Workshop / 11. September 2023 – 12. September 2023

International Hybrid Workshop "Fuel Cell MEA Characterization"

PERFORMANCE AND DURABILITY CHARACTERISATION OF MEA‘S

Dr. Pawel Gazdzicki, Dr. Jens Mitzel (DLR)

DLR

DLR
German Aerospace Center

- Research Institution
- Space Agency
- Project Management Agency

-Research Areas

- Aeronautics
- Space
- Transport
- Energy
- Space Agency
- Project Management Agency

Locations and employees

Approx. 10 000 employees across
55 institutes and facilities at
30 sites.

Offices in Brussels, Paris,

Tokyo and Washington.

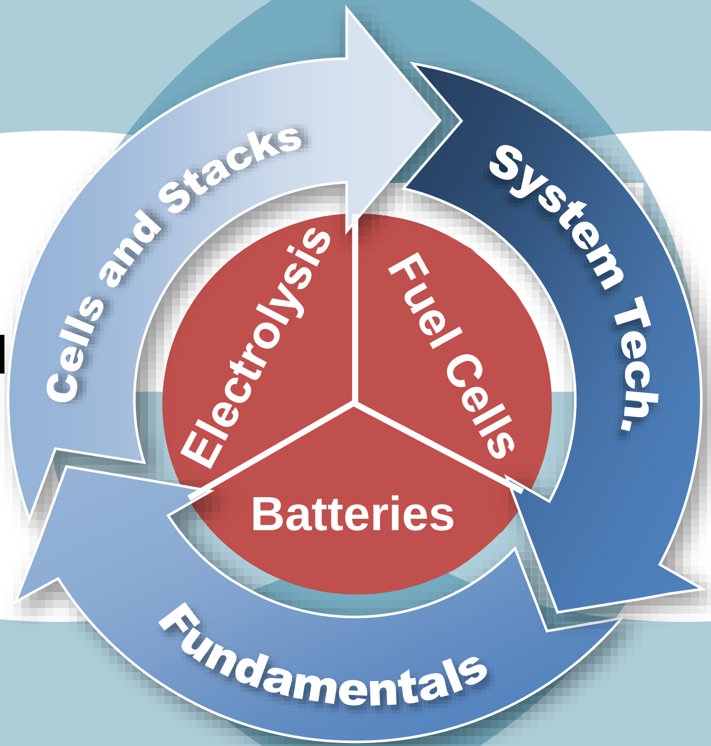
DLR – Institute of Engineering Thermodynamics

- Sites in Stuttgart, Oldenburg, Ulm, Hamburg, Köln (~200 staff)
- ~20 Mio. EUR annual budget with 50% third party funding
- Focus on energy storage and energy conversion based on renewable energies
- Director: Prof. André Thess

Electrochemical Energy Technology @ DLR

MODELLING

- Multiscale
- Nano-, Micro- and Mesostructure
- Prediction and Design
- Digital Twin



EXPERIMENT

- Operando Diagnostics
- Quality Control Diagnostics
- Validation
- Performance
- Breakthrough Innovations

Applications

Energy

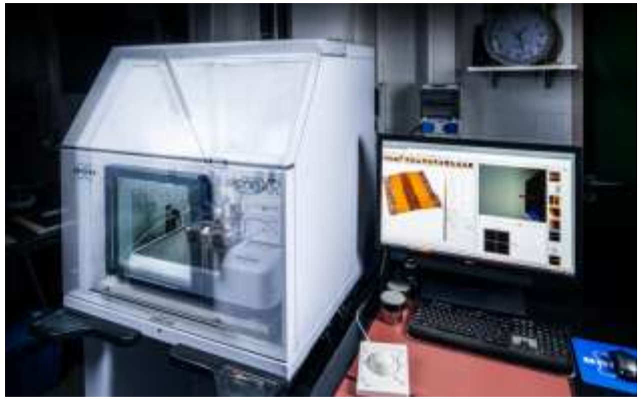
Transport

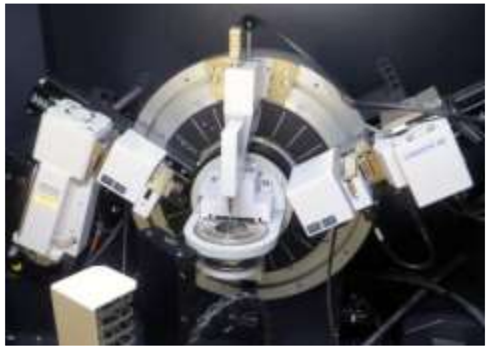
Aeronautics

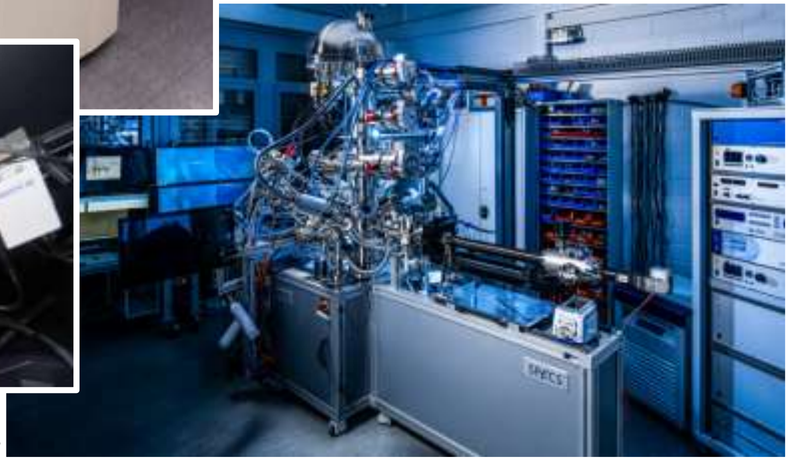
Space

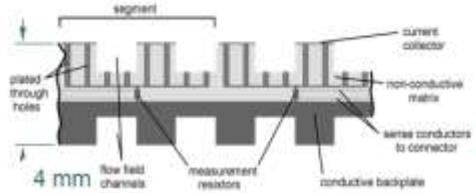
Three Departments: ECE, CEC, ESI

Infrastructure used for PEMFC research





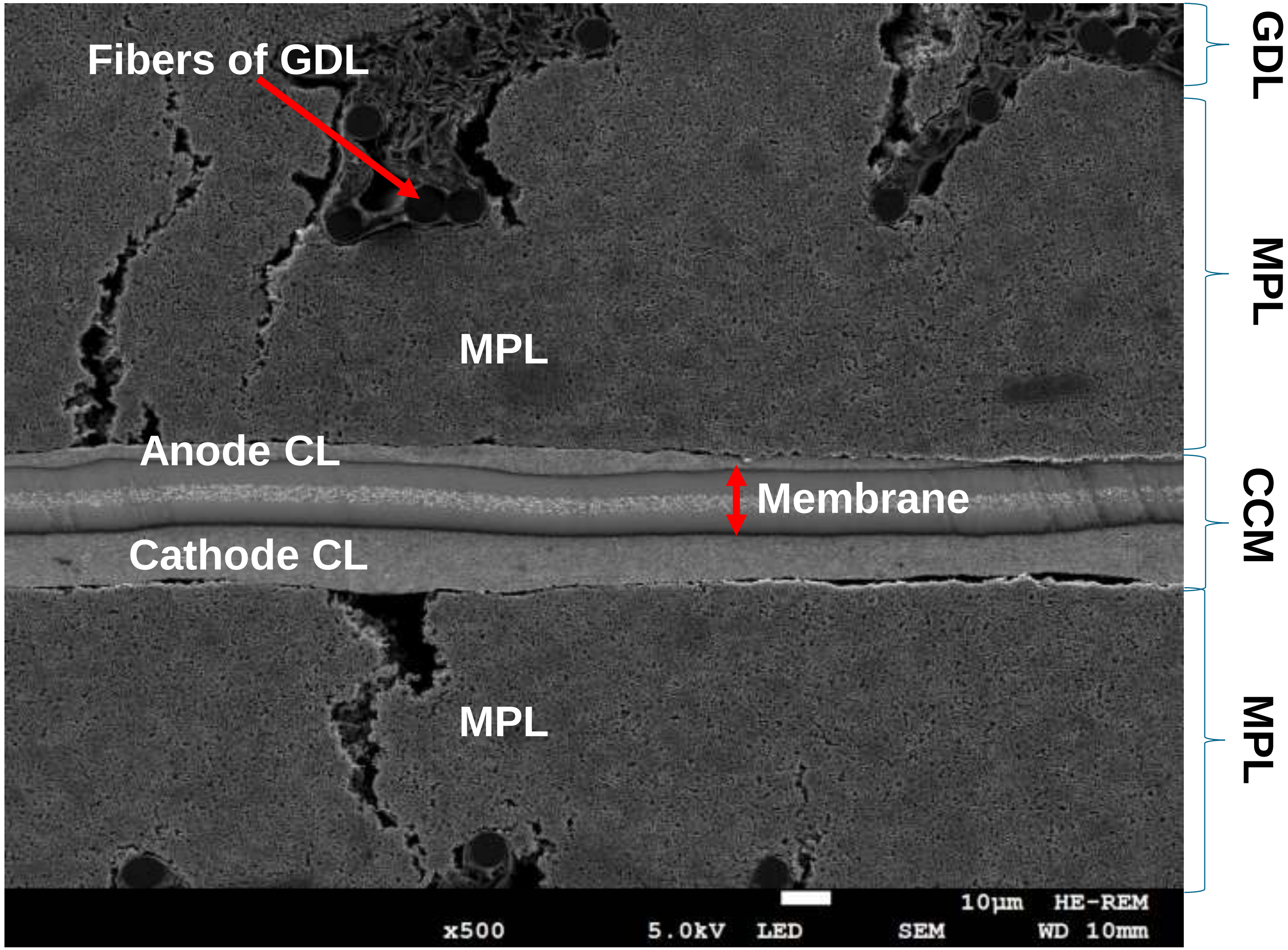


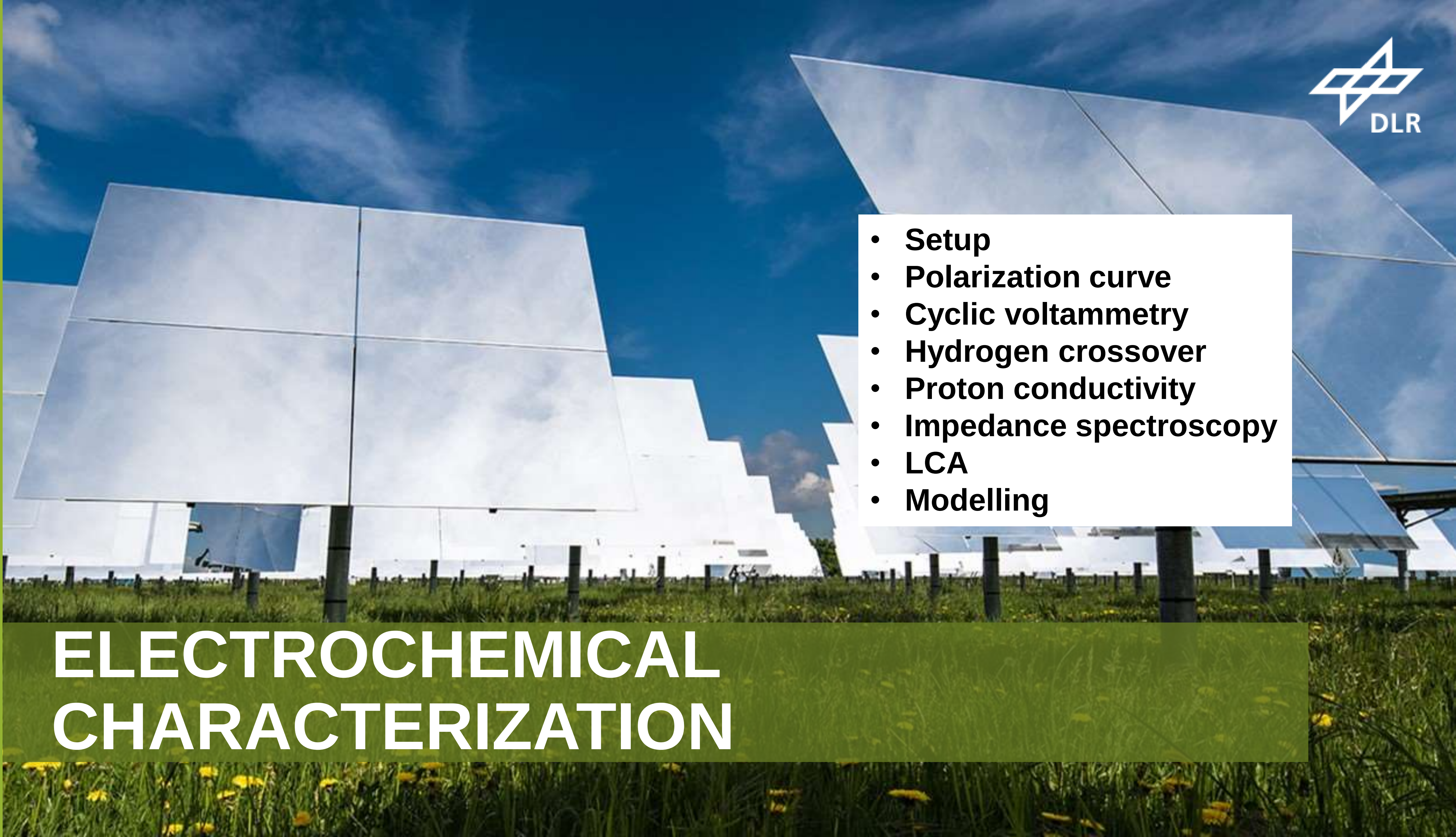


DLR's Segmented Cell as standard tool for monitoring current density and temperature distribution

Common analyses of MEAs

- **Operando and in-situ investigations (EIS, CV, LSV, LCA, …)**
- Dimensional investigations of cross sections (SEM or AFM)
- Compositional investigations (EDX, XPS, XRD …)
- 3D-structure investigations (CT, FIB-SEM)





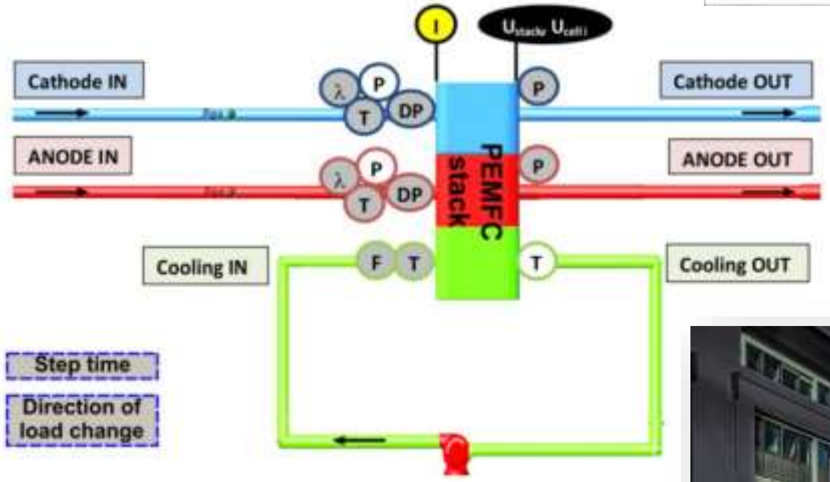
ELECTROCHEMICAL CHARACTERIZATION

- Setup
- Polarization curve
- Cyclic voltammetry
- Hydrogen crossover
- Proton conductivity
- Impedance spectroscopy
- LCA
- Modelling

Experimental Setup

Important for reliable FC operation:
- Test station with good control of operating conditions
  - ➢ Temperature
  - ➢ Pressure
  - ➢ Humidity
  - ➢ Gas flow
- Clear definition of all parameter sensors
- Beneficial:
  - ➢ Humidity sensors
  - ➢ Integrated HFR measurement

Important for reliable EC testing:
- High quality potentiostat
  - ➢ Current/voltage range to be adapted to cell/stack
- H_2/N_2 atmosphere on counter and working electrode WITHOUT O_2 traces
  - ➢ Be aware of O_2 traces in water for humidification
- Electrical noise: Inductivity of setup for EIS measurement
  - ➢ Twisted cables
  - ➢ Separate current and sense cables



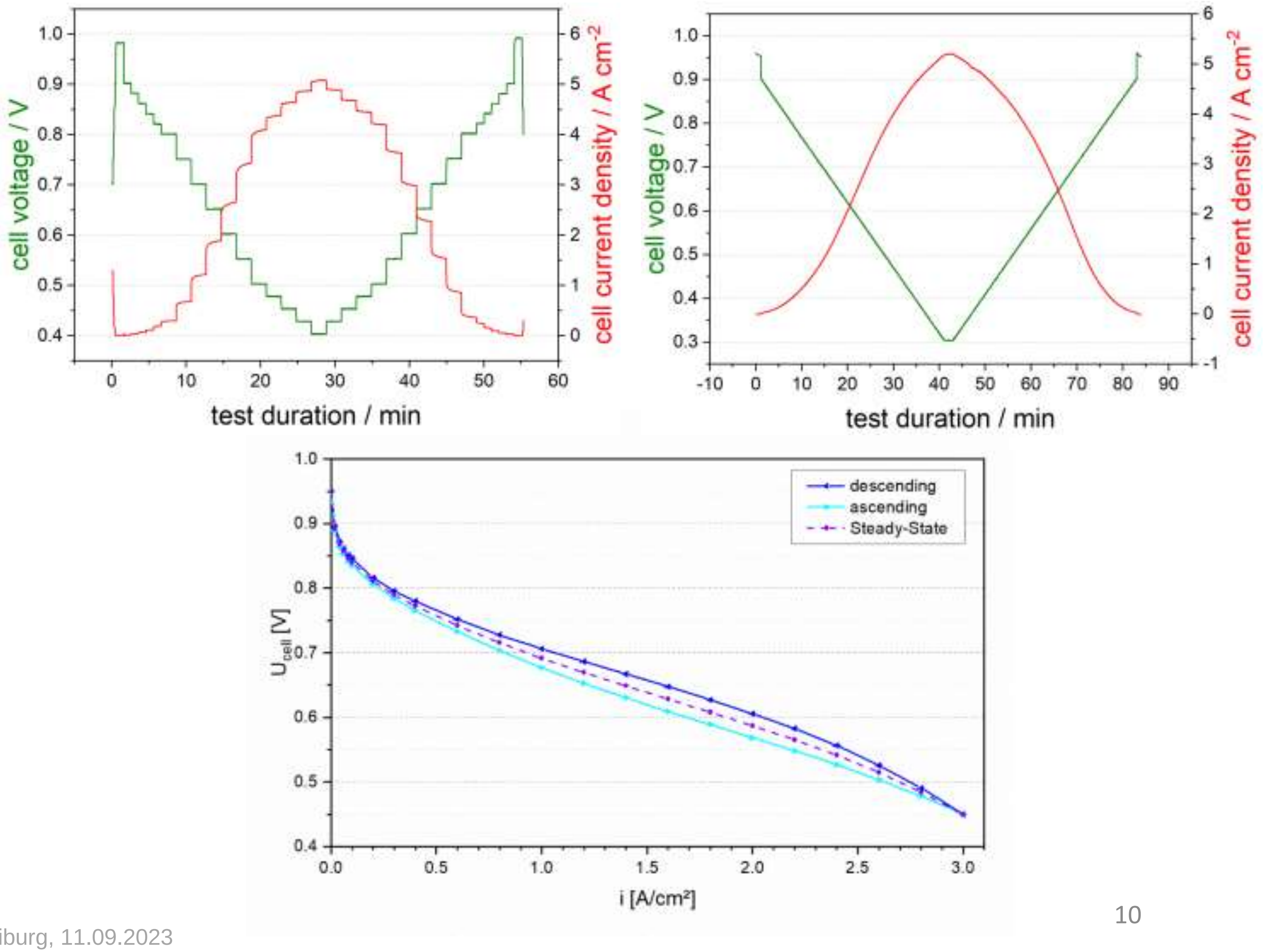
Polarization Curve

Standard method to determine:
- **MEA performance**
- Information about fuel cell performance in operating conditions for different applications

Different possibilities to measure:
- Staircase or dynamic ramp
- Galvanostatic or potentiostatic

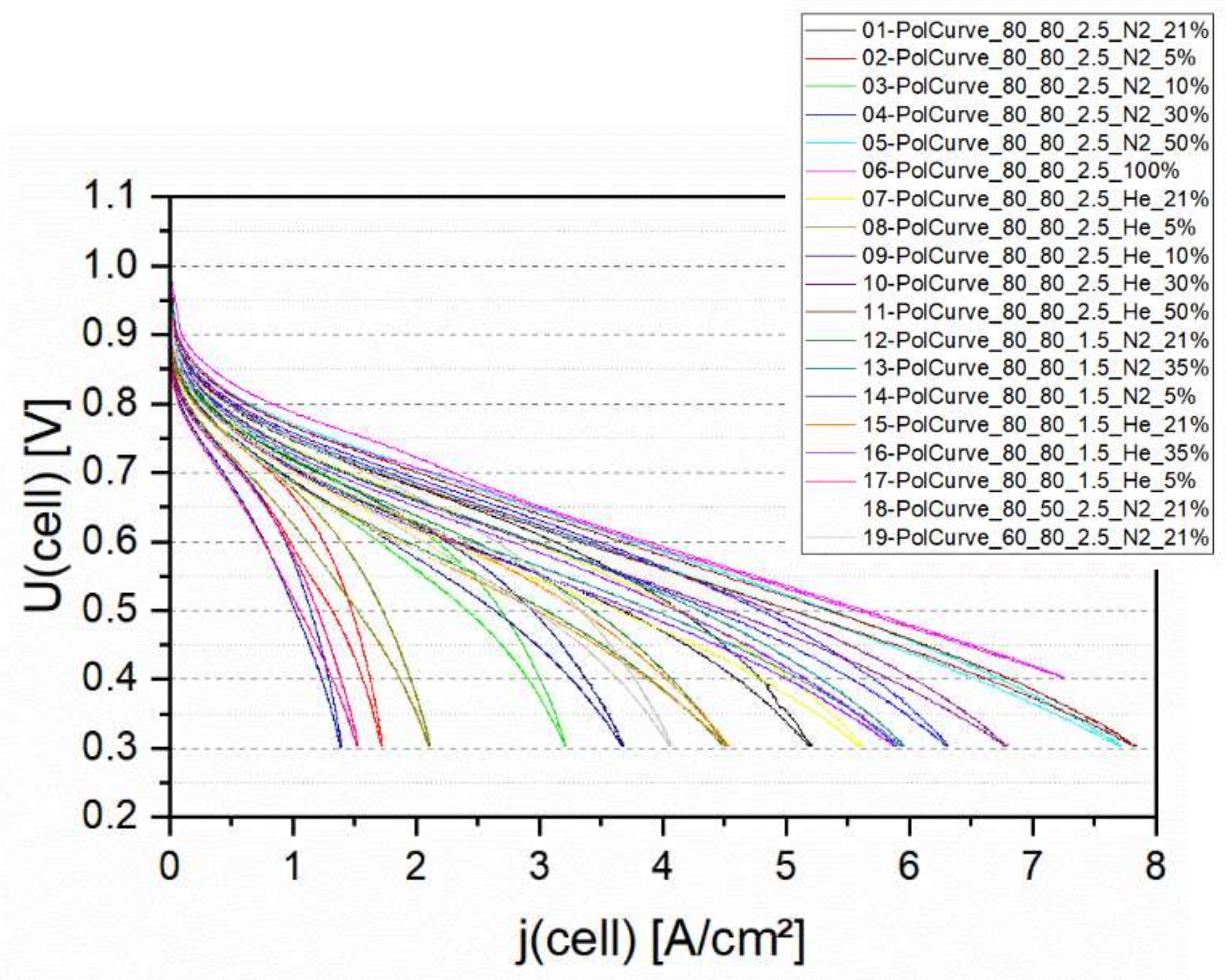
Clear definition of test protocol:
- Conditions
- Current/voltage steps
- Dwell time and stability criteria
- Analysis time

Hysteresis between measurement with increasing or decreasing current due to
- Water management
- Pt surface oxidation state



Polarization Curve

Parameter studies
- Parameter studies can provide information about performance using different operating conditions
- Parameter variation can improve validation of simulation models
  - ➢ Temperature
  - ➢ Pressure
  - ➢ Relative humidity
  - ➢ Oxygen concentration
  - ➢ Carrier gas (N2, He)

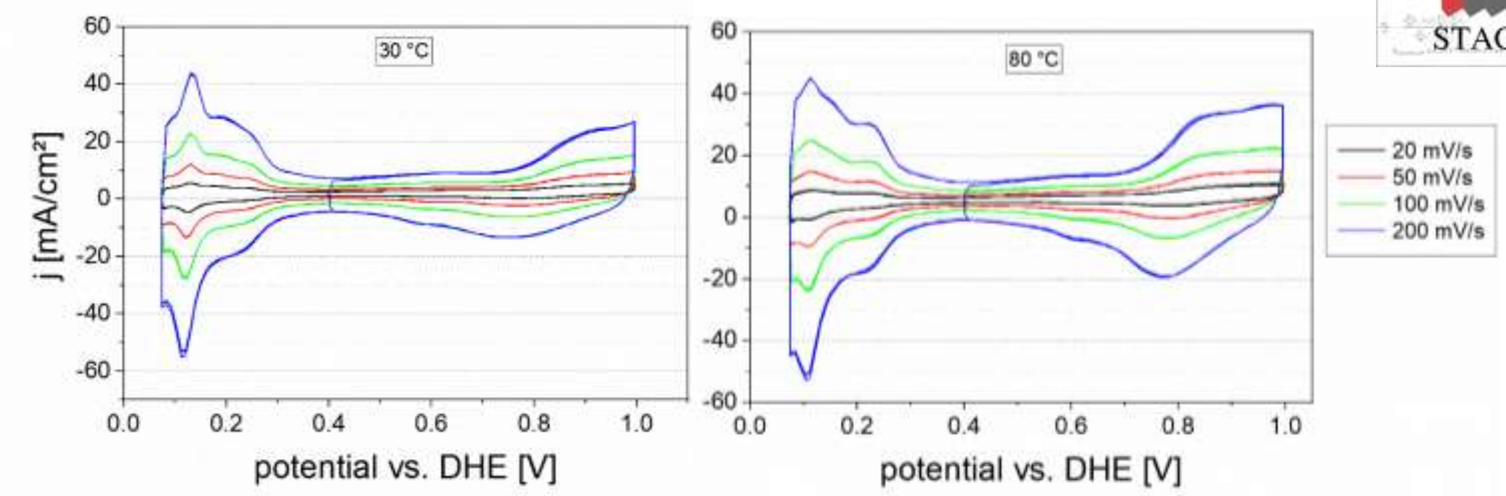


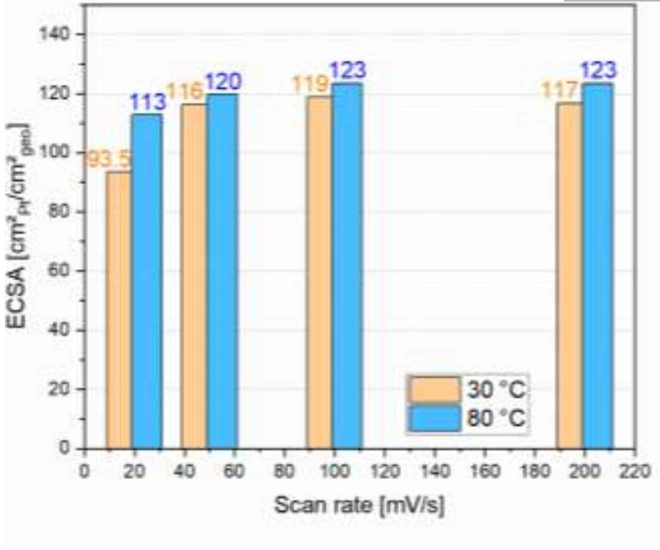
Cyclic Voltammetry (CV)

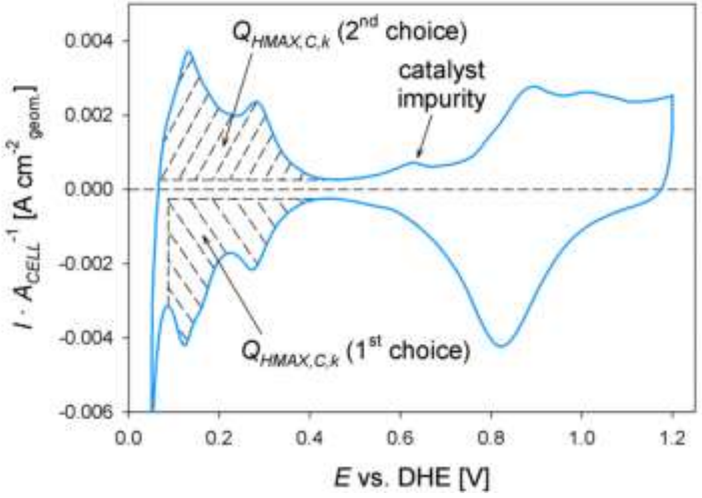
Standard method to determine:
- **Catalyst** properties
- ElectroChemically active Surface Area (ECSA)
- Contaminants

Important for reliable validation:
- Proper choice of vertex potentials depend on conditions
- Proper choice of scan speed (e.g. 100 mV/s)

Important for reliable analysis:
- Correct integration of hydrogen adsorption/desorption peak
- Gaseous H_2 evolution can result in over-estimation of ECSA







Cyclic Voltammetry (CV)

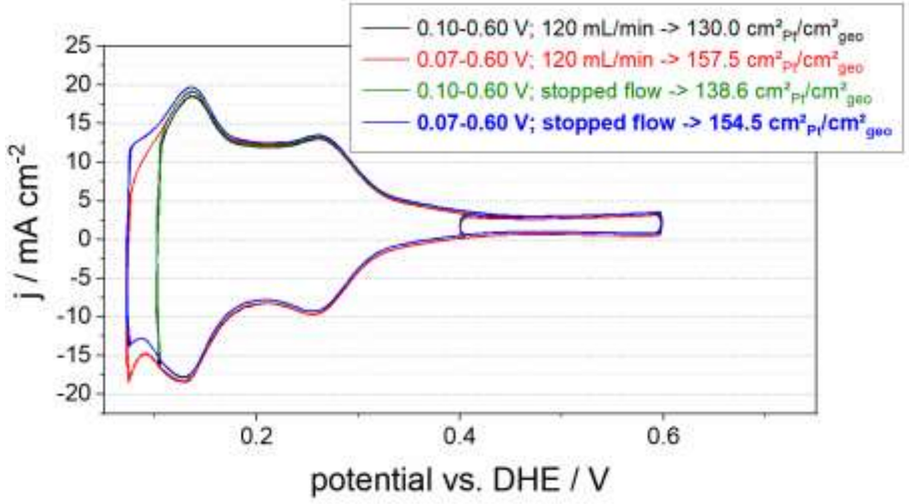
„Standard temperature conditions“

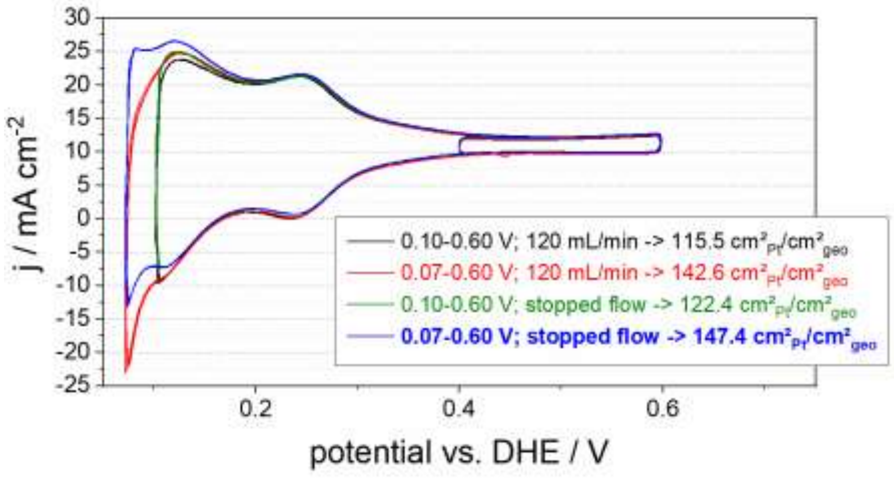
	Conditions
Cell temperature [°C]	30
Gas composition	H_2/N2
Pressure anode / cathode [bar_{abs}]	atm/atm
Gas inlet temperature anode/cathode [°C]	35/35
RH anode / cathode [%]	100/100
Gas flow H2/N2 [mL/(min*cm²)]	10/10

Important for reliable validation:
- Correct choice of vertex potentials:
  - ➢ High potential: loss of catalyst elements due to oxidation and dissolution (PtCo)
  - ➢ Low potential: gaseous H_2 evolution and over-estimation of ECSA
- Low gas flow or even stopped flow during CV -> surface sensitive method
- Choice of conditions: standard or application

"Application conditions (HD)"

	Conditions air inlet
Cell temperature [°C]	90
Gas composition	H_2/N2
Pressure anode / cathode [bar_{abs}]	2.5/2.5
Gas inlet temperature anode/cathode [°C]	95/95
RH anode / cathode [%]	100/100
Gas flow H2/N2 [mL/(min*cm²)]	10/10





The project is supported by the Clean Hydrogen Partnership and its members Hydrogen Europe and Hydrogen Europe Research.

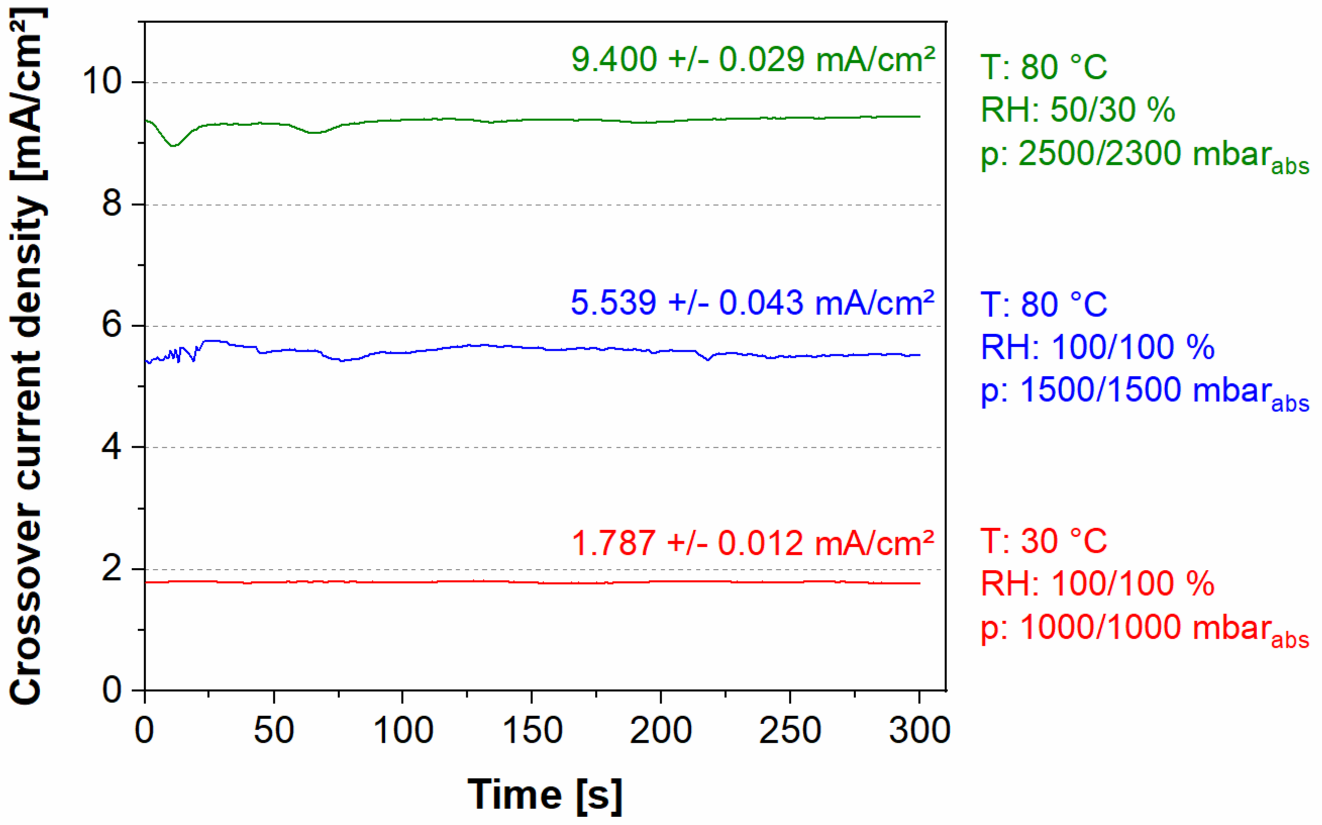
Hydrogen Crossover

Standard method to determine:

- **Membrane** properties
- Hydrogen crossover rate through membrane

Important for reliable validation:

- Measurement at 0.4 V
  - Free Pt surface for complete oxidation of H_2
- Sufficient time for conditioning and stabilization
  - Strongly depends on membrane state (water uptake)
- Hydrogen partial pressure difference is the driving force
  - Proper control of H_2 pressure on counter electrode
  - Be aware of water partial pressure for humidification

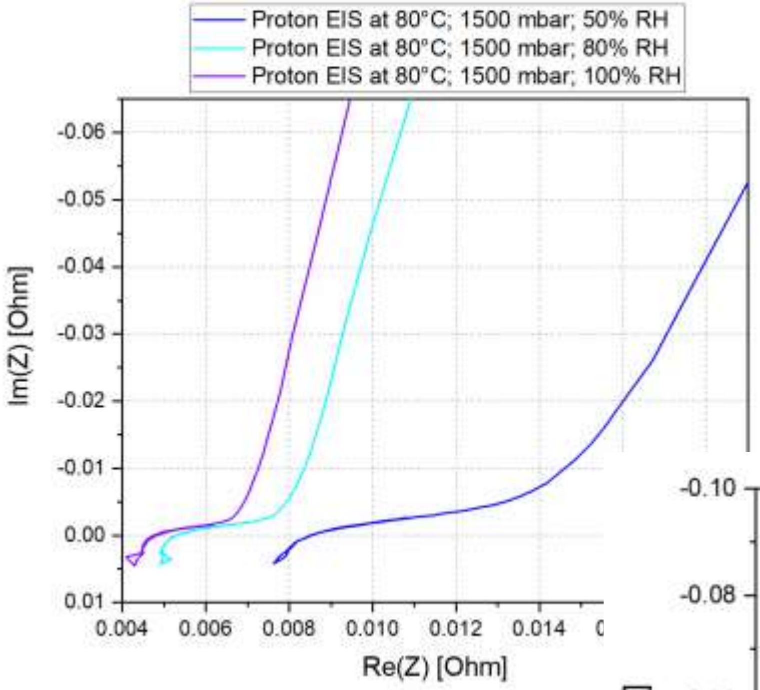


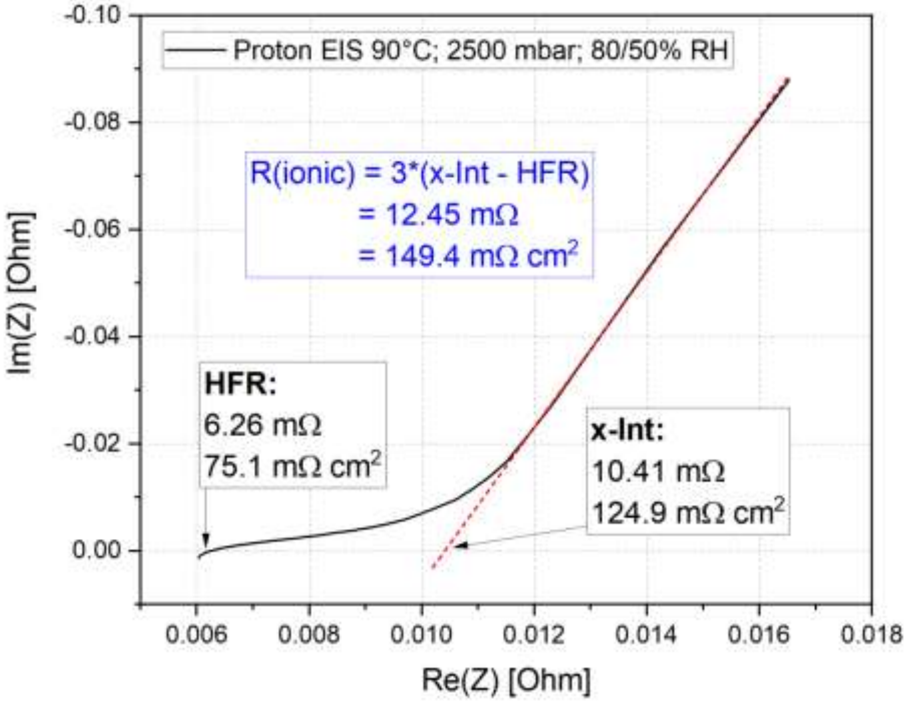
Proton conductivity

Standard method to determine:
- **Catalyst layer** properties
- Effective proton transfer resistance in catalyst layer

Important for reliable validation:
- Measurement at UDC = 0.4 V
- Typical amplitude: 5 mV
  - ➢ Check linearity
- Typical frequency: 50 kHz – 10 Hz
- Sufficient time for conditioning and stabilization
  - ➢ Strongly depends on catalyst layer humidity

Important for reliable analysis:
- Data evaluation by:
  - ➢ Transmission line model by Pickup et al.[1]
  - ➢ EIS fitting (equivalent circuits)



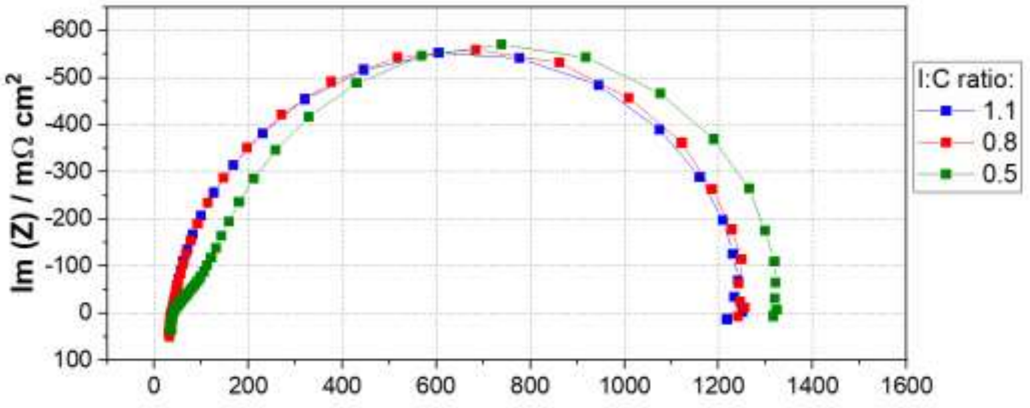


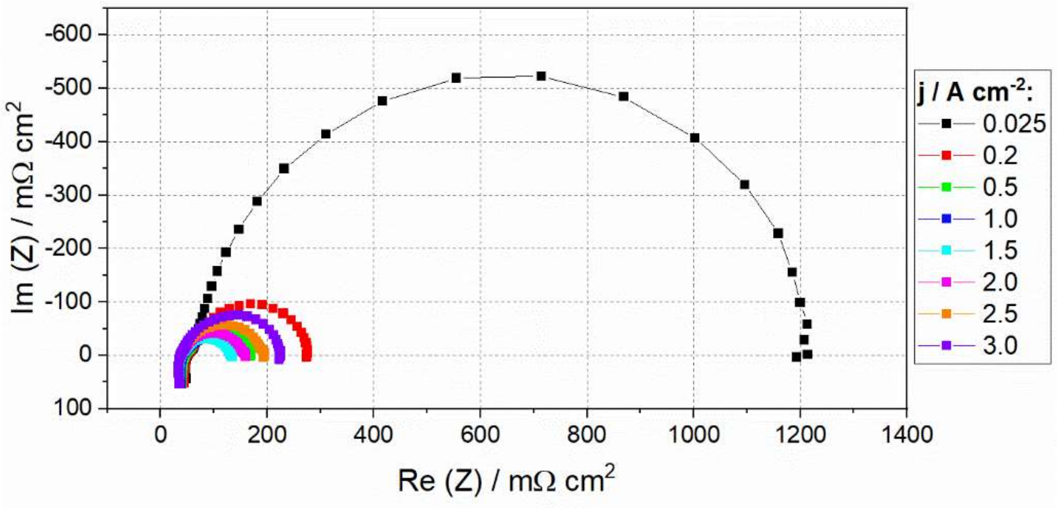
Ref [1]:
https://doi.org/10.1149/1.1390804
https://doi.org/10.1149/1.1611493

Electrochemical impedance spectroscopy

Standard method to determine:

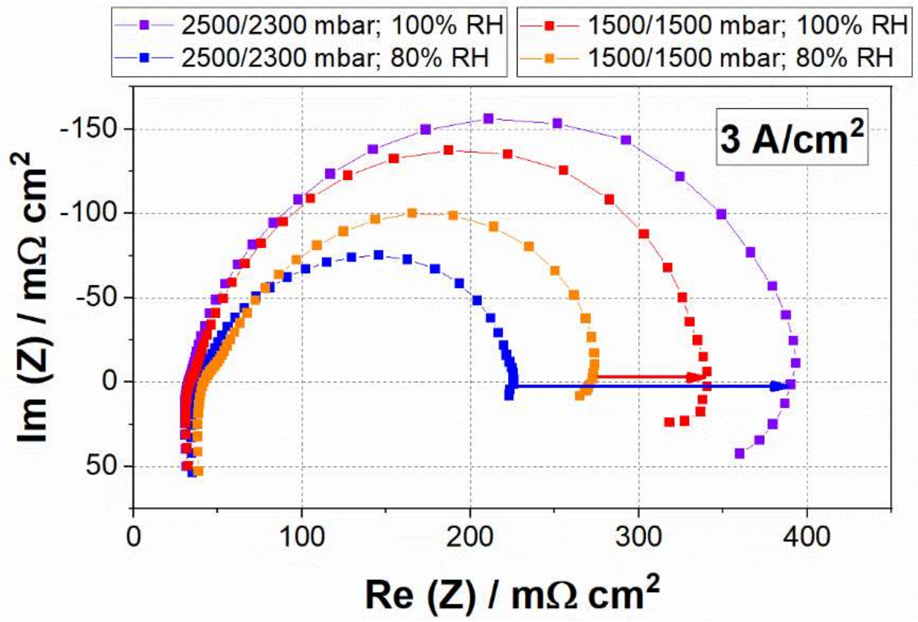
- **MEA properties under operating conditions**
- Information about:
  - Ohmic resistance of cell
    - Dominated by membrane
    - Can be impacted by contact resistance
  - Charge transfer resistance of hydrogen oxidation (HOR) and **oxygen reduction reaction (ORR)**
  - Transport resistances: oxygen and protons

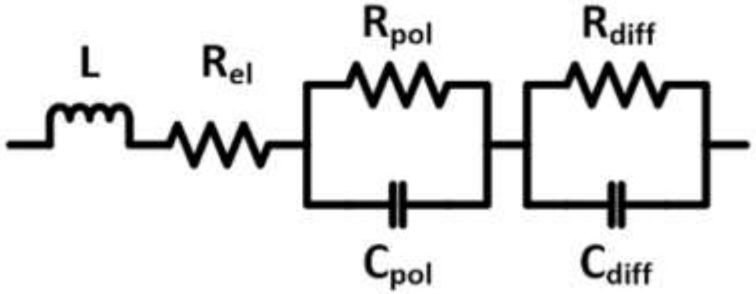




Electrochemical impedance spectroscopy

Important for reliable validation:
- AC amplitude in the range of 5-10 % of DC current
  - ➢ Linearity check!
- Avoid harmonic oscillations
- Minimize impact of test bench and electric noise
- Proper control of operating conditions
- Data analysis is very important
  - ➢ EIS fitting using equivalent circuits
  - ➢ **Avoid over-interpretation EIS!**

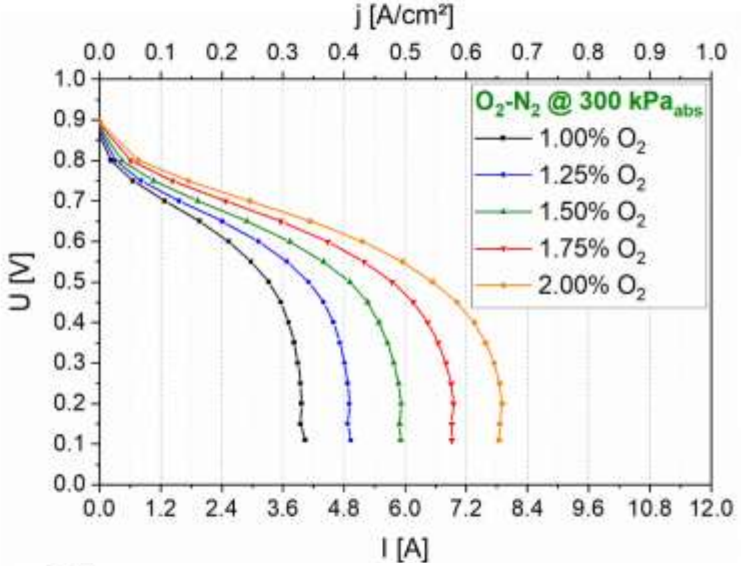


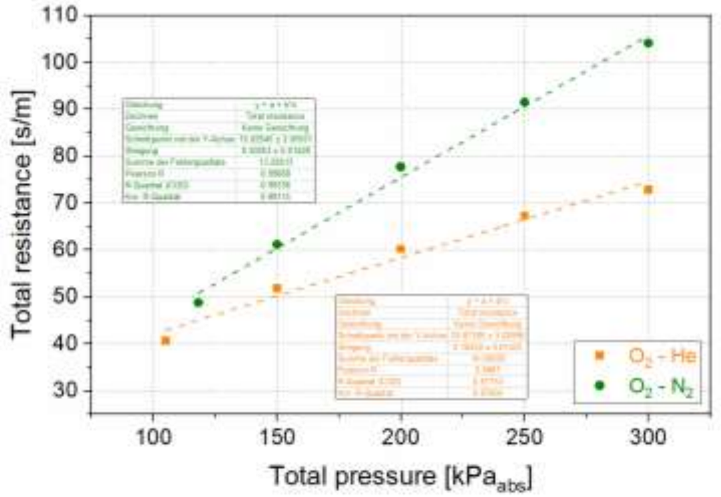


Limiting Current Analysis (LCA)

Method to determine:
- **Oxygen transport properties**
- Cathode electrode properties
- Variation of:
  - ➢ pressure
  - ➢ oxygen concentration (low concentration to consume all O2 at catalyst surface)

Analysis of O2 transport mechanisms:
- Pressure independent transport resistance:
  - ➢ Knudsen diffusion in small pores
  - ➢ Diffusion through water and ionomer films
  - ➢ Mainly in CL/MPL
- Pressure dependent transport resistance
  - ➢ Molecular diffusion in larger pores
  - ➢ Mainly in GDL



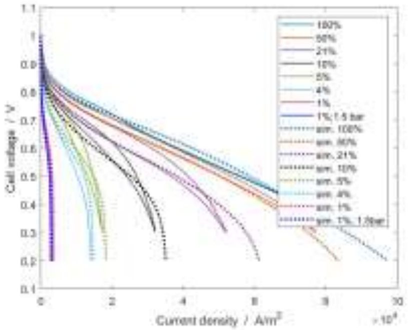


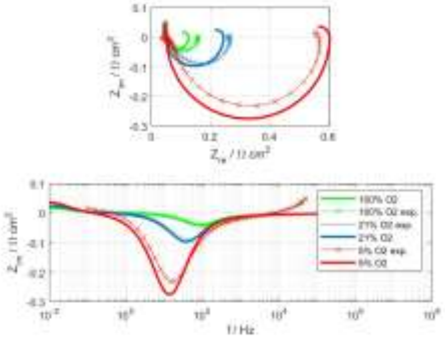
Physical modeling for interpretation of electrochemical characterization (NEOPARD-X)

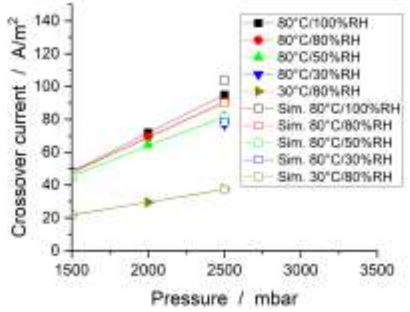
Approach:
- **Description of relevant processes (transport, electrochemistry) in form of a set of partial differential equations**
- Transient simulation of different electrochemical tests (polarization curves, EIS, LCA, …) with a single physical model

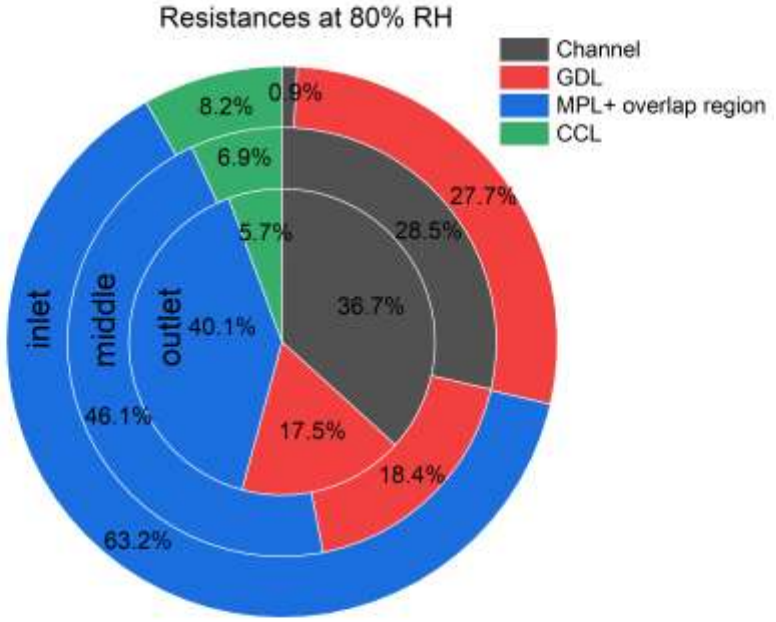
Results:
- Provides physically consistent interpretation of measurement results
- Gives insights on experimentally inaccessible properties (e.g. potential or species distributions within the MEA)
- Allows quantification of individual contributions to performance losses

Contact: Dr. T. Jahnke (DLR-TT/CEC)

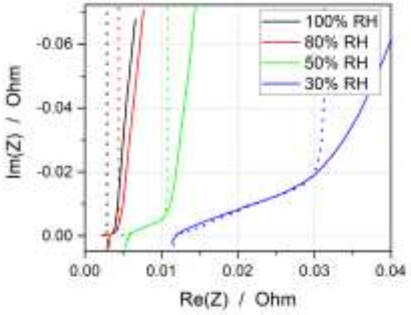


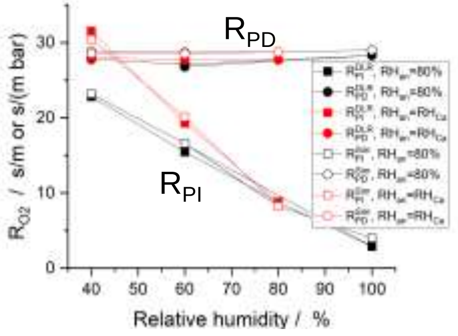






Result for differential cell!





PEMTASTIC

Clean Hydrogen Partnership
Co-funded by the European Union
The project is supported by the Clean Hydrogen Partnership and its members Hydrogen Europe and Hydrogen Europe Research.

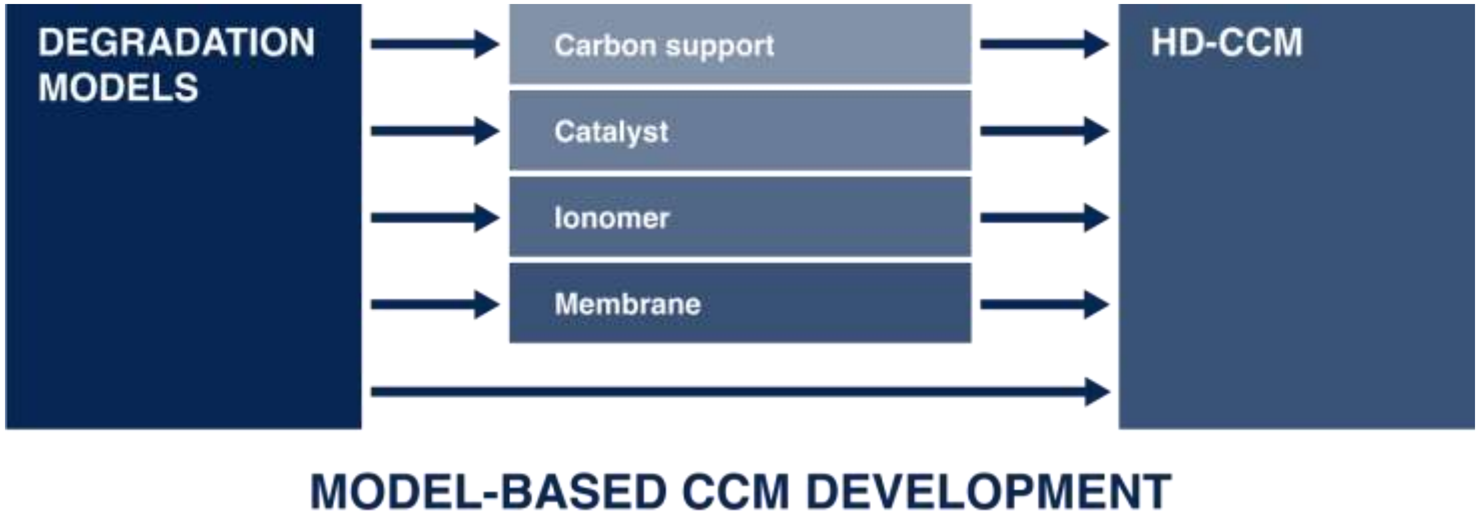
DLR

ROBUST PEMFC MEA DERIVED FROM MODEL-BASED UNDERSTANDING OF DURABILITY LIMITATIONS FOR HEAVY DUTY APPLICATIONS

http://pemtastic-project.eu/ https://www.linkedin.com/showcase/pemtastic Contact: pawel.gazdzicki@dlr.de

Concept

The R&D project PEMTASTIC aims to meet the key technical challenges to increase durability of membrane-electrode assembly (MEAs) for heavy-duty (HD) applications.

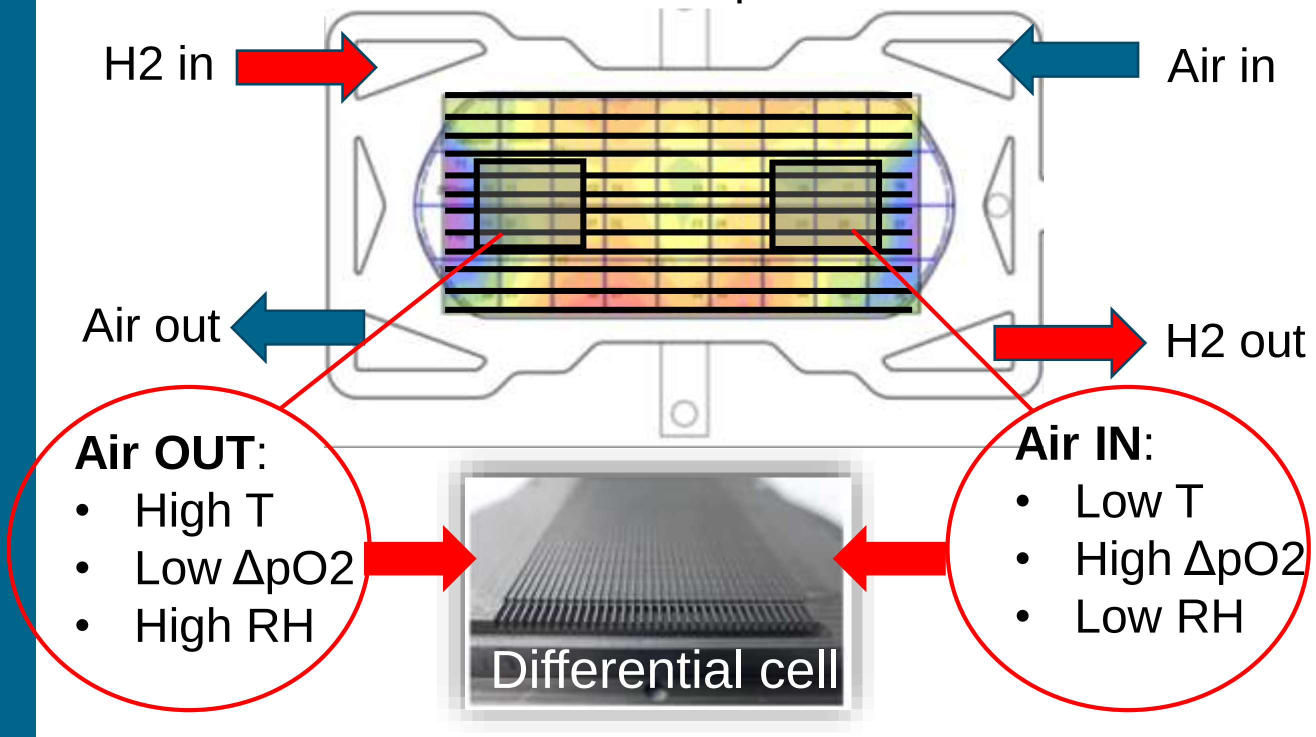


	Clean Hydrogen JU SRIA KPIs			
	SoA 2020[18]	Targets 2024	Targets 2030	PEMTASTIC targets
Durability / h	15,000	20,000	30,000	20,000
PGM loading / gkW^{-1}	0.4	<0,3	<0.25	0.3
Power density / Wcm^{-2}	1.0 @ 0.65V	>1,2@0,650V	>1,2@0,650V	1.2 @ 0.65V
Additional Project KPIs				
Operation temperature / °C	80-85			95-105 at low RH

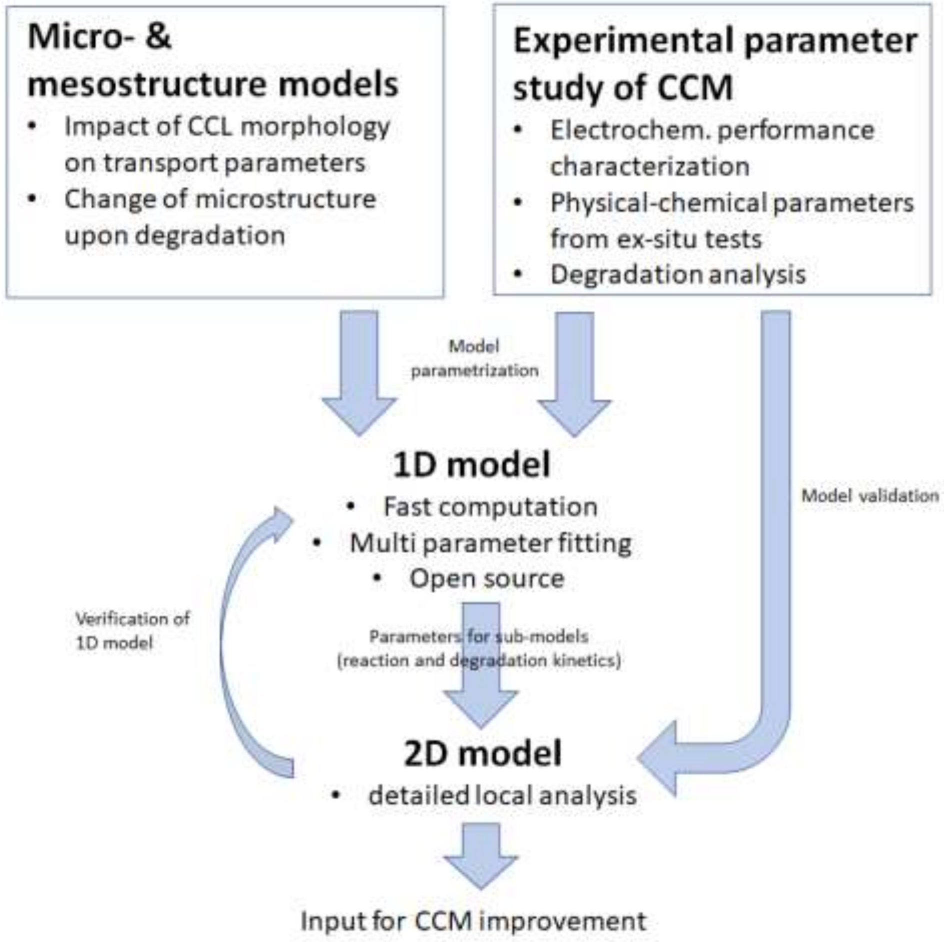
The project is supported by the Clean Hydrogen Partnership and its members Hydrogen Europe and Hydrogen Europe Research.

Methodology

Stack flow field with paralle channels



Reference MEA and final MEA will be tested in short stack

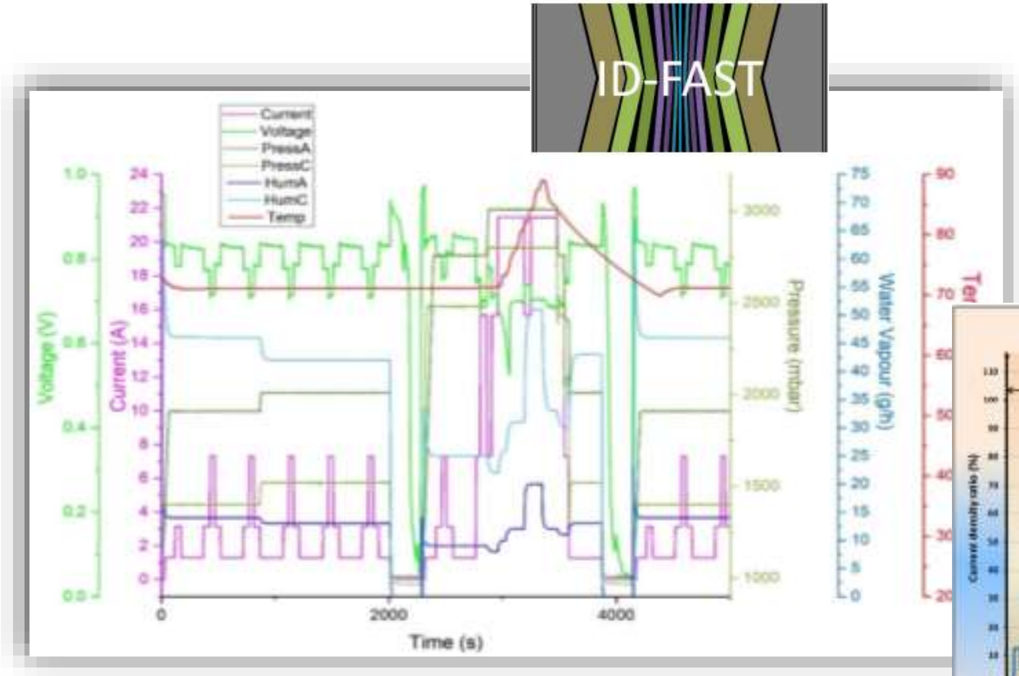


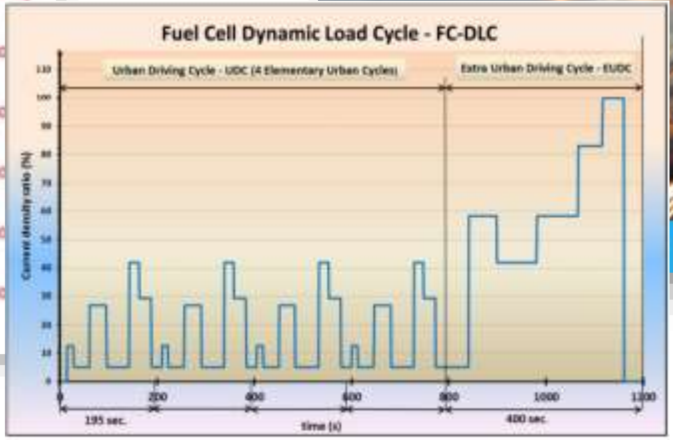
Clean Hydrogen Partnership
Co-funded by the European Union

The project is supported by the Clean Hydrogen Partnership and its members Hydrogen Europe and Hydrogen Europe Research.

Durability testing and characterization

- Harmonized testing protocols available for automotive application, but not for heavy-duty






European Commission

JRC SCIENCE FOR POLICY REPORT

EU HARMONISED TEST
FOR PEMFC MEA TESTIN
SINGLE CELL CONFIGUR
AUTOMOTIVE APPLICAT

Georgios Tsotridis, Alberto Pilenga


FUEL CELLS AND HYDROGEN JOINT UNDERTAKING

STACK-TEST

Stack-Test Master Document – TM 2.00

Project reference: FCH JU 303445
Topic: SP1-JTI-FCH.2011.5.4 "Development of industry wide uniform performance test procedures for PEM fuel cell stacks"

Version 1.01 (October 19th, 2015)

Author:
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Bérangère Guicherd (SymbioFCell)
Jens Mitzel (DLR)
Jürgen Hunger (ZSW)
Thomas Jungmann (FhG ISE)

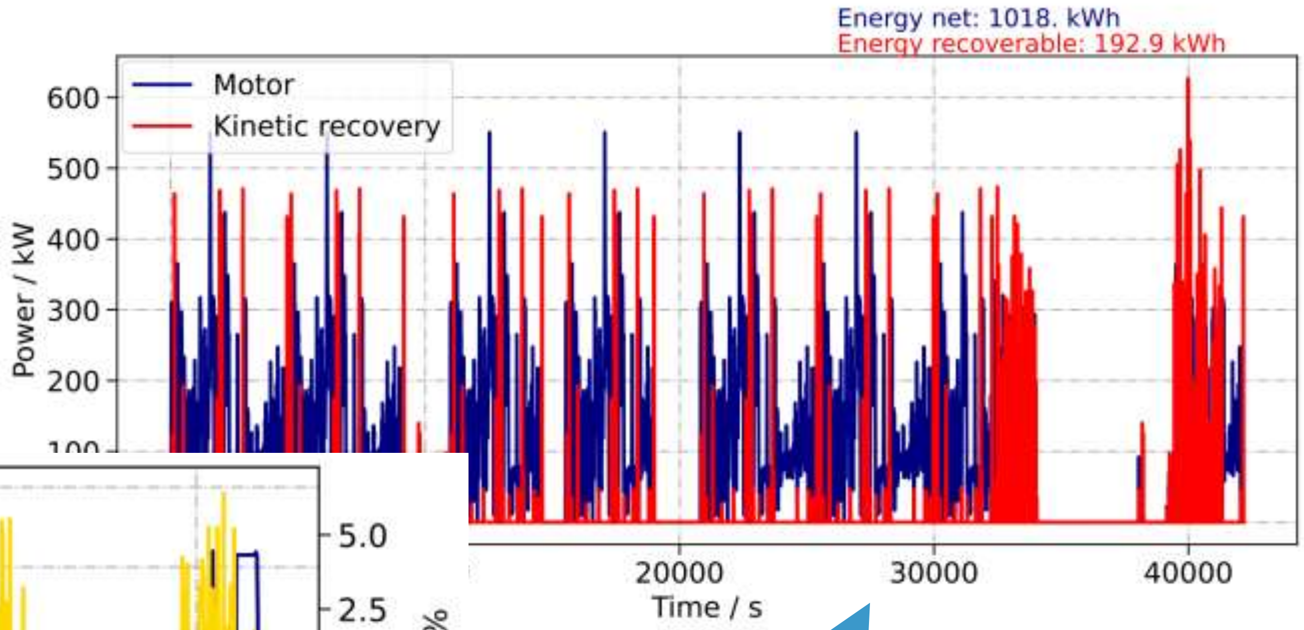
Definition of FC testing protocols for heavy duty application

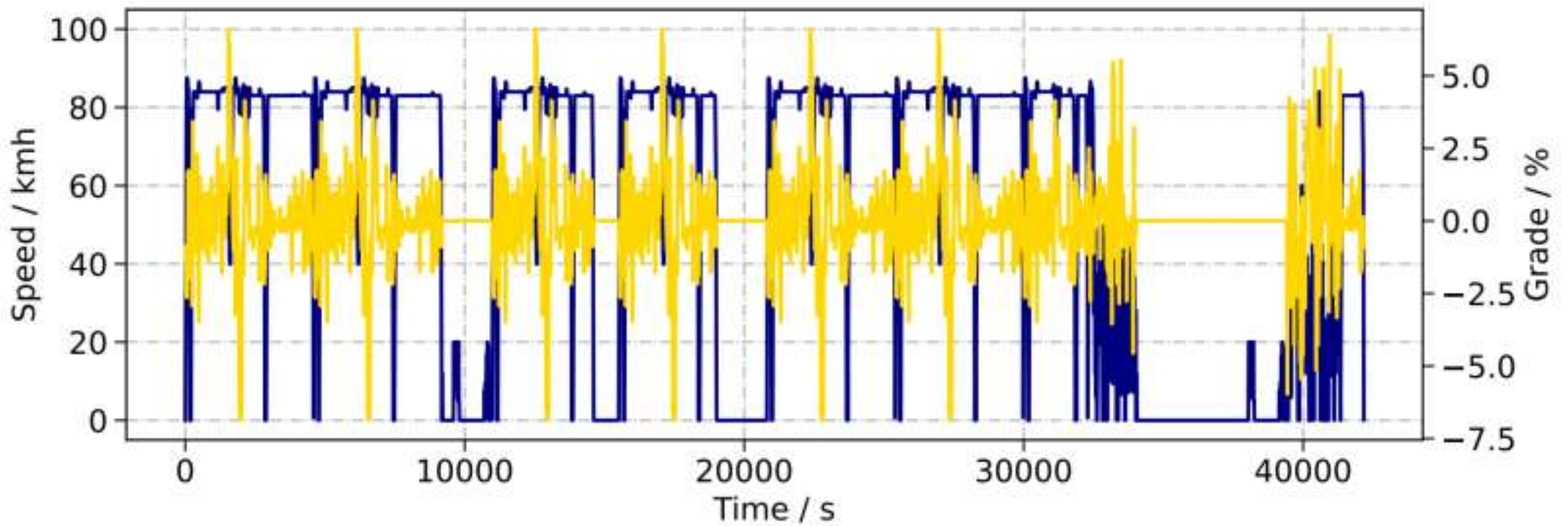
J. Sanchez-Monreal (DLR-TT)

Clean Hydrogen Partnership

Co-funded by the European Union

GVW	35029 kg
Vehicle	15729 kg
Load	19300 kg
$A \times C_d$	5.3 m^2
C_r	0.006





Mechanical simulation

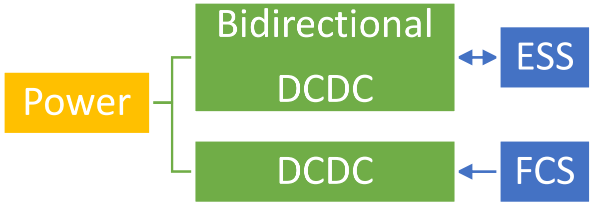
The project is supported by the Clean Hydrogen Partnership and its members Hydrogen Europe and Hydrogen Europe Research.

Definition of FC testing protocols for heavy duty application

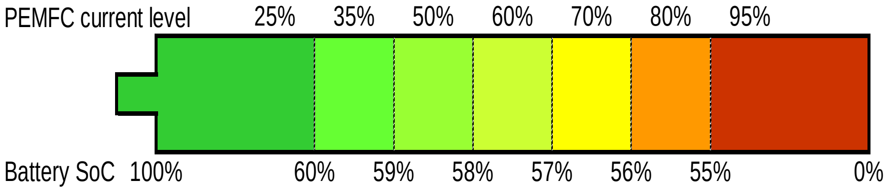
J. Sanchez-Monreal (DLR-TT)

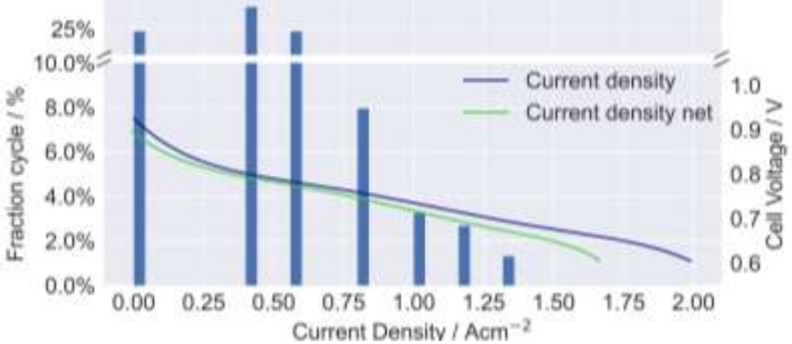
Clean Hydrogen Partnership
Co-funded by the European Union

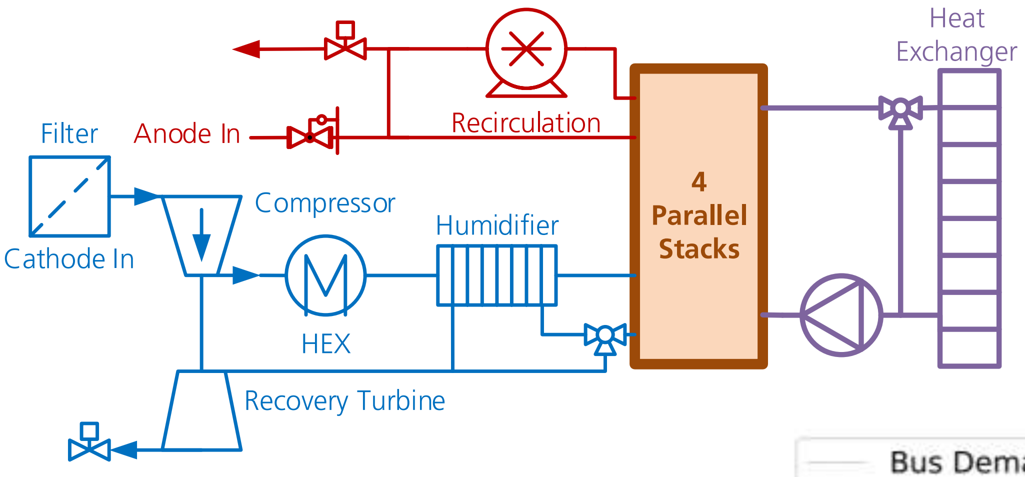
Hybrid Architecture

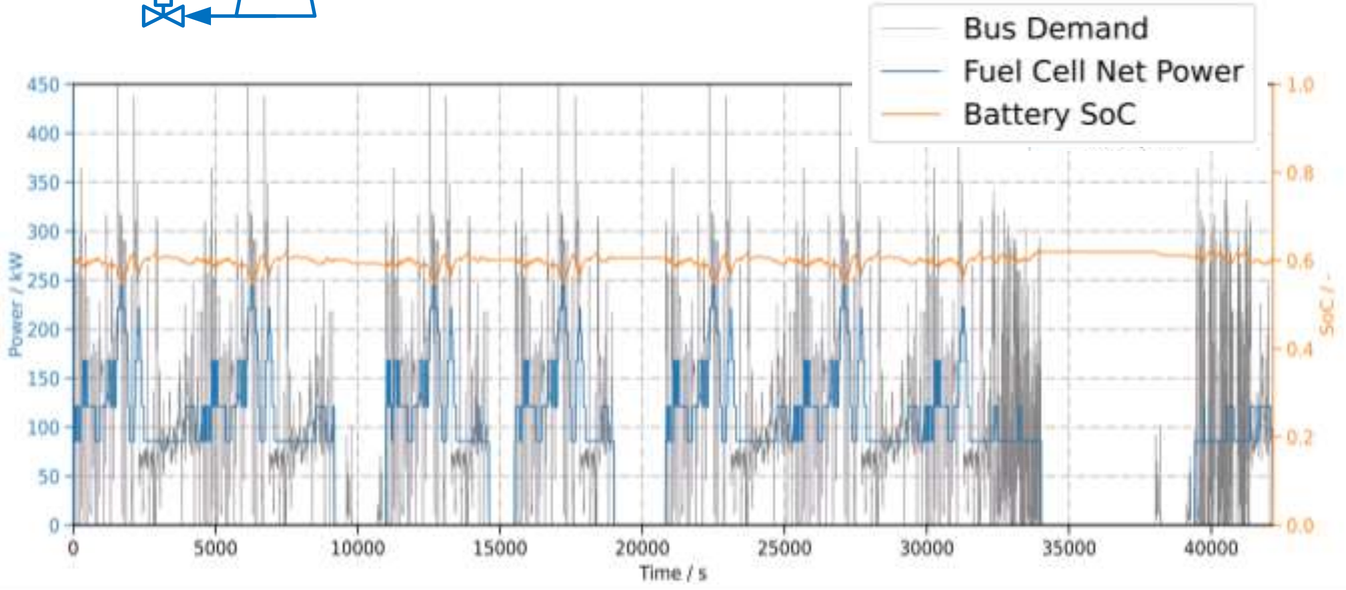


Hybridization Strategy







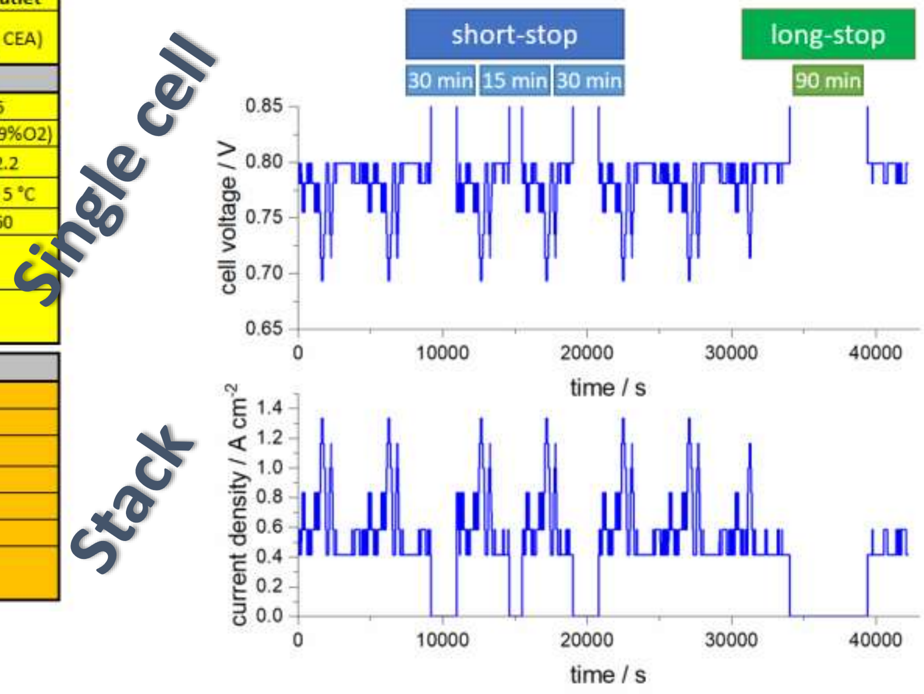


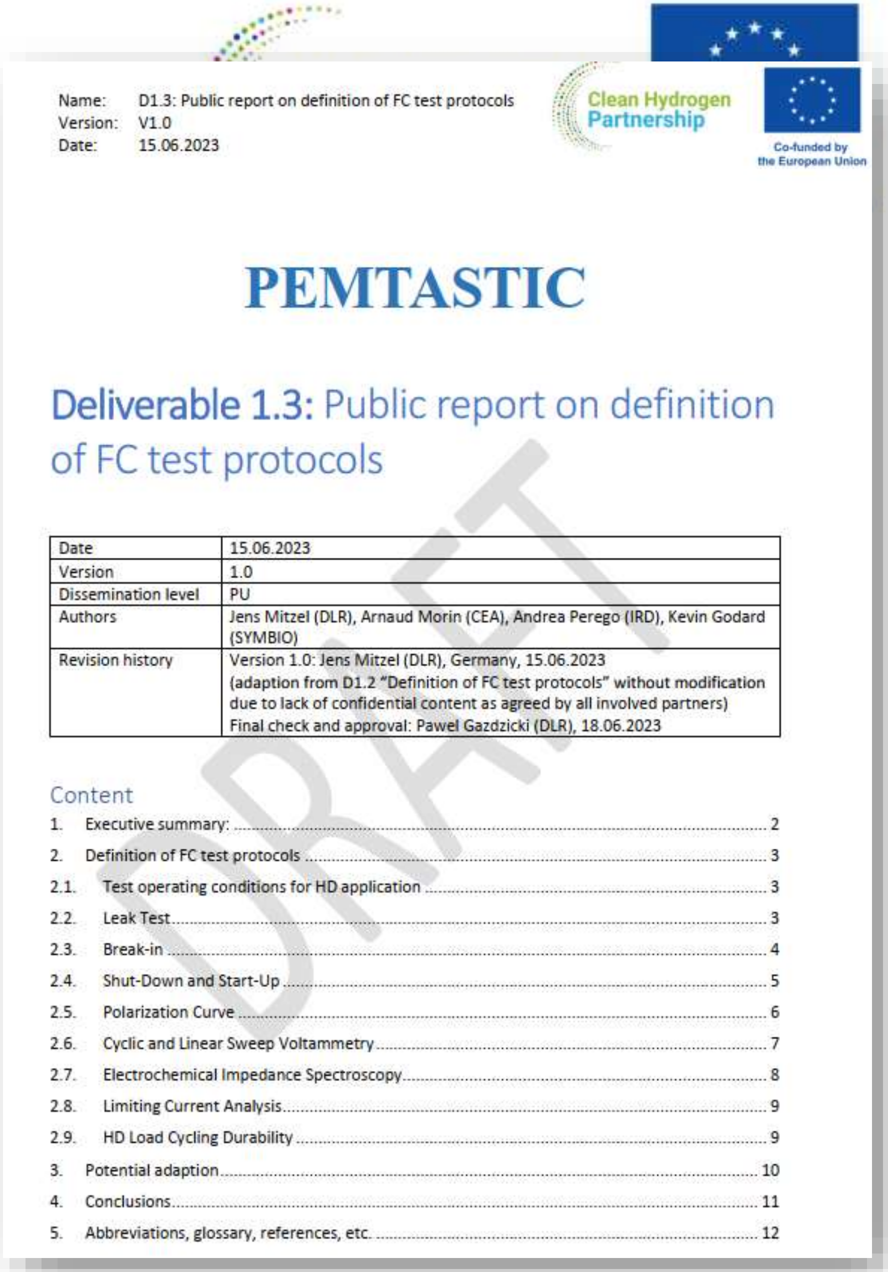
Project 101101433 — PEMTASTIC

The project is supported by the Clean Hydrogen Partnership and its members Hydrogen Europe and Hydrogen Europe Research.

Definition of FC testing protocols for heavy duty application

Proposed by	PEMTASTIC HD condtions	
Source	At air inlet	At air outlet
Comment		(calc by CEA)
Differential Cell		
Cell temperature [°C]	90	105
Gas composition	H2/air	H2/(N2+9%O2)
Outlet pressure anode / cathode [bar_{abs}]	2.5/2.5	2.6/2.2
Gas inlet temperature anode/cathode [°C]	Cell temperature + 5 °C	
RH anode / cathode [%]	80/50	35/60
H2 and O2 stoichiometry for 4 cm channel length [-]	10/10	
Fixed gas flow according to current density [A/cm²]	3.0	
Stack / technical single cell		
Coolant inlet temperature [°C]	90	
Gas composition	H2/air	
Inlet pressure anode / cathode [bar_{abs}]	2.6/2.5	
Gas inlet temperature anode/cathode [°C]	95	
RH anode / cathode [%]	50/35	
Stoichiometry integral cell / stack [-]	1.2/1.8	
Fixed gas flow according to current density [A/cm²]	0.2	





Date	15.06.2023
Version	1.0
Dissemination level	PU
Authors	Jens Mitzel (DLR), Arnaud Morin (CEA), Andrea Perego (IRD), Kevin Godard (SYMBIO)
Revision history	Version 1.0: Jens Mitzel (DLR), Germany, 15.06.2023 (adaption from D1.2 "Definition of FC test protocols" without modification due to lack of confidential content as agreed by all involved partners) Final check and approval: Pawel Gazdzicki (DLR), 18.06.2023

Content

The project is supported by the Clean Hydrogen Partnership and its members Hydrogen Europe and Hydrogen Europe Research.

Summary

- Typical electrochemical MEA charcterization
- For model paramterization/validation differential cells are used
- Durability to be investigated in relevant conditions
- HD testing protocols are proposed in frame of PEMTASTIC
- Important aspect is when and how often characterization is carried out in frame of durability testing

Clean Hydrogen Partnership

Co-funded by the European Union

PEMTASTIC

*The project **PEMTASTIC** is supported by the Clean Hydrogen Partnership and its members Hydrogen Europe and Hydrogen Europe Research under the Grant Agreement 101101433.*

*The project **Further-FC** has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 875025. This Joint Undertaking receives support from the European Union's Horizon 2020 Research and Innovation program, Hydrogen Europe and Hydrogen Europe Research.*

Thank you for your attention

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