HYDROGEN ADVANCED WATER SPLITTING TECHNOLOGY PATHWAYS WORKSHOP

Protocol for harmonized measurements and reproducibility

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The Fraunhofer Institute for Solar Energy Systems ISE

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Staff: approx. 1,300

Budget 2019: 102.6 Mio. €

Established: 1981

Photovoltaics

Energy Efficient Buildings

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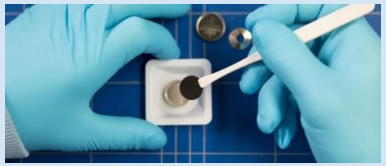
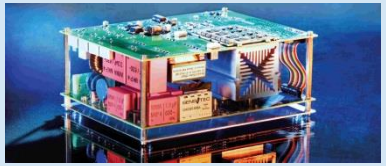


Hydrogen Technologies and Electrical Energy Storage



Power Electronics, Grids and Smart Systems

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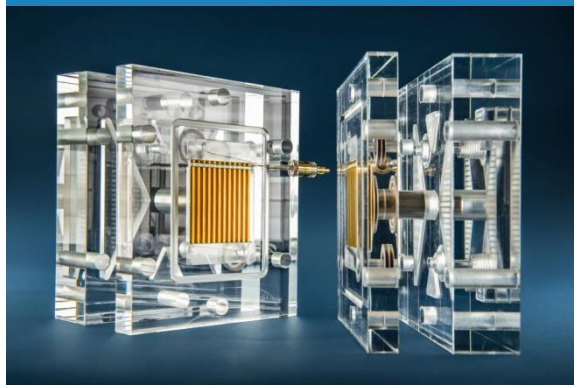
Research Topic @ Fraunhofer ISE

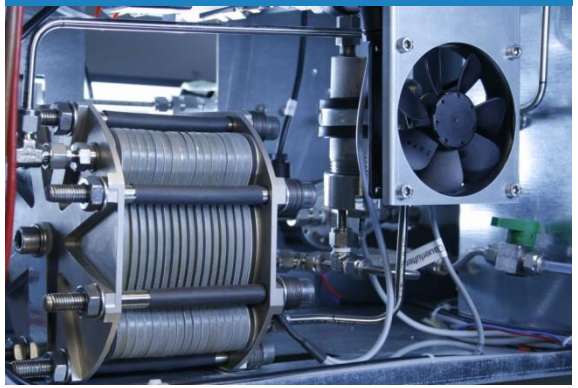
Electrolysis and Power to Gas

Characterisation of Materials and Components



- Elektrochemical Characterisation
- Investigation of life-time / Accelerated stress tests
- Ex-situ analysis

Development of PEM Water Electrolysis Systems



- New Cell concepts
- Laboratory PEM stacks
- Energy-optimised balance of plant
- Control strategies

Power to Gas

- Dynamic system modelling of PtG systems
- Development of system and plant concepts
- H_2 yield assessment

Hydrogen infrastructure

- Technology consulting
- Techno economical analysis /market survey
- Roll out H_2 technologies
- Life cycle assessment

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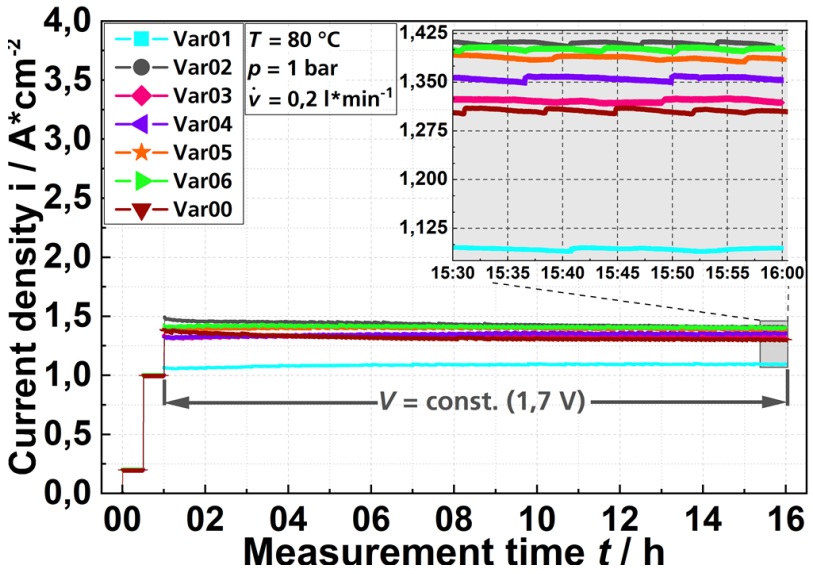
OUTLINE OF THE TALK

- Harmonized test protocol
  - Hardware
  - Cell conditioning
  - Polarization curves
  - Impedance spectroscopy
- Some results
  - Polarization curves and influence of parameter
  - Variation of pressure on active area
  - Reproducibility in-house
- Summary & conclusion

Harmonized Test Protocol
Hardware: test bench

- No harmonized test bench
  - Who could and who would afford it, anyway?
- Individual responsibility that criteria can be met.
  - But: settings can have impact on results



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Harmonized Test Protocol
Hardware: test cell

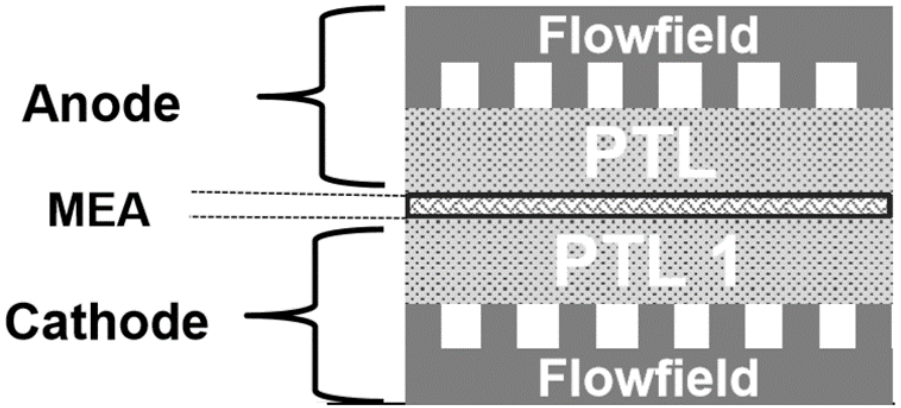
- Small, simple, reliable, easy to use
  - Two half cells, put on top of each other and compressed
  - 4 cm² active area
  - Pocket depth adaptable via PEEK frames
  - Clamping force applied and measured via external cage unit
- Admittedly: Doesn't include all interesting features
  - No reference electrode
  - Pocket depth isn't adjustable, new frame needed for any new material
  - Clamping force is applied to all: Active area, hard stop plus gasket
- Nonetheless: Good value for money, successfully used on four out of five continents!

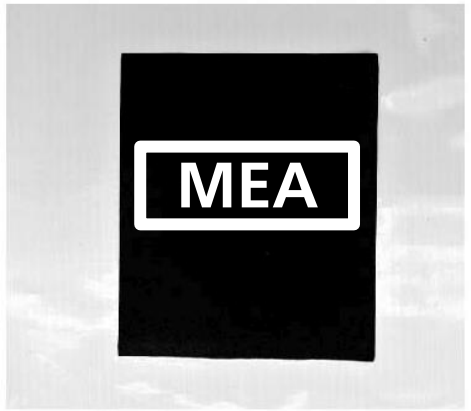
Harmonized Test Protocol
Hardware: mode of operation

- Reproducibility and simplicity beats slightly better performance
  - Flooded operation on anode and cathode side

    (T-control, no need for humidity control on cathode side)
  - Use of commercial reference materials, i.e. MEA, & PTLs

    (identical quality is crucial)
    - Use of mature and well tested MEA (Greenerity)
    - Use of Ti-PTLs (Bekaert) rather than carbon paper on cathode (less different materials + gold coating no longer necessary)
  - Initially dry assembly (same reference point for swelling), by now pre-swelling in water (better access for water to membrane)





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Harmonized Test Protocol
Cell conditioning

- Any measurement needs a reproducible break-in procedure
  - Particularly necessary if assembled in dry state
  - Water drag of proton conductivity through membrane seems to be responsible for observed break-in effect
  - Performed at 80 °C to increase kinetics
  - In our case at least 9 hours (0.5h + 0.5h + 8h)
- Aim: generation of steady state with defined maximum drift.

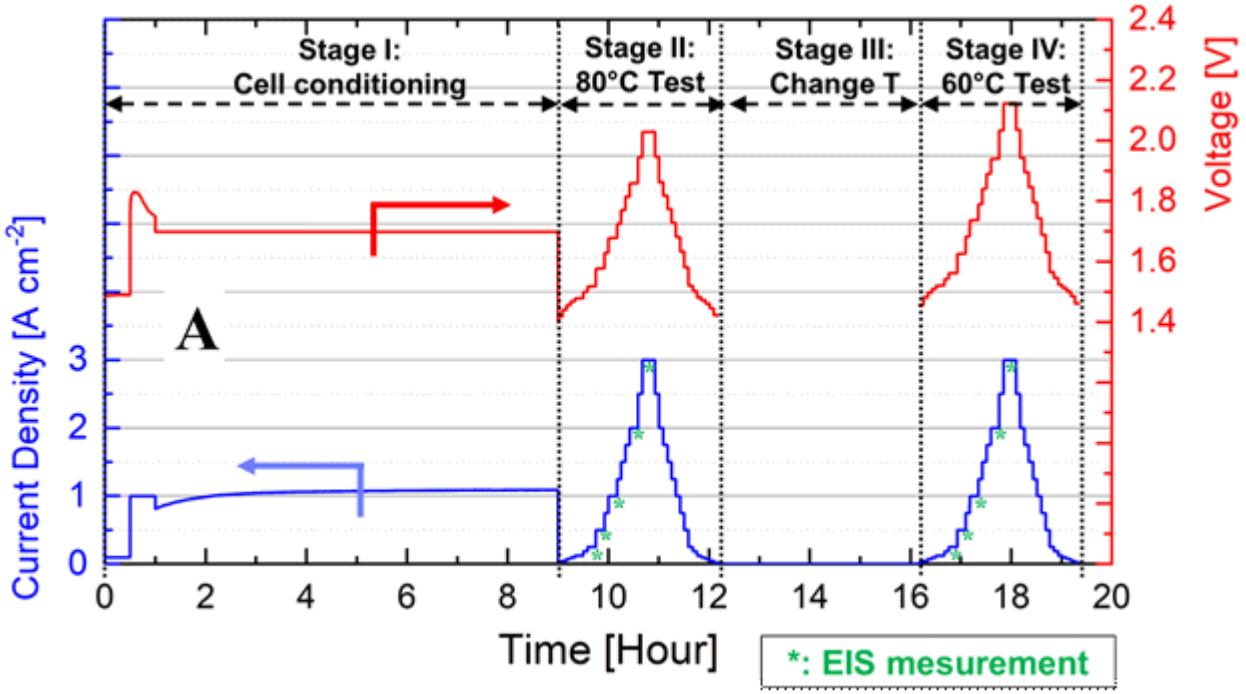

Figure with courtesy of NREL

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Harmonized Test Protocol
Polarization curves

- Current density-lead measurements
  - Smaller steps at lower current densities
  - Holding time per step: 5 mins
  - First at 80 °C, then 60°C
  - 2 measurements at each point (up and down)
  - N.b.: Up and down protocol is NOT the same as two individual measurements.
- Aim: Reproducible polarization curve with ΔV=10 mV @ 1 A*cm^{-2}, ΔV=20 mV @ 2 A*cm^{-2}

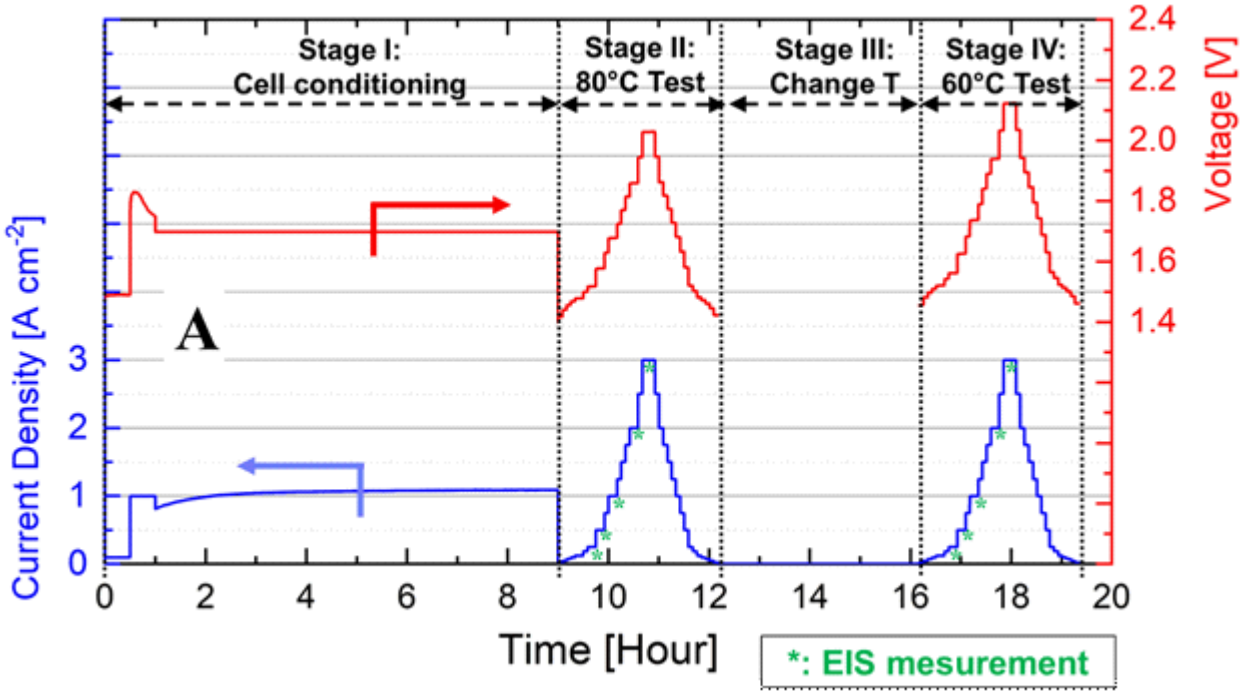

Figure with courtesy of NREL

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Harmonized Test Protocol
EIS measurements

- A polarization curve is the culmination of MANY different effects.
- Also “identical” polarization curves can have different underlying reasons and mechanisms.
- Electrochemical Impedance spectroscopy can help to analyze individual contributions to the overall signal.
  - Important: The EIS measurements (in principle) do not influence the polarization curve.
  - But if used for analysis, they have to be as stringent as the measurement protocol.

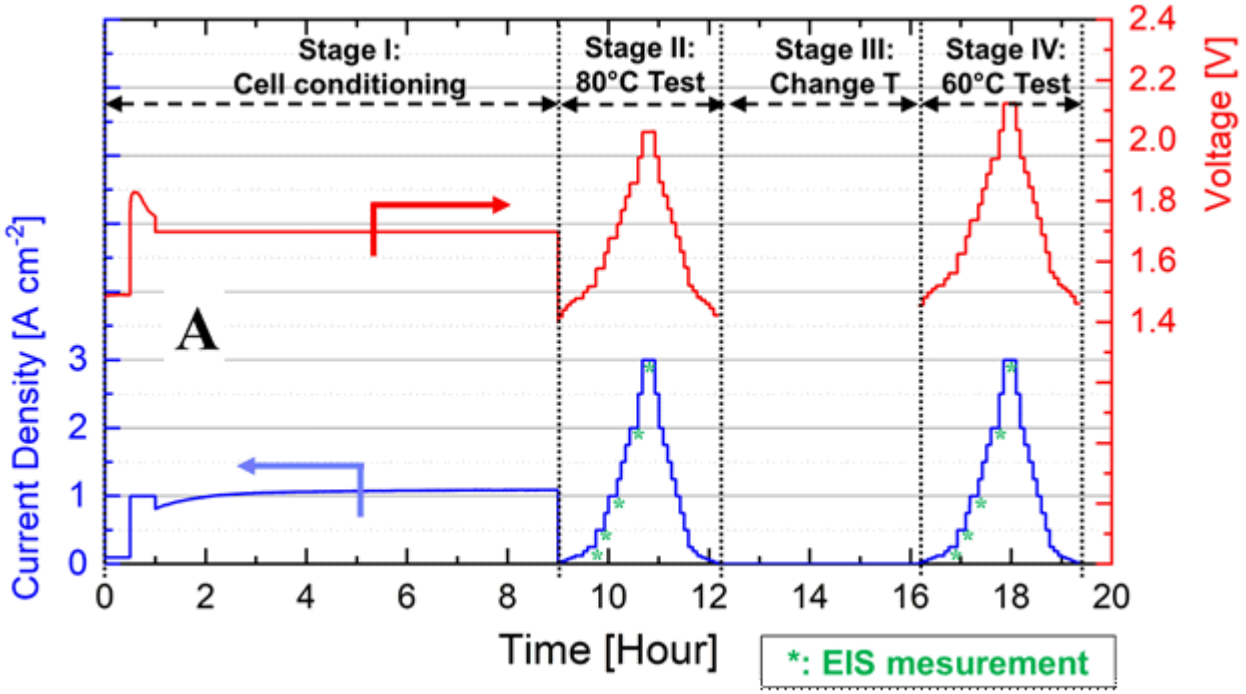

Figure with courtesy of NREL

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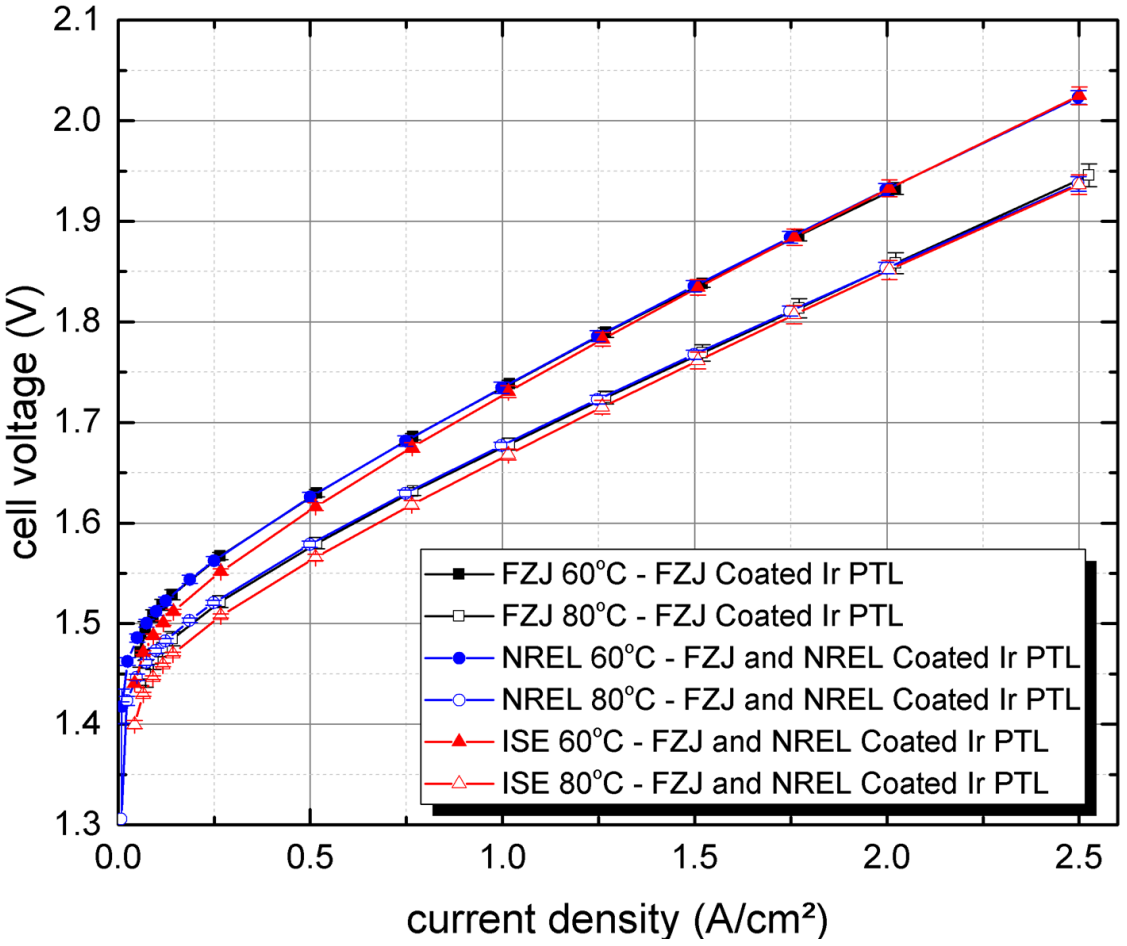
Some Results

Polarization curves and influence parameter

- Despite all efforts, there are still issues to be solved
  - While deviation at 1 A*cm^{-2} is acceptable, and at 2 A*cm^{-2} results are “spot on”.

    BUT: there is a significant and hence not acceptable deviation in region < 1 A*cm^{-2}
  - Two out of three partners have an almost perfect match
  - Unfortunately, it’s our results deviating from the other partners’
  - Reasons for deviation is not straight forward



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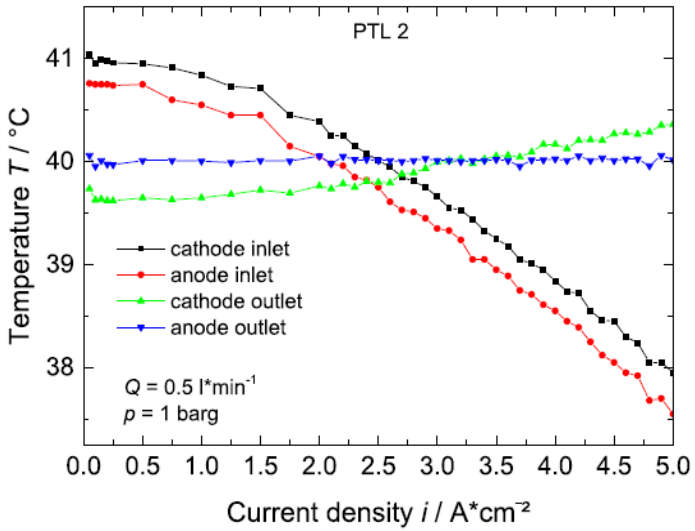
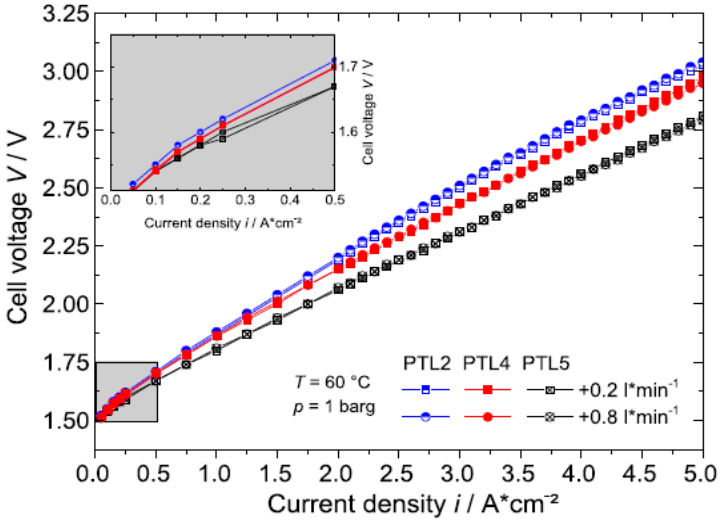
Some Results

Polarization curves and influencing parameter

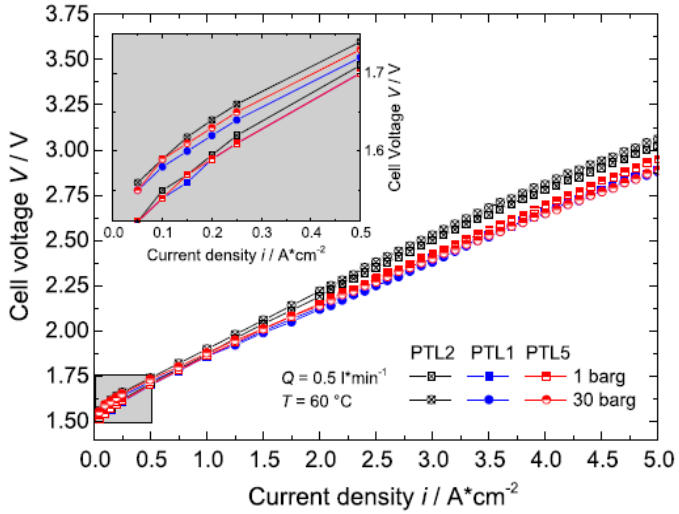
Temperature*



Flow rate*



Gas pressure*



- Position of temperature sensor will have an impact on the temperature distribution in the cell
  - Reference cell now comes with a build-in temperature sensor
- While temperature is known to have a major impact on performance, it cannot be the only reason (cp. gradient of the curve)

*obtained with different test cell: Lickert et al. *International Journal of Hydrogen Energy* **2020,** 45 (11) 6047-6058.

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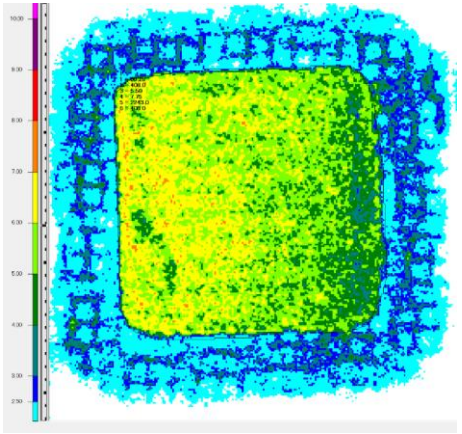
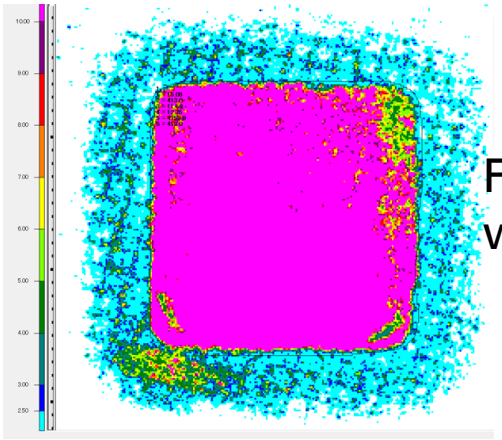
Some Results

Variation of pressure on active area

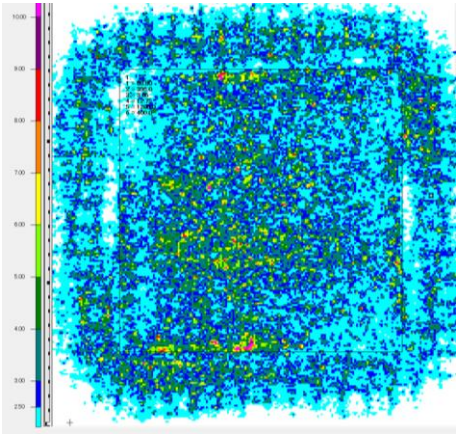
- While the clamping force (Fc = 4 kN) was properly defined, this doesn't necessarily mean, the pressure on the active area is identical.
  - Hard to circumvent without major changes to the cell
- Tests can be done with pressure paper. However: It's not a fool proof procedure either
- The production accuracy of the PEEK frame and the PTL can cause different pressure distributions between active area and gasket.
- Swelling of the MEA can significantly changes this picture (see figures)



Fc = 4 kN,
no MEA



Fc = 4 kN,
with MEA



Fc = 2.5 kN,
no MEA

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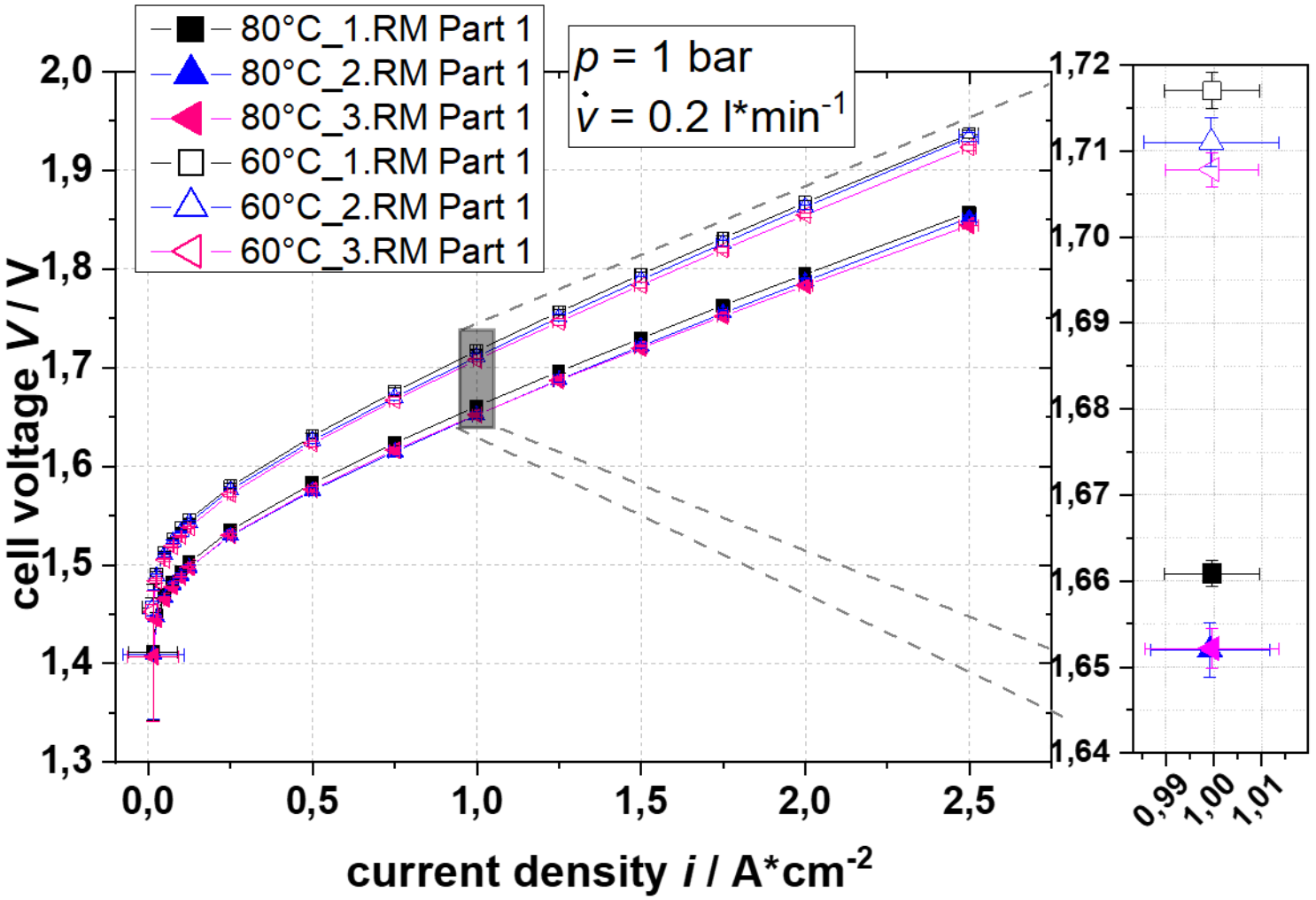
Results

Reproducibility in-house

- Fc = 2.5 kN → avg. pressure on active area ~ 3 MPa
- Results for “fresh” components
  - ΔV = 8 mV @ 0,25 A*cm^{-2}, 60 °C
  - ΔV = 5 mV @ 0,25 A*cm^{-2}, 80 °C
  - ΔV = 9 mV @ 1 A*cm^{-2}, 60 °C
  - ΔV = 9 mV @ 1 A*cm^{-2}, 80 °C
  - ΔV = 12 mV @ 2 A*cm^{-2}, 60 °C
  - ΔV = 12 mV @ 2 A*cm^{-2}, 80 °C



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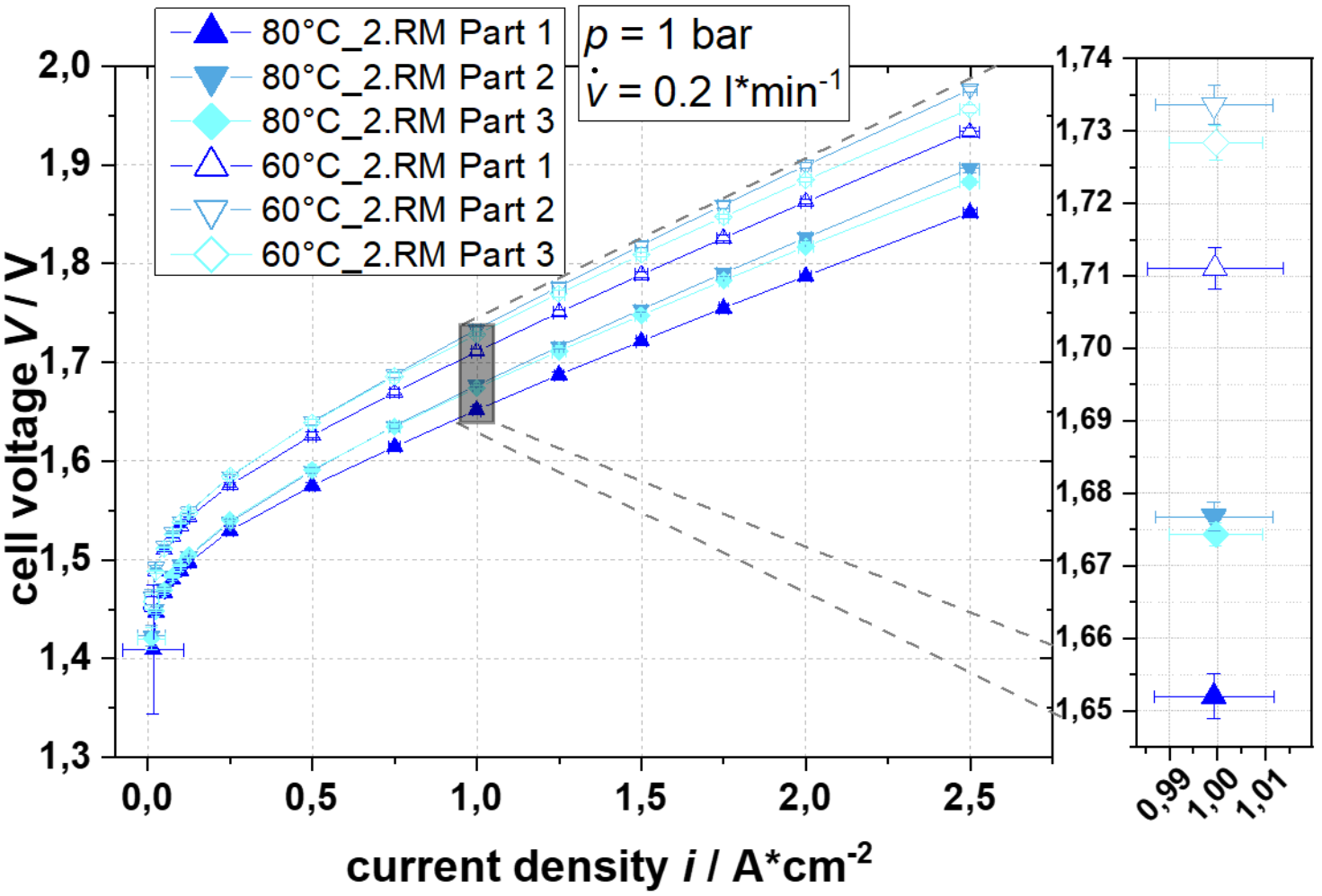
Results

Reproducibility in-house

- Fc = 2.5 kN → avg. pressure on active area ~ 3 MPa
- Results for reassembling with the same (used) components
  - ΔV = 9 mV @ 0.25 A*cm^{-2}, 60 °C
  - ΔV = 10 mV @ 0.25 A*cm^{-2}, 80 °C
  - ΔV = 17 mV @ 1 A*cm^{-2}, 60 °C
  - ΔV = 22 mV @ 1 A*cm^{-2}, 80 °C
  - ΔV = 37 mV @ 2 A*cm^{-2}, 60 °C
  - ΔV = 39 mV @ 2 A*cm^{-2}, 80 °C



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Summary and conclusion

- While people often stress the looong way to go, there is INDEED progress
- There is no need for identical test bench hardware
  - Some adjustment data collection might be needed, but those are minor changes
- There is a widely accepted test cell for reference measurements
  - To be able to compare high accuracy measurements, this is unavoidable
  - The cell can provide the needed accuracy
- Established measurement protocol
  - Includes a lot of experience
  - Already good enough to act as “best practice” guide
  - With limited investigations in identified areas, it can be converted into a quantitative reference measurement procedure

Thanks a lot for your kind attention!

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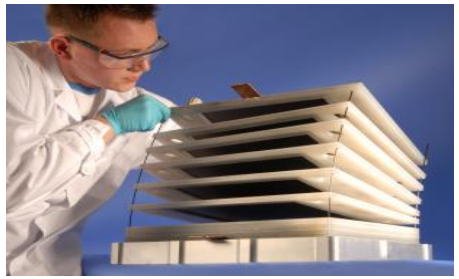

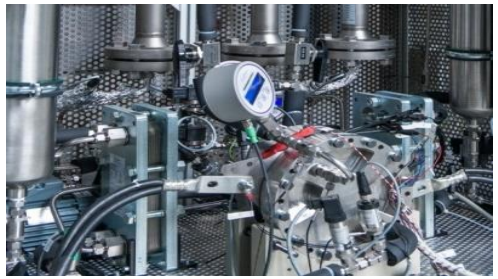
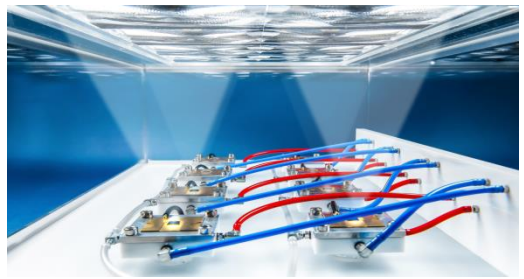


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