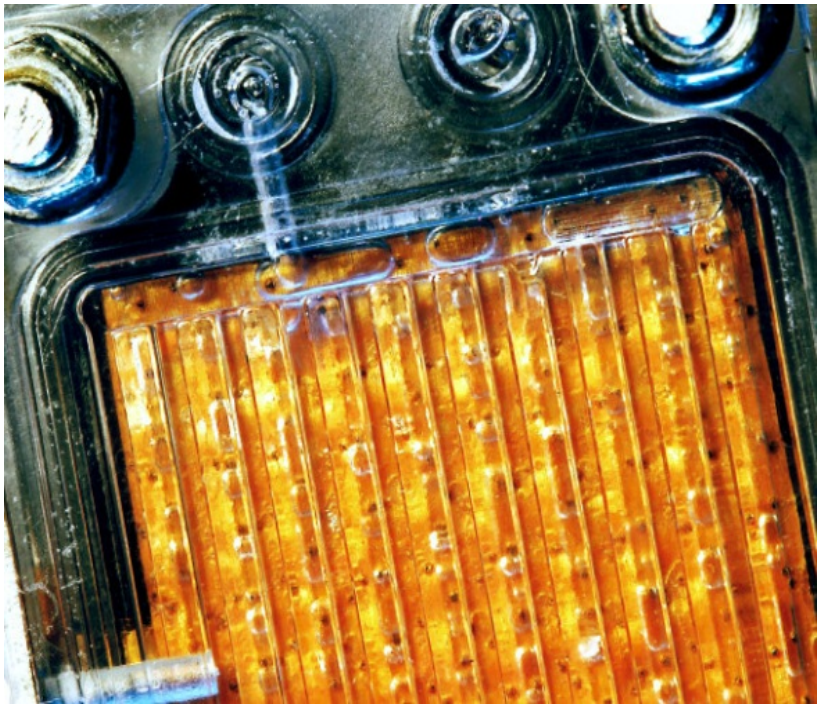
Round Robin Testing for Polymer Electrolyte Membrane Water Electrolysis – Phase 2

On the (long) way to harmonized testing protocols



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Outline

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- Round Robin Test – Phase 2
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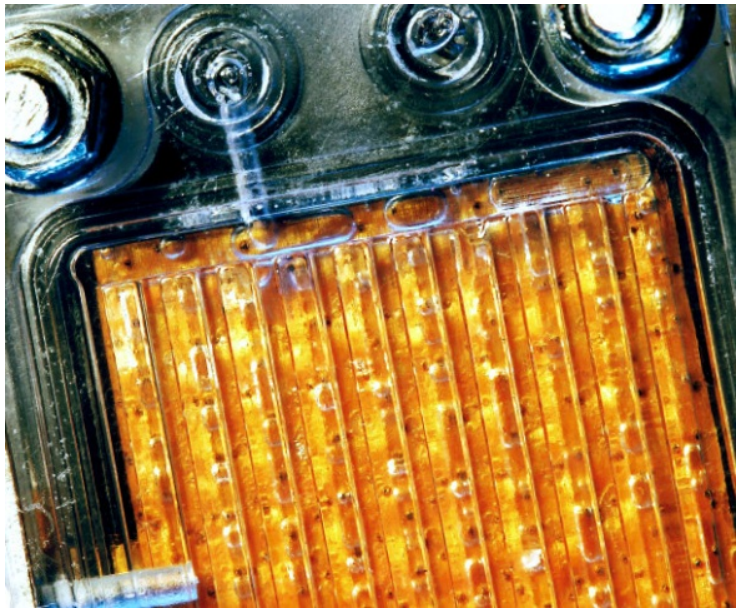
- Conclusion and next steps



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Working group IEA Annex 30 (Electrolysis)
Introduction

- One out of eight working groups in the Technology Collaboration Programme on Advanced Fuel Cells (AFC TCP), established in 2014
  - More information: http://www.ieafuelcell.com/
- Technologies covered
  - PEM water electrolysis
  - Alkaline water electrolysis
  - Solid oxide steam electrolysis
- General motivation
  - information sharing and learning between experts
  - accelerate development of WE towards commercialisation



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Technology Collaboration Programme on Advanced Fuel Cells
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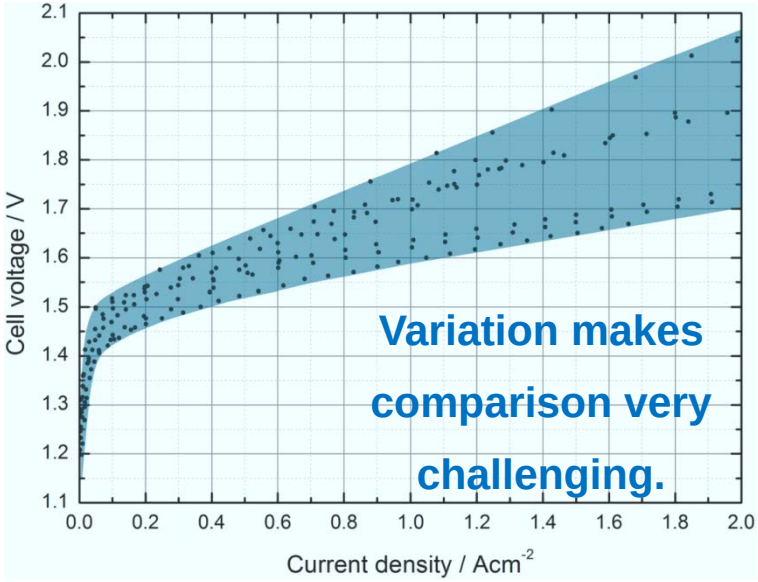
Working group IEA Annex 30 (Electrolysis)
Motivation for harmonized test procedures

- "We are comparing apples to pears"
  - Anybody
  - All over the world
- Establish the basis for accurate data comparison across laboratories to evaluate progress in R&D work
  - In-situ experiments → Vi curves
  - Ex-situ experiments
- Use this common basis to join research effort on degradation issues in water electrolysis



Comparison of different cell performance data from literature (source: M. Carmo [1])

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Harmonized Test Procedure For PEM Water Electrolysis

Standard operational conditions

Quantity	For PEM WE
Cell Temperature	60, 80 °C
Water Flow Temp. Anode	60, 80 °C (inlet)
Water Flow Temp. Cathode	60, 80 °C (inlet) (optional)
Water Flow Anode	Min 2 mL/min·cm^2 $\Delta T \leq 2$ K
Water Flow Cathode	2 mL/min·cm^2 (optional)
Pressure Anode	1 bar_abs @ outlet
Pressure Cathode	1 bar_abs @ outlet
Water quality	ASTM Type II (> 1 MOhm·cm)
Cell Area	25 cm^2 (still under discussion)

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Harmonized Test Procedure For PEM Water Electrolysis

Standard operational protocol (revised draft)

1	Achieve stabilized standard operational conditions (Q, T and p)
2	Apply relevant manufacturer break-in procedure
3	Apply 1.7 V until stationary condition (less than 0.5 % variation in 1h)
4	Perform polarization curve: * Galvanostatic mode, current density in/decreased with steps of 250 mA/cm² * Drift Control Protocol (further information see [1]) * Sequence for current steps - 1x from low current densities to high current densities - 1x from high current densities to low current densities * Cell voltage is limited to 2.0 V * Mean values of all "up & down" Vi data with indicating standard deviation
5a	- HFR at 1 kHz, amplitude: +/-5 % (and not over +/-10 mV) of the applied current (maximal phase shift of +- 5°) → iR-corrected Vi curves (0.2 – 3 A/cm²)
5b	- Impedance spectroscopy (EIS) from 10 kHz – 0.1 Hz +/-5 % I_DC amplitude, max +/- 10 mV @ 0.125 / 0.25 / 0.5 / 1.0 / 2.0 A/cm²

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Harmonized Test Procedure For PEM Water Electrolysis
Introduction to Round Robin Tests

- Definition, according to Wikipedia[1]
  - Is an interlaboratory test (measurement, analysis, or experiment) performed independently several times;
  - Identical samples are analyzed by identical methods to make statements about the measuring accuracy in general or about the measuring quality of the participating institutes;
- Motivation for RR tests in our Annex 30 (Electrolysis)
  - Validation of test protocols for harmonized test procedure.
  - Quantify testing variation across international PEM WE community

[1] *https://en.wikipedia.org/wiki/Round_robin_test*

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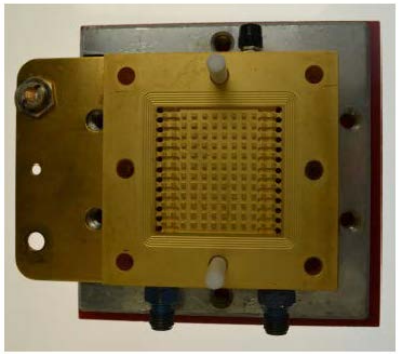
Round Robin Test – Phase 1
Objective and hardware

- Establish the basis for accurate data comparison across five laboratories:
  - NREL, FZJ, DLR, Fraunhofer ISE, Proton OnSite
- Polarization curves with commercial MEA, defined hardware and operating conditions
  - FZJ test cell with 25 cm² and pin cushion
  - Toray carbon paper as porous transport layer
  - 3-layer CCM from Grenerity E300 Gen1 (Five CCMs for each partner)
- More details:
  - *[1] M. Carmo: Benchmarking and Round Robin Testing for Polymer Electrolyte Membrane (PEM) Water Electrolysis. Oral presentation, 231st ECS Meeting, New Orleans/USA, 2017-05-30*
  - Joint publication coming soon!





Laboratory test cell for RRT-1 (FZJ)

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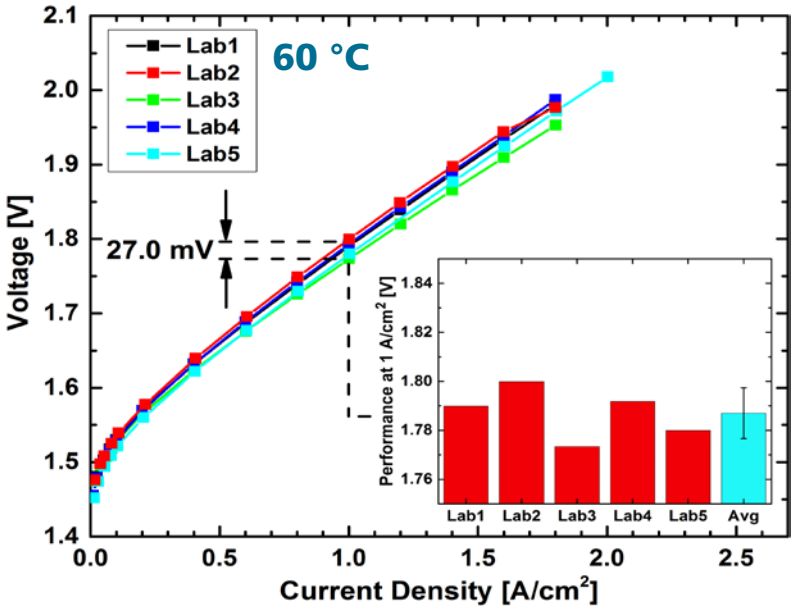
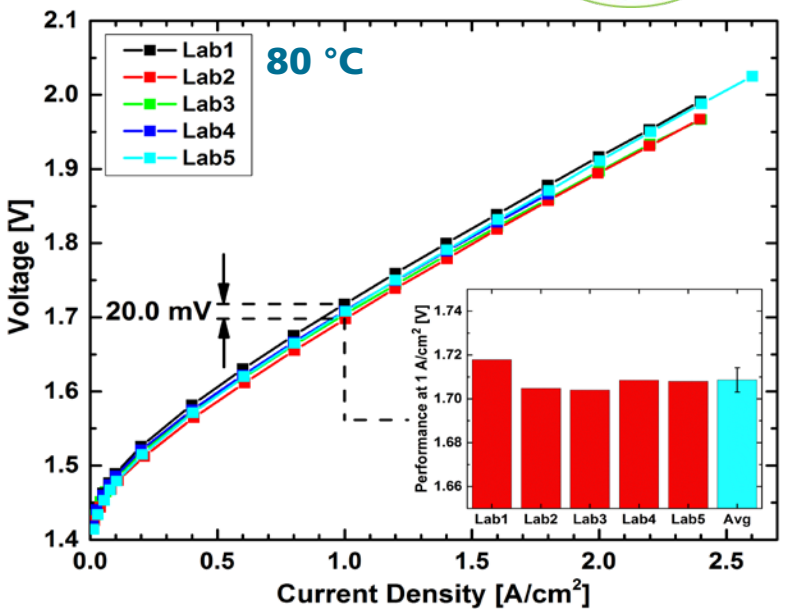
Round Robin Test – Phase 1

Main results: Reproducibility across all labs





- By factor 3 higher variation compared to the best repeatability in a single lab
- Outliers were already removed from the comparison
- Variation higher at lower temperatures

➔ **Too large variation as effect of temperature, reassembling and aging**

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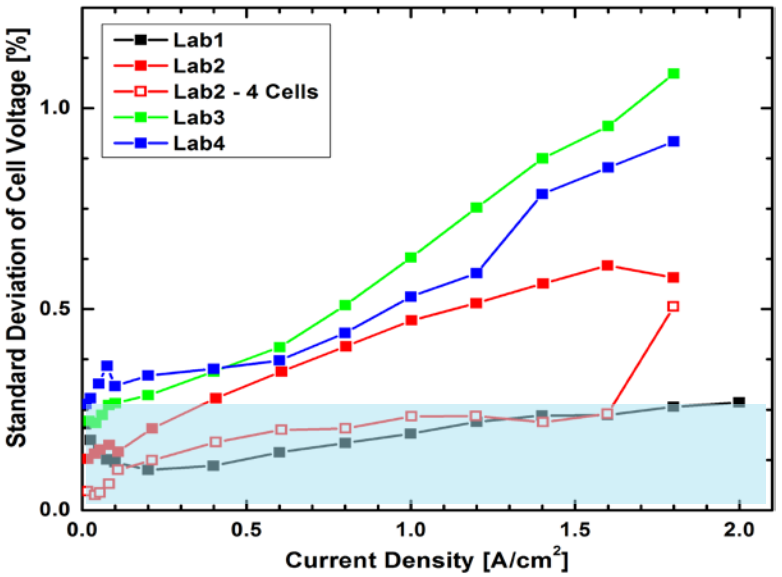
Round Robin Test – Phase 2

How can we achieve higher reproducibility?

- Round robin test phase 2 is set up among three institutes to achieve standard deviation < 0.25 % of cell voltage
- **#1: Tighten operating conditions**
  - temperature control (!) and TE calibration
  - pressure control, (water flow)
- **#2: New hardware**
  - New test cell with precise and adjustable clamping force
  - Titanium based porous transport layer
- **#3. Review and refine procedures**
  - Smoothing test protocol
  - Add EIS experiments (harmonize EIS procedure and data extraction)



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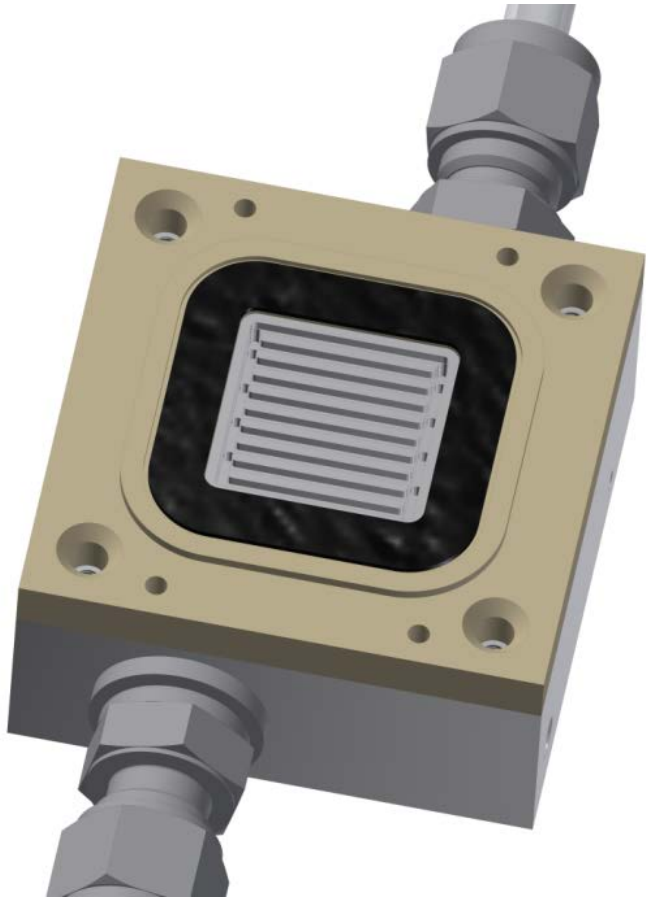
Round Robin Test – Phase 2

New laboratory test cell for higher reproducibility

- Active area: 4 cm² (rev0 → tbd)
- No reference electrodes (rev0 → tbd)
- Half cell bodies made from Au coated Ti
- Different PTL's can be tested (thickness) by replaceable cell frame made from plastic
- Sealing by flat sheet gasket
- Temperature conditioning via water flow circulation
- Inflow at the back of the PTL
  (no impact from the PTL edge)



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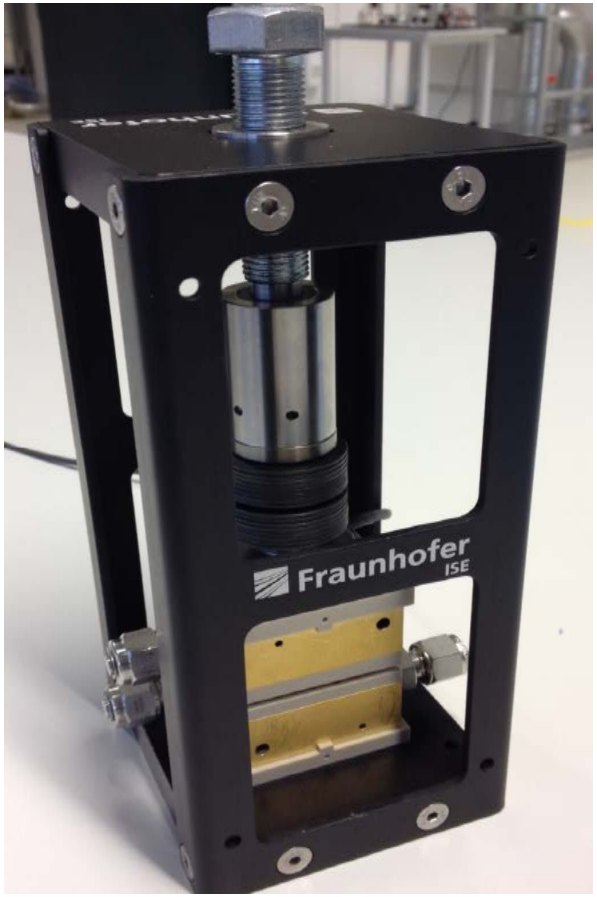
Round Robin Test – Phase 2

New laboratory test cell for higher reproducibility

- Two half cells are compressed and fixed by central punch/stamp
  - Force-displacement-decoupling by disc springs
- Force sensor to monitor clamping pressure during measurement (sealing & active area)
  - Adjustment of clamping force only manually possible
- Several voltage and temperature measurement loops
- Laboratory cell already provided to several institutions around the world
- ➔ Could serve as a standard hardware for harmonized test protocols



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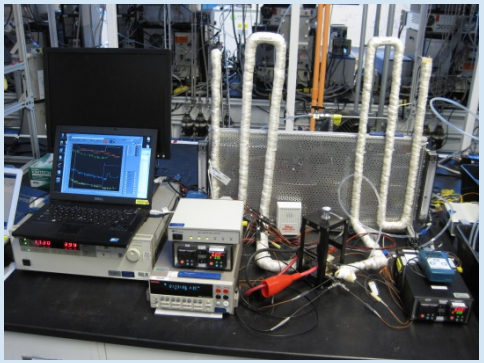
Round Robin Test – Phase 2

Different test benches are used across all labs



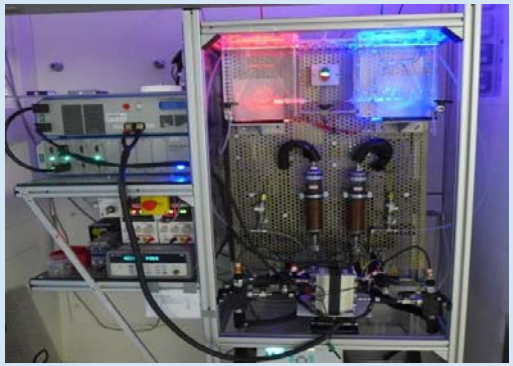
Research Center Jülich

- Commercial set up Greenlight G40
- 12V/100A power supply
- Up to 30 bar operation
- Anode and cathode water circulation loop



National lab NREL

- In-house construction
- 20V/120A power supply
- Up to 1.5 bar operation
- Back pressure control to compensate altitude
- Single-pass water flow



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- In-house construction
- 4x 10V + FRA power supply & 100A booster
- Atmospheric operation
- Anode and cathode water circulation loop

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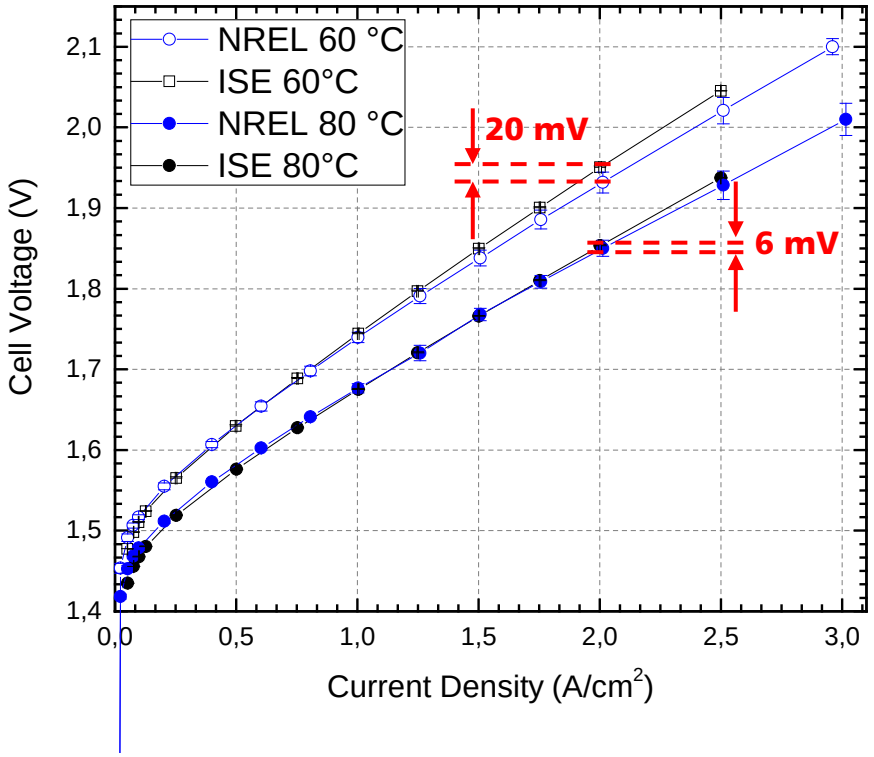
Round Robin Test – Phase 2

Interim results: Polarisation curve

- Interim results from only two labs (on-going measurements)
- Already small variation for 2 A/cm²
  - 20 mV @ 60 °C
  - 6 mV @ 80 °C
- Identical temperature control at inlet of anode
- Water flow at anode and cathode
  - But lower flow rate at NREL
  - More heat is retained at NREL's measurements → higher temp.



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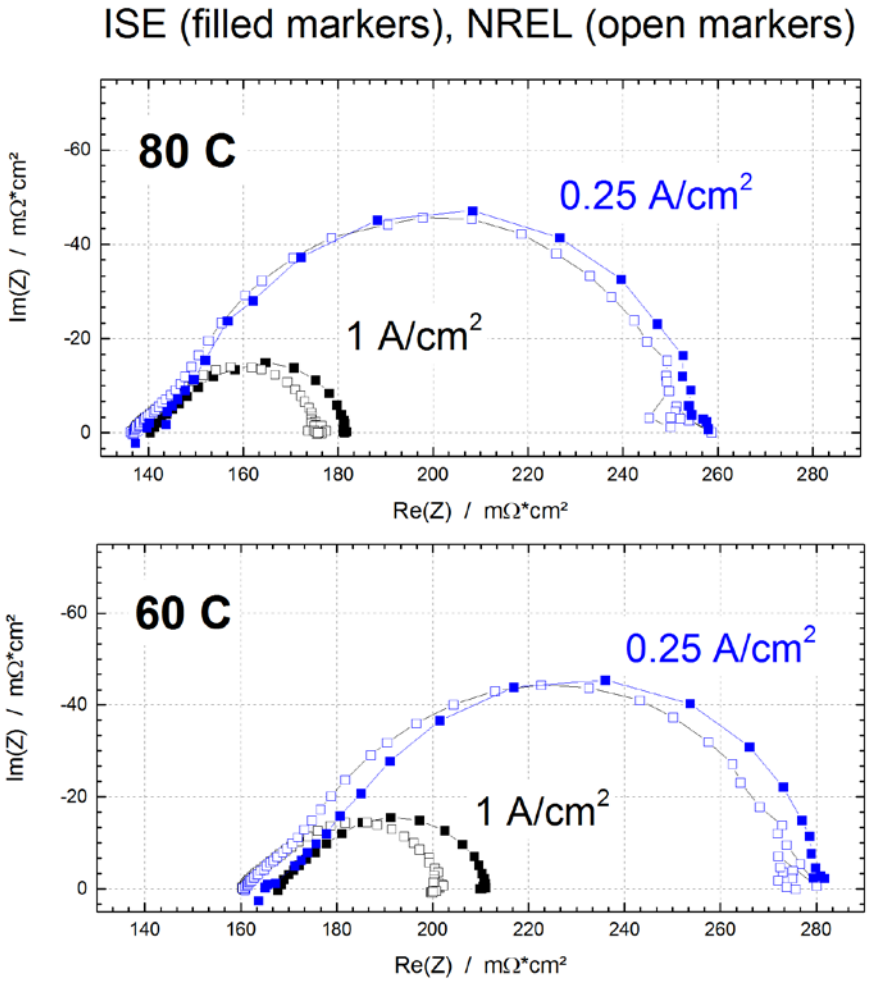
Round Robin Test – Phase 2

Interim results: Nyquist plot from impedance spectroscopy

- Interim results from only two labs (on-going measurements)
- Impedance spectroscopy is powerful tool to evaluate performance and variation
- Typical Nyquist plot
  - Temperature shift
  - Current density dependance
  - ➔ High frequence resistance (HFR) can be extracted
- Test protocol and data extraction still under discussion



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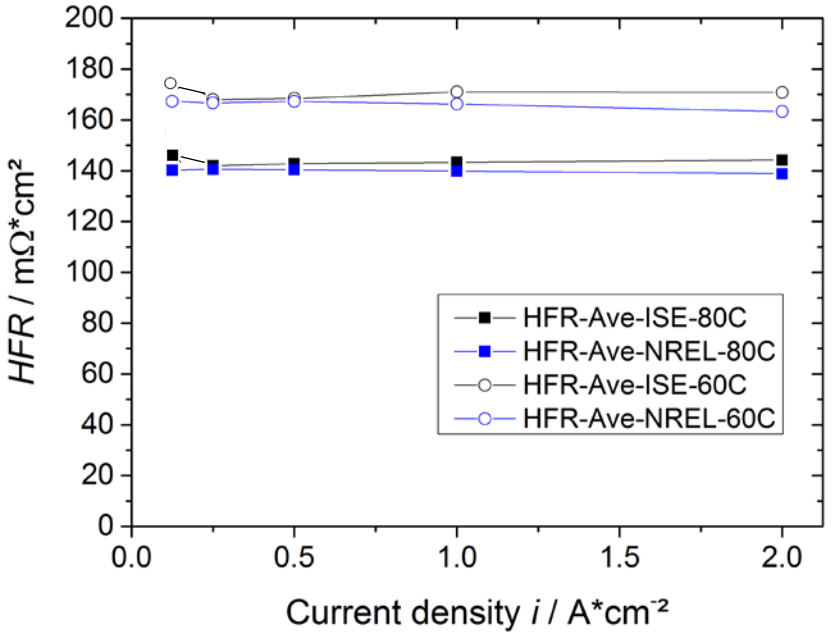
Round Robin Test – Phase 2

Interim results: High frequence resistance (HFR)

- Interim results from only two labs (on-going measurements)
- Extracted HFR from EIS measuremnet corresponds well to Vi curves
  - Membrane dominates HFR
    - Temperature control
    - Water management



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Round Robin Test – Phase 2
Preliminary conclusions

- Improvement measures of 2nd phase resulted in smaller variation
  - Successful deployment of new test cell with incompressible Ti PTLs
  - Promising agreement between two labs
- 2nd phase still not finished
  - Further understanding/investigating differences will continue to drive protocol development
  - Temperature control location(s) needs further harmonization (inlet vs. outlet, anode-only vs. anode and cathode)
  - Define PTL "state" (i.e. cleaning procedure, degree of oxidation)
- Next steps after 2nd phase of round robin tests:
  - Finishing paper for 1st phase of round robin tests
  - Define standard testing protocols to access cell degradation
  - Define accelerated stress tests (ASTs)

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Thanks a lot for your kind attention!

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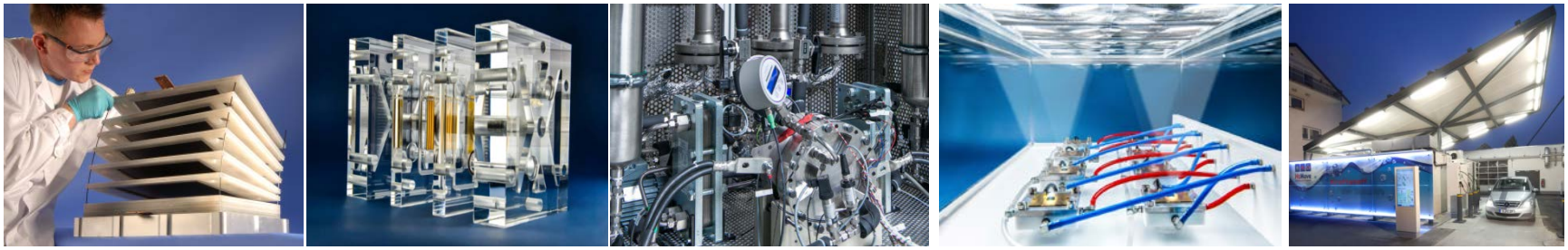

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