DEGRADATION, HARMONIZATION AND DYNAMIC TESTING IN AEL AND AEM

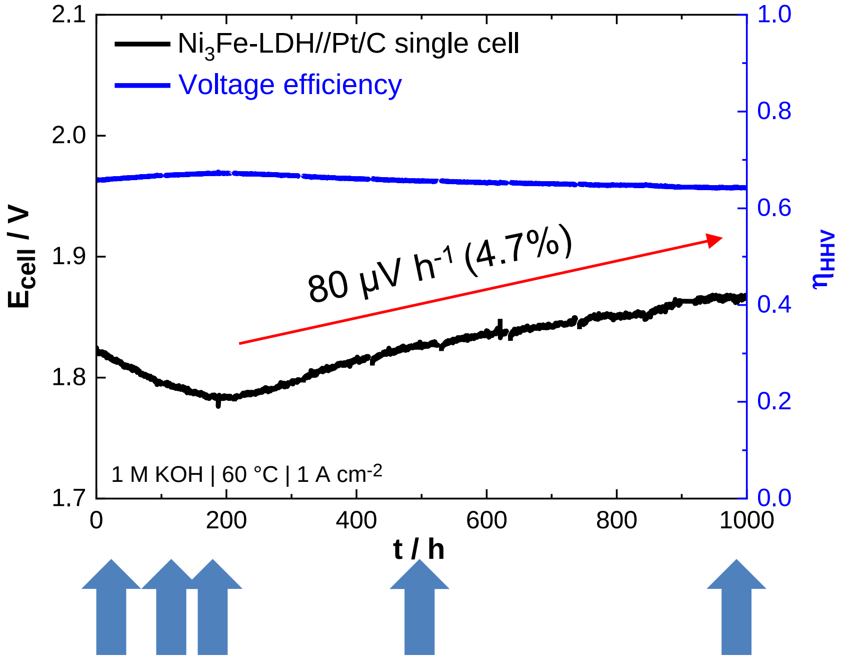
SEP. 29TH 2023 I F. LOHMANN-RICHTERS, I. GALKINA, M. MÜLLER, S. SUNDE, L. RITZ, S. PAPE

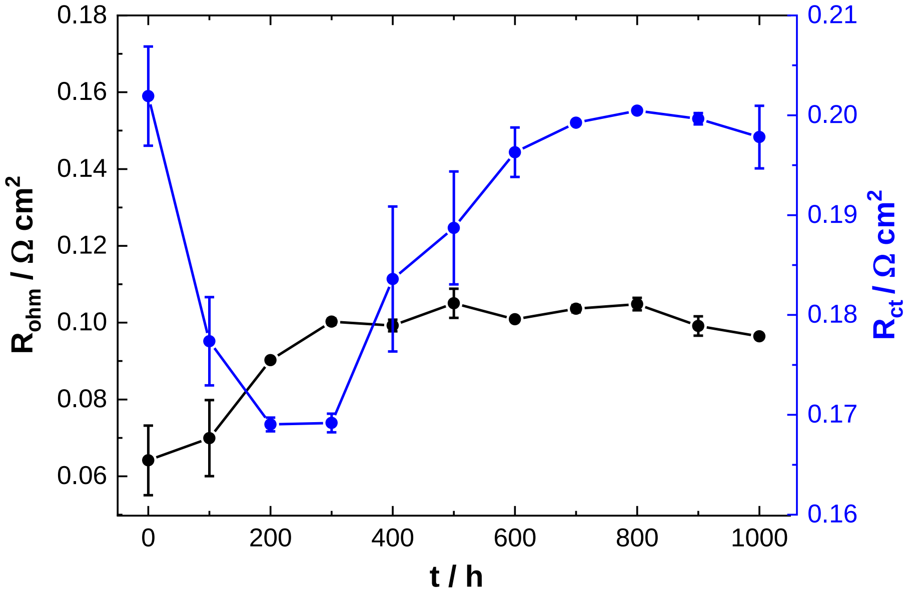
f.lohmann-richters@fz-juelich.de

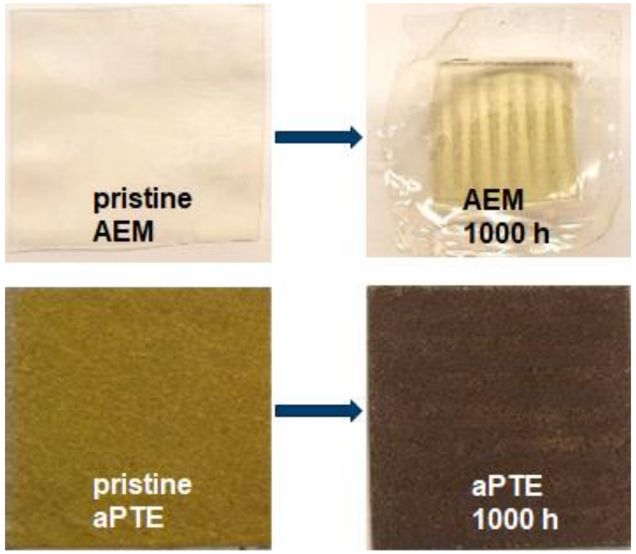
IEK-14: Institute of Electrochemical Process Engineering

PERFORMANCE & STABILITY OF THE Ni_3Fe-LDH ANODE

1000 h durability test and anode degradation tracking





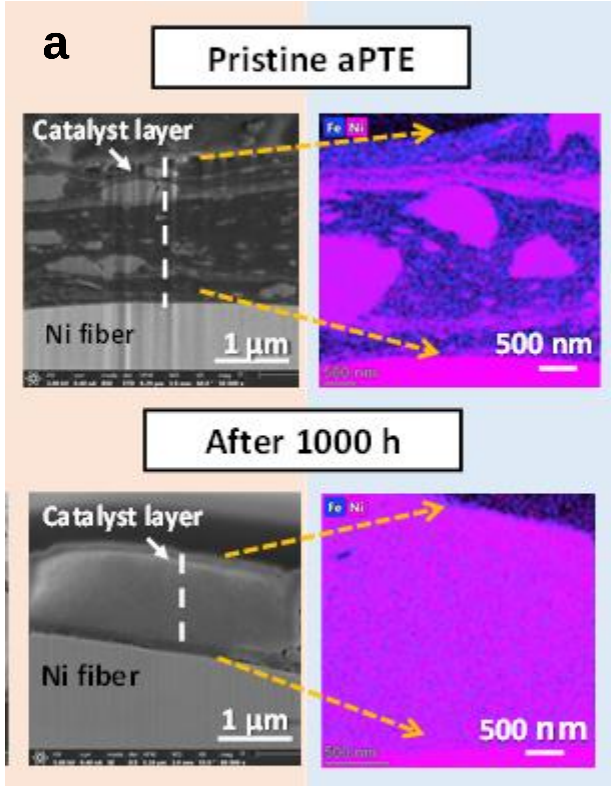


- Degradation tracking of the anode with ex-situ techniques after 15, 100, 200, 500, 1000 h
- Minor degradation rate of 80 µV h^{-1} (200-1000h)

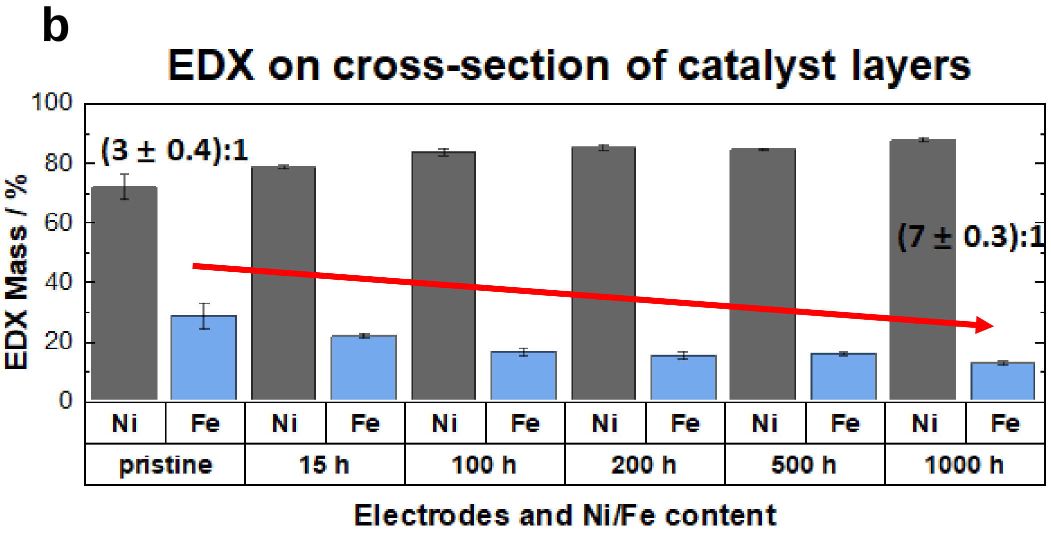
- R_{ohm} stabilized fast, indicating potential minor AEM degradation and stable catalyst layer-AEM connection
- R_{ct} decreases due to catalyst structure activation first, increase due to partial loss in electrocatalytic activity/ECSA

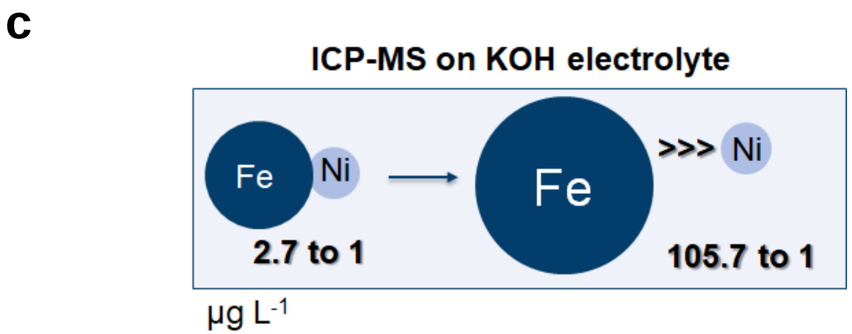
PERFORMANCE & STABILITY OF THE Ni_3Fe-LDH ANODE

Anode degradation tracking: SEM, EDX, ICP-MS…



fib-SEM STEM-EDS





- **Deep reconstruction** of catalyst layer: from agglomerates to sponge-like structure
- EDX and ICP-MS: Fe leaching from electrode

- Despite known degradation phenomena **stable performance**

PROJECT CHANNEL: ROLE OF HARMONIZATION

Comparison of test protocols

Test protocol in CHANNEL

1. **before assembly: soak electrodes and membrane in 1M KOH for 3h**
2. **cell assembly**
3. **Electrolyte feed:** 1M KOH (85%) feed rate: 250 mL/min, T=60 °C
4. **Break-in:**
   - start: 0.01 A/cm2
   - end: 1.5 A/cm2
   - step size: 0.1 A/cm2
   - hold: 250 s
   - cut-off: 2.1 V
5. **Constant current:**
   - 1 A/cm2 for 4 h
6. **Polarization curve**
   - start: 0.008 A/cm2
   - end: 1.5 A/cm2
   - step size:
     - 8 mA/cm2 until 0.12 A/cm2, Juelich: every 2nd
     - 20 mA/cm2 until 0.32 A/cm2
     - 40 mA/cm2 until 1.5 A/cm2
7. **GEIS**
   - 0.2 A/cm2, 0.5 A/cm2, 1 A/cm2, 1.5 A/cm2
   - 100 kHz to 100 mHz
   - 10 points/dec
   - amplitude: 10% of applied current density
8. **OCP 3 h**
9. **Polarization curve as before**
10. **GEIS as before**

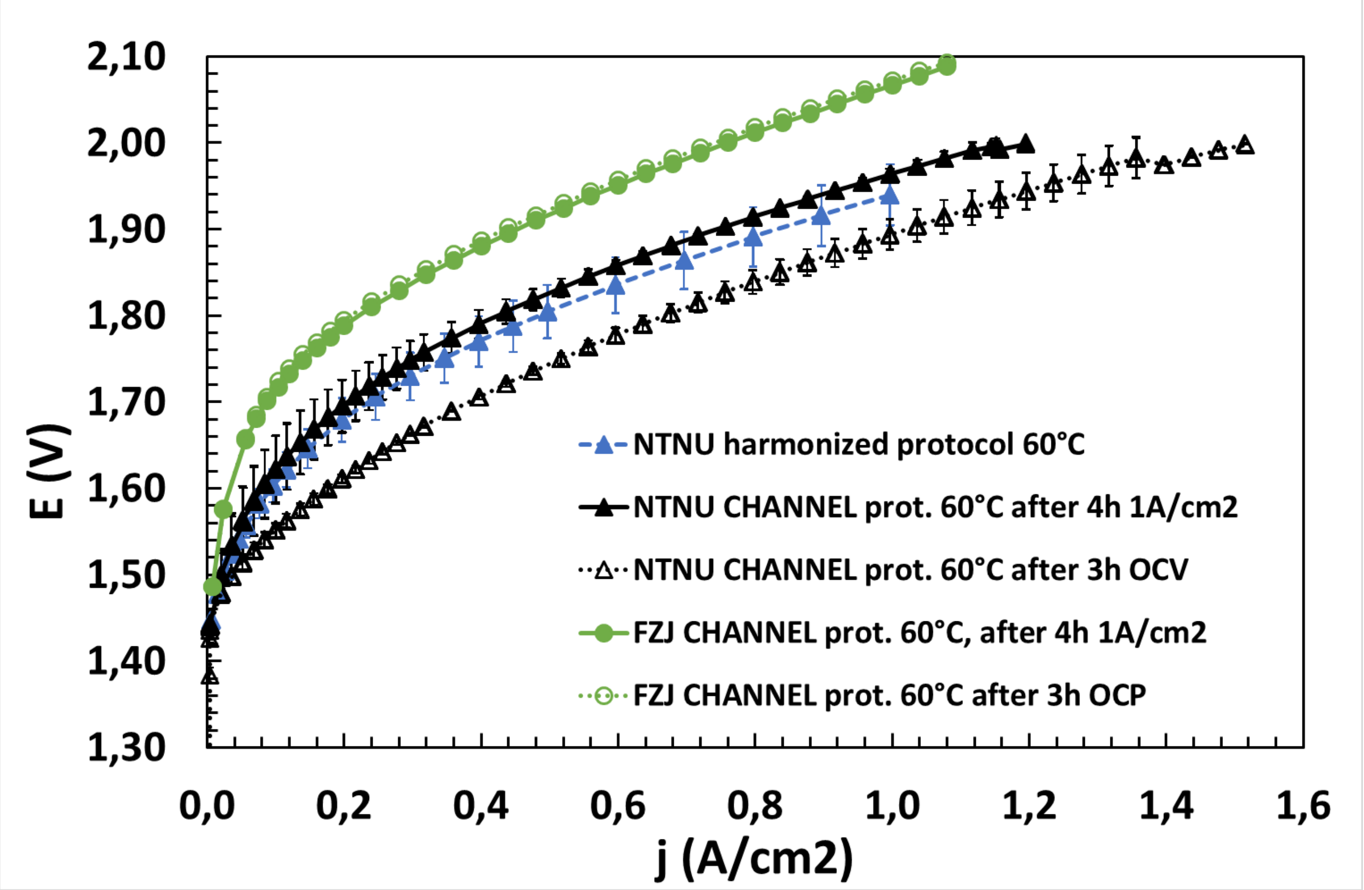
Harmonized protocol

1. **cell assembly**
2. **Electrolyte feed**: H_2O, 0.2M or 1M KOH; feed rate: 1 mL cm^{-2} min^{-1}, T=50 °C
3. **Conditioning**
   - 0.2 A/cm2 for 2 h (for KOH)
   - 0.05 A/cm2 for 2 h (for water)
4. **GEIS**
   - 0.2 A/cm2, 1 A/cm2
   - 100 kHz to 100 mHz
   - amplitude: 5% of applied current density
5. **Stabilize selected T (measured at outlet) and p at maximum current density**
6. **Polarization curve**
   - if maximum operating current density known: measure in descending fashion
   - start: 0.01 A/cm2
   - end: 1 A/cm2
   - step size:
     - 8 mA/cm2 until 0.10 A/cm2
     - 10 mA/cm2 until 0.06 A/cm2
     - 20 mA/cm2 until 0.15 A/cm2
     - 50 mA/cm2 until 0.6 A/cm2
     - 100 mA/cm2 until 1 A/cm2
7. **Polarization curve in ascending fashion (check for hysteresis)**
8. **Repeat at other T or p**

PROJECT CHANNEL: ROLE OF HARMONIZATION

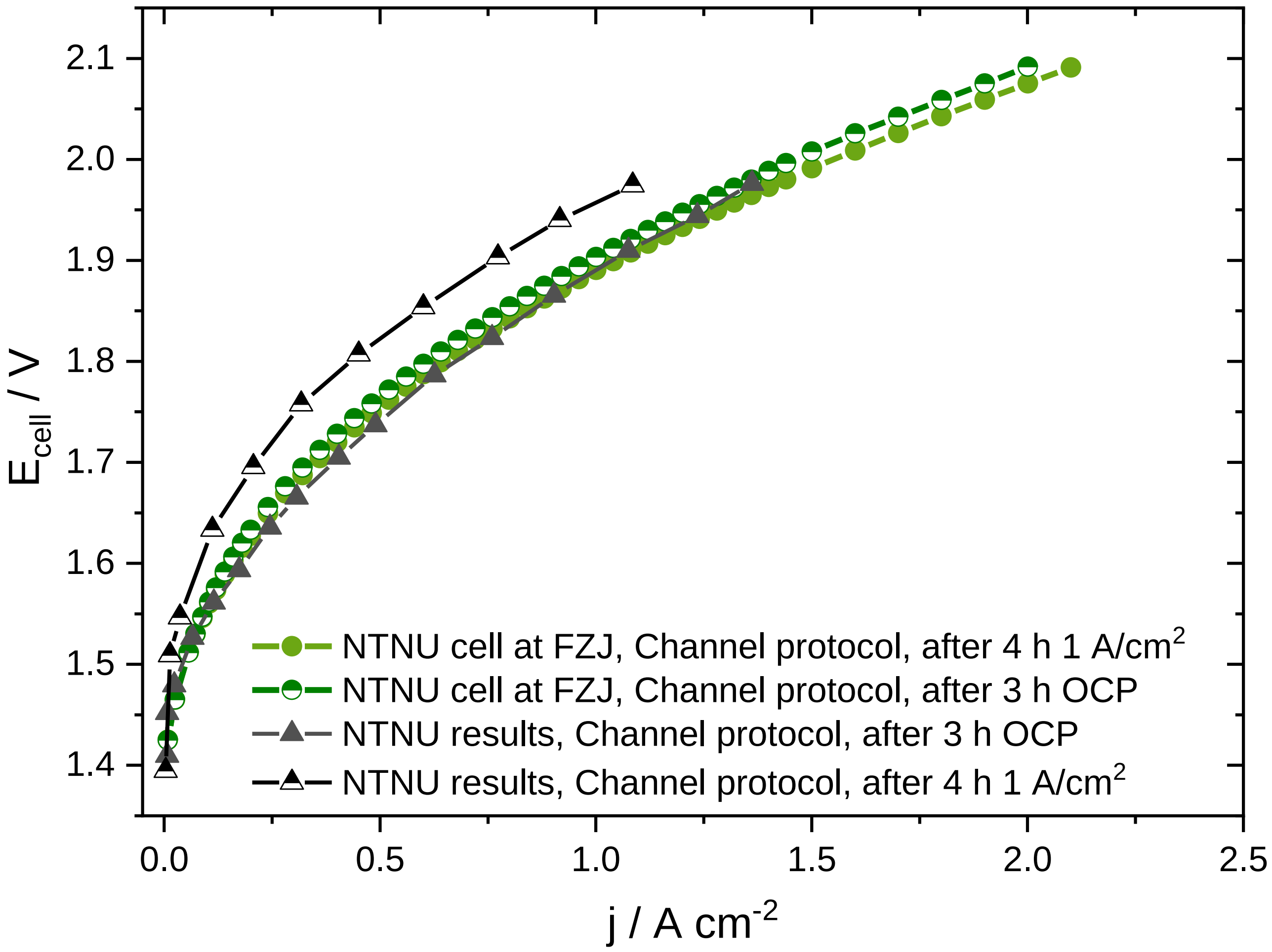
- Similar performance CHANNEL vs. harmonized protocol
- Lower performance observed at FZJ
- Influence of OCP not consistent

- No significant influence of flow rate observed

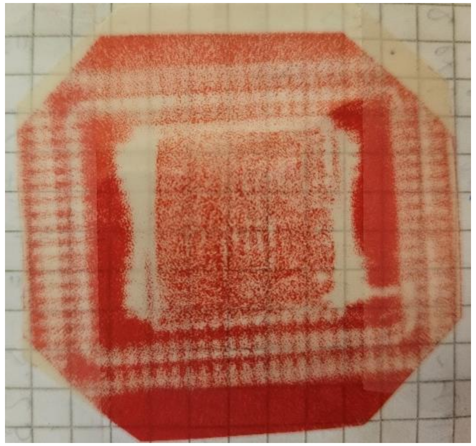


PROJECT CHANNEL: ROLE OF HARMONIZATION

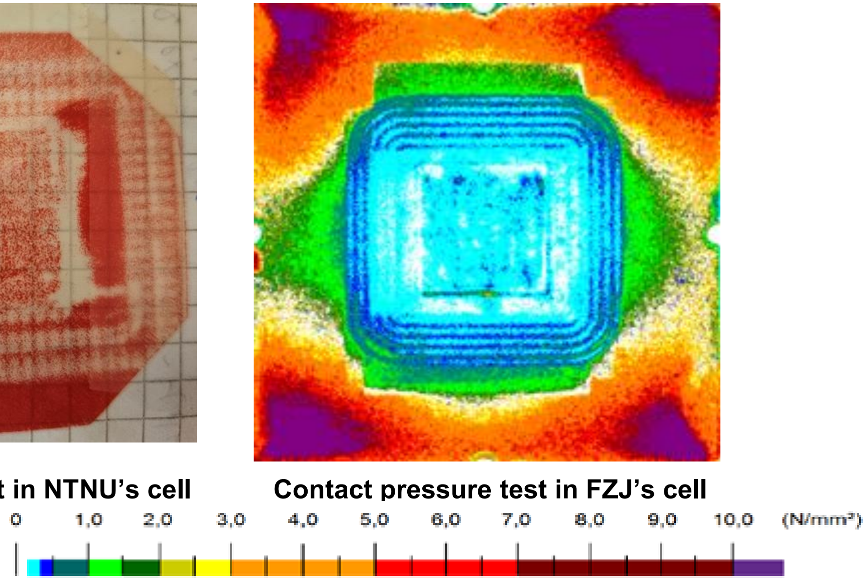
Impact of the single cell hardware



- Comparable results with same single cell
- Homogenous pressure distribution inside the active area for both used cells



Contact pressure test in NTNU's cell

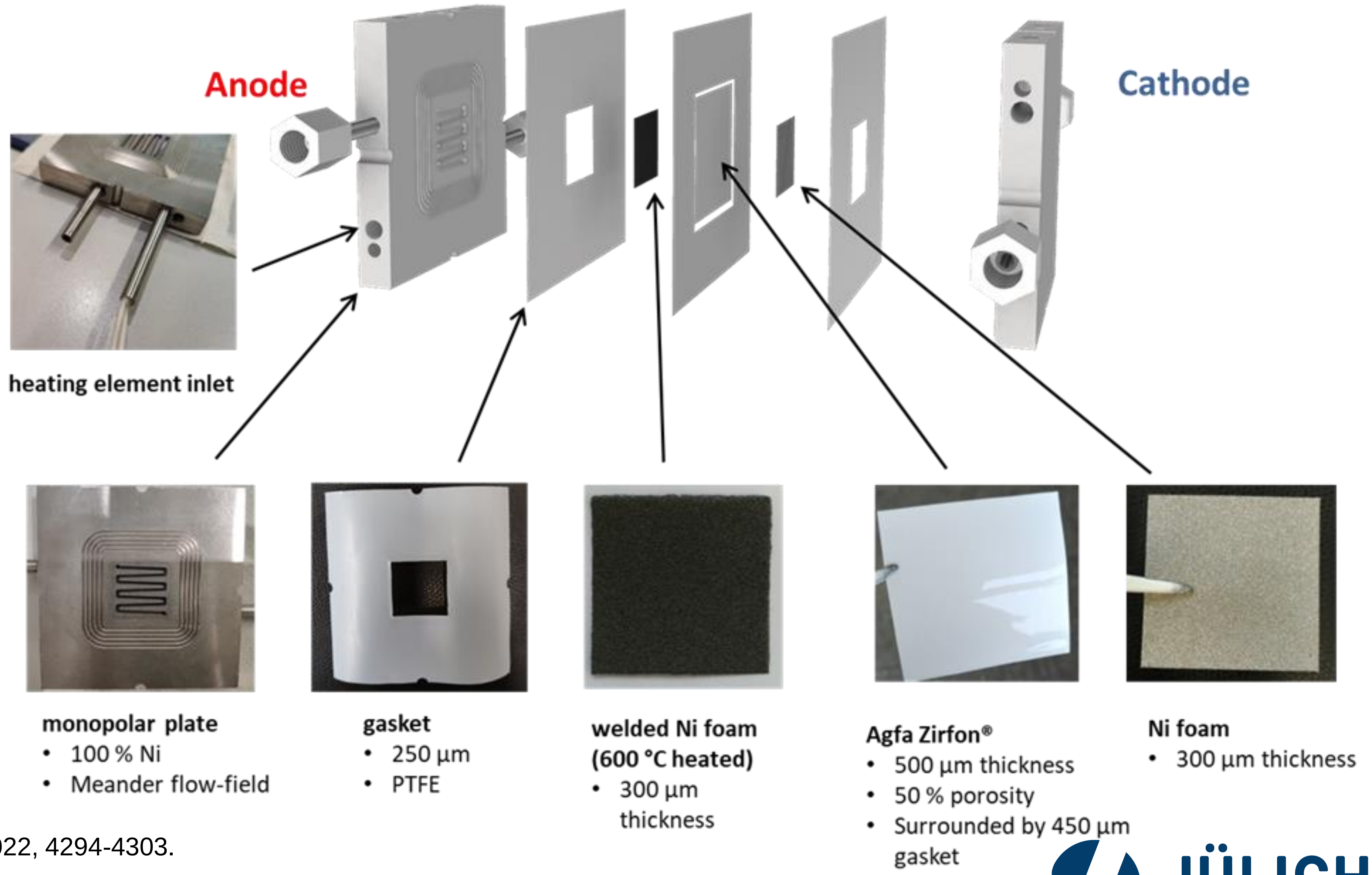


Contact pressure test in FZJ's cell

JÜLICH Forschungszentrum

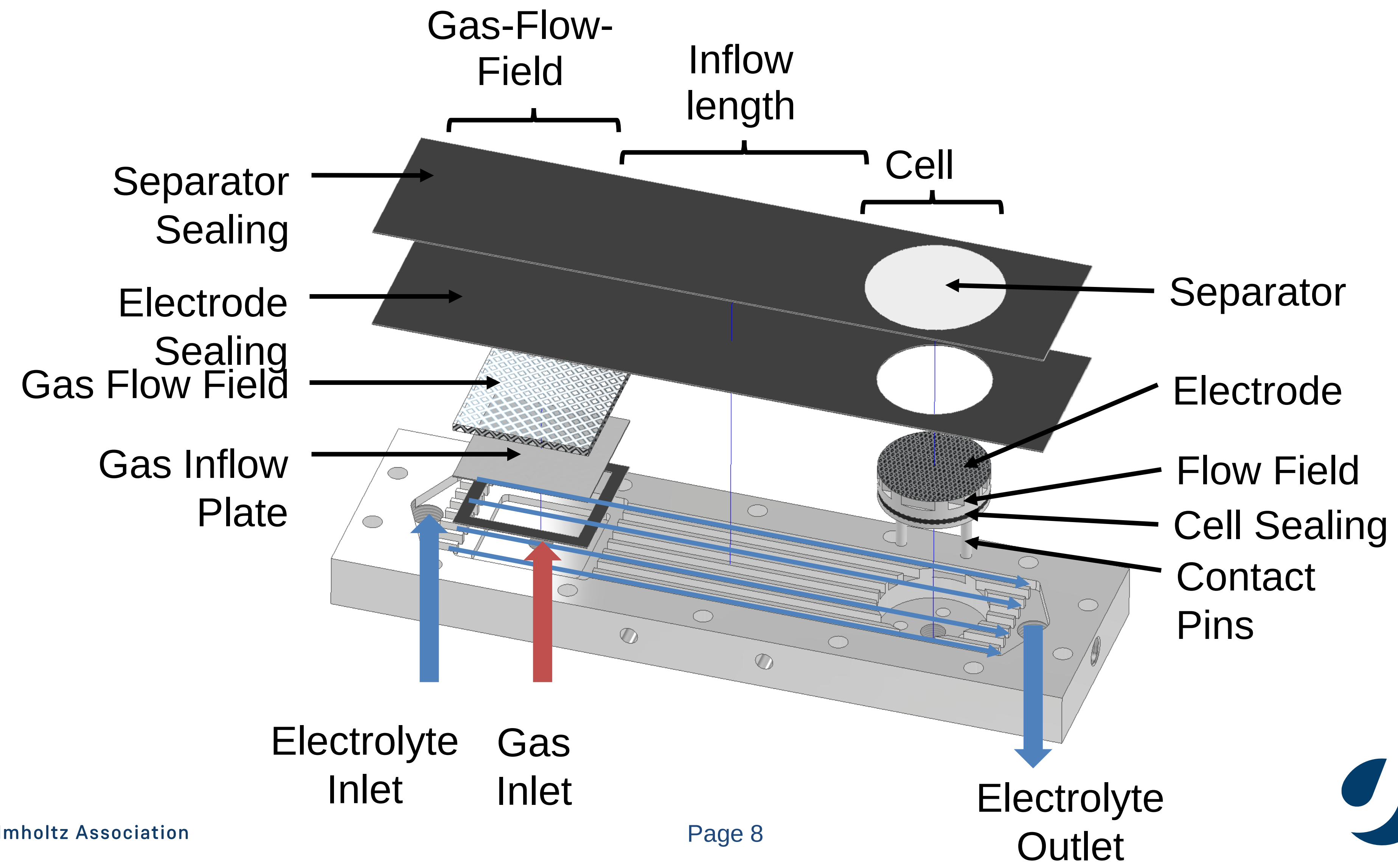
AWE BENCHMARK CELL

- Compression defined by hard stop gaskets
- Different compression requires reassembly of the cell



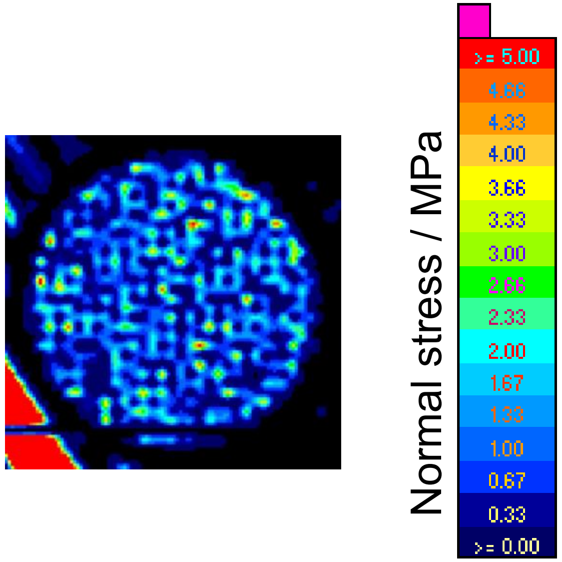
C. Karacan, *et al.*, *Int. J. Hydrogen Energy 47*, 2022, 4294-4303.

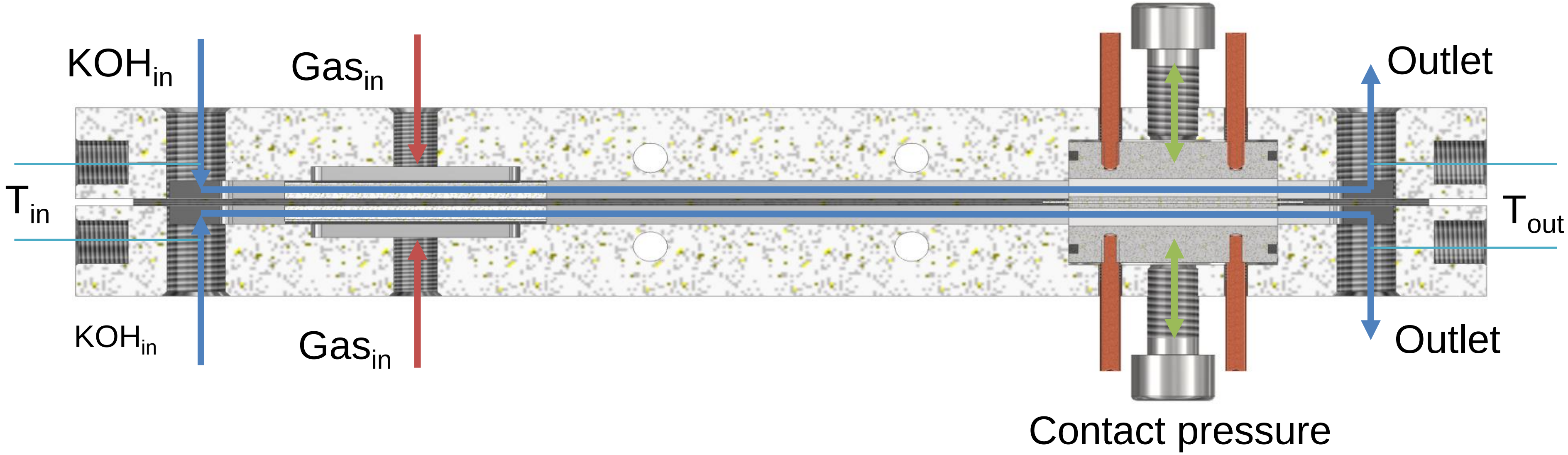
CONTACT PRESSURE CELL



CONTACT PRESSURE CELL

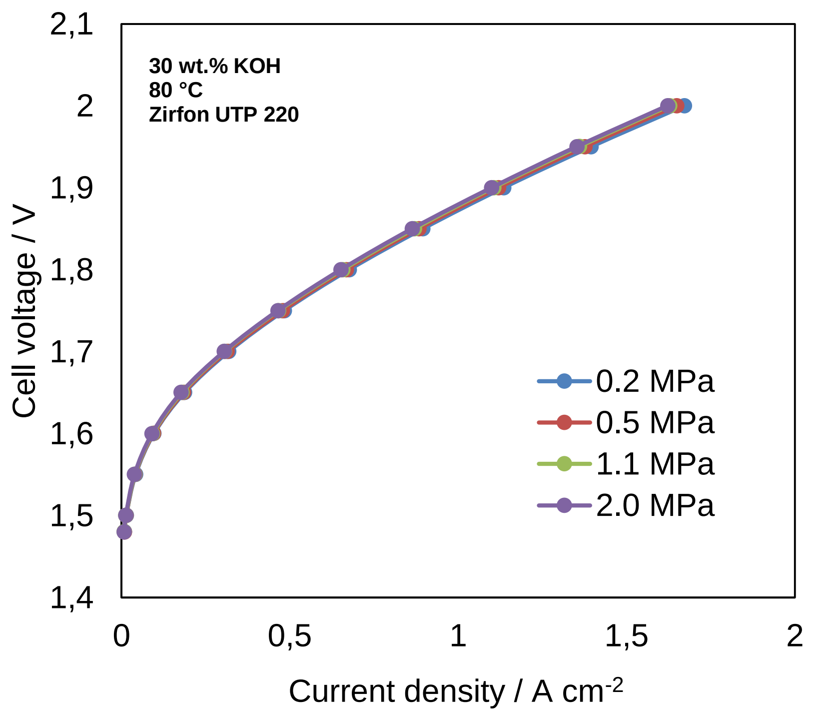
- Cell design to allow easy and precise control of compression

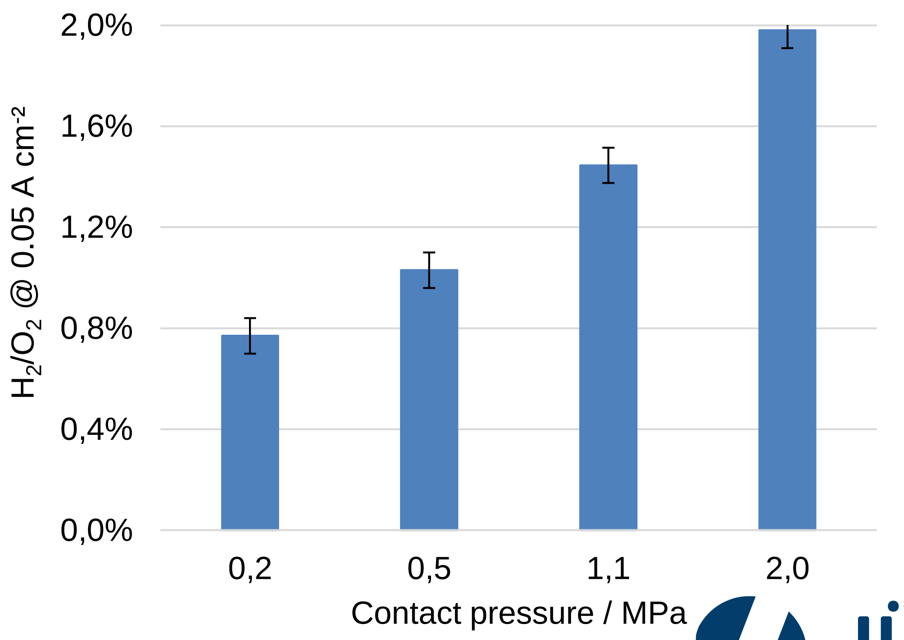




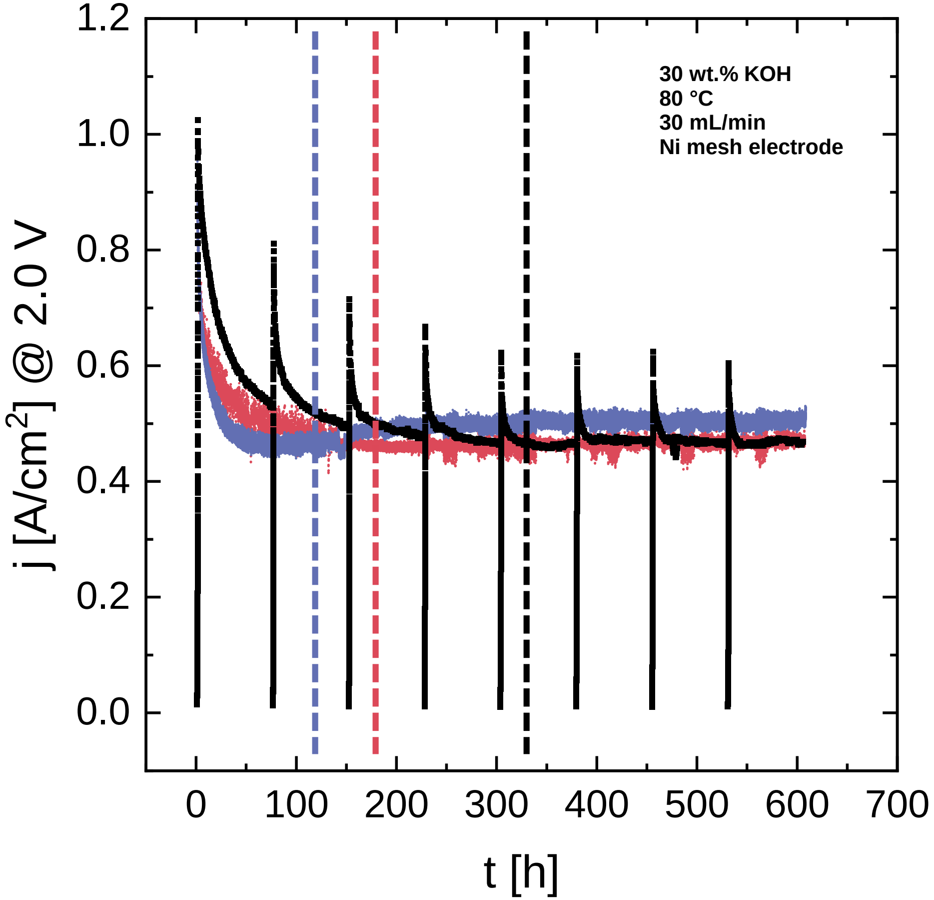
CONTACT PRESSURE IMPACT

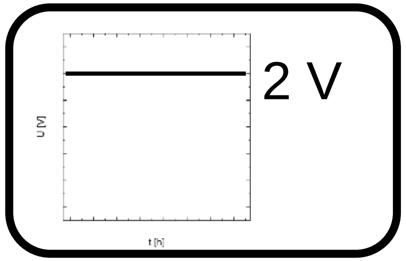
- Polarization curve is independent of compression
- Crossover depends strongly on contact pressure
- Crossover coparison requires **precise control of compression**



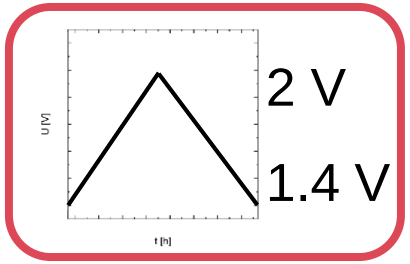


DYNAMIC CONDITIONING AND DEGRADATION

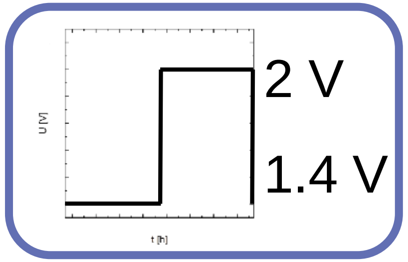




Stationary

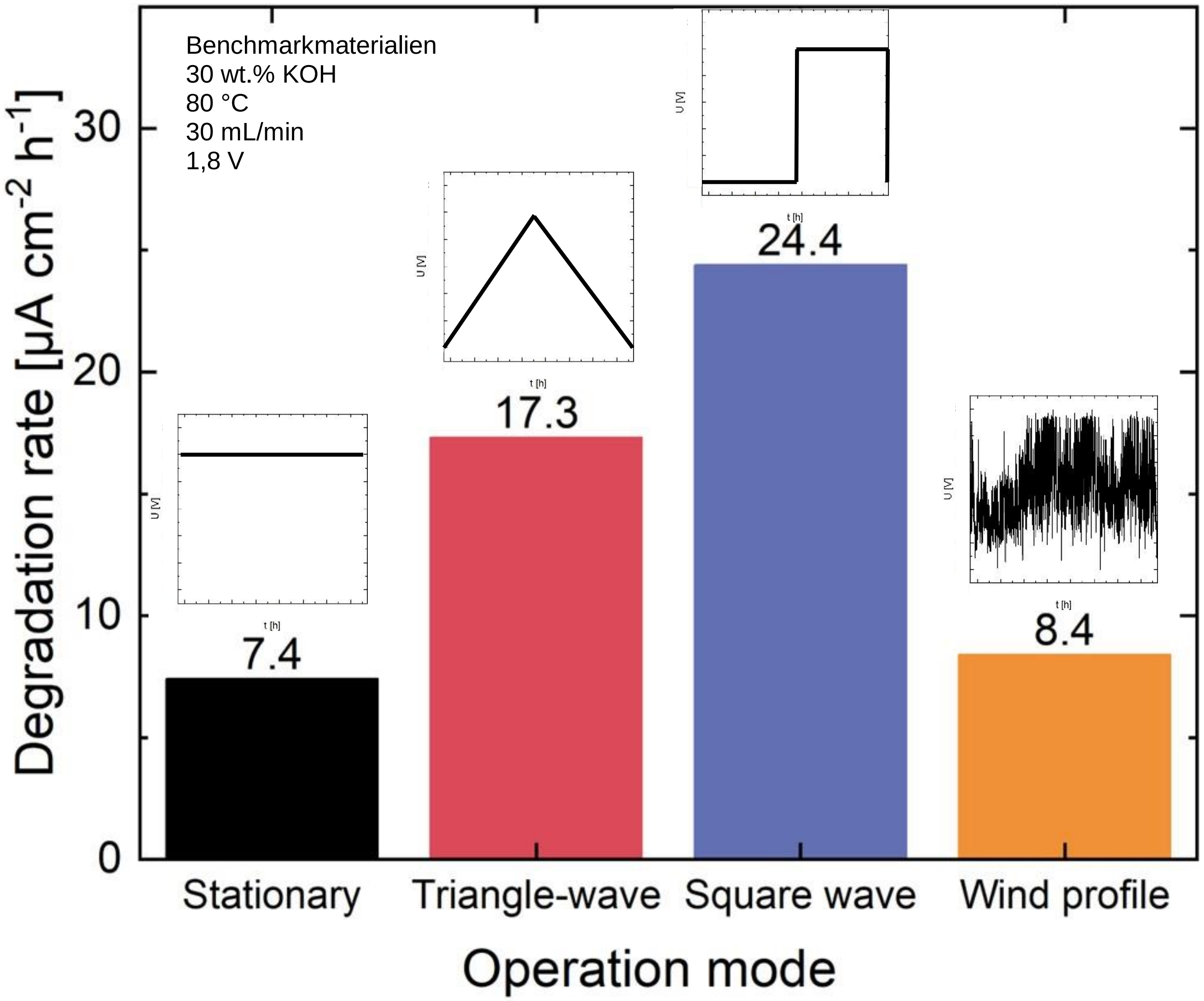


Triangle wave



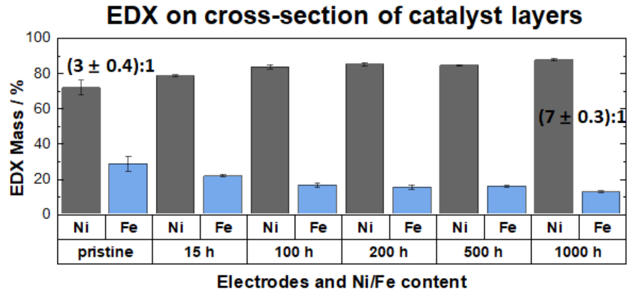
Square wave

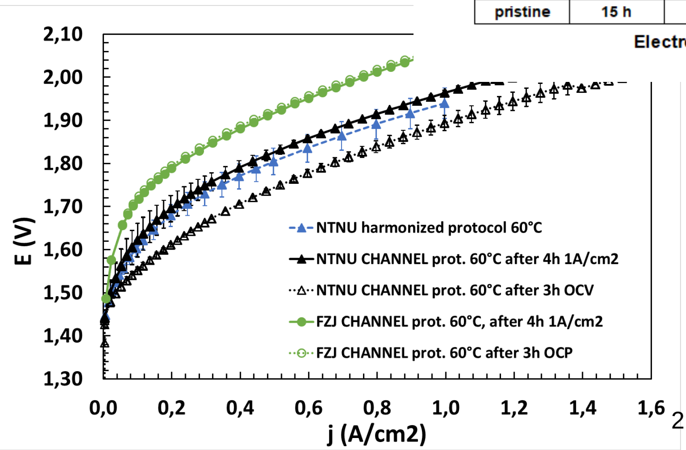
DYNAMIC CONDITIONING AND DEGRADATION

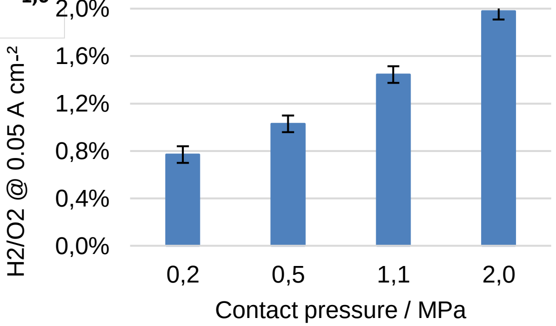


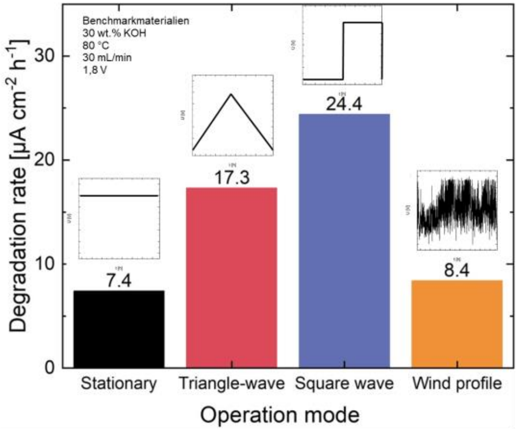
SUMMARY

- Degradation tracking in AEMEL anode
- Harmonization of protocol and single cell needed
- Contact pressure has important influence on crossover
- Dynamics influence conditioning and degradation









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ACKNOWLEDGEMENTS

CHANNEL

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THANK YOU FOR LISTENING!

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