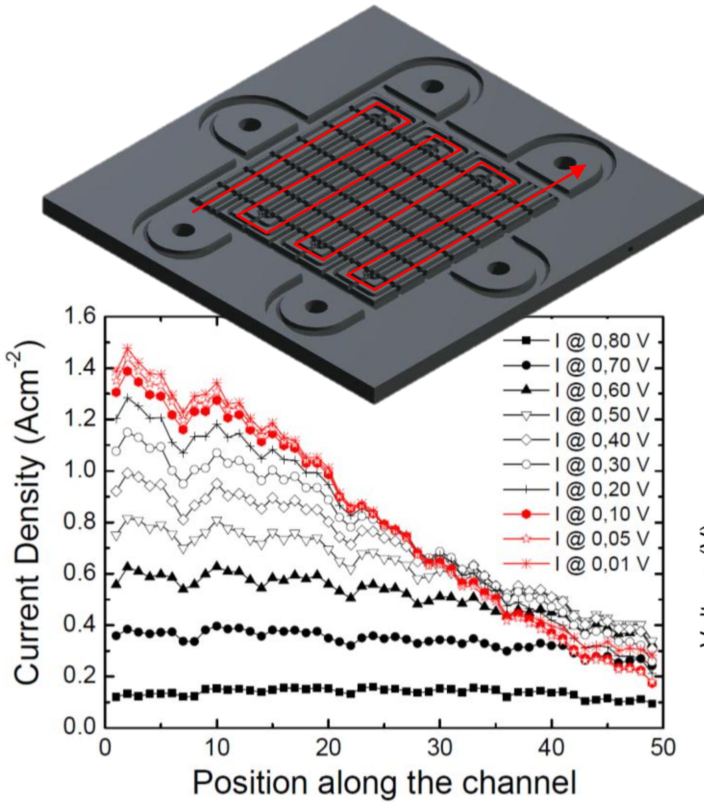
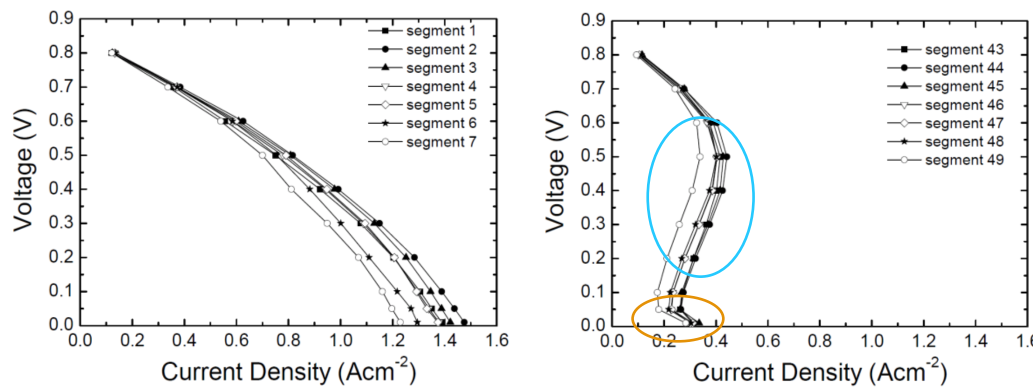
A bistable behavior in the performance of the cell is measured under the steady state condition

An increase in current is measured despite depletion of oxygen $\lambda_{o2} < 1$

- Spatially resolved polarization curves with steady-state operation
  - Bending back of the current due to coupling to the upstream segments
  - Current recovery at low but **positive** voltage





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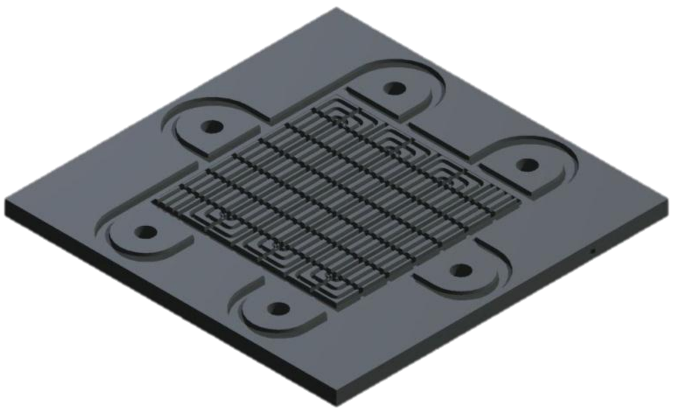
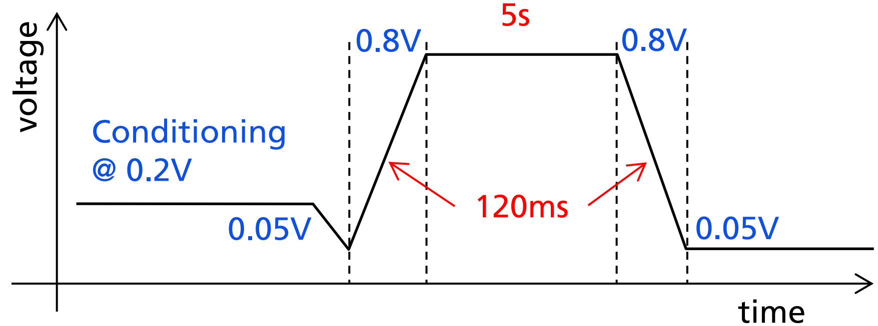
Under transient conditions, transport limitations can be minimized

Description of Experiment



- Transient operation means:



- Conditioning @ 0.2V: establishing saturation distribution
- Super-fast pol-curve (upwards)
- Dwell time 5s at high voltage: equalization of c_{O2} distribution in active area → gas residence time ~230ms
- Super-fast pol-curve (downwards)

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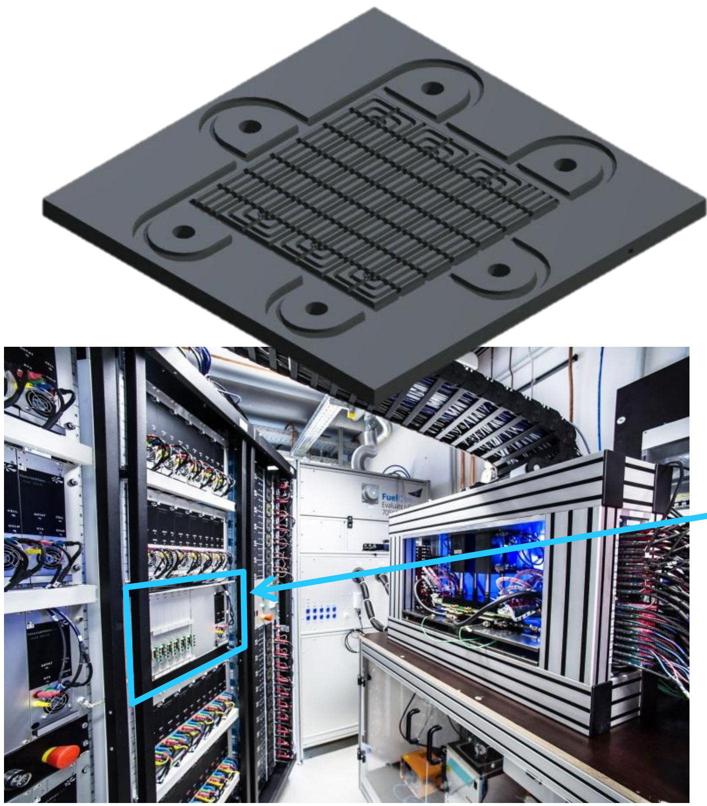

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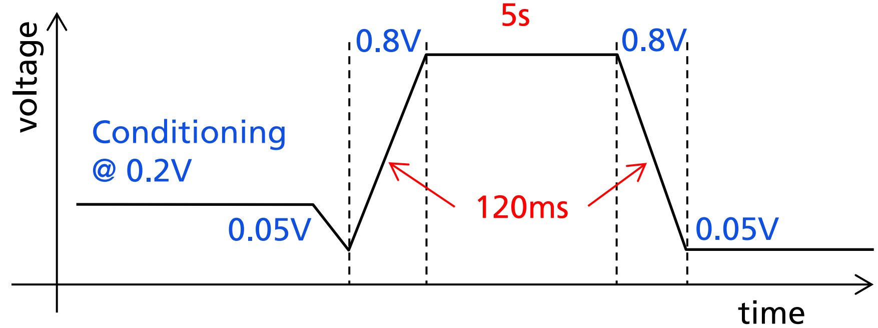

- Multichannel-FRA used for fast data sampling (rate: 6kHz)

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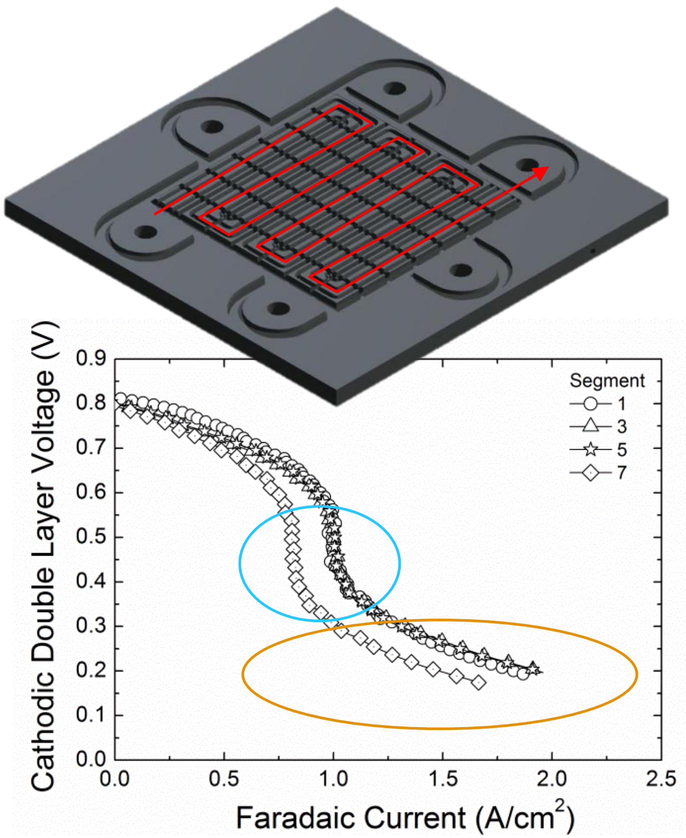
A bistable behavior in the performance of the cell is measured under transient operation

The recovery occurs at a higher voltage



- Spatially resolved polarization curves with transient operation
  - Again: Bending back of the current curve
  - Again: Current recovery at relatively high positive voltage
- Characteristics are the same for all segments (independent of region: inlet, middle, outlet)

INLET SEGMENTS

Zamel et al. Int. J. Hydrogen Energy, 38 (2013) 15318-15327

A bistable behavior in the performance of the cell is measured under transient operation

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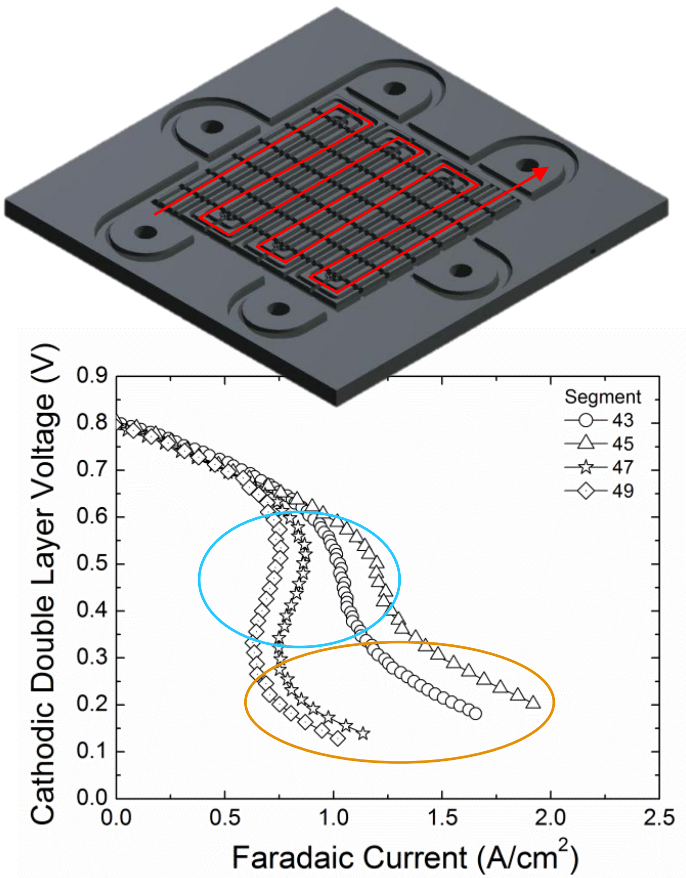

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OUTLET SEGMENTS

Zamel et al. Int. J. Hydrogen Energy, 38 (2013) 15318-15327

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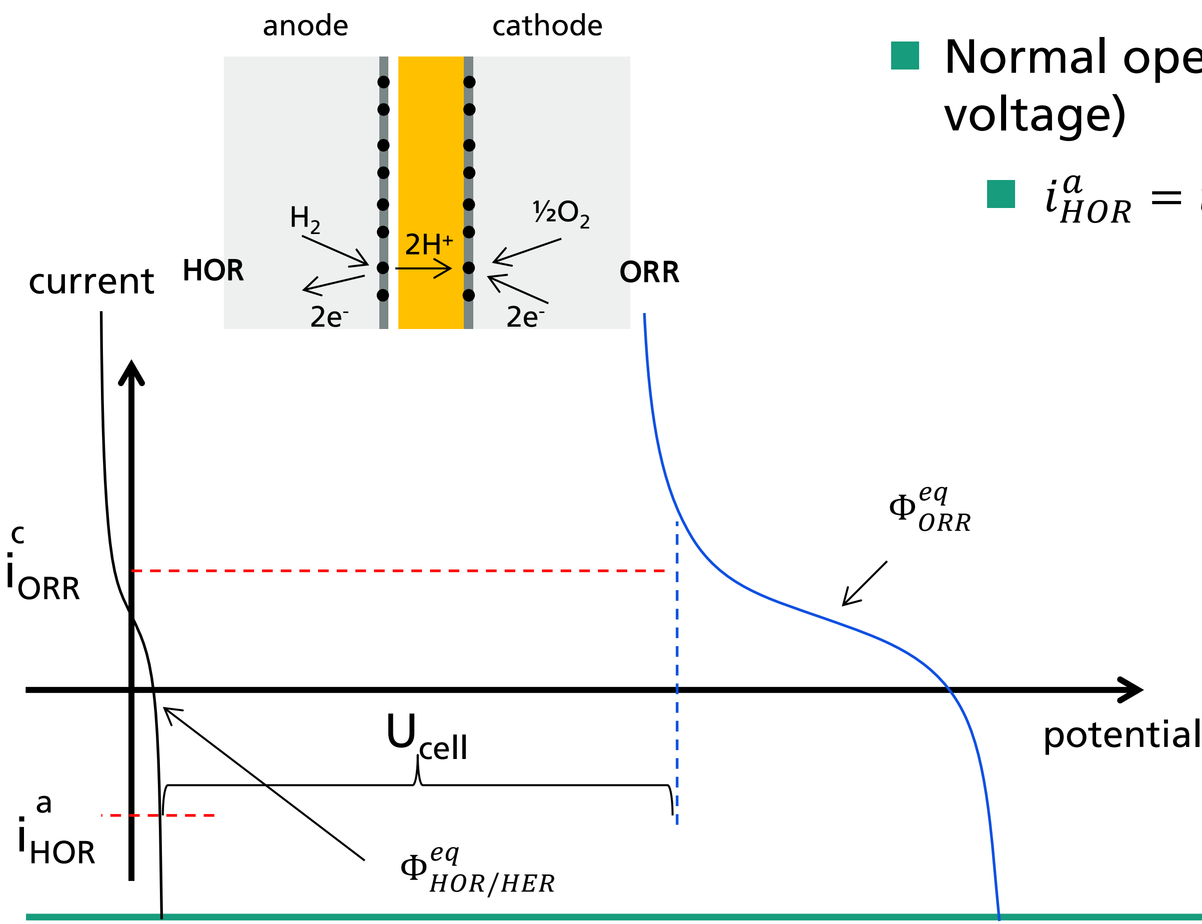
Summary of the experimental findings
what can we learn from the measured polarization curves?

- Based on experimental measurements the following is true:
  - Vertical branch in the i-U curve of the cell corresponds to the stoichiometric possible current from the oxygen fed to the cell
  - Current density in the tail of the i-U curve lies a little bit above limiting current
- The following can then be concluded:
  - The NDR branch originates from a cathodic process
  - This tail originates from an unexpected second Faradaic process

Explanation for the measured findings (steady-state)

theory of HER on the cathode



- Normal operation (high/medium voltage)
  - $i^a_{HOR} = i^c_{ORR}$

Zamel et al. Int. J. Hydrogen Energy, 38 (2013) 15318-15327

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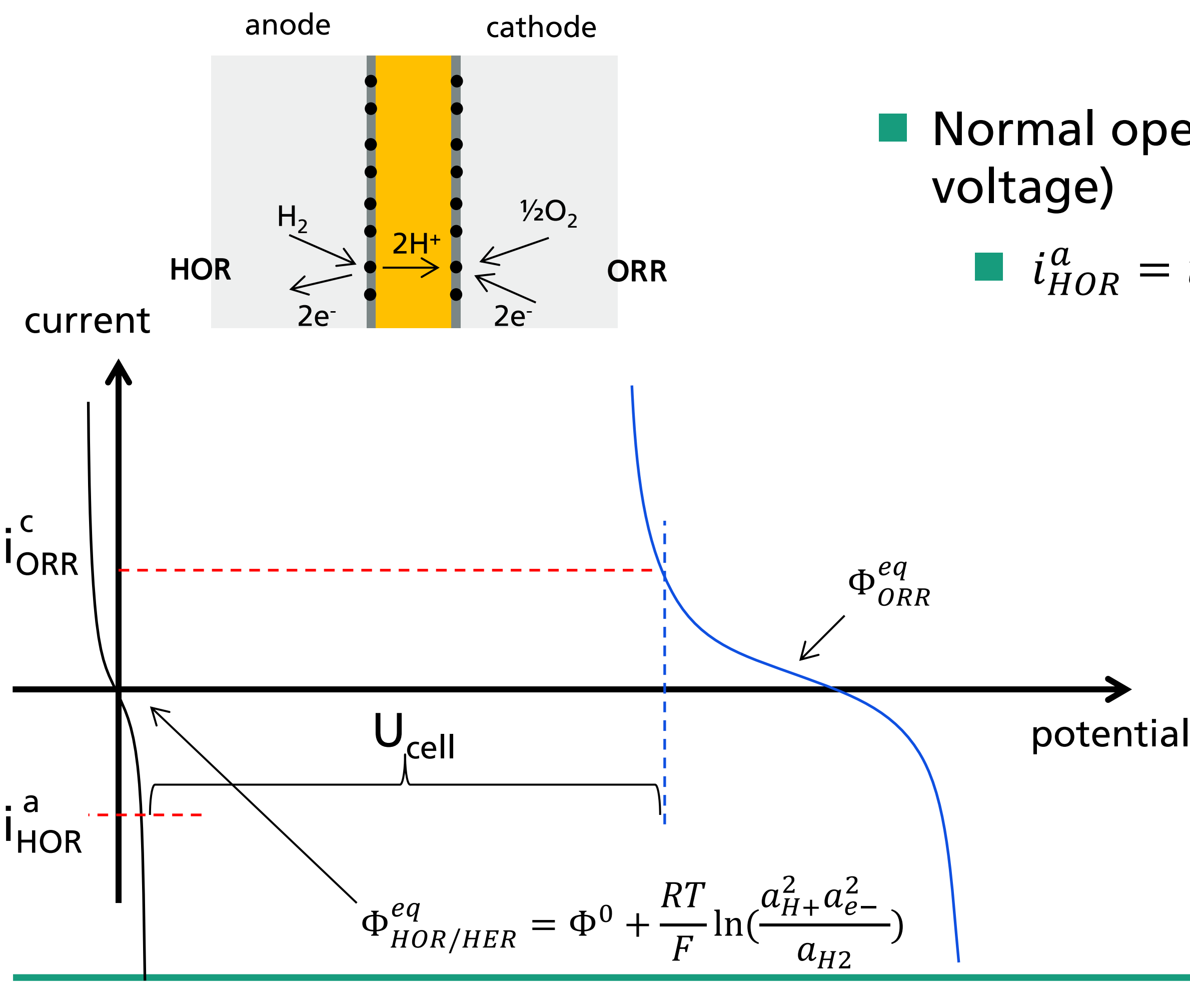

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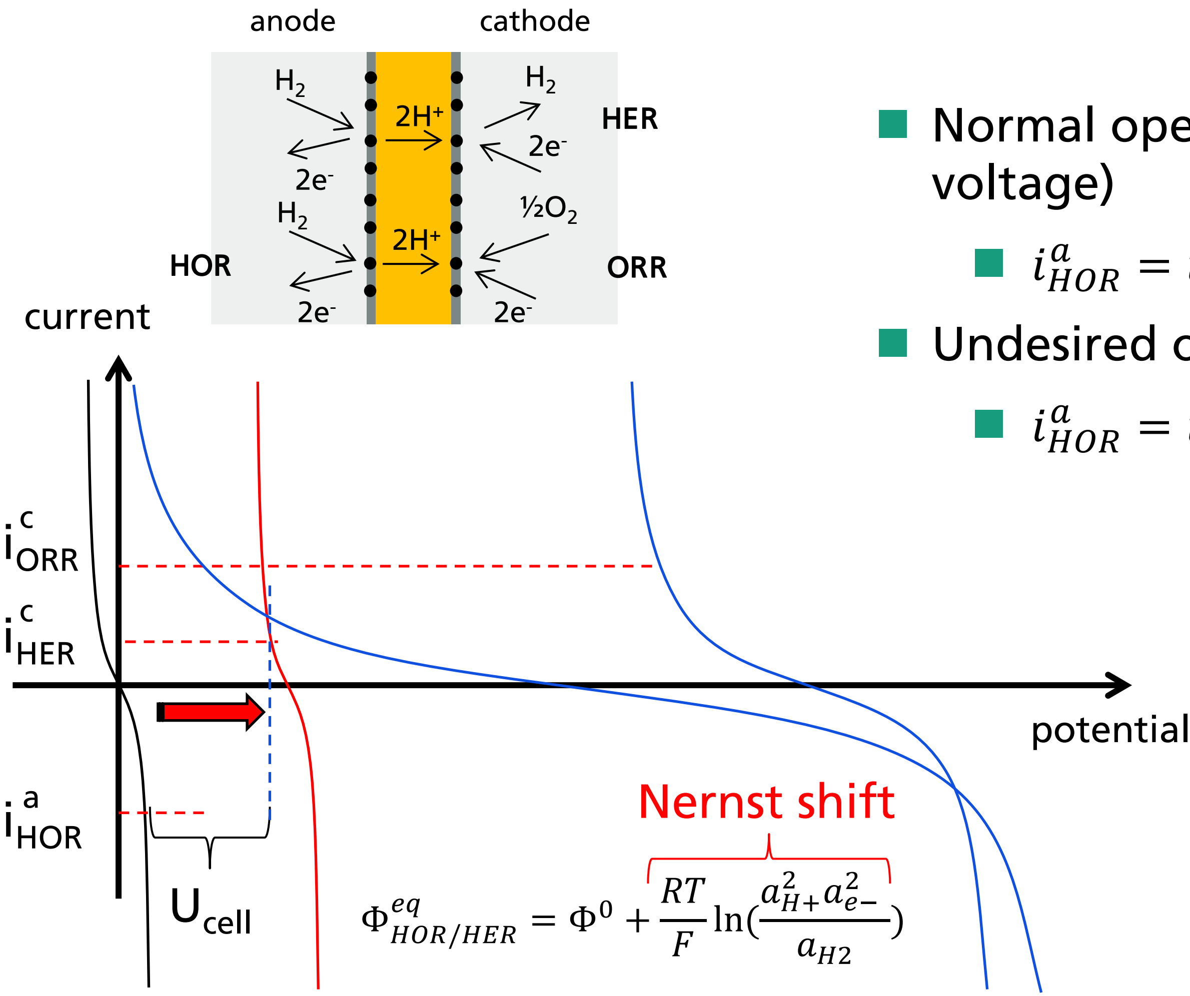

$$\Phi^{eq}_{HOR/HER} = \Phi^0 + \frac{RT}{F}\ln\left(\frac{a^2_{H+}a^2_{e-}}{a_{H2}}\right)$$

- Normal operation (high/medium voltage)
  - $i^a_{HOR} = i^c_{ORR}$
- Undesired operation (low voltage)
  - $i^a_{HOR} = i^c_{ORR} + i^c_{HER}$

Zamel et al. Int. J. Hydrogen Energy, 38 (2013) 15318-15327

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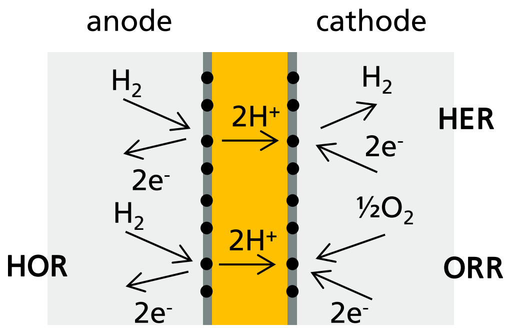
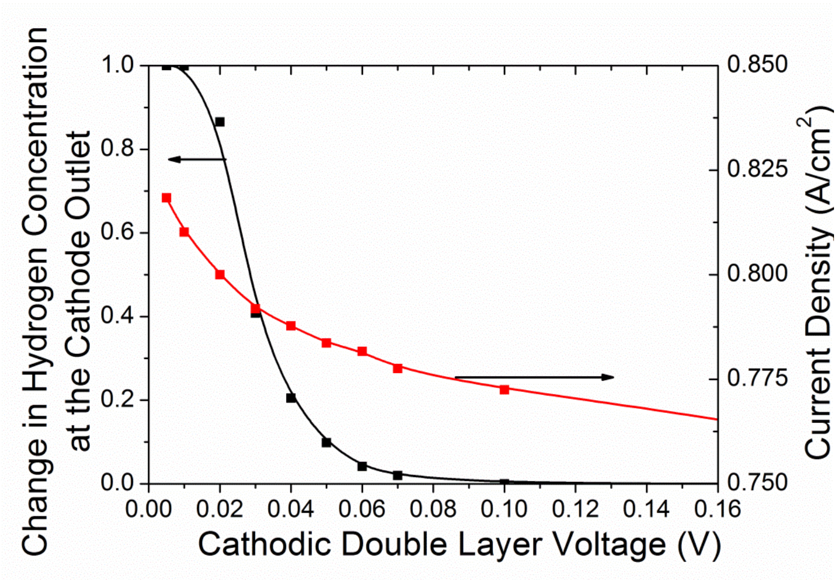
Explanation for the measured findings
theory of HER on the cathode





- Normal operation (high/medium voltage)
  - $i_{HOR}^{a} = i_{ORR}^{c}$
- Undesired operation (low voltage)
  - $i_{HOR}^{a} = i_{ORR}^{c} + i_{HER}^{c}$

Zamel et al. Int. J. Hydrogen Energy, 38 (2013) 15318-15327

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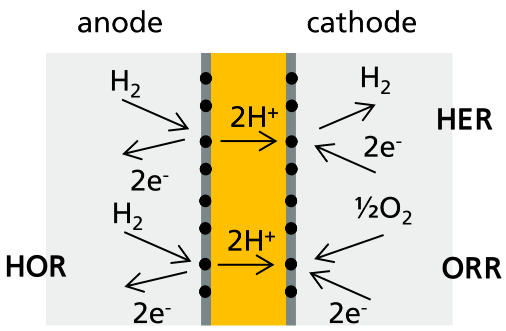
But, how can we explain the transient behaviour?

Explanation of the transients
description of time-dependent cathode model

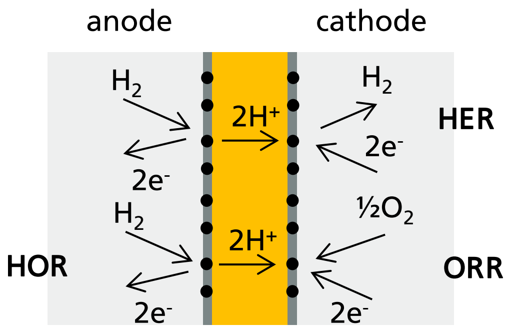


- HER mechanism: Volmer – Heyrovsky
  - $\boldsymbol{Pt^* + H^+ + e^- \rightleftharpoons Pt - H_{upd}}$
  - $\boldsymbol{Pt - H_{upd} + H^+ + e^- \rightleftharpoons Pt^* + H_2}$

- Measureable current under dynamic operation
  - $i_{meas} = \boxed{i_{ORR}} + i_{HER} + i_{DL-charg}$
- ORR described by Tafel approach

- $i_{ORR}[t] = 4 * F * i_0 * c_{O2}[t] * Exp\left[\frac{\Phi_e[t] - \Phi_p[t] - VOC}{b}\right]$

Explanation of the transients
description of time-dependent cathode model



- HER mechanism: Volmer – Heyrovsky
  - $\boldsymbol{Pt^{*} + H^{+} + e^{-} \rightleftharpoons Pt - H_{upd}}$
  - $\boldsymbol{Pt - H_{upd} + H^{+} + e^{-} \rightleftharpoons Pt^{*} + H_2}$

- Measureable current under dynamic operation
  - $i_{meas} = i_{ORR} + \boxed{i_{HER}} + i_{DL-charg}$
- HER described by Volmer-Heyrovsky steps

- $i_V[t] = k * F * ((1 - \theta_H[t]) * Exp\left[-\frac{\Phi_e[t] - \Phi_p[t] - \Delta\Phi}{b2}\right] - \theta_H[t] * Exp\left[\frac{\Phi_e[t] - \Phi_p[t] - \Delta\Phi}{b2}\right])$

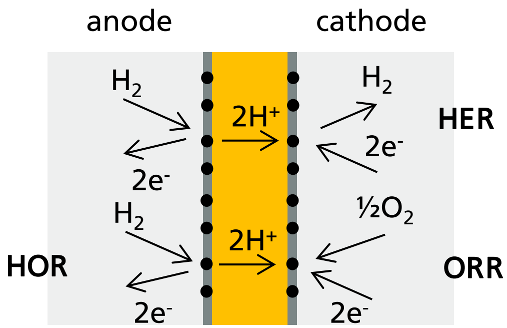
with equilibrium potential $\Delta\Phi = \Phi^0 + \frac{RT}{F}\ln(\frac{a_{H+}(1-\theta)}{\theta})$

- $i_H[t] = h * F * (\theta_H[t] * Exp\left[-\frac{\Phi_e[t] - \Phi_p[t] - \Delta\Phi}{b2}\right] - (1 - \theta_H[t] *) * c_{H2}[t] * Exp\left[\frac{\Phi_e[t] - \Phi_p[t] - \Delta\Phi}{b2}\right])$

Fraunhofer ISE

Explanation of the transients

description of time-dependent cathode model

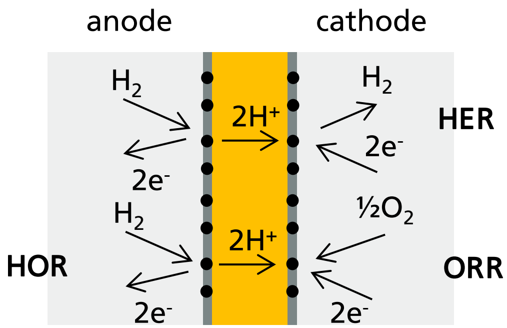


- HER mechanism: Volmer – Heyrovsky
  - $\boldsymbol{Pt^* + H^+ + e^- \rightleftharpoons Pt - H_{upd}}$
  - $\boldsymbol{Pt - H_{upd} + H^+ + e^- \rightleftharpoons Pt^* + H_2}$

- Measureable current under dynamic operation
  - $i_{meas} = i_{ORR} + i_{HER} + \boxed{i_{DL-charg}}$
- Charging of the double layer capacitance

• $i_{DL-charg}[t] = C_{DL} * \partial_t(\Phi_e[t] - \Phi_p[t])$

Explanation of the transients
Description of time-dependent cathode model



- Differential equation system with the solving variables Φ_p, C_{O2}, Θ_H
  - Assuming no anode polarization → Φ_p @ anode = 0V
    - $\Phi_p[t] = -R_{Ohm} * \left(i_{ORR}[t] + i_{HER}[t] + i_{DL-charg}[t]\right)$
  - Fickian Diffusion in GDL, no spatial resolution of the CCL
    - $\varepsilon_{GDL} * \partial_t c_{O2}[t] = -\frac{i_{ORR}[t]}{4*F} + D_{GDL}^{eff} * \frac{c_{O2}^{inlet} - c_{O2}[t]}{L_{GDL}}$
  - Hydrogen as solely adsorbate
    - $\Gamma * \partial_t \Theta_H[t] = i_V[t] - i_H[t]$

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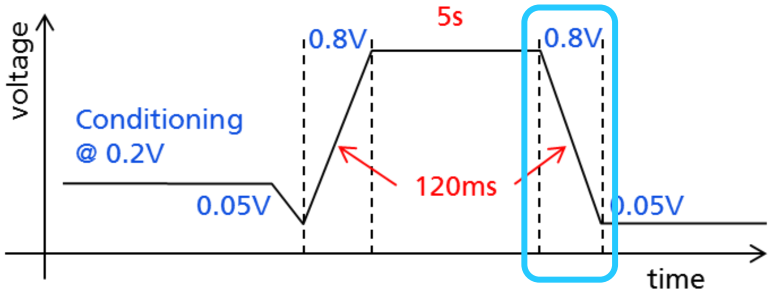
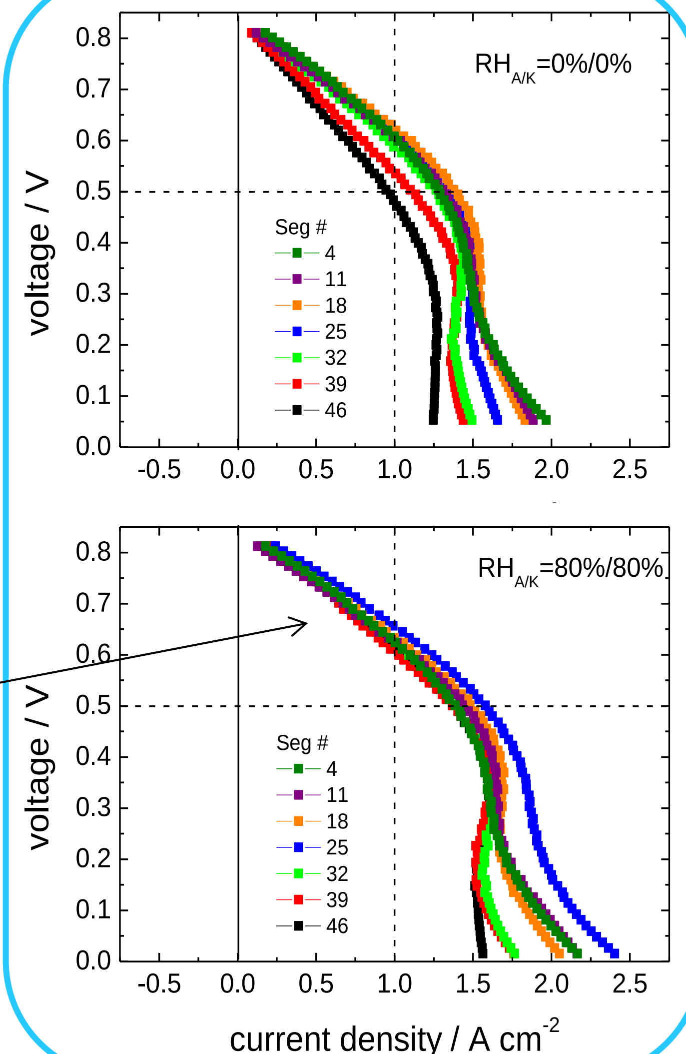
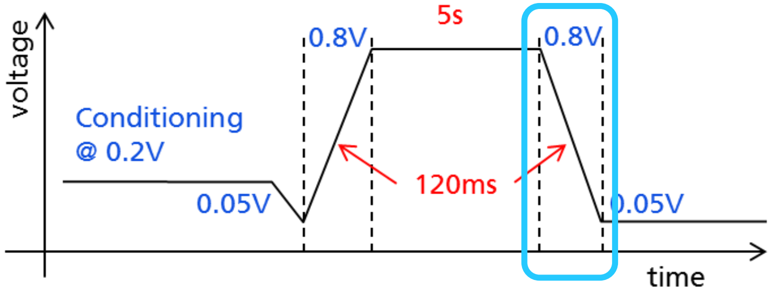
Transient polarization curves at two humidity conditions



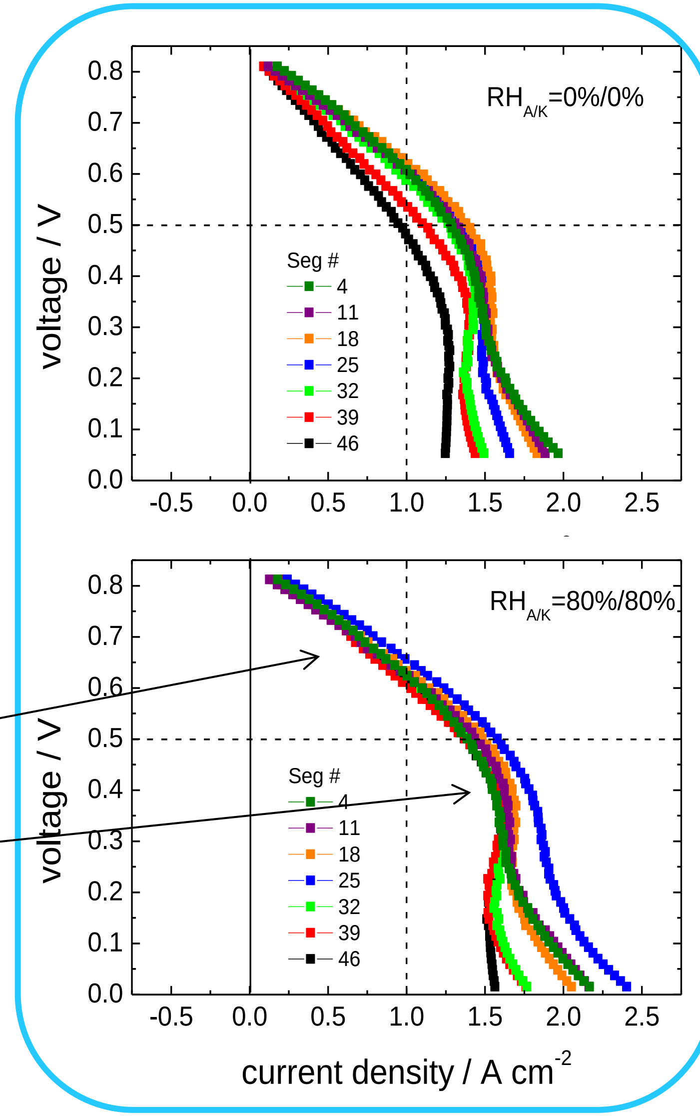
- Operation
  - Counter-flow
  - High to low voltage sweep
- Curve characteristics
  - Linear drop



Transient polarization curves at two humidity conditions

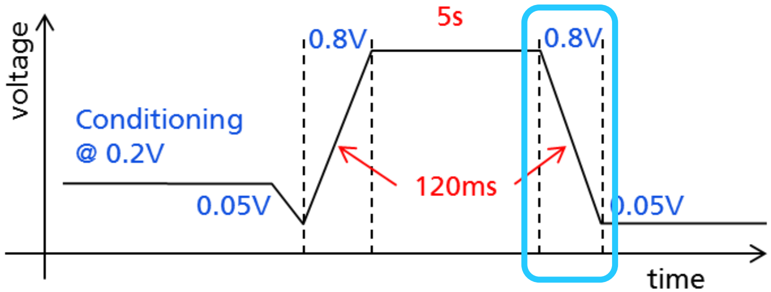


- Operation
  - Counter-flow
  - High to low voltage sweep
- Curve characteristics
  - Linear drop
  - Steep drop

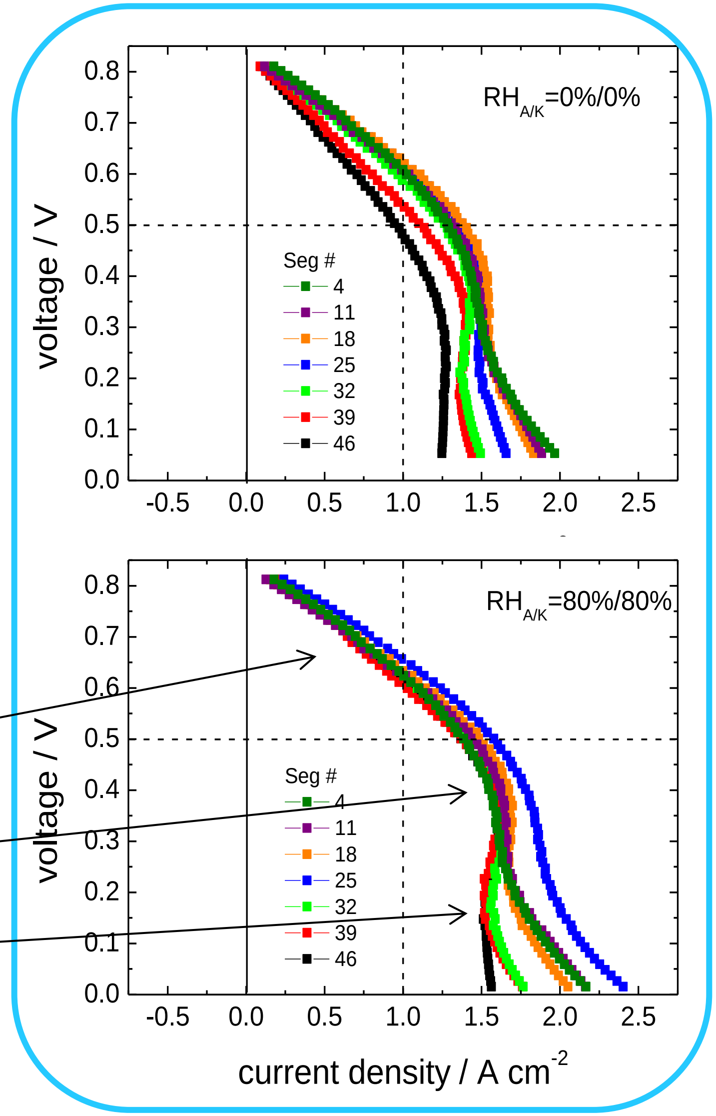


Fraunhofer ISE

Transient polarization curves at two humidity conditions

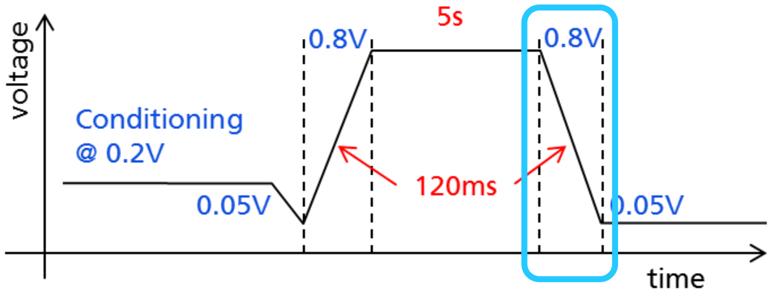


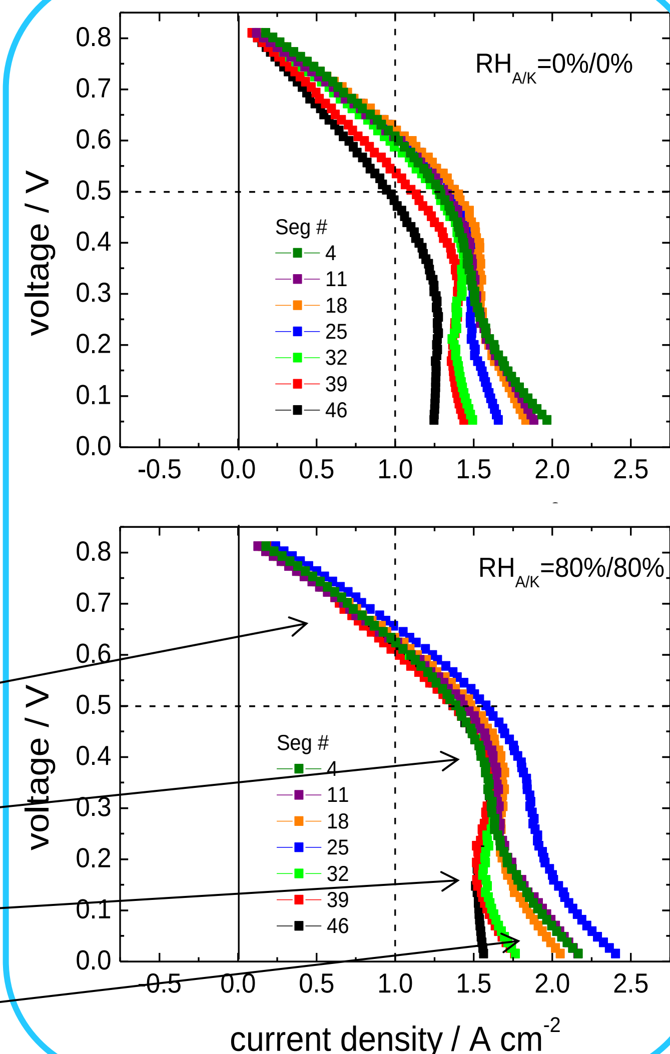
- Operation
  - Counter-flow
  - High to low voltage sweep
- Curve characteristics
  - Linear drop
  - Steep drop
  - Back bending



Fraunhofer ISE

Transient polarization curves at two humidity conditions

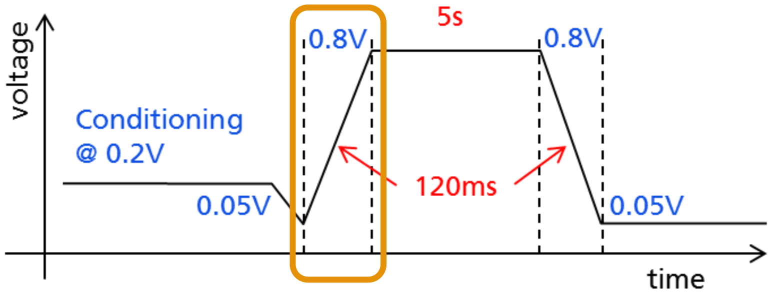




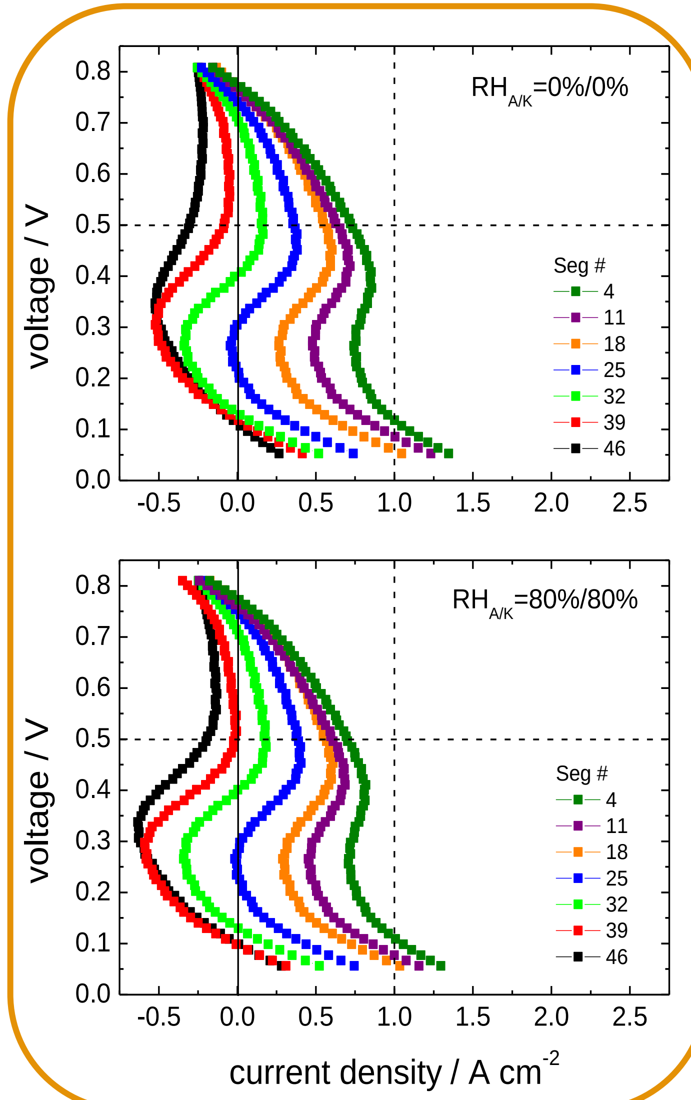
- Operation
  - Counter-flow
  - High to low voltage sweep
- Curve characteristics
  - Linear drop
  - Steep drop
  - Back bending
  - Recovery (not @ dry anode inlet)

Fraunhofer ISE

Transient polarization curves at two humidity conditions



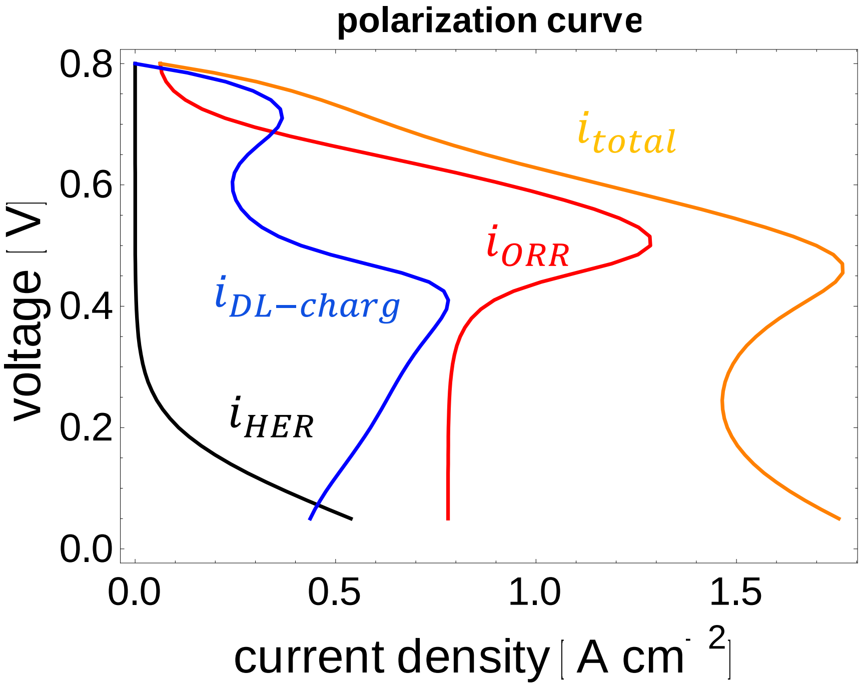
- Operation
  - Counter-flow
  - Low to high voltage sweep
- Curve characteristics
  - More pronounced recovery branch
  - Negative currents
  - Influence of local position visible at entire pol-curve



Fraunhofer ISE

Can the model predict the trends?

- The simulation results show the characteristics of back bending and recovery



Fraunhofer ISE

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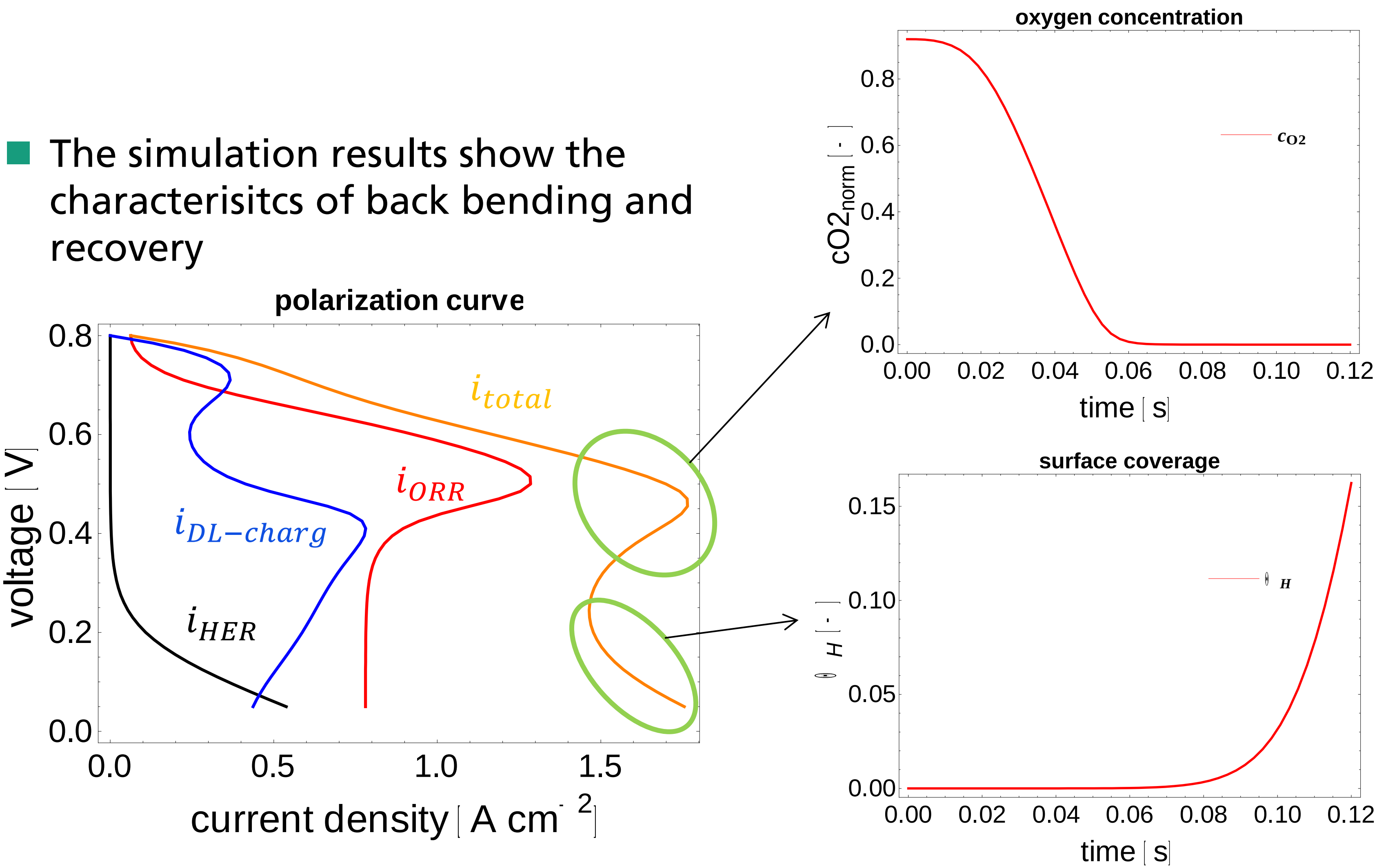
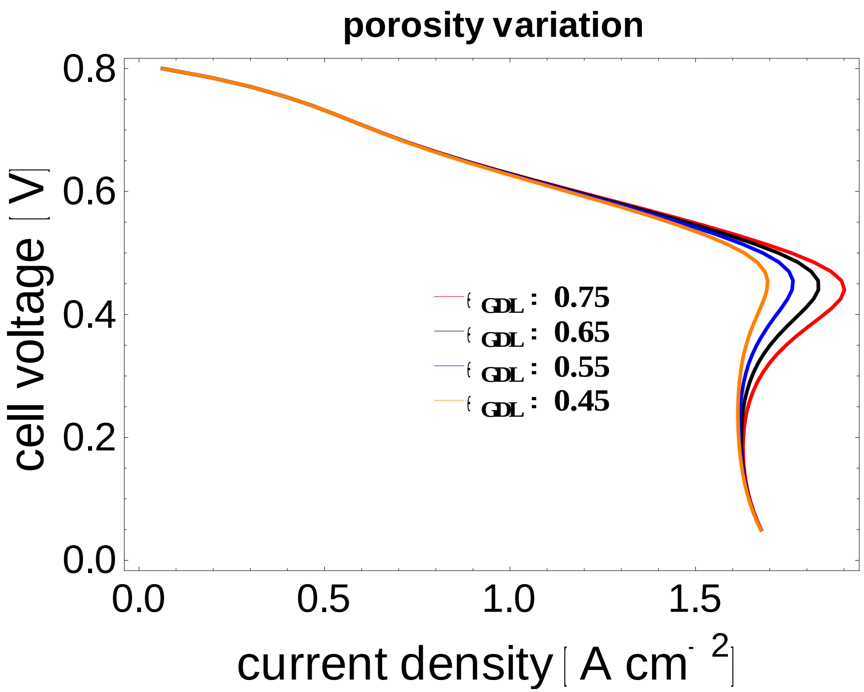
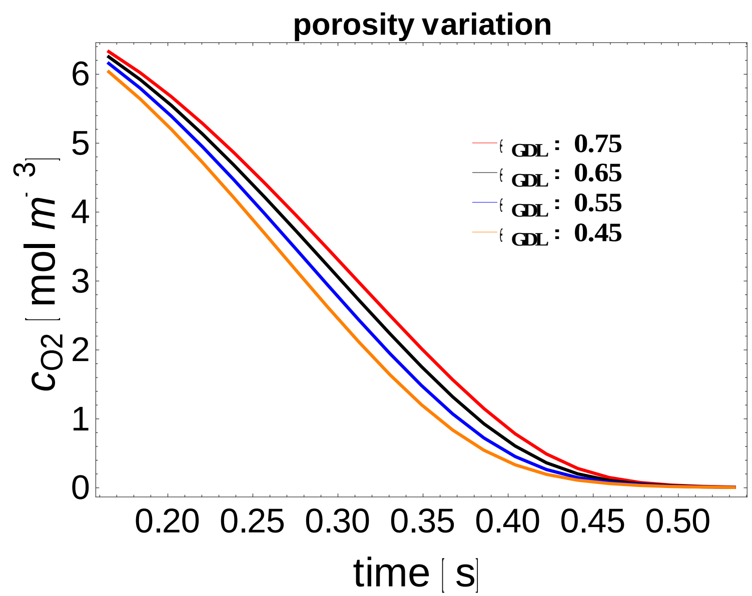


Fraunhofer ISE

Impact of the model parameters
analysis of the GDL porosity

- Porosity determines the current overshoot due to the oxygen storage capacity

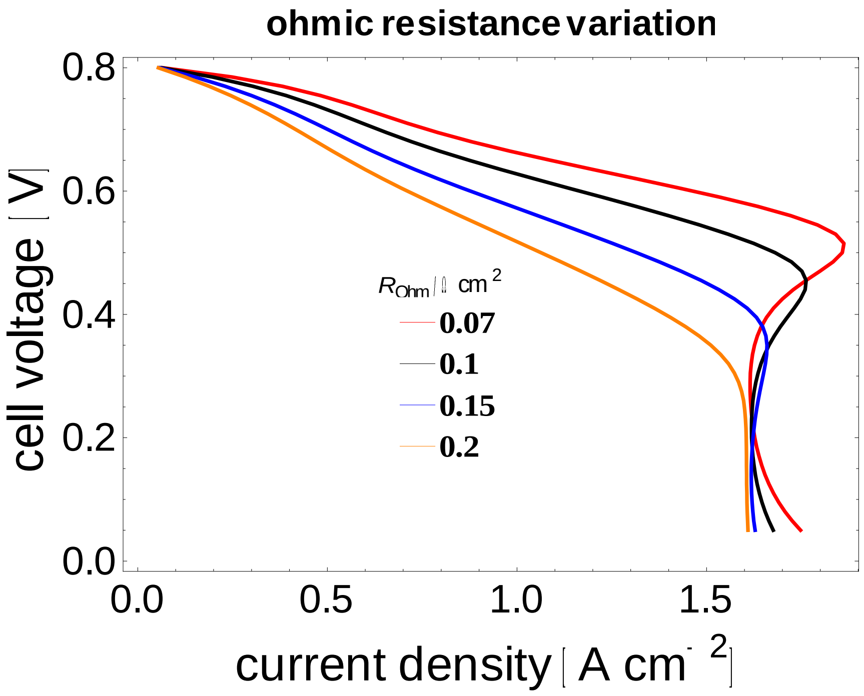




Fraunhofer ISE

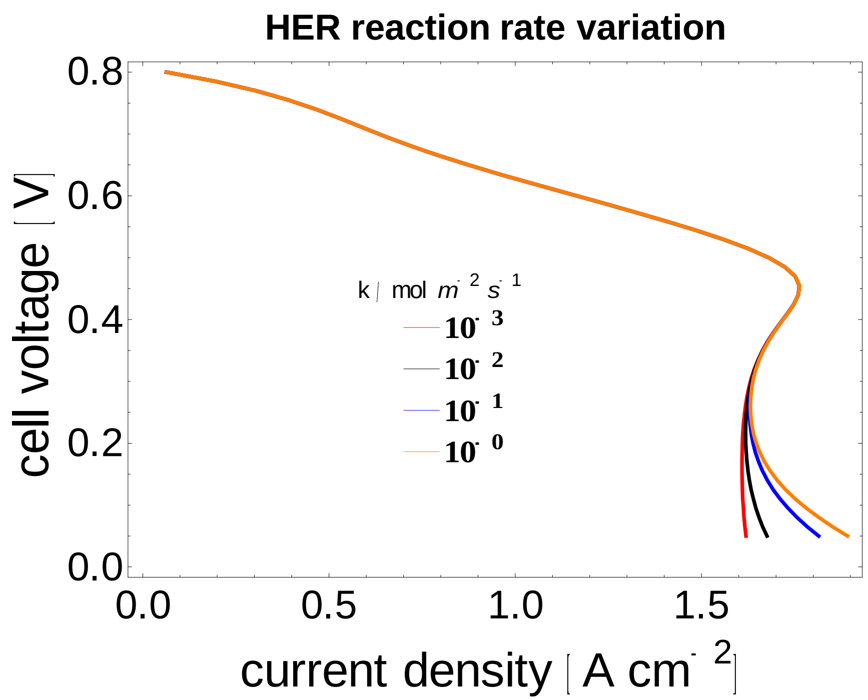
Impact of the model parameters
analysis of the ohmic resistance (mainly R_{mem})

- Ohmic resistance impacts the entire characteristic
- High resistance suppresses the HER → cathodic Galvani potential not low enough



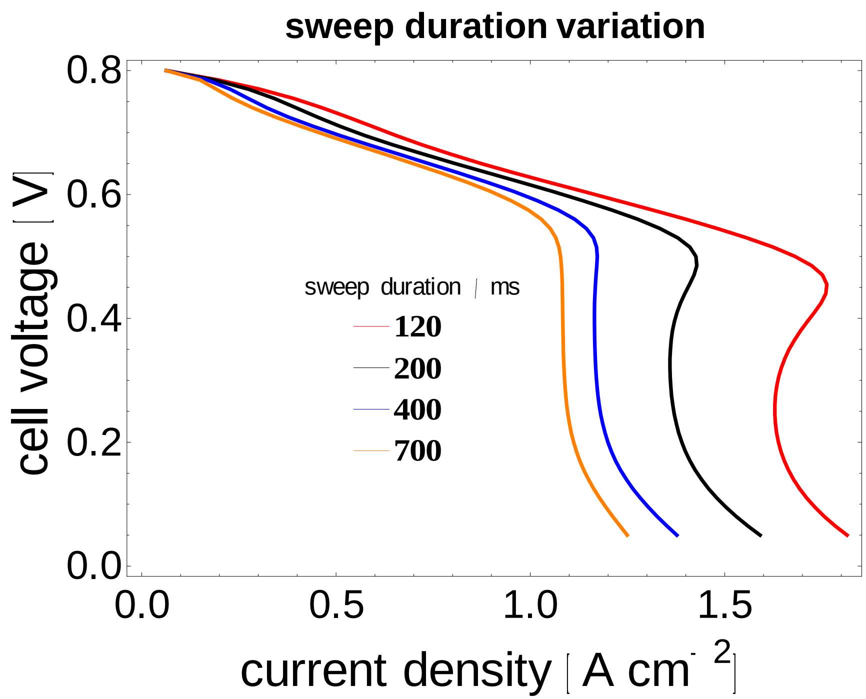
Impact of the model parameters
analysis of the reaction rate constant of the Volmer step

- Reaction rate constant has a strong influence on the current recovery characteristics



Impact of the model parameters
analysis of the sweep duration

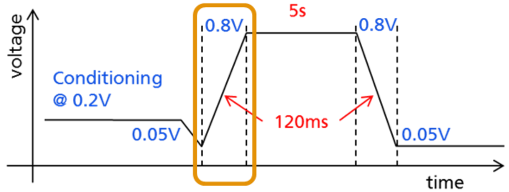
- Considering the gas storage capacity, convergence to steady-state is reached @ ~ 2s

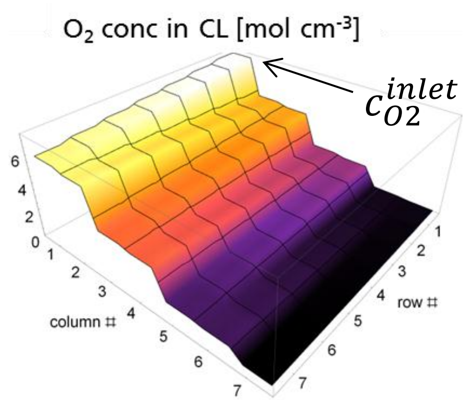


Impact of the sweep direction

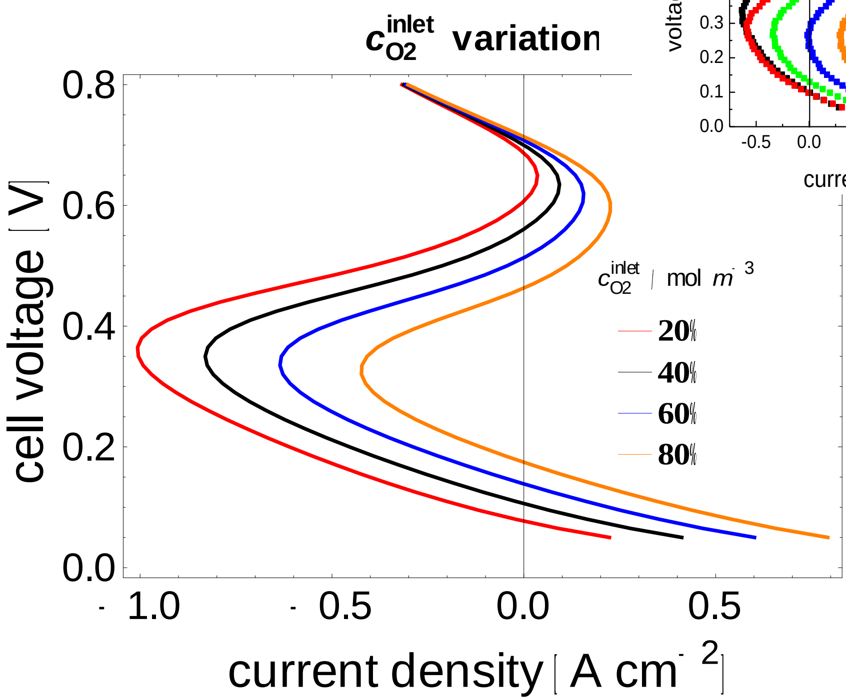
model predicts the partly negative current @ upward sweep

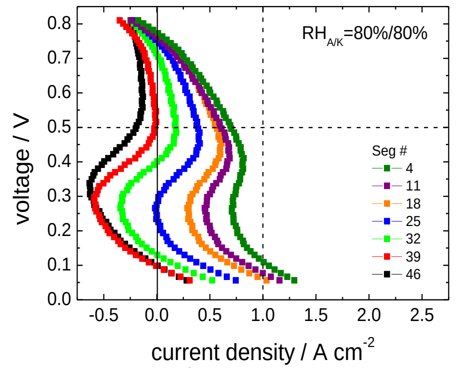
- Mimic the interfacial condition at channel/GDL





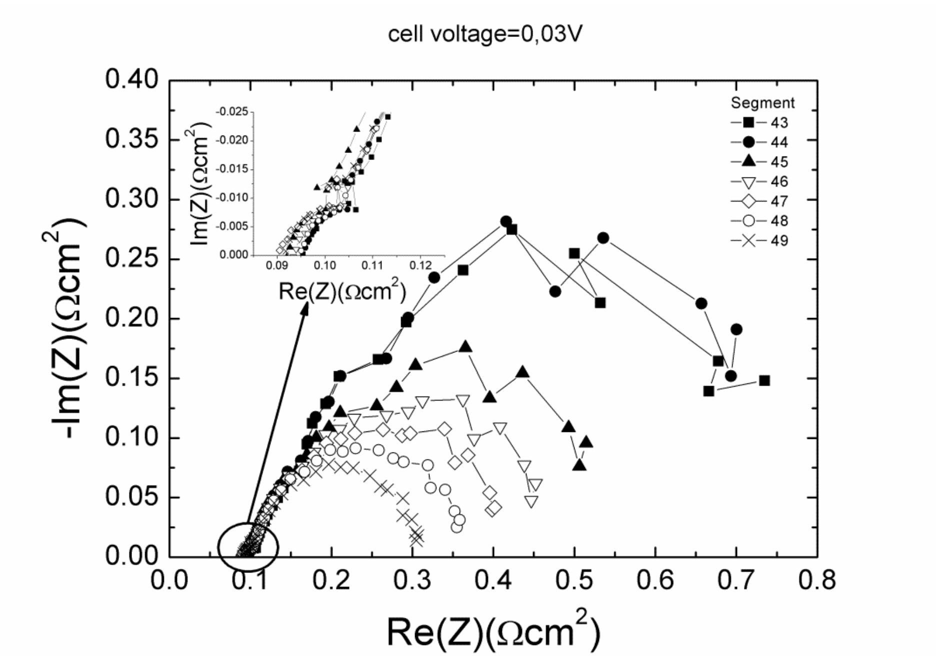
- Recovery of c_{O2} with time affects the characteristics





Can the HER be used to explain the trends of the impedance spectra towards the outlet of the air stream?

a second arc is measured despite local perturbation



- Operation
  - Counter-flow
  - Potentiostatic mode
  - Cell temperature kept at 60°C
  - i-U curve collected for a voltage range of 800 mV – 50 mV
  - 30 minutes at each voltage increment
  - Air/hydrogen flow rates 600/600
  - Inlet relative humidity of air/hydrogen 80%
  - Local perturbation of cell is used

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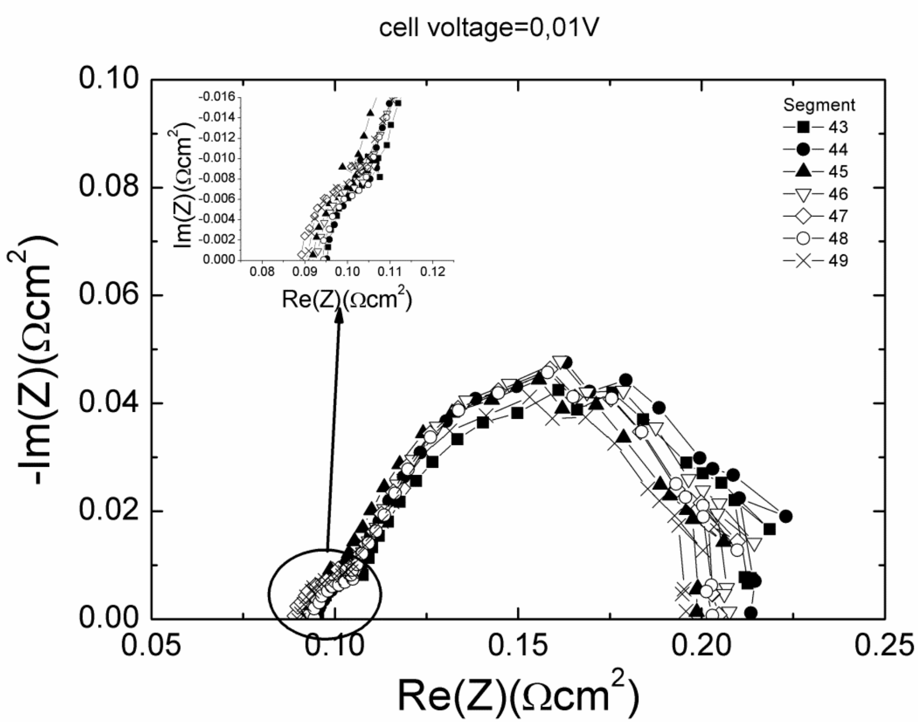

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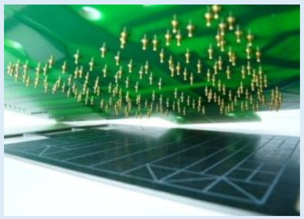
Conclusion



- Due to the various and coupled processes, inhomogeneities are inevitable within the cell area
  - Spatially resolved characterization is needed to disclose these inhomogeneities
  - With the 68 channel system, characterization of various cell areas is possible
  - Spatially resolved characterization allows for the investigation of specific trends
  - Measurements with the MCCS can be coupled with our single cell EIS monitoring of short stacks, also under extreme climate conditions
  - This helps to optimize design, material, and operation strategy

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Thank you for your attention!

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Fraunhofer Institute for Solar Energy Systems ISE

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www.h2-ise.com

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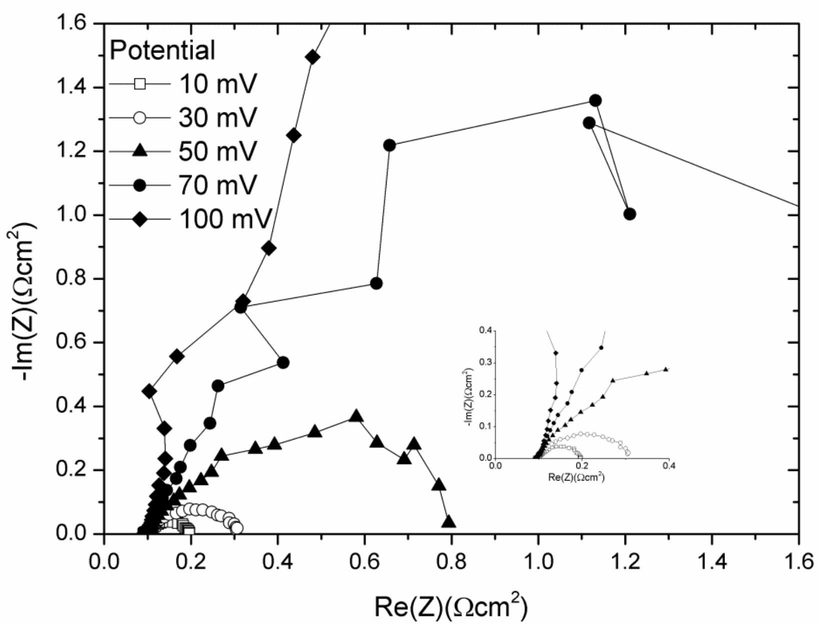
a second arc is measured despite local perturbation



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  - Local perturbation of cell is used

Thank You Very Much for Your Attention!

Acknowledgement:

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Canadian-German Co-operation on Kinetics and mass transport Optimization in PEM fuel cells **GECKO**

www.gecko-fuelcell.com

Fraunhofer Institute for Solar Energy Systems ISE

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Conclusion and Outlook

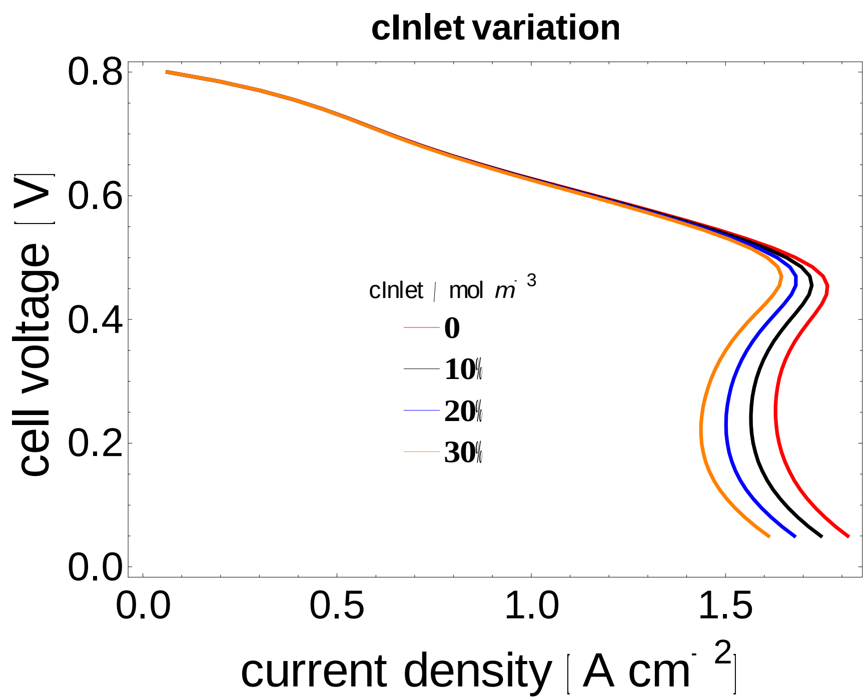
- Theoretical explanation is given for the measured N-shaped polarization curves in steady-state as well as dynamic operation
- A simple 0-dim model that accounts for the oxygen storage capacity of the GDL, the ORR as well as HER on the cathode predicts the current back bending and recovery very well
- Fast transient measurements can be used to decompose mass transport losses from the residual losses (kinetics, proton migration, contact resistance,…) but
  - the sweep rates have to be adjusted to minimize in-plane effects (high sweep rates) and to minimize the HER on the cathode (low sweep rates)

Conclusion and Outlook

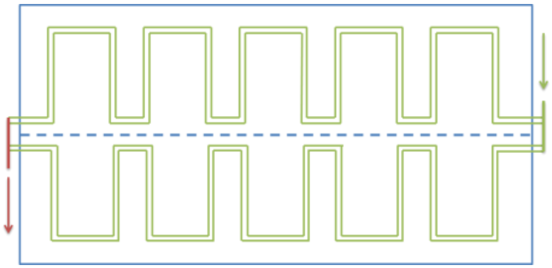
- It has to be clarified if at low cell voltage the HER on the cathode affects the cathode impedance spectrum
- If hydrogen on the cathode side (generated at fast load steps or at undesired oxygen starvation operation) has an impact on cathode degradation has to be discussed

Impact of the model parameters
Assuming a linear drop of the oxygen concentration at channel | GDL boundary

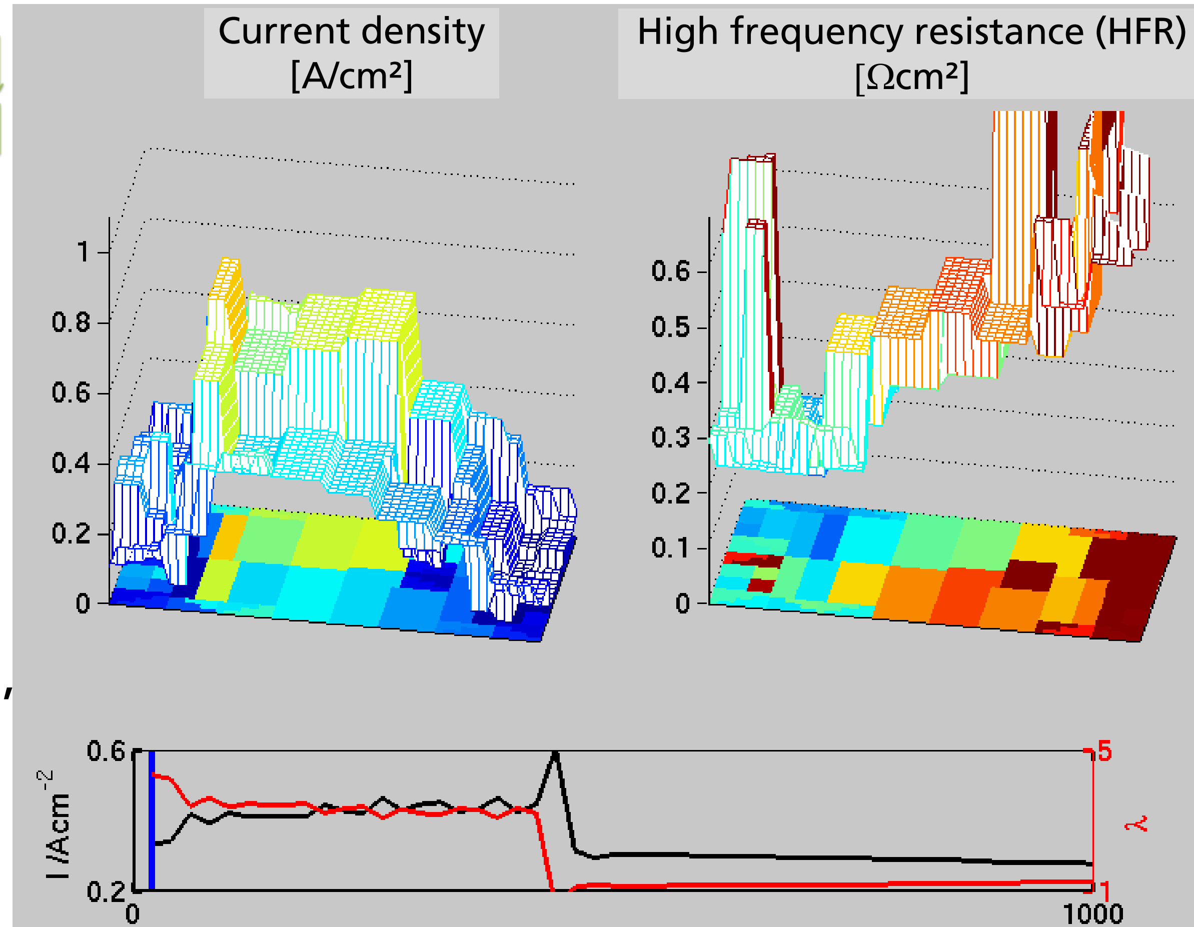
- Since gas residence time (230ms) is in the same range as the sweep time (120ms) downstream effects along the channel have to be considered



Spatially resolved characterization of a specific single cell



- PEMFC with 200 cm² active area
- Co-flow with meandric flowfield
- Characterization at 0.6 V, r.H. 50% (H_2/air), T=60°C
- Variation of stoichiometry



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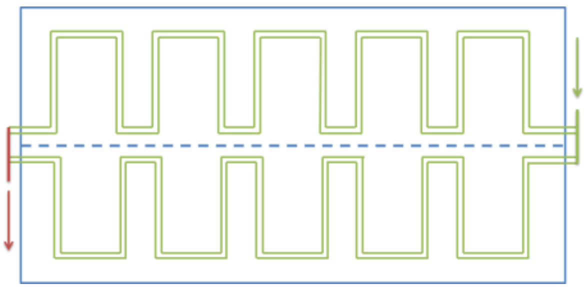
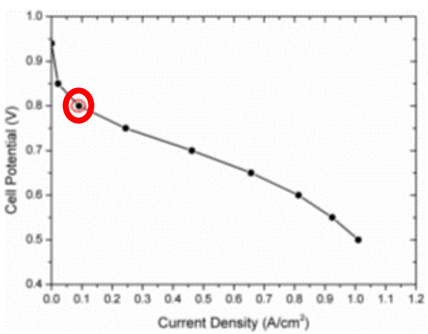
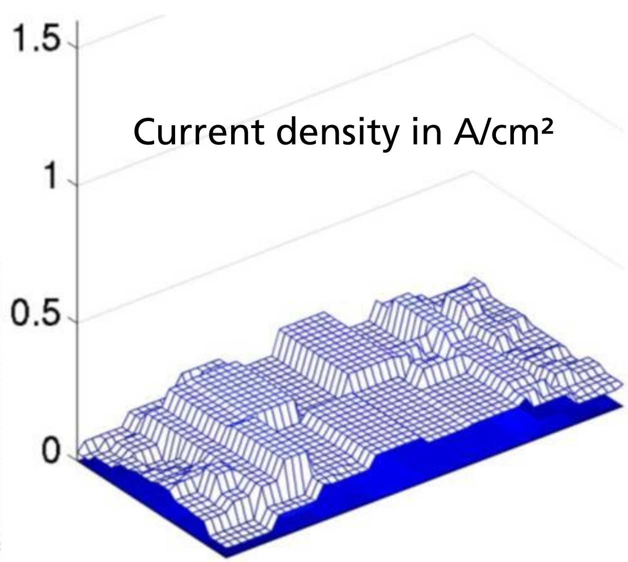
Spatially resolved characterization of a specific single cell

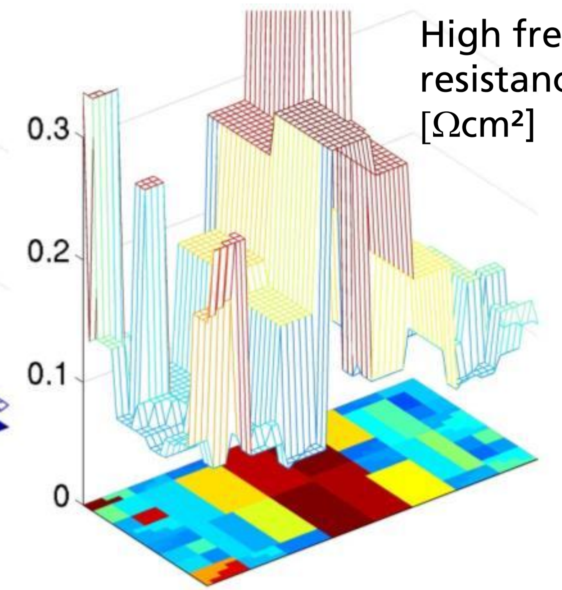


- PEMFC with 200 cm² active area (for stationary applications)
- Co-flow with meandric flowfield
- Characterization at 0.8 V, r.H. 95% (H_2/air) stoichiometry of 24 /25 (H_2/air)

I-V-curve







High frequency resistance (HFR) [Ωcm²]

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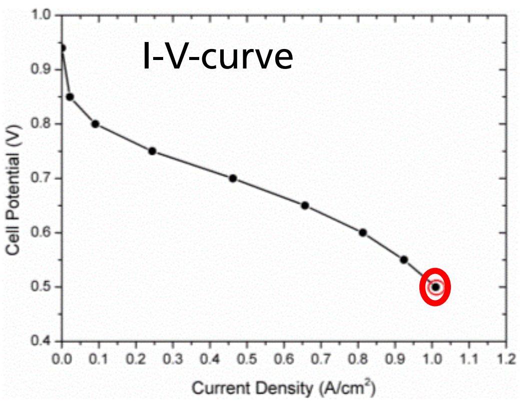
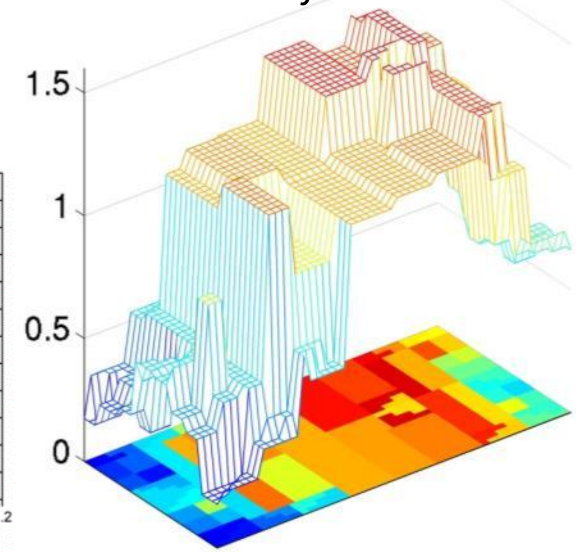
Spatially resolved characterization of a specific single cell

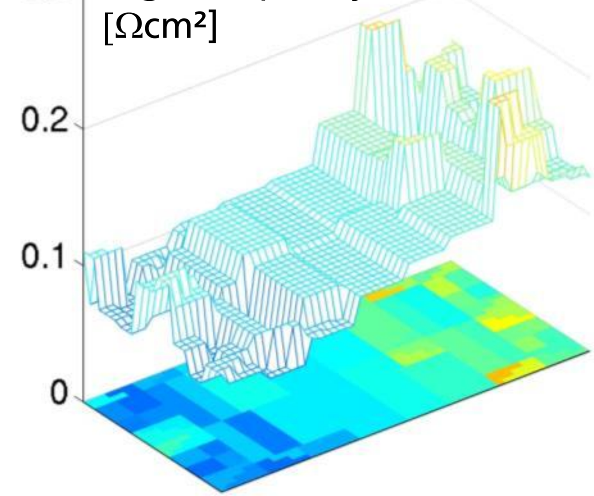
- Characterization at 0.5 V, r.H. 95% (H_2/air); stoichiometry of 2.2 /2.3 (H_2/air)



Current density in A/cm²



High frequency resistance (HFR) [Ωcm²]



- Current mapping shows very low current production at inlet and outlet
- HFR indicates dehydration at the inlet
- Symmetric behaviour with respect of flow field design

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