

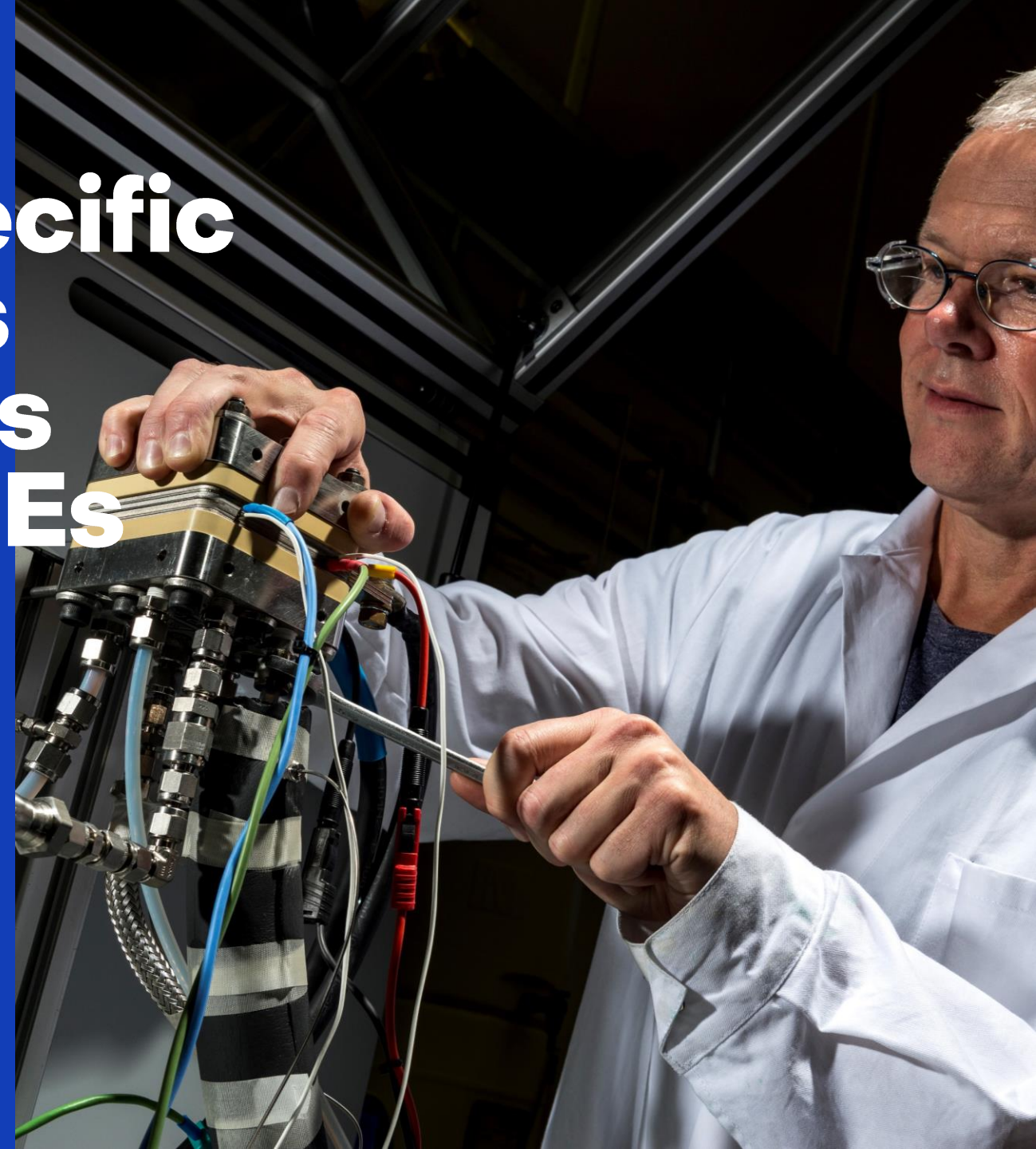
A Component-specific approach towards Accelerated stress testing for PEMWEs

29th September 2023

Dr. Johanuurma | Scientist johan.buurma@tno.nl

Dr. Giulia Marcandalli | Consultant giulia.marcandalli@tno.nl

Dr. Arend de Groot | Sr. Consultant arend.degroot@tno.nl



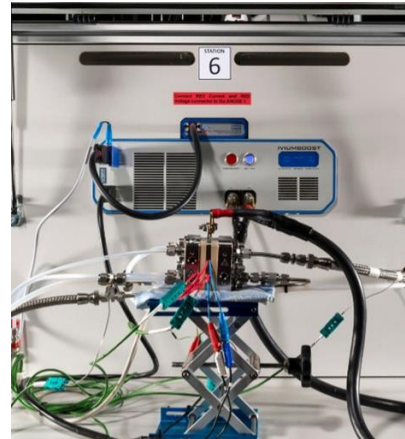
TNO: Netherlands Organisation for Applied Scientific Research



1 cm²

ASTs Testing stations

10 cm²



R&D CELL MANUFACTURING & CELL/SHORT STACK TESTING

PEM, SOE, AEM + testing for Alkaline

Hydrohub
Groningen

Faraday Lab
Petten



2500 cm²



STACK AND BALANCE-OF-PLANT TESTING
PEM (50 kW, full-scale cells) and Alkaline in consortium with end-users

Accelerated Stress and Life Tests

Understand and Prevent **cell degradation**

ACCELERATED STRESS TESTS (ASTs)

- I. Harsh stressor
- II. Target specific components
- III. Trigger one degradation mechanism on a shorter time scale (500 hours)



Screening for the best electrolyzer component
(e.g. vs. state-of-art component)



Estimate the Durability of the component (needs
"acceleration multiplier")

ACCELERATED LIFE TESTING (ALTs)

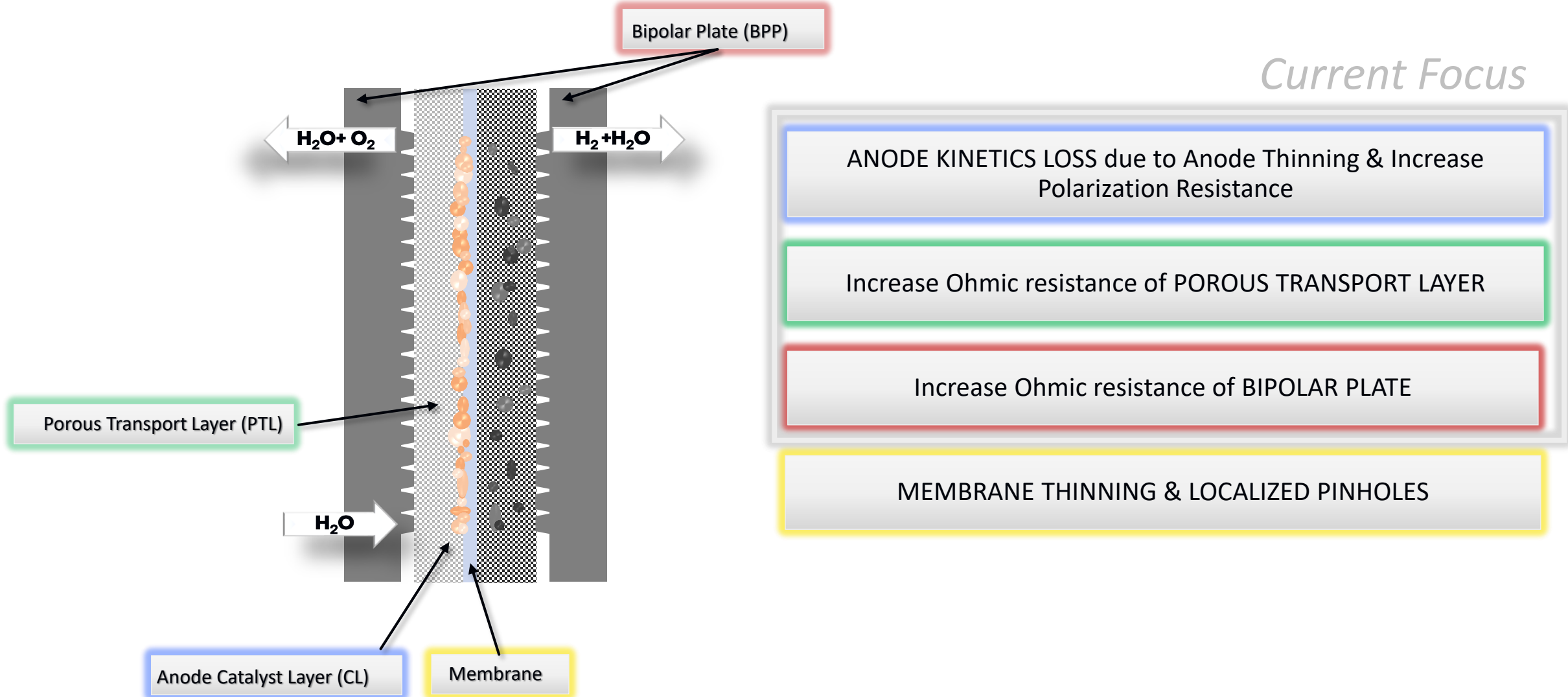
- I. Mild stressors
- II. Generic cell test
- III. Mimic real life conditions for one use case on a longer time scale (> 1000 hours).



Estimate the Durability of the whole cell (needs
"acceleration multiplier")

Current Focus

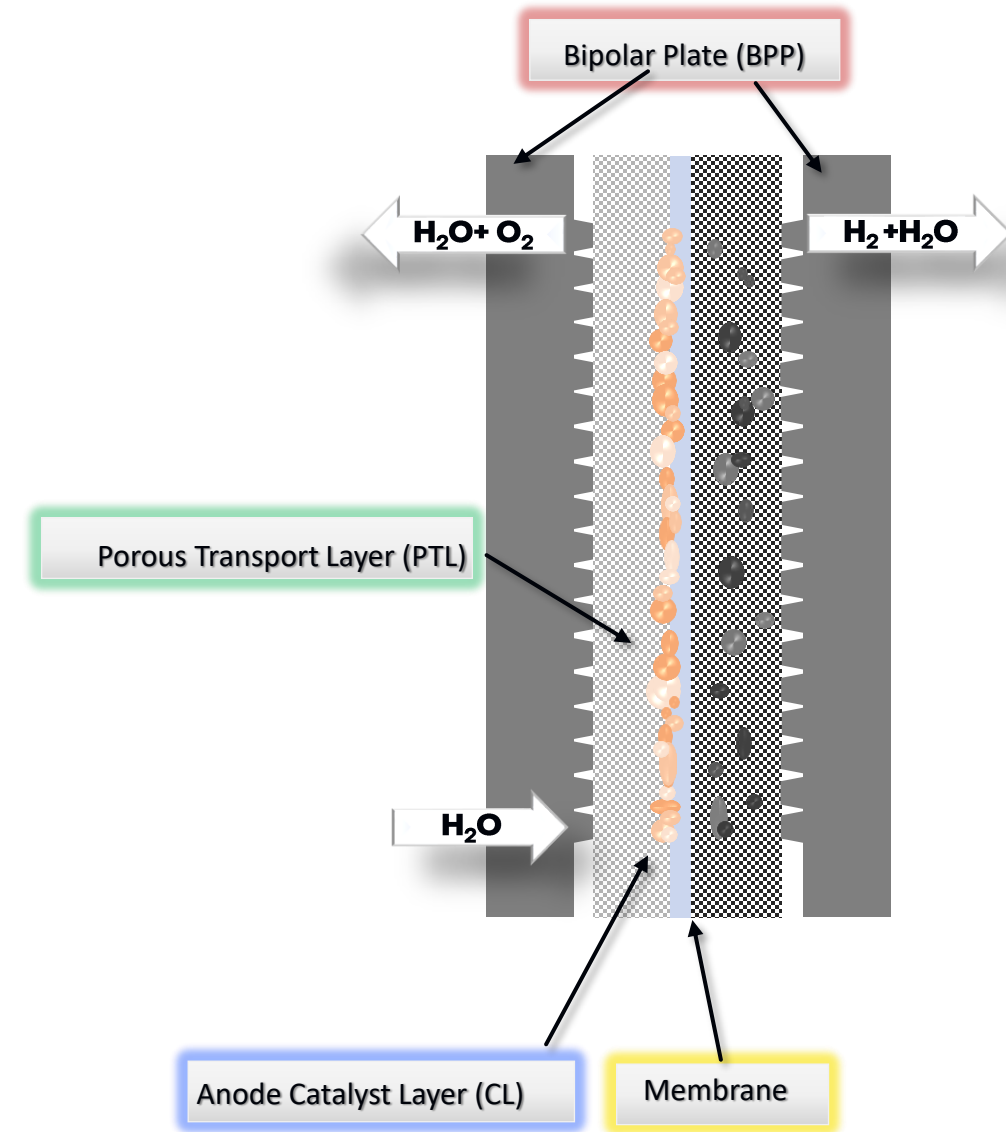
Component-Specific Irreversible Losses



*Cathode kinetics is rapid and low degradation is expected, hence outside the current scope.

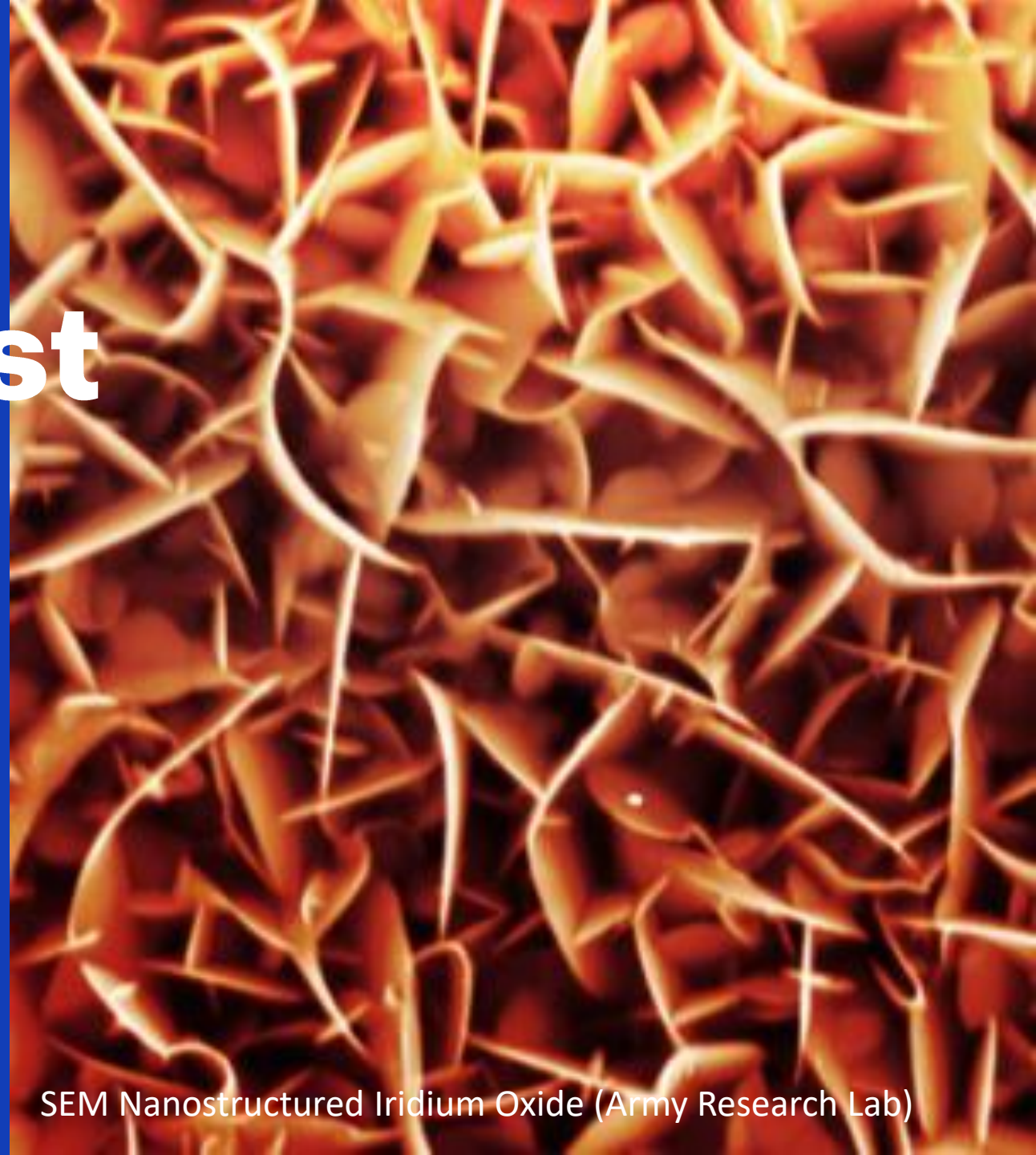
Stressors and Targets

High temperature ($> 60^{\circ}\text{C}$)	Anode CL	PTL	BPP	Membrane
Low current ($\leq 0.3 \text{ A cm}^{-2}$)	Anode CL			Membrane
Dynamic (On/Off)	Anode CL			Membrane
I-V cycling ($1.5\text{V} - 2.2 \text{ V}$)	Anode CL			
High voltage ($> 2.2. \text{ V}$)	Anode CL	PTL	BPP	
Low humidity	Anode CL			Membrane



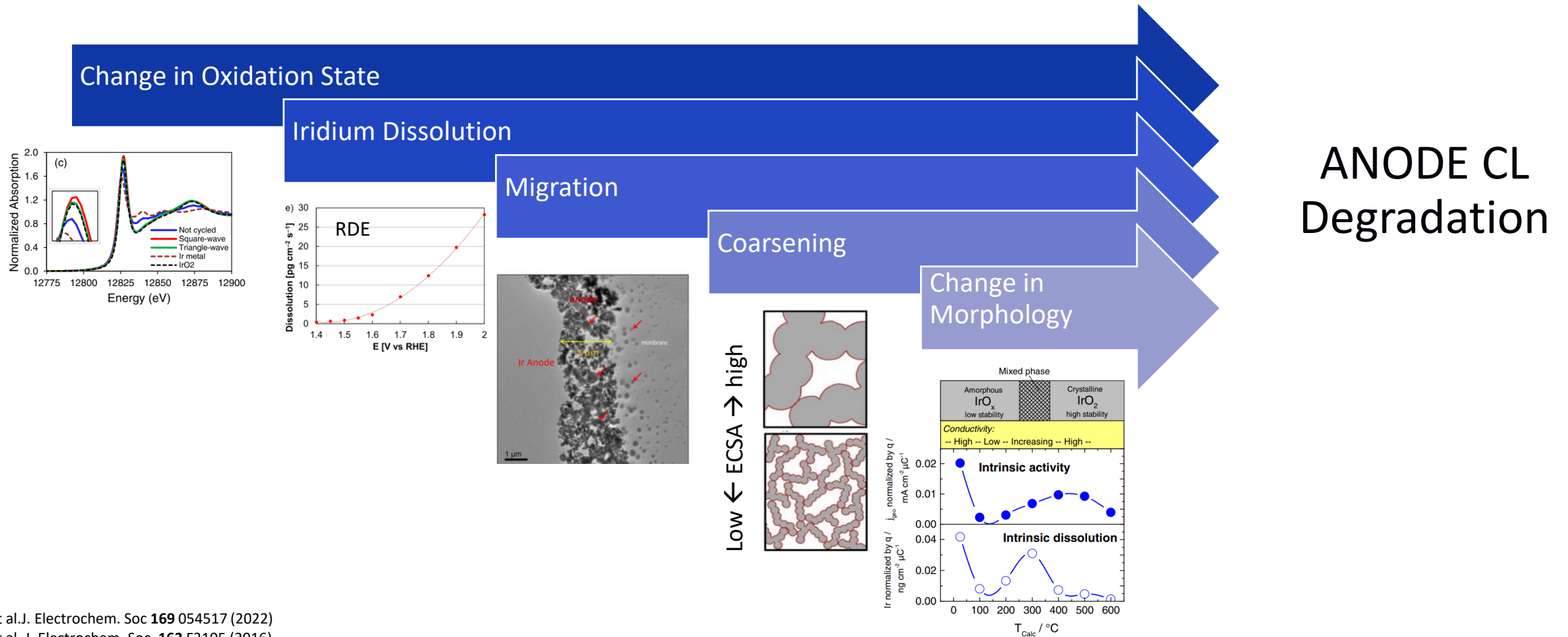
Anode Catalyst Layer

ASTs



SEM Nanostructured Iridium Oxide (Army Research Lab)

AST Protocol Anode CL- Anticipated Effects



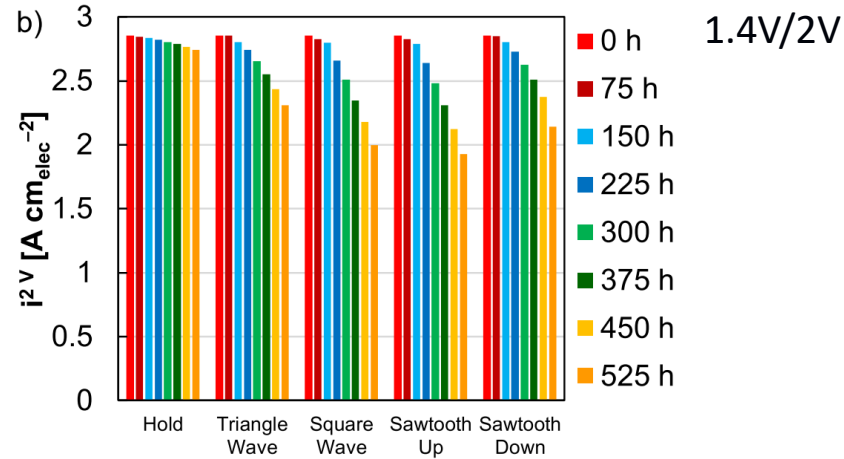
- Alia et al. J. Electrochem. Soc. **169** 054517 (2022)
 Alia et al. J. Electrochem. Soc. **163** F3105 (2016)
 Alia et al. J. Electrochem. Soc. **166** (15) F1164-F1172 (2019)
 Ayers et al. J. Electrochem. Soc. Interface **30** 67 (2021)
 Padgett et al. J. Electrochem. Soc., **170** 084512 (2023)
 Geiger et al. 2016 J. Electrochem. Soc. 163 F3132
 Weiß et al. Journal of The Electrochemical Society, **166** (8) F487 (2019)

- I. Harsh stressor
- II. Target-specific components
- III. Trigger one degradation mechanism on a shorter time scale (500 hours)

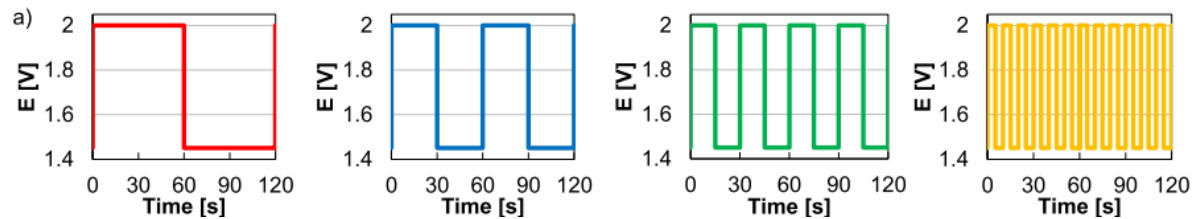
NREL

Load cycling

Stressor waveforms



Cycle time



Technische Universität München

Load cycling

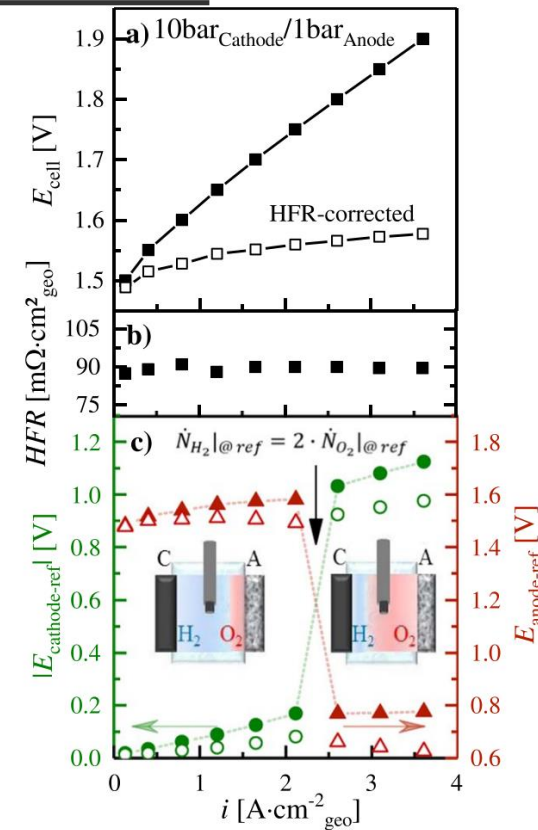
START & STOP

3.0 Acm⁻²

10min

0.1 Acm⁻²

OCV

Weiß et al. Journal of The Electrochemical Society, **166** (8) F487 (2019)Weiß et al. Journal of The Electrochemical Society, **168** 114511 (2021)Alia et al. J. Electrochem. Soc., **166** (15) F1164-F1172 (2019)

TNO's AST Protocol: Anode CL

- Anode CL: 0.8 mg/cm². Needs to be optimized to lower loading.
- Cathode CL: 0.5 mg/cm² Pt
- PTL and BPP: Pt-coated Titanium.
- Membrane: Nafion115 , at T < 80°C, and MilliQ water.

APPROACH

- Evaluate effect of load cycling
- Shutdown behaviour

Anode CL

EX-SITU measurements

Pristine Witness sample
SEM,TEM,
XPS/XANES,
XRF
Conductivity

CONDITIONING

1. Heat to 40°C for 0.5 h
2. Heat to 80°C for 5 h
3. Apply 2 V for 24 h
4. Record an *I-V* curve (80°C)
5. Cool down to T_x for 0.5 h

80°C,
2 barg

IN-SITU measurements

I-V: 1.55 – 2.275 V
PEIS: 1.55 V, 1.65V, 25-
0.5 kHz

60°C,
2 barg (BoL)

STRESSOR

I-V Square waves
1.4 ⇔ 2.275 V
t_{step} = 60 s
5000 cycles (166 h)



x6 (1000 hours)

60°C, 2 barg

IN-SITU measurements

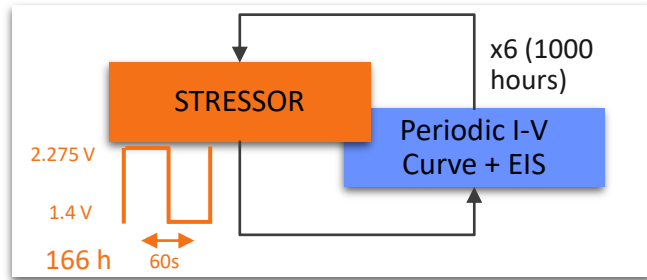
I-V: 1.55 – 2.275 V
PEIS: 1.55 V, 1.65V, 100-
0.5 kHz
ICP-MS, ICP-OES

60°C,
2 barg (EoL)

EX-SITU measurements

Aged sample
SEM,TEM,
XPS/XANES,
XRF
Conductivity

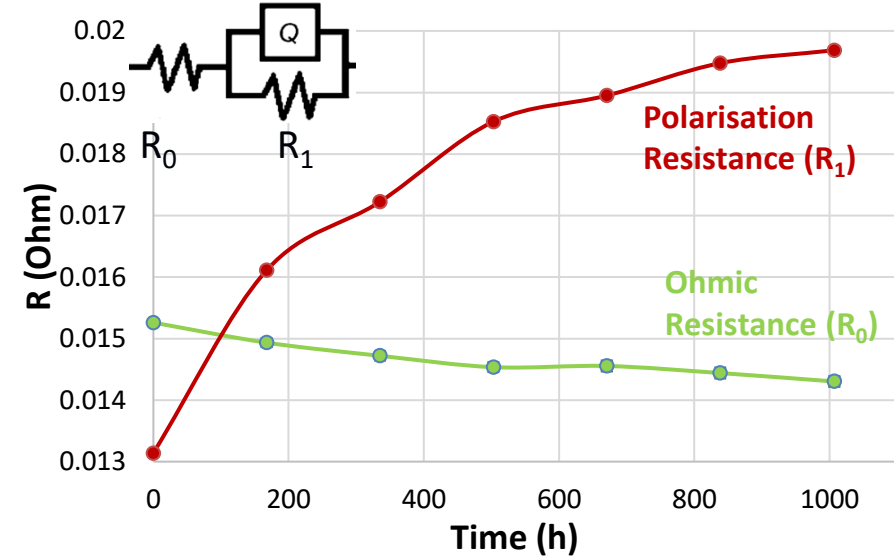
Results 1000h AST- Anode CL



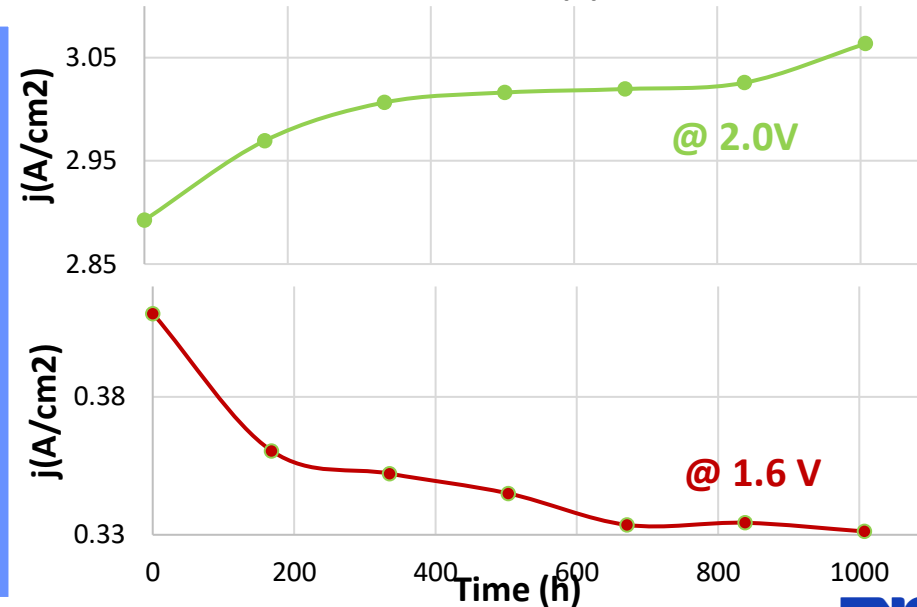
Over time, we measured two opposite trends:

- **Polarization resistance** increases (Current decreases) → kinetic activity loss
- **Ohmic resistance** decreases (Current increases) → membrane thinning/creep or PTL/CL interface conductivity

EIS @ 1.55 V



I-V Curves



Conclusions & Future Perspective

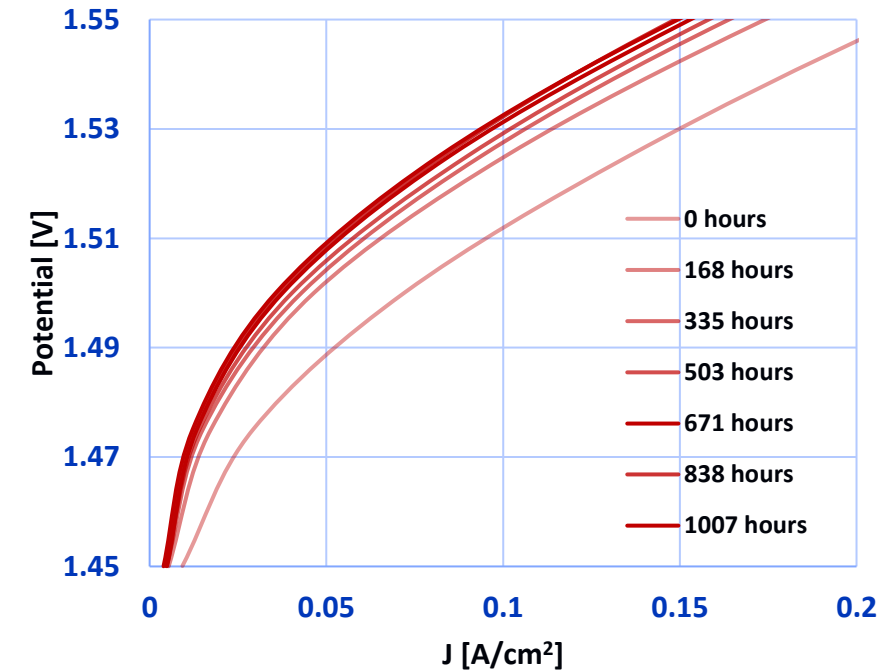
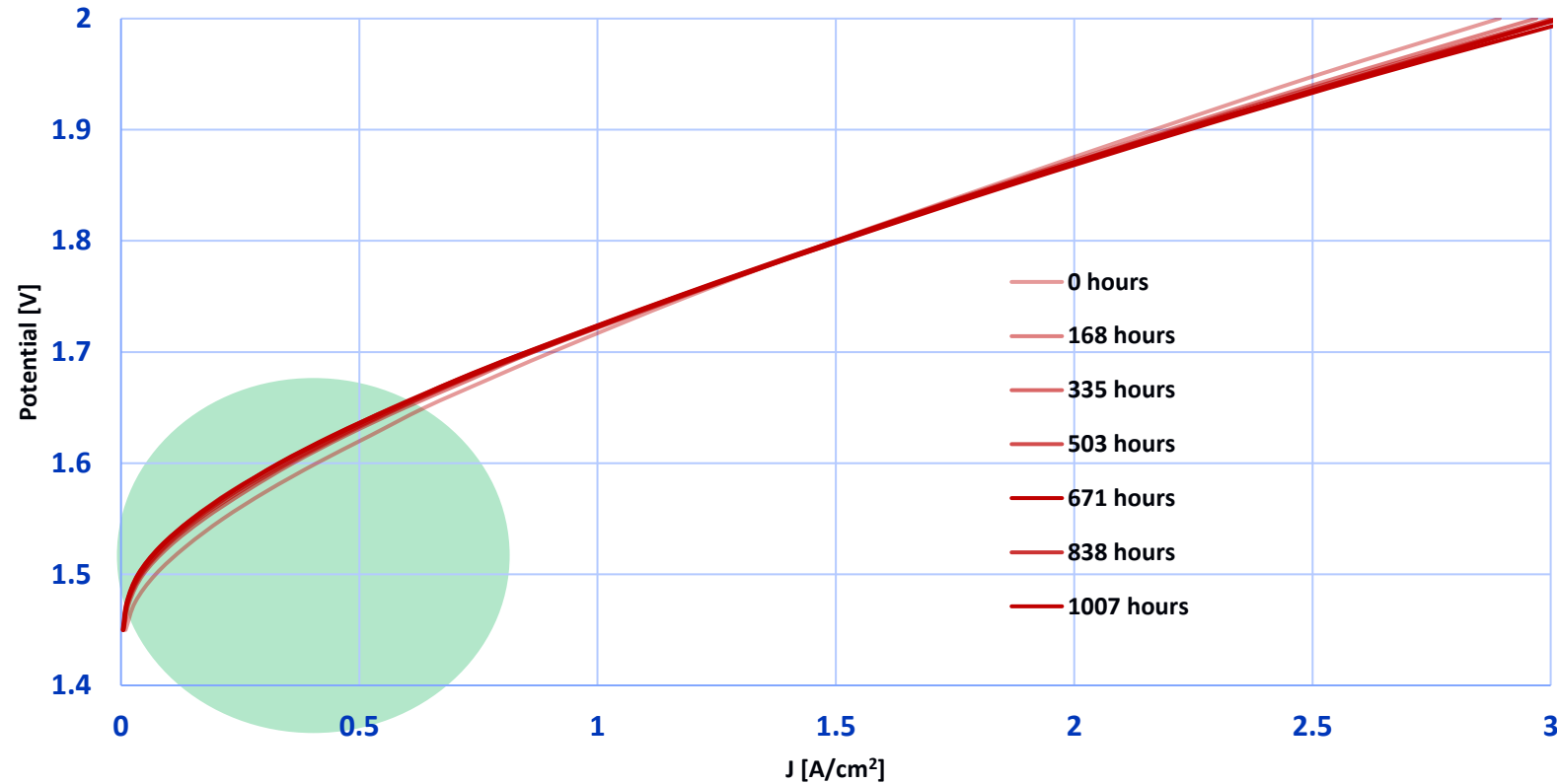
- We have developed component-specific ASTs to steer component R&D
- Efficient anode catalyst utilization (lower loading) is important for scale up.
- Catalyst durability will become an increasingly important topic for lower catalyst loadings.
- TNO's AST for the Catalyst Layer with Voltage cycling effectively degrades the anode CL

Next steps

- In-situ measurements will be complemented by ex-situ characterisations.
- Cycling frequency of stressor waveform.
- Further reduction in catalyst loading.
- Study the effect of shutdown behavior.



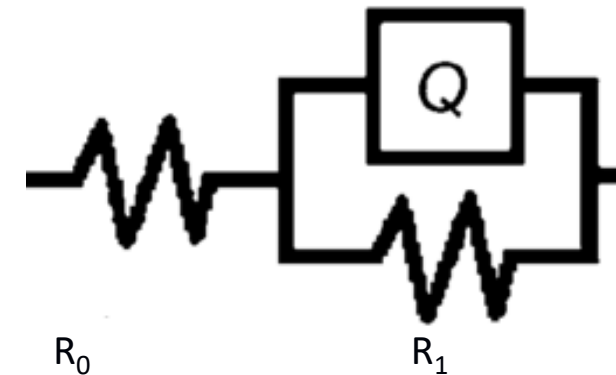
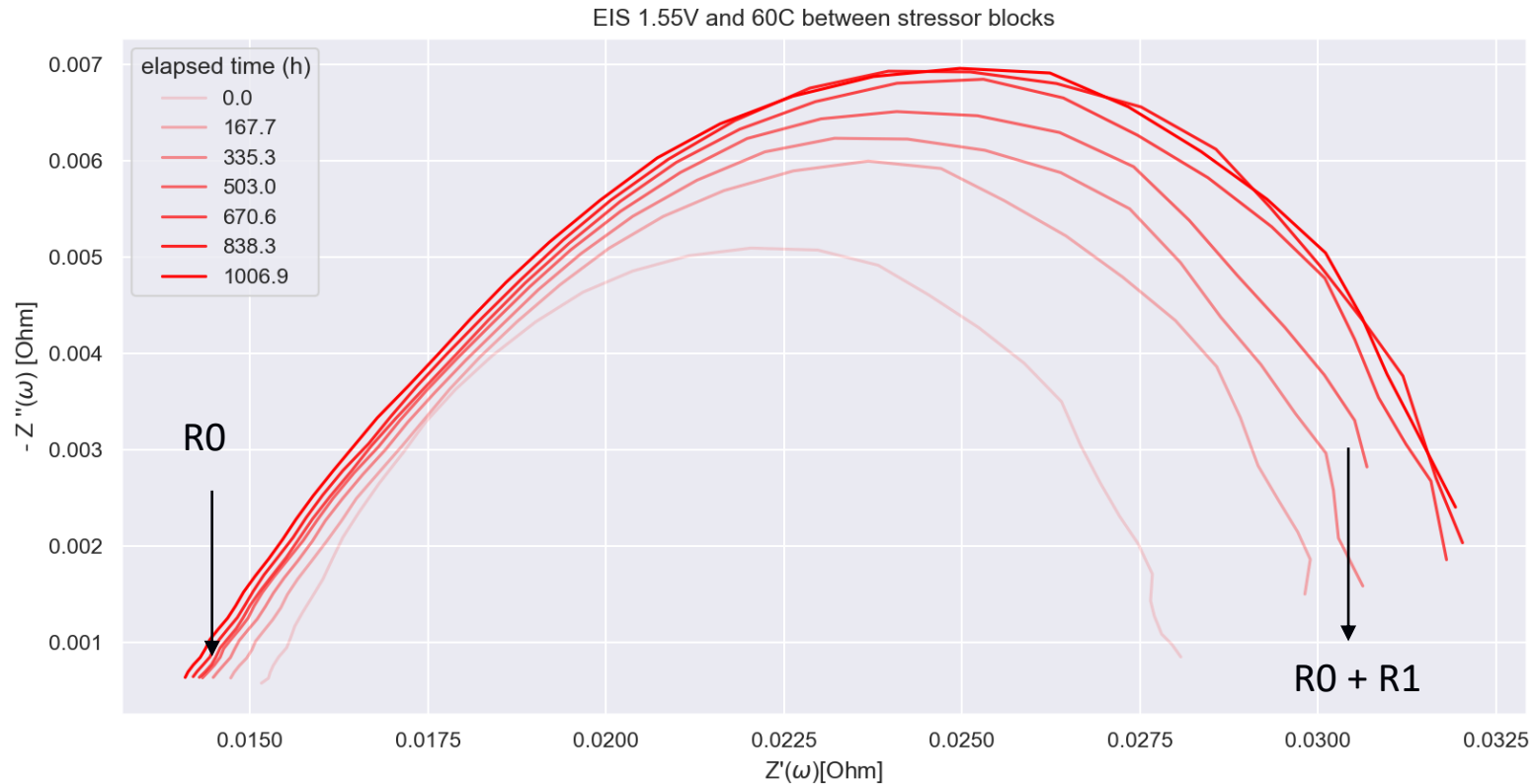
Results 1000h AST – Polarisation Curves



Two opposite trends are observed for the measured current:

1. Repeated cycling increases kinetic overpotential
2. Ohmic losses decrease over time

Results 1000h AST – Impedance Spectra



Two opposite trends are observed for the measured resistance:

- Polarization resistance increases \rightarrow kinetic loss.
- Ohmic resistance decreases \rightarrow membrane thinning/creep or PTL/CL interface conductivity