DEVELOPMENT AND CHARACTERIZATION OF ELECTRODES AND MEAS FOR HT-PEM FUEL CELLS

INVITED TALK IN SESSION A "FUEL CELL ELECTROCATALYST ACTIVITY AND DURABILITY"

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German Aerospace Center (DLR), Institute of Engineering Thermodynamics

ECS PRiME 2024, 10th October 2024

DLR

German Aerospace Center (DLR)

AERONAUTICS

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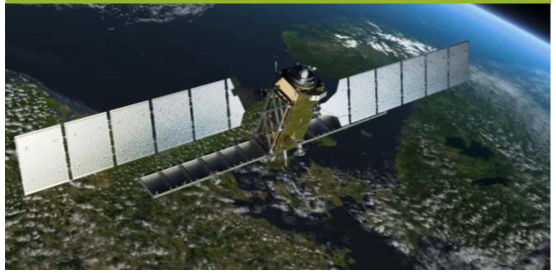

ENERGY

TRANSPORT

SECURITY
Civil Security & Defence Research

DIGITALISATION, QUANTUM TECHNOLOGY AND SYSTEM MODELLING

- Europe's largest research centre for aeronautics and space
- 54 institutes and facilities across 30 sites
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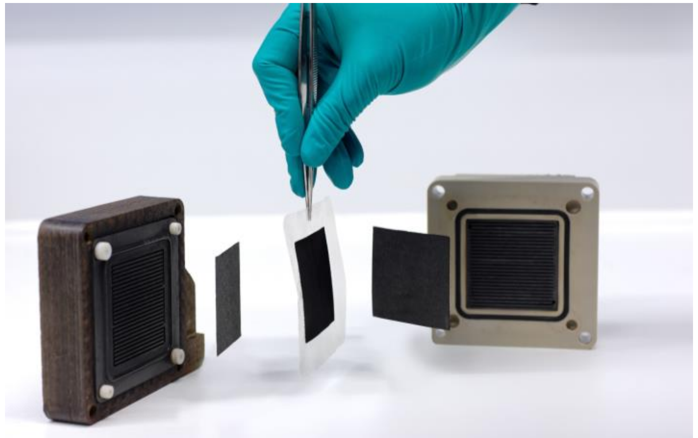
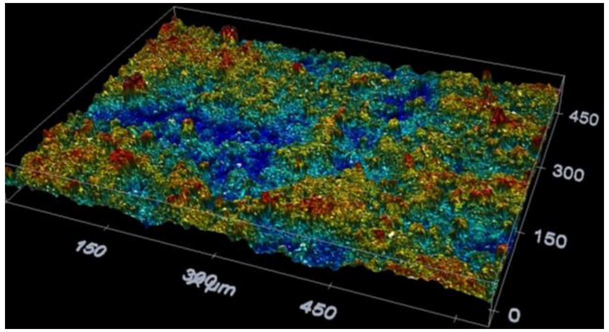
DLR Institute of Engineering Thermodynamics



Material Development

Long-term stable, efficient components for HT-PEM fuel cells

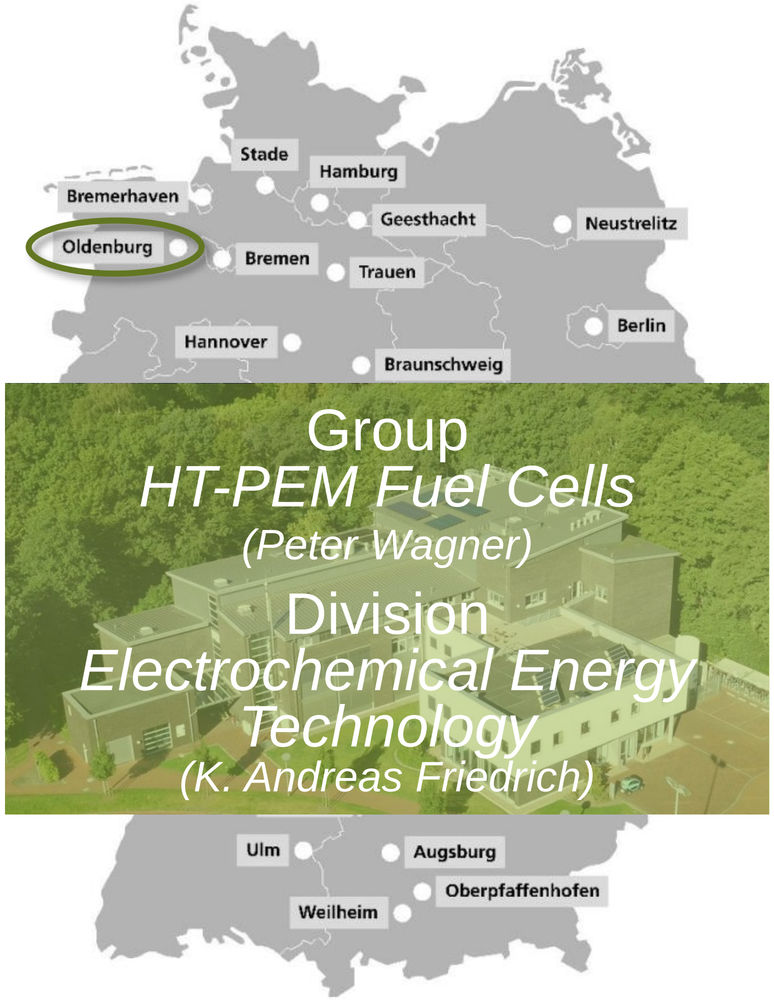
Catalysts, membranes, electrodes, membrane-electrode-assemblies



Analytics and Quality Control

On gas diffusion layers and bipolar plates after fabrication

Electrochemical, physico-chemical, imaging methods



Group

HT-PEM Fuel Cells

(Peter Wagner)

Division

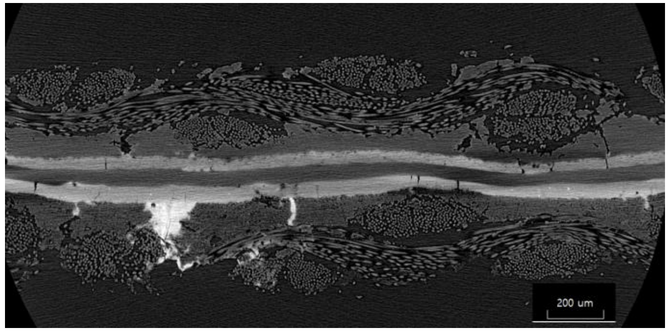
Electrochemical Energy Technology

(K. Andreas Friedrich)

Performance Studies

From thin-film analysis to PEM fuel cells

Activity and degradation, contamination effects, accelerated stress tests



Cost-efficient Electrodes

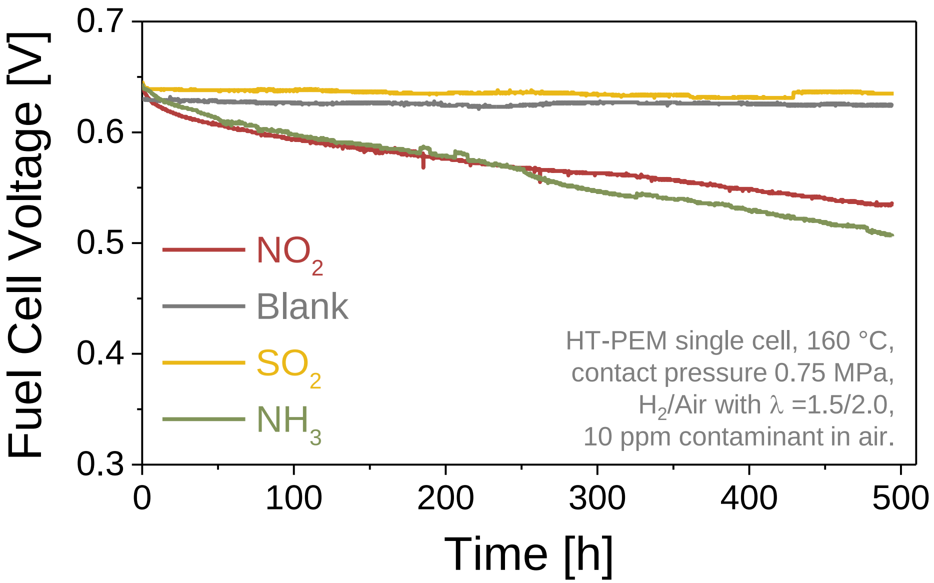
for PEM fuel cells

Reduced PGM-contents, use of M-N-C catalysts, sintered paper-based electrodes

HT-PEM Fuel Cells

Advantages

- Increased tolerance towards contaminants like CO or H_2S due to **160 °C**
- Direct use of industrial quality H_2 or reformates
  → **Application flexibility**



HT-PEM single cell, 160 °C, contact pressure 0.75 MPa, H_2/Air with λ =1.5/2.0, 10 ppm contaminant in air.

Challenges

- Lower cell performances and graphitic bipolar plates → Larger stack sizes compared to LT-PEMFCs
- Limited lifetime due to corrosion of components
- Phosphate poisoning of catalytic active sites
  → Higher Pt loading of **up to 1 $mg_{Pt}cm^{-2}$** per electrode

Increasing
Corrosion Resistance

Reducing PGM-Contents
in PEM Fuel Cells

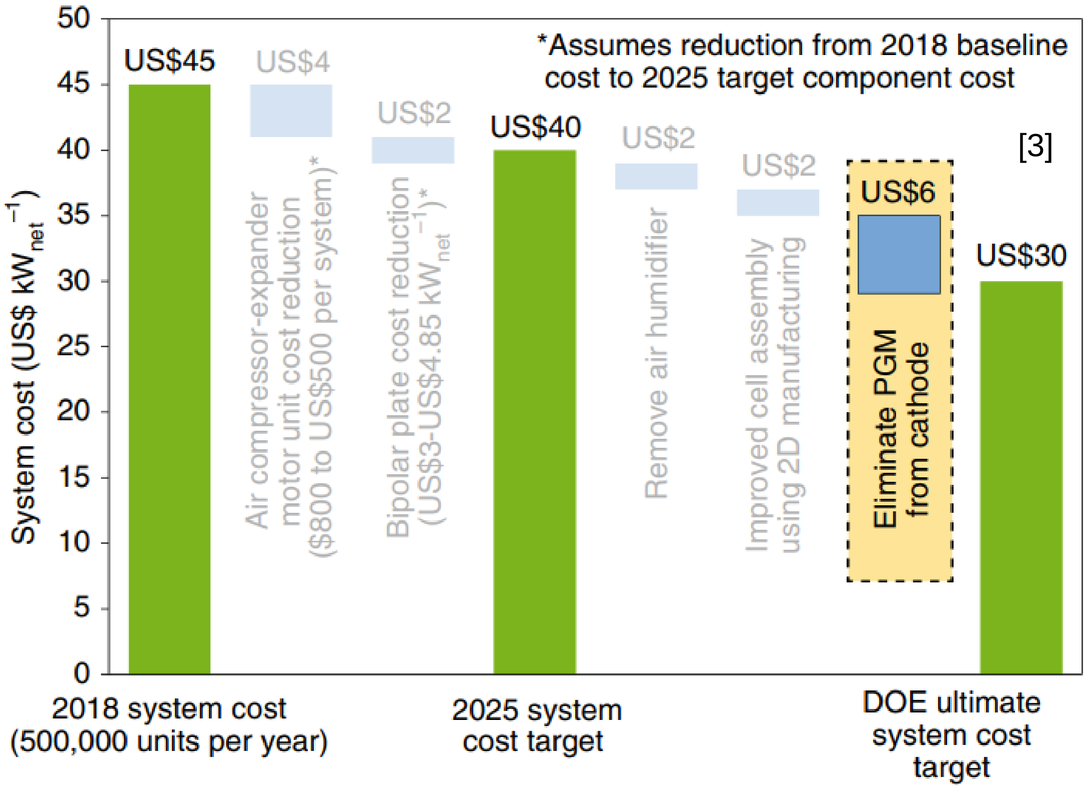
D. Schonvogel et al., J. Power Sources **2021**, *High temperature polymer electrolyte membrane fuel cell degradation provoked by ammonia as ambient air contaminant*, 109, 401.

D. Schonvogel et al., Int. J. Hydrog. Energy **2021**, *Impact of air contamination by NOx on the performance of high temperature PEM fuel cells*, 46, 33934.

D. Schonvogel et al., Int. J. Hydrog. Energy **2021**, *Effect of air contamination by sulfur dioxide on the high temperature PEM fuel cell*, 46, 6751.

PEM FC Cost Issues

- Elimination of Platinum in PEM FC cathode significant for system cost reduction
- Current Pt loadings per electrode
  - HT-PEM FC: 0.70-1.0 mg_{Pt} cm^{-2} [1]
  - LT-PEM FC: 0.05-0.3 mg_{Pt} cm^{-2} [2]
- Most promising alternative: M-N-C (Metal-Nitrogen-Carbon) catalyst [3]
  - Fe-N-C active for the oxygen reduction reaction (ORR)



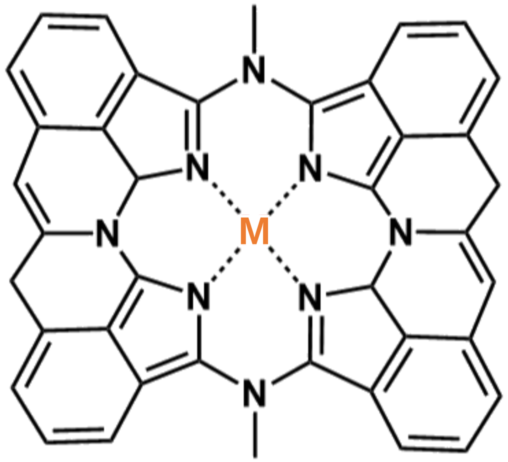
System costs for light-duty vehicle PEMFC system.
This cost reduction pathway is based on system cost analysis.

[1] N. Seselj, S. M. Alfaro, E. Bompolaki, L. N. Cleemann, T. Torres, K. Azizi, *Adv. Mater.* **2023**, 2302207.
[2] Y. Wang, D. F. Ruiz Diaz, K. S. Chen, Z. Wang, X. C. Adroher, *Mater. Today* **2020**, 32, 178.
[3] S. T. Thompson, D. Papageorgopoulos, *Nature Catalysis* **2019**, 2, 558.

M-N-C for Oxygen Reduction Reaction

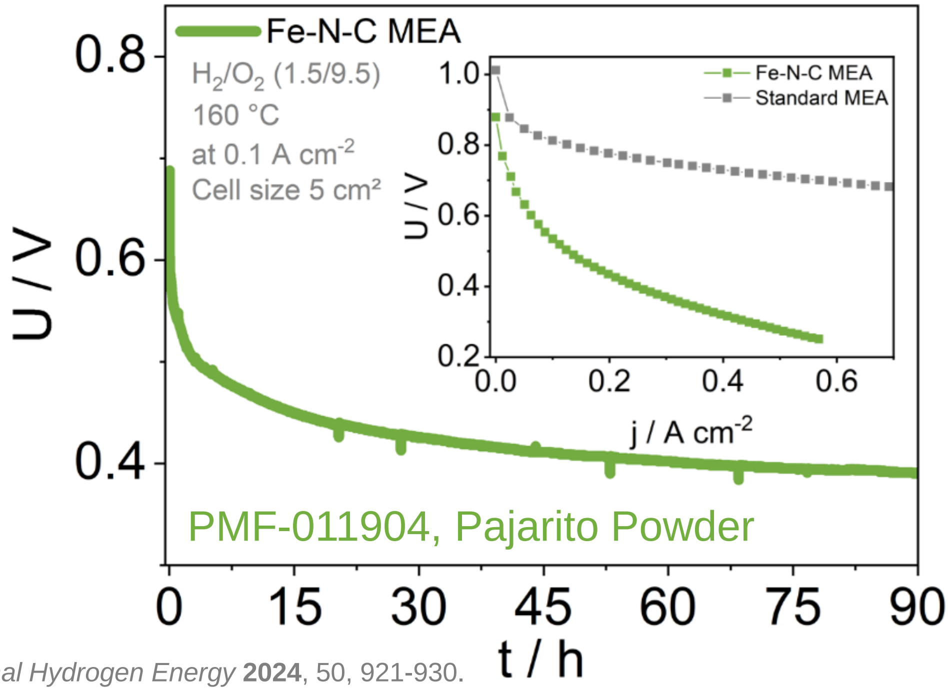
Advantages

- M-N-C costs 200 times less than Pt-based catalyst (0.142 mg_{Pt} cm^{-2}) [1]
- No catalyst poisoning by phosphates in HT-PEM FCs [2,3]



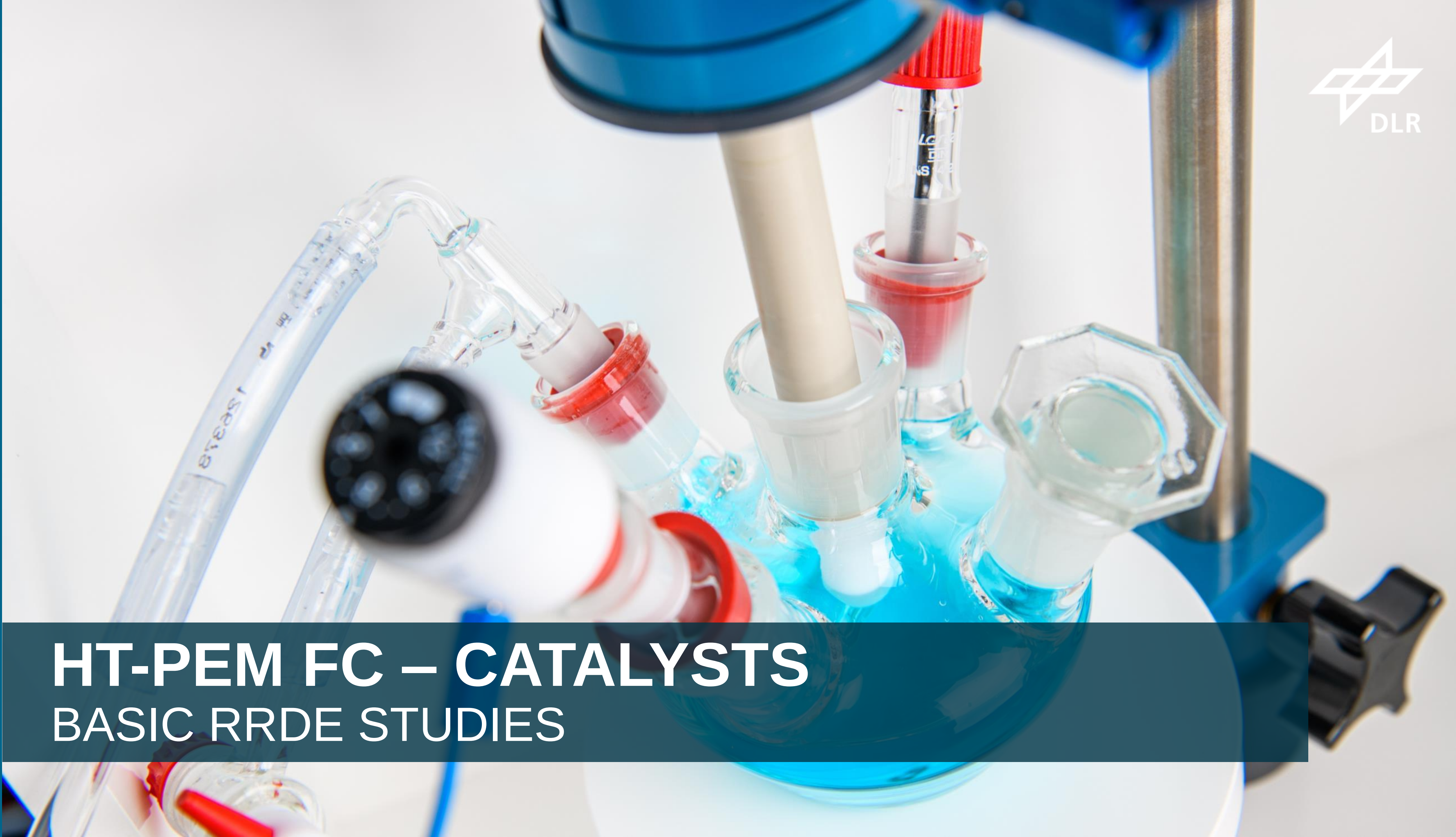
Challenges

- Volumetric activity lower compared to Pt/C [1,2]
  - Thicker catalyst layers (60-100 µm versus 3-5 µm for PGM in LT-PEM FC) [1]
- Stability insufficient in LT- and HT-PEM FC [4, 5]



PMF-011904, Pajarito Powder

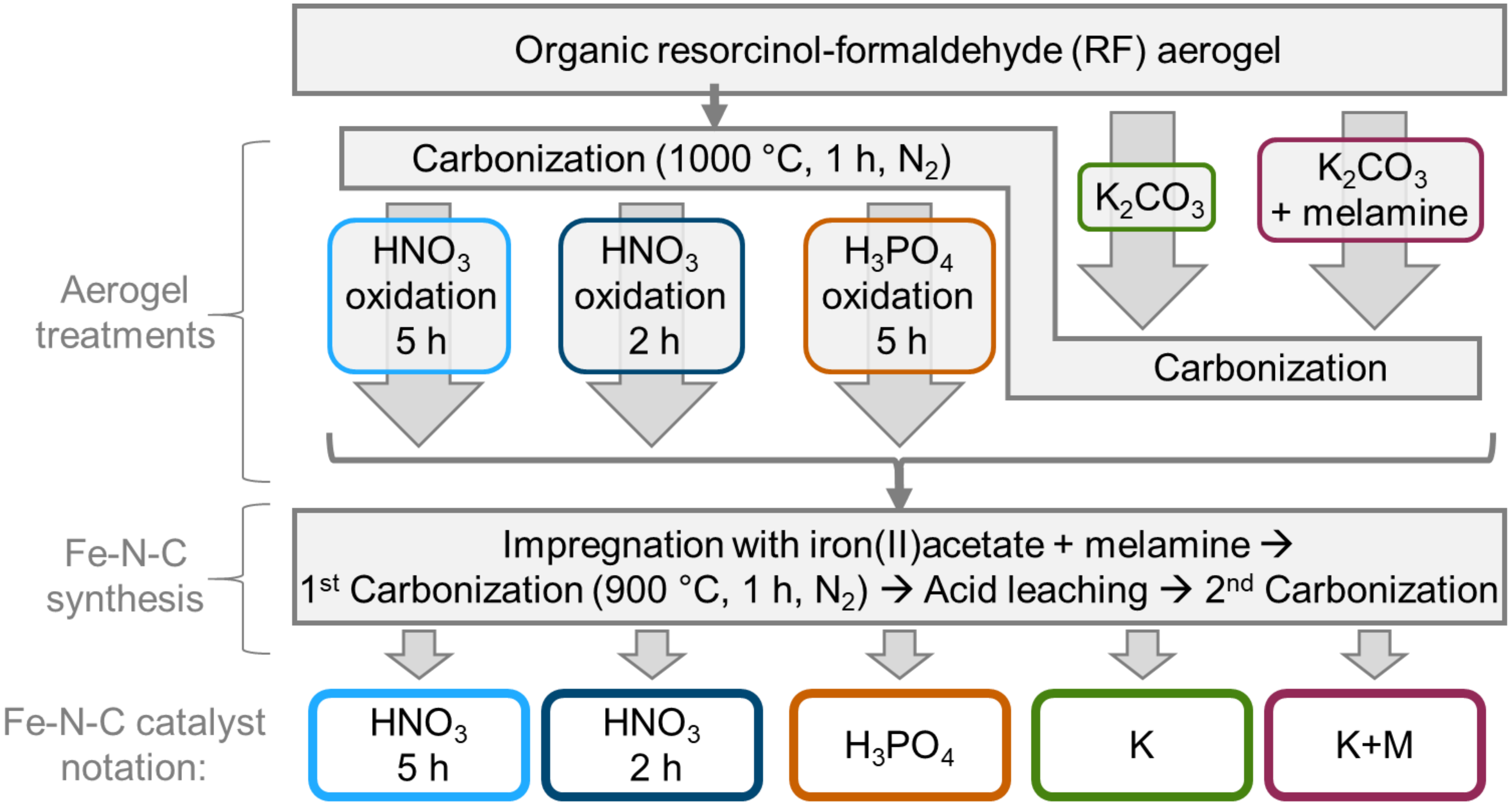
[1] S. T. Thompson, D. Papageorgopoulos, Nature Catalysis 2019, 2, 558.
[2] Q. Meyer, C. Yang, Y. Cheng, C. Zhao, *Electrochem. Energy Rev.* **2023**, 6, 16.
[3] Y. Hu, J. O. Jensen, C. Pan, L. N. Cleemann, I. Shypunov, Q. Li, *Appl. Catal., B* **2018**, 234, 357.
[4] K. Kumar, L. Dubau, F. Jaouen, and F. Maillard, *Chem. Rev.* **2023**, 123, 9265.
[5] J. Müller-Hülstede, H. Schmies, D. Schonvogel, Q. Meyer, Y. Nie, C. Zhao, P. Wagner, M. Wark, *Int. Journal Hydrogen Energy* **2024**, 50, 921-930.



HT-PEM FC – CATALYSTS

BASIC RRDE STUDIES

Fe-N-C Catalysts based on Carbon Aerogels



Organic resorcinol-formaldehyde (RF) aerogel

Aerogel treatments

Carbonization (1000 °C, 1 h, N_2)

HNO_3 oxidation 5 h

HNO_3 oxidation 2 h

H_3PO_4 oxidation 5 h

K_2CO_3

K_2CO_3 + melamine

Carbonization

Fe-N-C synthesis

Impregnation with iron(II)acetate + melamine →
1st Carbonization (900 °C, 1 h, N_2) → Acid leaching → 2nd Carbonization

Fe-N-C catalyst notation:

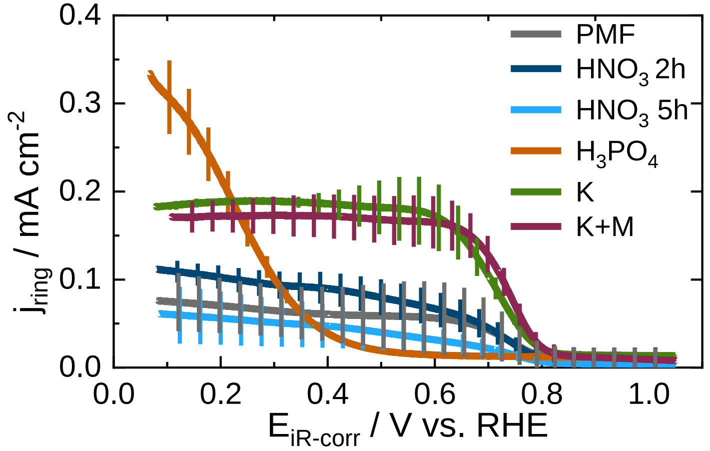
HNO_3 5 h	HNO_3 2 h	H_3PO_4	K	K+M

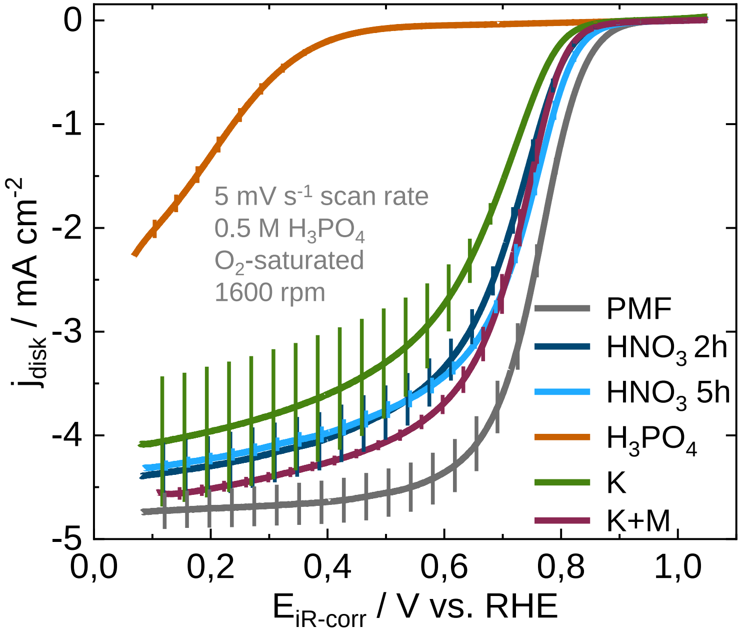
T. Zierdt et al., ChemSusChem **2024**, *Impact of Aerogel Modification for Fe N C Activity and Stability towards Oxygen Reduction Reaction in Phosphoric Acid Electrolyte*, under revision.

Fe-N-C Catalysts based on Carbon Aerogels

Effect of Aerogel Treatment

RRDE Study
0.5 M H_3PO_4
Room Temperature





- H_3PO_4 → Treatment not beneficial
- K and K+M → Lower selectivity
  - Higher graphitic N content
  - Lower pyrrolic/pyridinic N contents than HNO_3
- HNO_3 2h → Incomplete oxidation
  - Lower ORR activity and selectivity
- HNO_3 5h → Highest activity and selectivity

Increase of pyrrolic/pyridinic N content

→ Higher ORR activity with **higher pyrrolic/pyridinic N** required for Fe-N_x sites

T. Zierdt et al., ChemSusChem **2024**, *Impact of Aerogel Modification for Fe N C Activity and Stability towards Oxygen Reduction Reaction in Phosphoric Acid Electrolyte*, under revision.

Fe-N-C Catalysts based on Carbon Aerogels

Optimized Synthesis Route

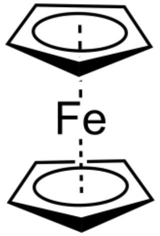
Organic resorcinol-formaldehyde (RF) aerogel monolith

Ball milling, 30 s
3x15 mm Balls

Milled RF-Aerogel

\+

Ferrocene (Fe)



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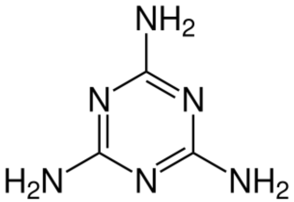
Melamine (M)



1st Heat treatment
1 h, 1000 °C

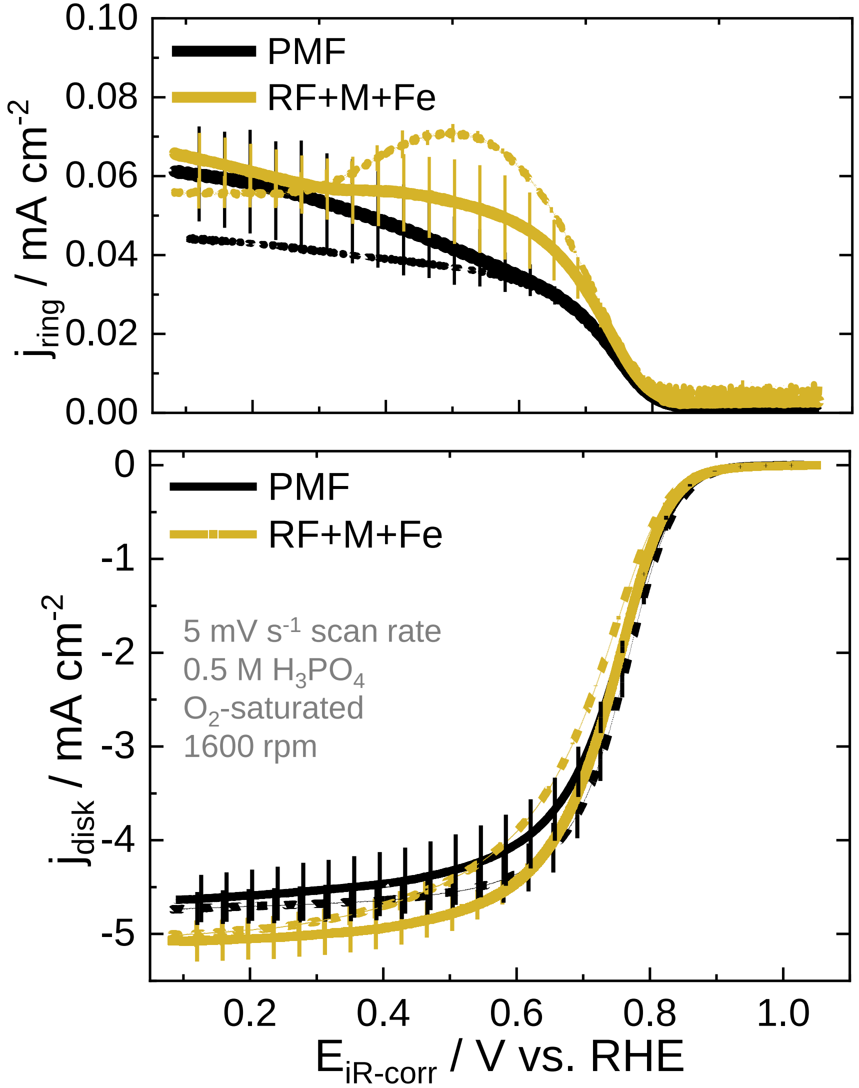
2nd Heat treatment
900 °C, 1h
(N_2 atmosphere)

RF+M+Fe

Fe-N-C Catalysts based on Carbon Aerogels

ORR Activity and Stability

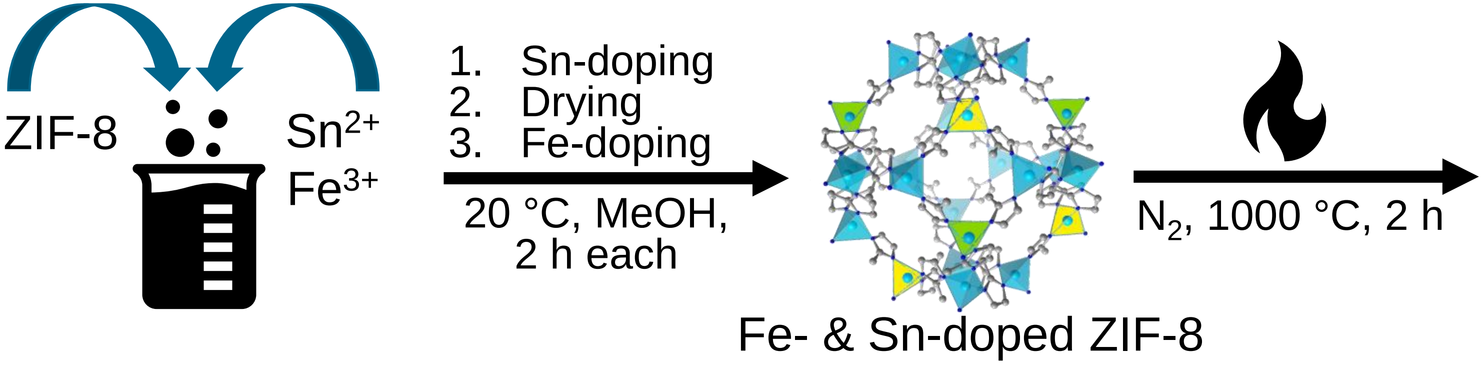
RRDE Study
0.5 M H_3PO_4
Room Temperature



Catalyst	Mass activity @ 0.8 V / A g^{-1}	Mass activity @ 0.8 V after AST / A g^{-1}	Loss / %
Commercial Fe-N-C (PMF)	3.9 ± 0.2	2.6 ± 0.6	-34
RF+M+Fe	2.7 ± 0.2	2.1 ± 0.1	-25

- Faster and cheaper catalyst fabrication than template or carbon support synthesis
- Sufficient activity and stability close to commercial Fe-N-C
- Next step: Synthesis upscaling to 20 g and HT-PEM electrode fabrication

Bimetallic Fe-Sn-N-C Catalysts based on MOFs
Synthesis and ICP-MS


ZIF-8

Sn^{2+}

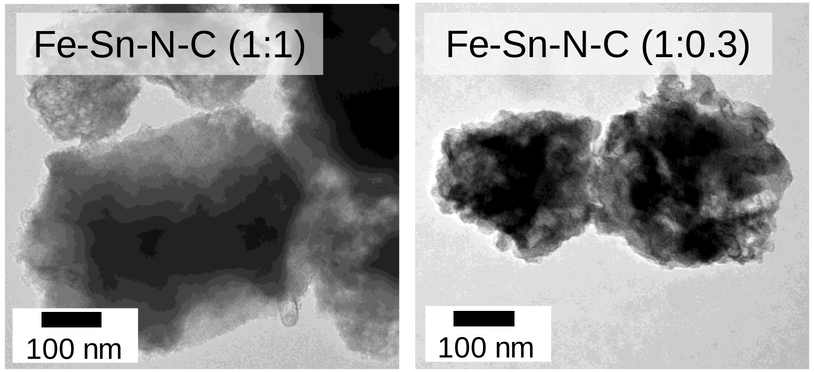
Fe^{3+}

1. Sn-doping
2. Drying
3. Fe-doping

20 °C, MeOH,
2 h each

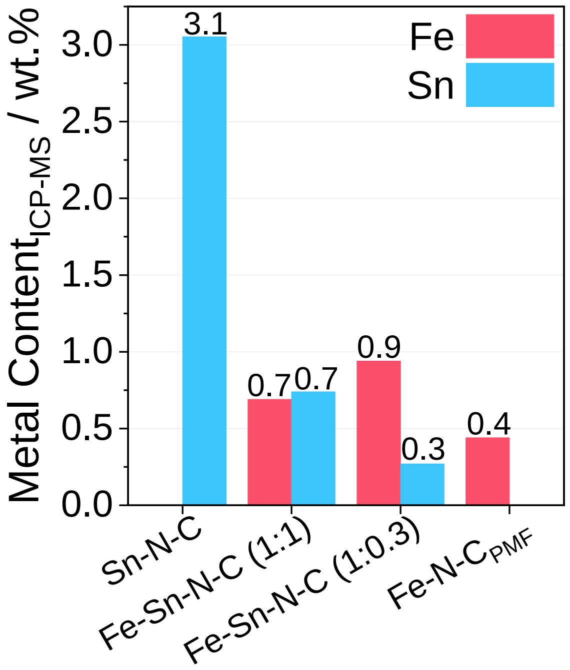
Fe- & Sn-doped ZIF-8

N_2, 1000 °C, 2 h


Fe-Sn-N-C (1:1)

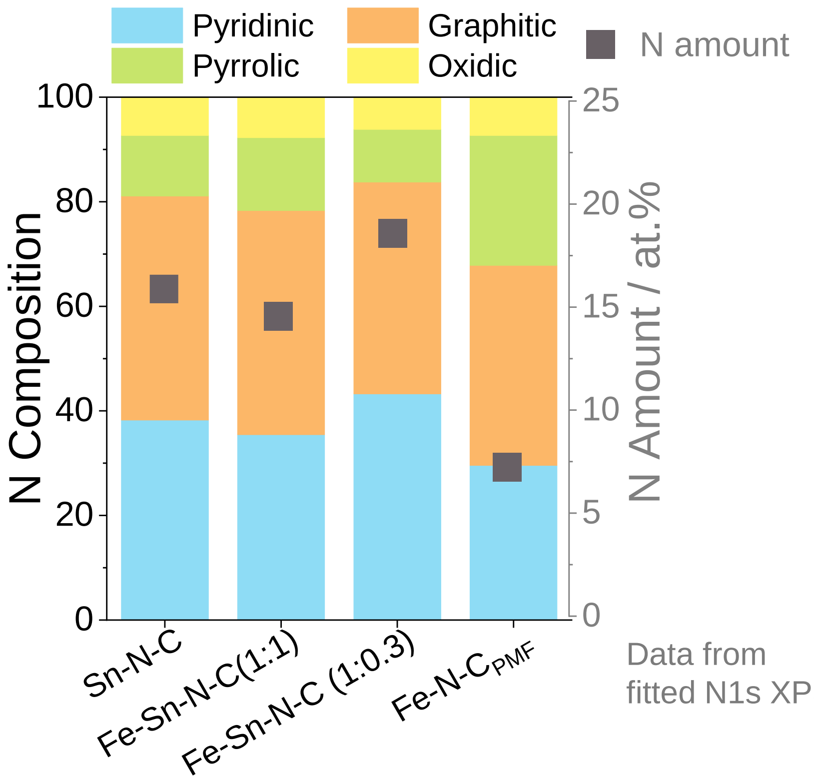
Fe-Sn-N-C (1:0.3)

- Names refer to metal ratios determined by ICP-MS
- Fe-N-C_{PMF} → PMF-D14401 (Pajarito Powder) for comparison
- Low Fe and Sn amounts in Fe-Sn-N-Cs
  - Second doping in MeOH probably causes Sn being washed out
- No acid leaching or template removal necessary
  - Fewer steps, easier upscaling

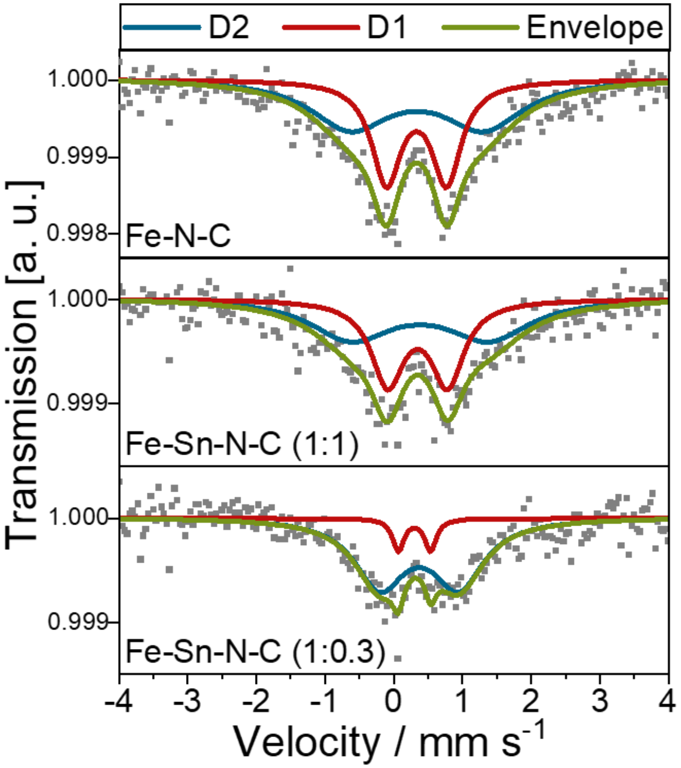


Bimetallic Fe-Sn-N-C Catalysts based on MOFs

XPS and Mössbauer Spectroscopy



Data from fitted N1s XPS



^{57}Fe-Mössbauer spectra at room temperature

Measurements performed at Renz Group, Institute of Inorganic Chemistry, Leibniz University Hannover.

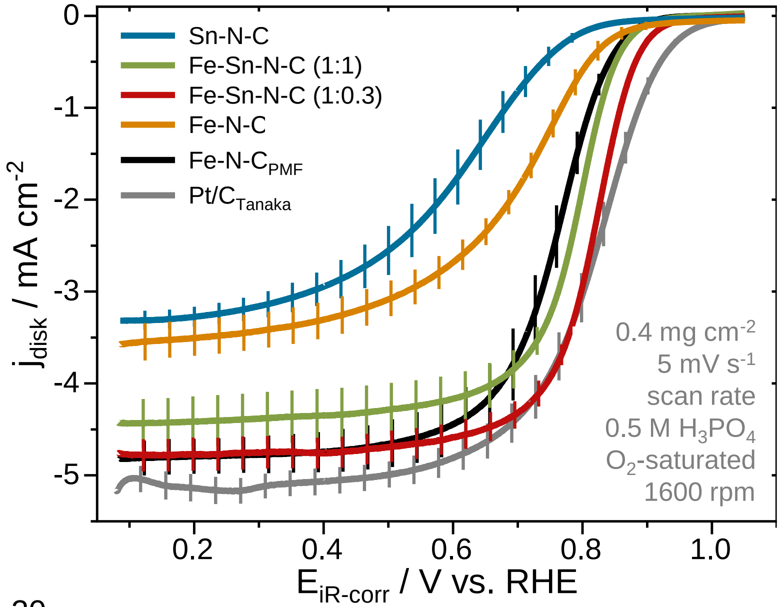
- Significant contents of pyridinic, pyrrolic, graphitic N species
  - M-N$_x$ sites selective for preferred 4e$^-$ pathway and non-metallic N sites catalyzing 2x2e$^-$ pathway
- Similar Mössbauer results for Fe-N-C and Fe-Sn-N-Cs
  - Comparable Fe coordination environments indicating bimetallic character without neighbored Fe and Sn

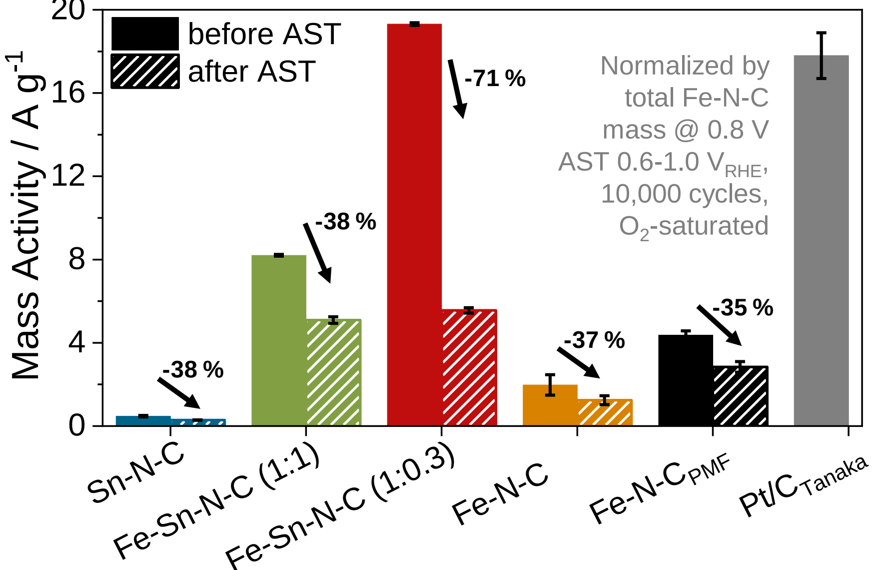
Bimetallic Fe-Sn-N-C Catalysts based on MOFs
ORR Activity and Stability

RRDE Study
0.5 M H_3PO_4
Room Temperature

- Low mass activity for monometallic Sn-N-C and Fe-N-C
- High mass activity for Fe-Sn-N-Cs
  - Exceeding commercial Fe-N-C (PMF, Pajarito Powder)
  - Fe-Sn-N-C (1:0.3) surpassing commercial Pt/C (40 wt% Pt/C, TEC10E40E, Tanaka)
- Good stability of Fe-Sn-N-C (1:1) under harsh AST
  - Comparable to commercial Fe-N-C
  - Much lower stability of Fe-Sn-N-C (1:0.3) compared to (1:1)
  - Indicating stability enhancing effects of Sn

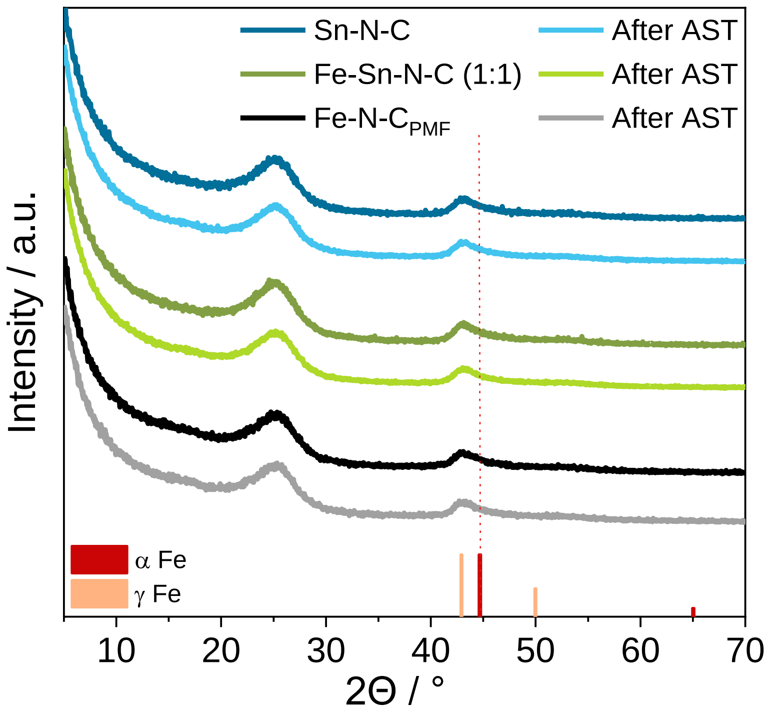
→ Significant impact of Sn on ORR activity



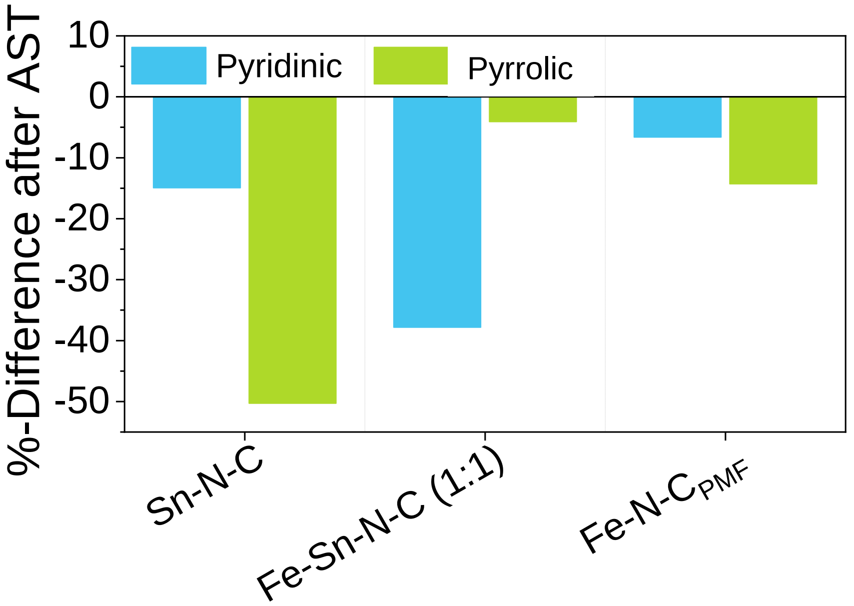


Bimetallic Fe-Sn-N-C Catalysts based on MOFs
XRD and XPS before and after Stress Testing

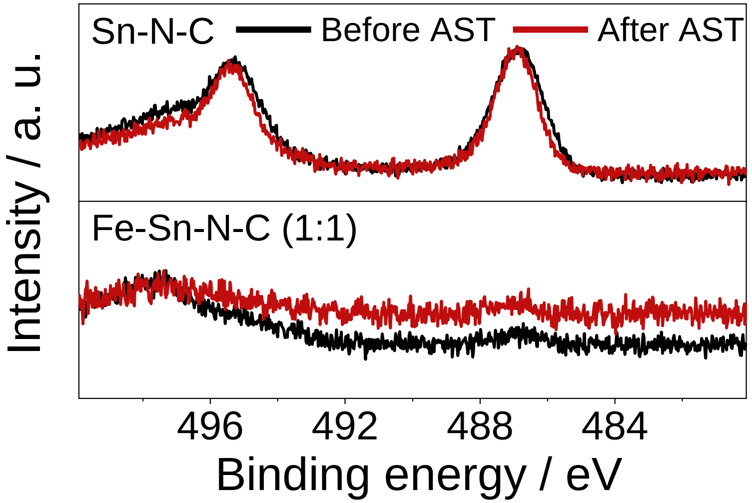
RRDE Study
0.5 M H_3PO_4
Room Temperature



XRD before and after AST



Determined loss of proportions of N-species (pyridinic and pyrrolic) before and after the AST



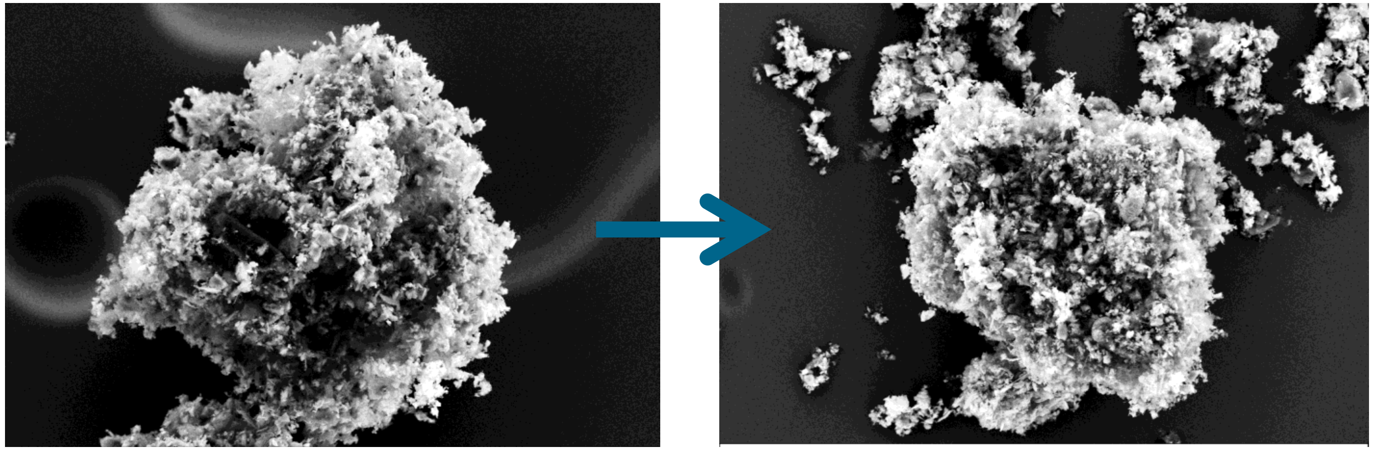
Sn3d XPS before and after AST

- In XRD no changes of crystal structure or metallic particle formation
  - Visible peaks belonging to the glassy carbon disc
- In XPS fitting for N1s, however, due to Nafion® no fitting for Fe, C and O
  - Pyridinic N decreased after AST showing loss of active sites
- In XPS no significant differences for Sn3d

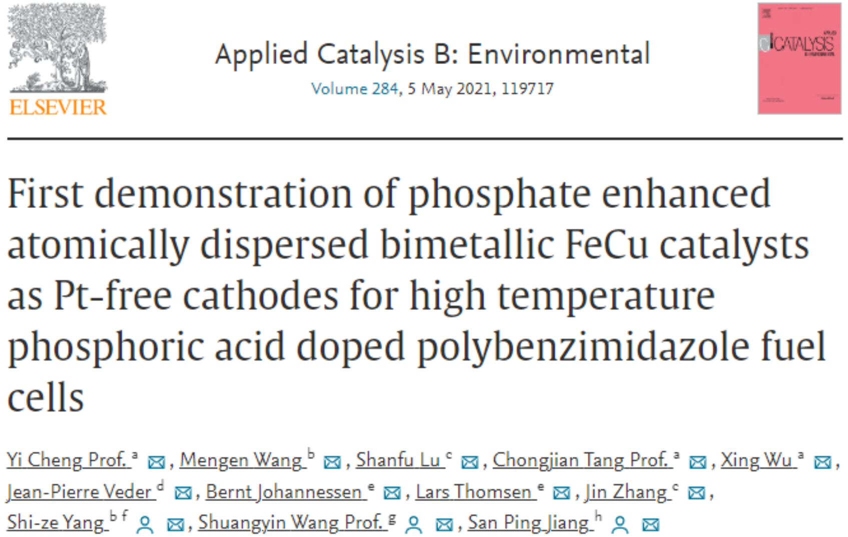
→ XRD and XPS suggesting that mostly non-metallic active sites are lost

Bimetallic M-M-N-C Catalysts based on SiOC

- Choice of metal combinations embedded into SiOC material
    1. Fe and Co (most widely studied combination for ORR in FC applications)
    2. Fe and Cu (paper by Prof. Cheng group and their superior FeCu catalyst)
    3. Fe and Mn (Mn good ORR catalyst in acidic medium)
- SiOC materials prepared in Advanced Ceramics group at University of Bremen
- Modification with ZIF-8 using pyrolysis



SEM of FeMn-based material before (-PDC) and after modification with ZIF-8 via pyrolysis at 950 °C (-N-SiOC).



ELSEVIER

Applied Catalysis B: Environmental

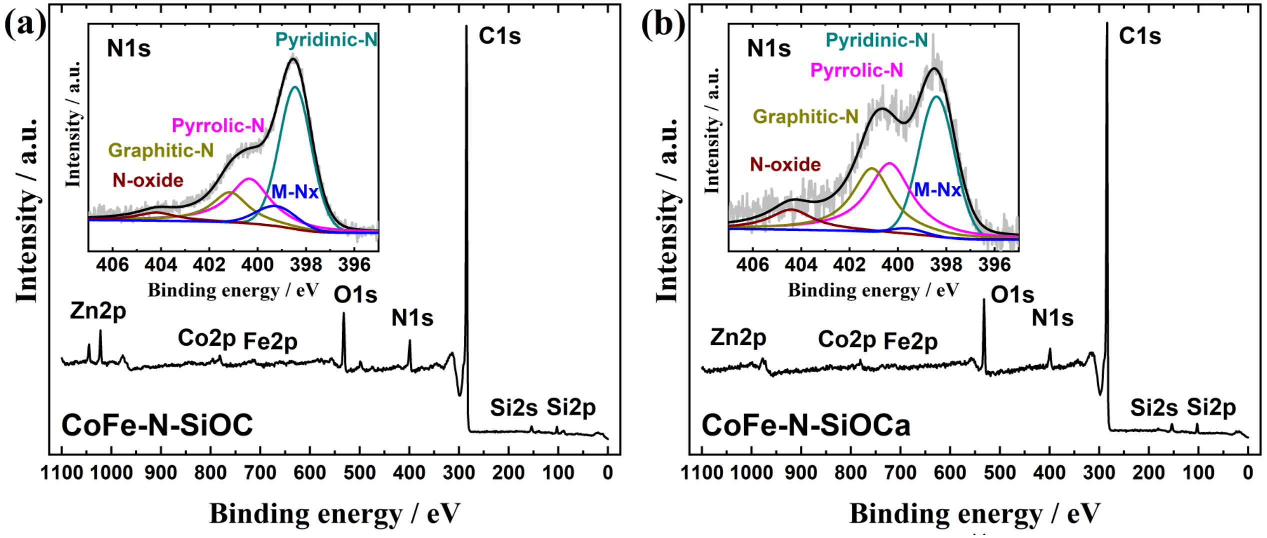
Volume 284, 5 May 2021, 119717

First demonstration of phosphate enhanced atomically dispersed bimetallic FeCu catalysts as Pt-free cathodes for high temperature phosphoric acid doped polybenzimidazole fuel cells

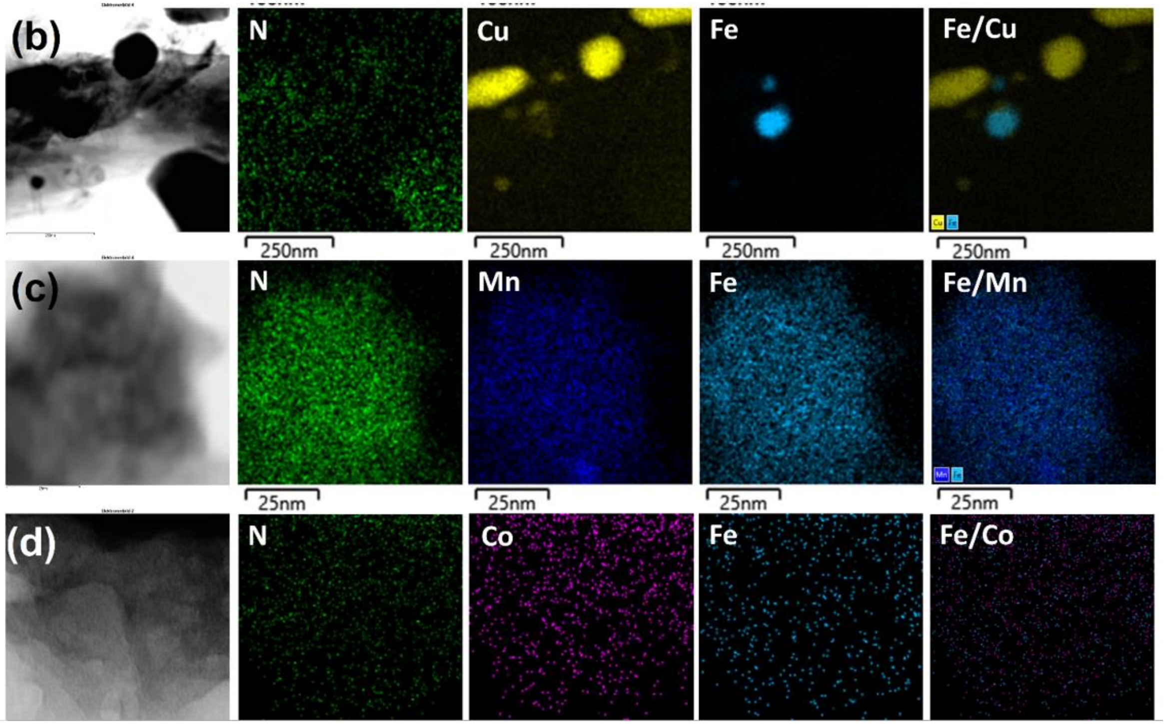
Yi Cheng Prof.[a], Mengen Wang[b], Shanfu Lu[c], Chongjian Tang Prof.[a], Xing Wu[a], Jean-Pierre Veder[d], Bernt Johannessen[e], Lars Thomsen[e], Jin Zhang[c], Shi-ze Yang[b f], Shuangyin Wang Prof.[g], San Ping Jiang[h]

M. Mooste et al., Electrochimica Acta **2024**, *Binary transition metal and ZIF-8 functionalised polymer-derived ceramic catalysts for high temperature PEM fuel cell cathode*, submitted.

Bimetallic M-M-N-C Catalysts based on SiOC
Physical Characterisation



XPS of a) pristine CoFe-N-SiOC and (b) after acid treating (2 M sulfuric acid for 16 h at 90 °C) and second pyrolysis.

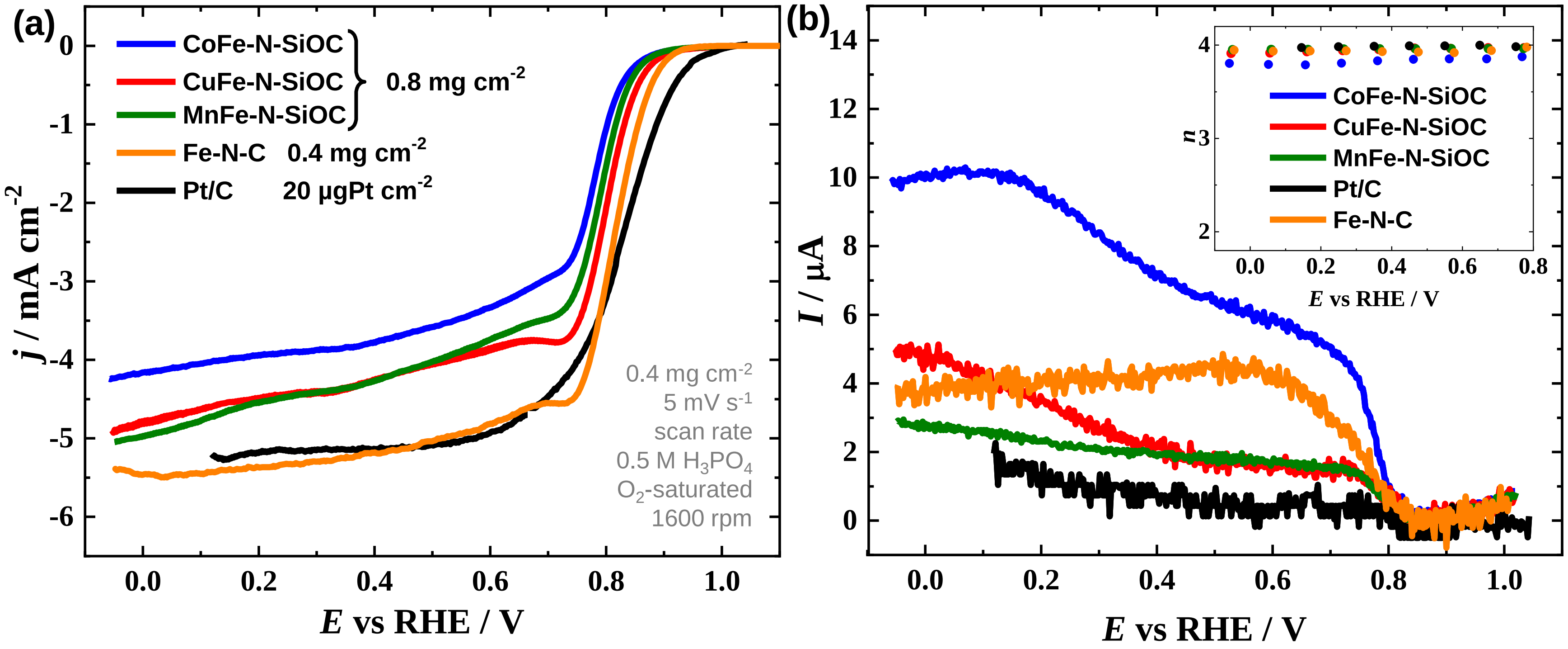


Scanning transmission electron microscopy (STEM) with EDS

M. Mooste et al., Electrochimica Acta **2024**, *Binary transition metal and ZIF-8 functionalised polymer-derived ceramic catalysts for high temperature PEM fuel cell cathode*, submitted.

Bimetallic M-M-N-C Catalysts based on SiOC
ORR Activity and Stability

RRDE Study
0.5 M H_3PO_4
Room Temperature



Catalyst	$E_{1/2}$	$MA_{0.80V}$
CoFe-N-SiOC	770 ± 3	1.66 ± 0.10
MnFe-N-SiOC	774 ± 2	2.65 ± 0.07
CuFe-N-SiOC	781 ± 6	3.55 ± 0.14
Fe-N-C	808 ± 2	17.15 ± 0.15
Pt/C (20 wt% Pt)	824 ± 3	74.35 ± 2.91

Fe-N-C - PMF-0011904, Pajarito Powder

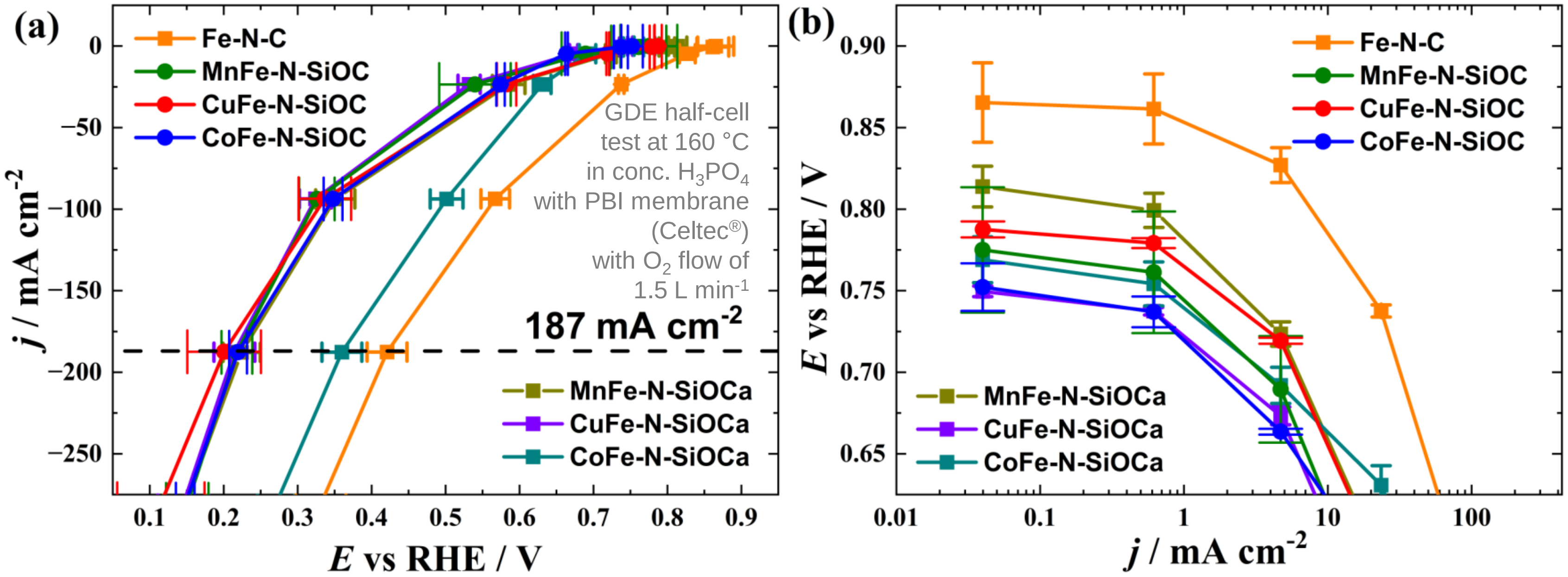
Mass activity (MA, A $g_{Catalyst}^{-1}$) at 0.8 $V_{RHE.}$

M. Mooste et al., Electrochimica Acta **2024**, *Binary transition metal and ZIF-8 functionalised polymer-derived ceramic catalysts for high temperature PEM fuel cell cathode*, submitted.

Bimetallic M-M-N-C Catalysts based on SiOC

GDE Performance

GDE Study
Conc. H_3PO_4
160 °C



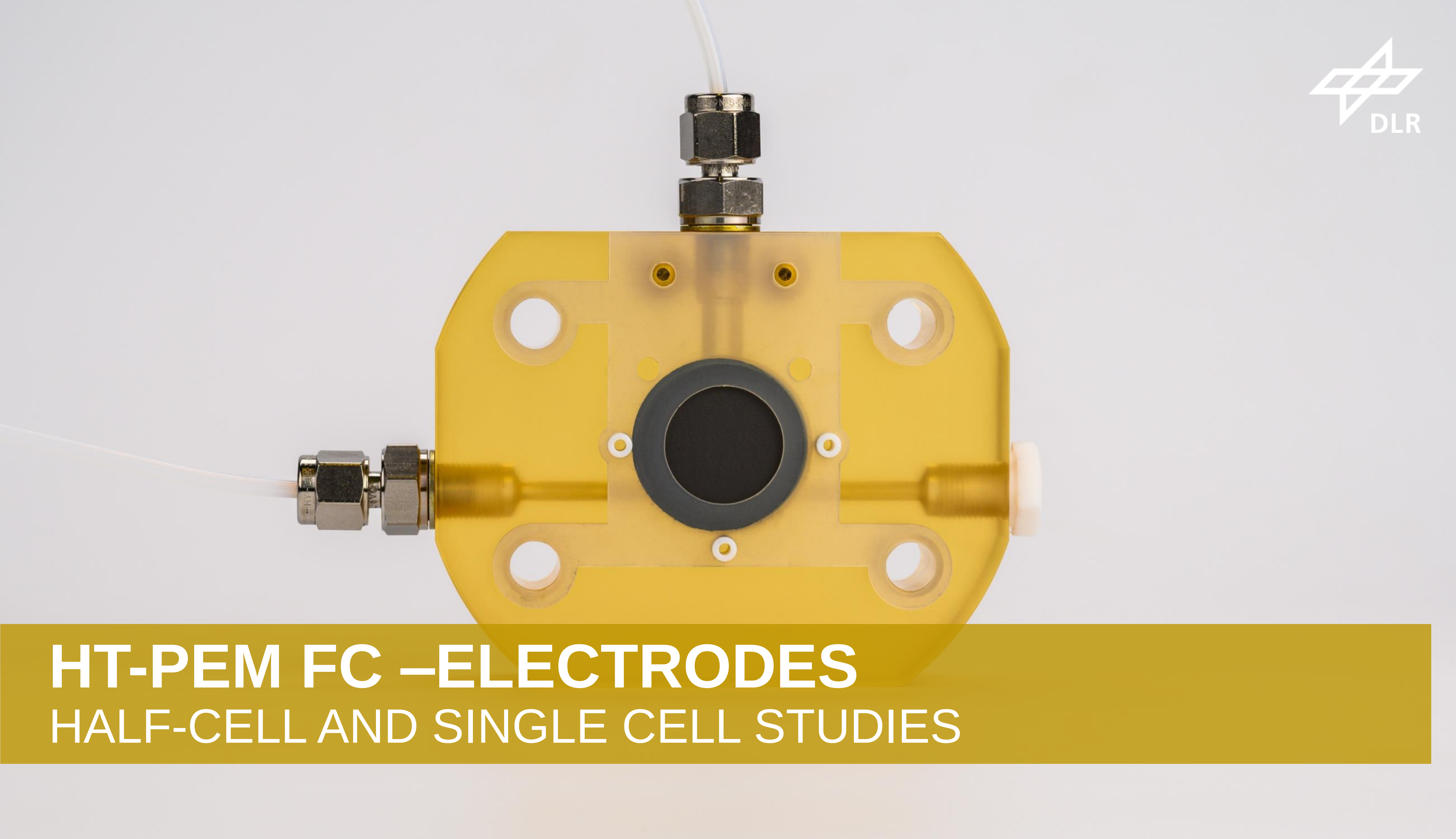
(a) Polarisation curves of ZIF-8-modified and further acid leached (in 2 M sulfuric acid for 16 h at 90 °C with second pyrolysis) catalyst based GDEs and (b) semi-logarithmic plots.

Catalyst	OCP	E_{187}
CoFe-N-SiOC	752 ± 15	219 ± 12
CoFe-SiOCa	**17 mV** ↑ 769 ± 14	**64%** ↑ 360 ± 27
MnFe-N-SiOC	775 ± 38	218 ± 21
MnFe-SiOCa	**39 mV** ↑ 814 ± 12	**~3%** ↑ 224 ± 25
CuFe-N-SiOC	788 ± 15	201 ± 50
CuFe-SiOCa	**38 mV** ↓ 750 ± 13	**~6%** ↑ 214 ± 28
Fe-N-C	865 ± 24	421 ± 27

Fe-N-C - PMF-0011904, Pajarito Powder

Open circuit potential (OCP, mV$_{RHE}$) and potential value at 187 mA cm^{-2} (E187, mV$_{RHE}$) .

M. Mooste et al., Electrochimica Acta **2024**, *Binary transition metal and ZIF-8 functionalised polymer-derived ceramic catalysts for high temperature PEM fuel cell cathode*, submitted.

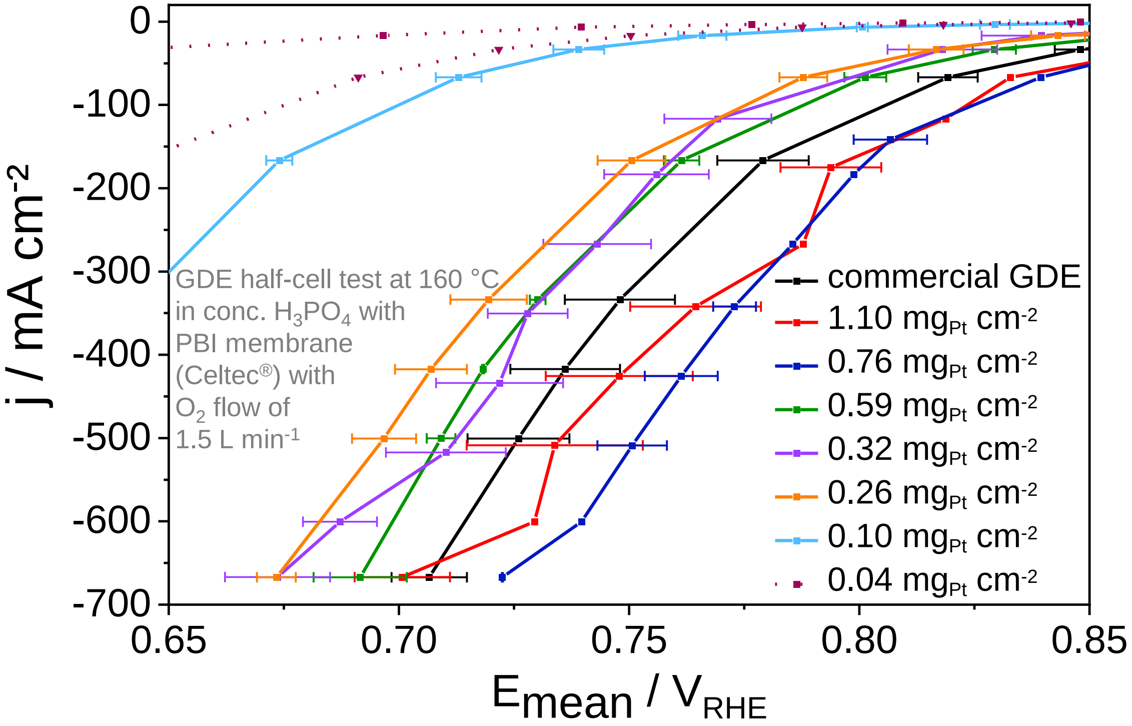


HT-PEM FC –ELECTRODES

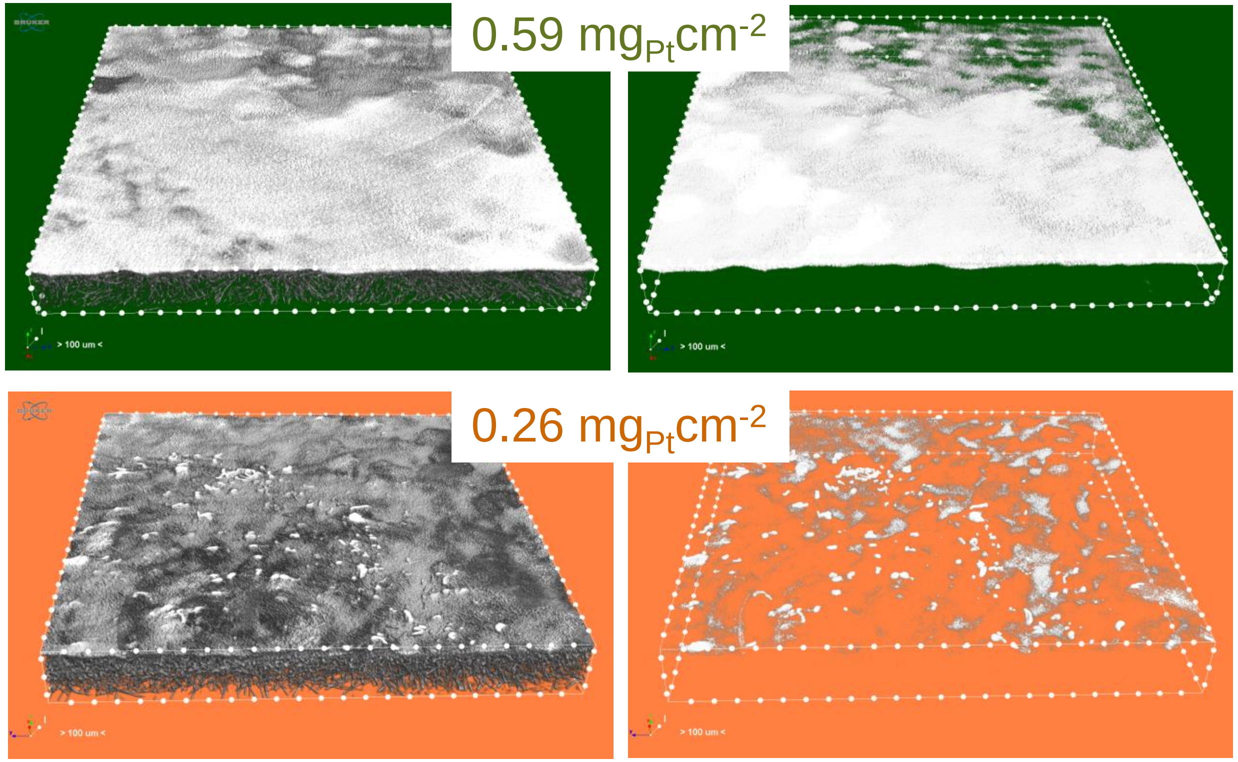
HALF-CELL AND SINGLE CELL STUDIES

Reduction of Platinum Contents in HT-PEM Electrodes

GDE Study
Conc. H_3PO_4
140 °C



GDE half-cell test at 160 °C in conc. H_3PO_4 with PBI membrane (Celtec®) with O_2 flow of 1.5 L min^{-1}

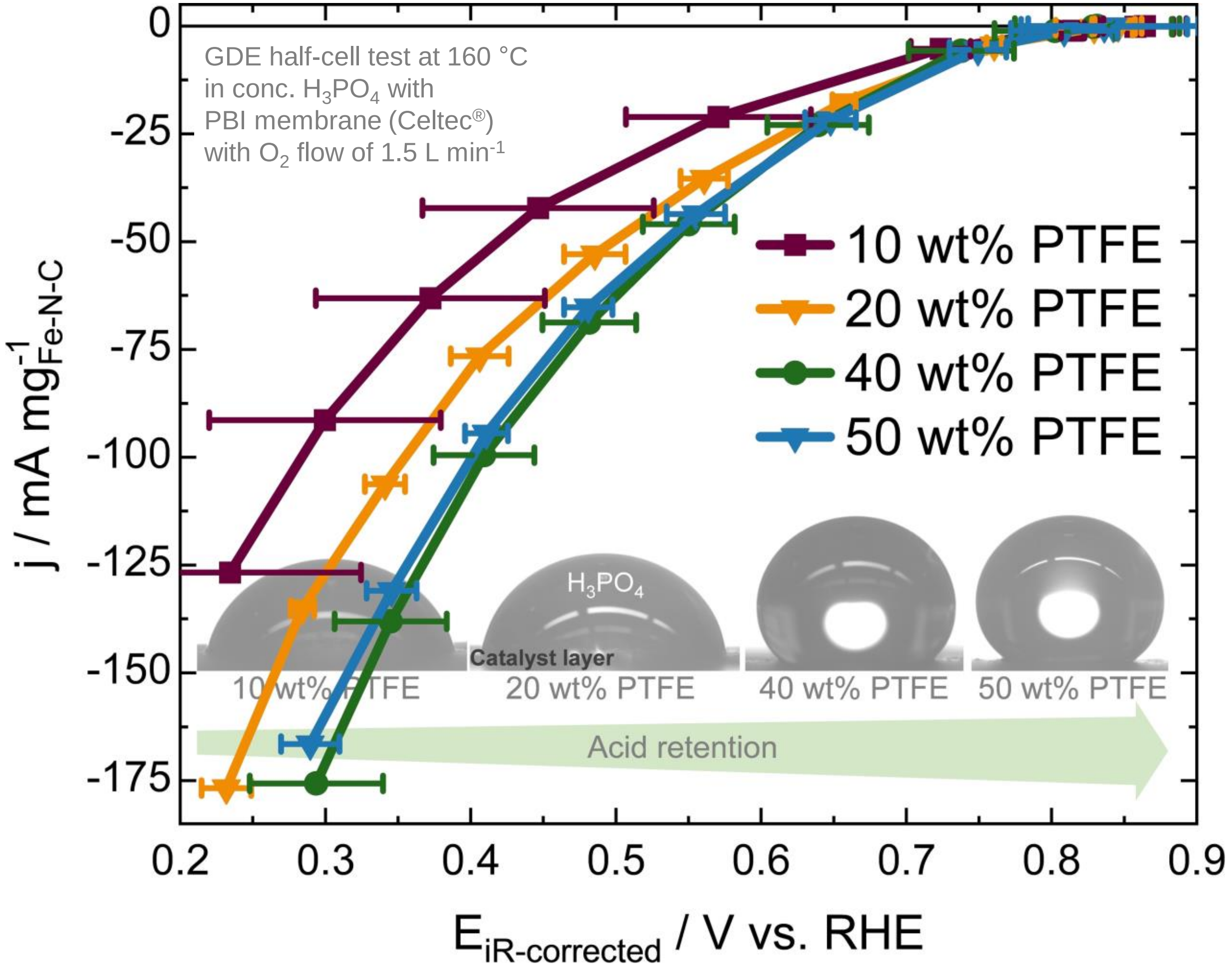


µ-Computed tomographic images of the whole GDEs (left) and catalyst layers only (right).

H. Schmies et al., J. Power Sources **2022**, Reduction of platinum loading in gas diffusion electrodes for high temperature proton exchange membrane fuel cell application: Characterization and effect on oxygen reduction reaction performance, 529, 231276.

Impact of PTFE of Fe-N-C-based HT-PEM Electrodes

GDE Study
Conc. H_3PO_4
160 °C

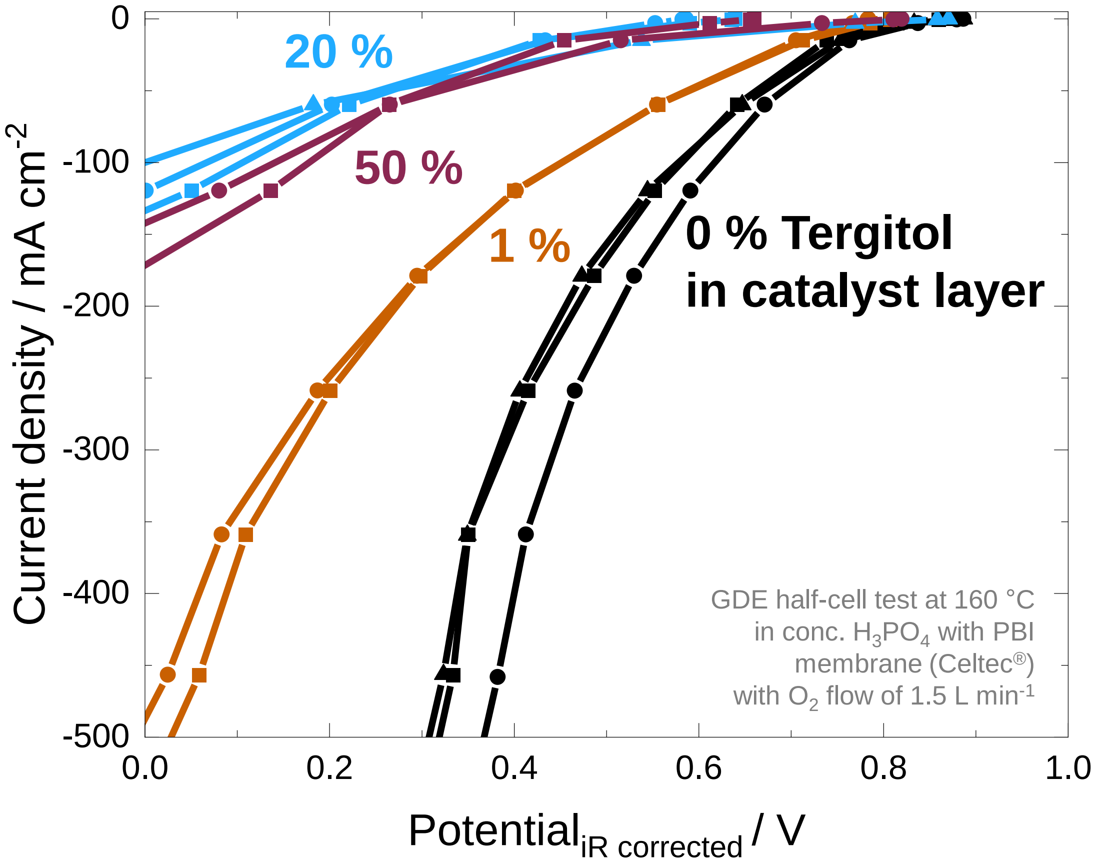


- Different PTFE contents in catalyst layer using commercial Fe-N-C (PMF-D14401, Pajarito Powder)
  - Minimum of 20 wt% PTFE in Fe-N-C catalyst layers mandatory
  - H_2O contact angle > 140 ° beneficial hydrophobic properties
  - Fe-N-C copes wide range of PTFE contents

T. Zierdt et al., ChemElectroChem **2024**, *Effect of Polytetrafluorethylene Content in Fe-N-C-Based Catalyst Layers of Gas Diffusion Electrodes for HT-PEM Fuel Cell Applications*, 11, e202300583.

Impact of Additive of Fe-N-C-based HT-PEM Electrodes

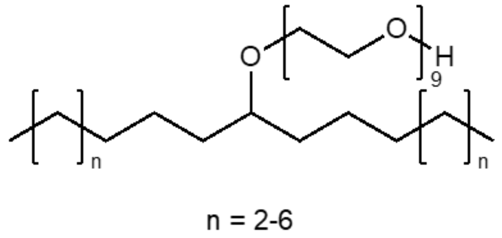
GDE Study
Conc. H_3PO_4
160 °C



GDE half-cell test at 160 °C in conc. H_3PO_4 with PBI membrane (Celtec®) with O_2 flow of 1.5 L min^{-1}

- Different additive contents in catalyst layer using commercial Fe-N-C (PMF-D14401, Pajarito Powder) and 50 wt% PTFE
  - Increase of surface hydrophilicity with increased Tergitol amount
  - Reduced conductivity and performance for GDEs with increased Tergitol amount

→ No positive effect for Fe-N-C based ink and catalyst layer



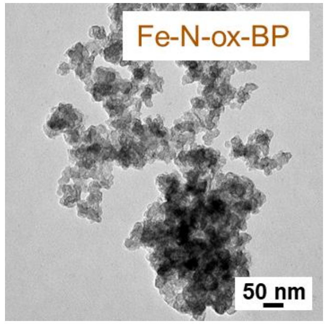
Tergitol™ 15-S-9

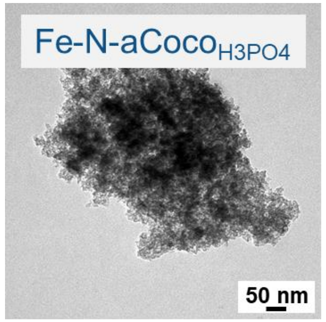
T. Zierdt et al., ECS Trans. **2024**, *Effect of Non-ionic Surfactant on Fe-N-C Catalyst Layers under HT-PEMFC Conditions*, in press.

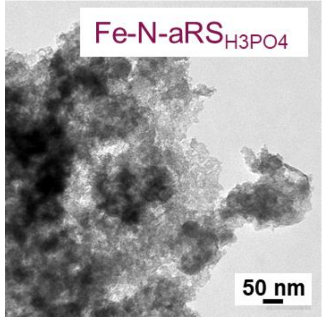
Effect of Carbon Support on Fe-N-C-based HT-PEM Electrodes

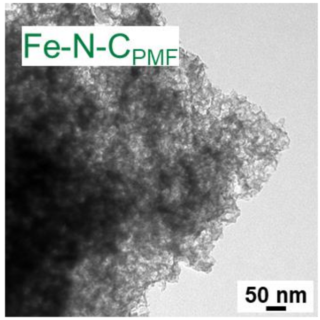
HT-PEM Single Cell Study
160 °C

Catalyst









C- Source

Black Pearls® 2000

H_3PO_4 activated Coconut Shell

H_3PO_4 activated Rye Straw

PAJARITO POWDER
FUEL CELL CATALYSTS

PMF-011904

Properties

	Support-based synthesis			Commercial
Fe:	1.3 wt.%	1.3 wt.%	1.2 wt.%	1.2 wt.%
Surface Area:	1280 m² g^{-1}	1117 m² g^{-1}	977 m² g^{-1}	588 m² g^{-1}
N+O content:	6.4 at.%	**19.7 at.%**	**19.3 at.%**	8.4 at.%
P content	-	**1.9 at.%**	**2.2 at.%**	-

Support-based synthesis

Commercial

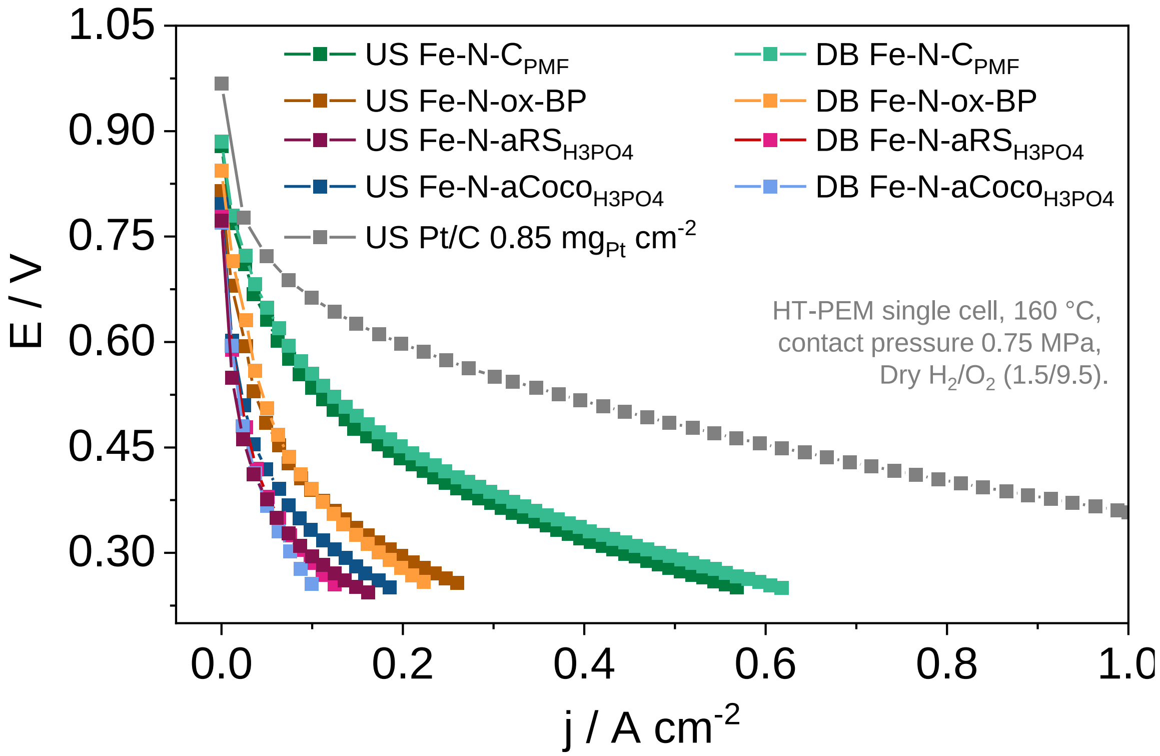
J. Hülstede et al., *Relevant Properties of Carbon Support Materials in Successful Fe-N-C Synthesis for the Oxygen Reduction Reaction: Study of Carbon Blacks and Biomass-Based Carbons*, Materials **2021**, 14, 1, 45.

J. Müller-Hülstede et al., ACS Appl. Energy Mater. **2021**, *Incorporation of Activated Biomasses in Fe-N-C Catalysts for Oxygen Reduction Reaction with Enhanced Stability in Acidic Media*, 4, 7, 6912.

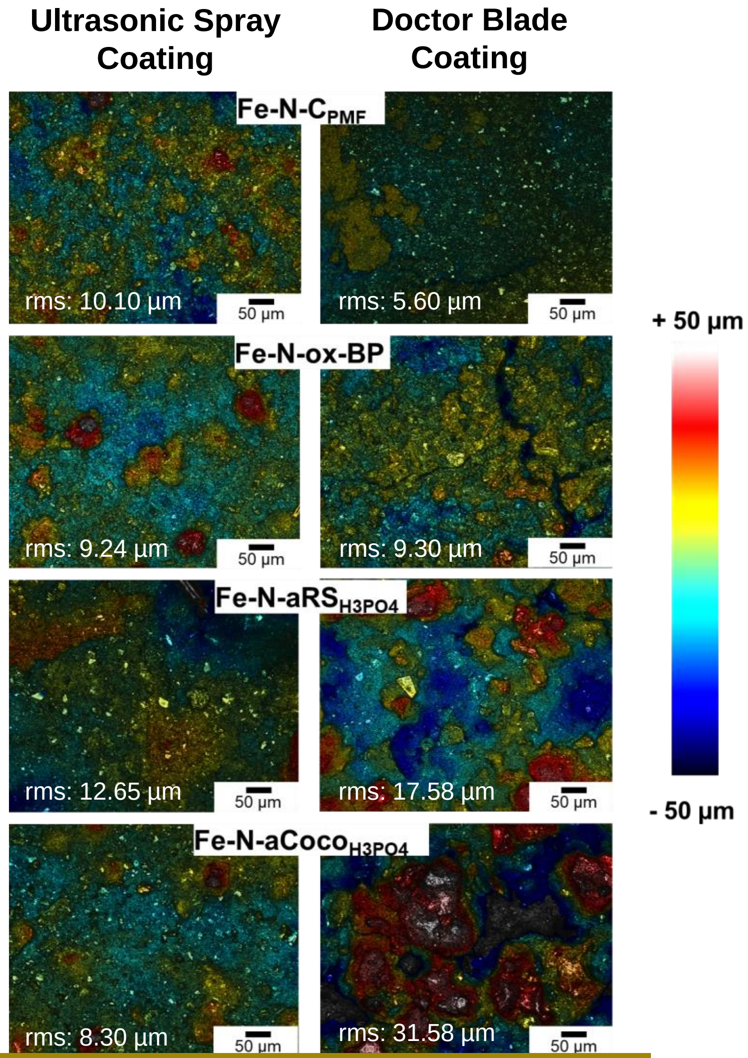
Effect of Carbon Support on Fe-N-C-based HT-PEM Electrodes

HT-PEM Single Cell Study 160 °C

- Implementing Fe-N-C catalysts in HT-PEM FC cathodes
  - Target loading of 3 mg cm^{-2}
  - 40 wt% PTFE
- Nature of the catalyst more relevant than electrode fabrication



HT-PEM single cell, 160 °C, contact pressure 0.75 MPa, Dry H_2/O_2 (1.5/9.5).



Confocal microscopic images of Fe-N-Cs.

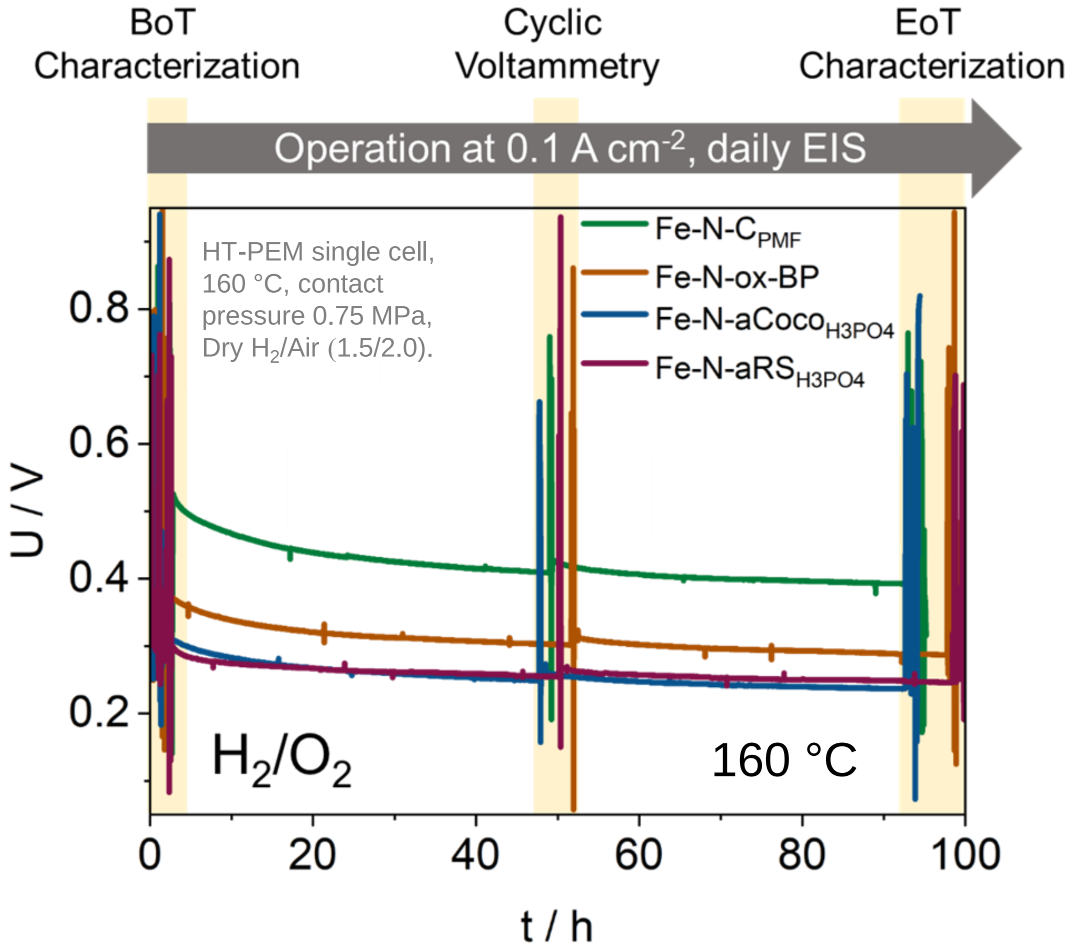
J. Müller-Hülstede et al., J. Power Sources **2022**, *Implementation of Different Fe-N-C Catalysts in High Temperature Proton Exchange Membrane Fuel Cells – Effect of Catalyst and Catalyst Layer on Performance*, 537, 231529.

Stability Limits of Fe-N-C-based HT-PEM FCs

HT-PEM Single Cell Study 160 °C

- Different initial performances
  - Catalyst layer inhomogeneity of biomass based Fe-N-Cs
- Rapid loss of voltage within first 24 h of operation for all MEAs (~ 27 %)
- Further losses 4-10 % mainly attributed to reactive oxygen species formation due to Fenton-like reaction

→Need for improved metal site and metal site incorporation and optimisation of catalyst layer



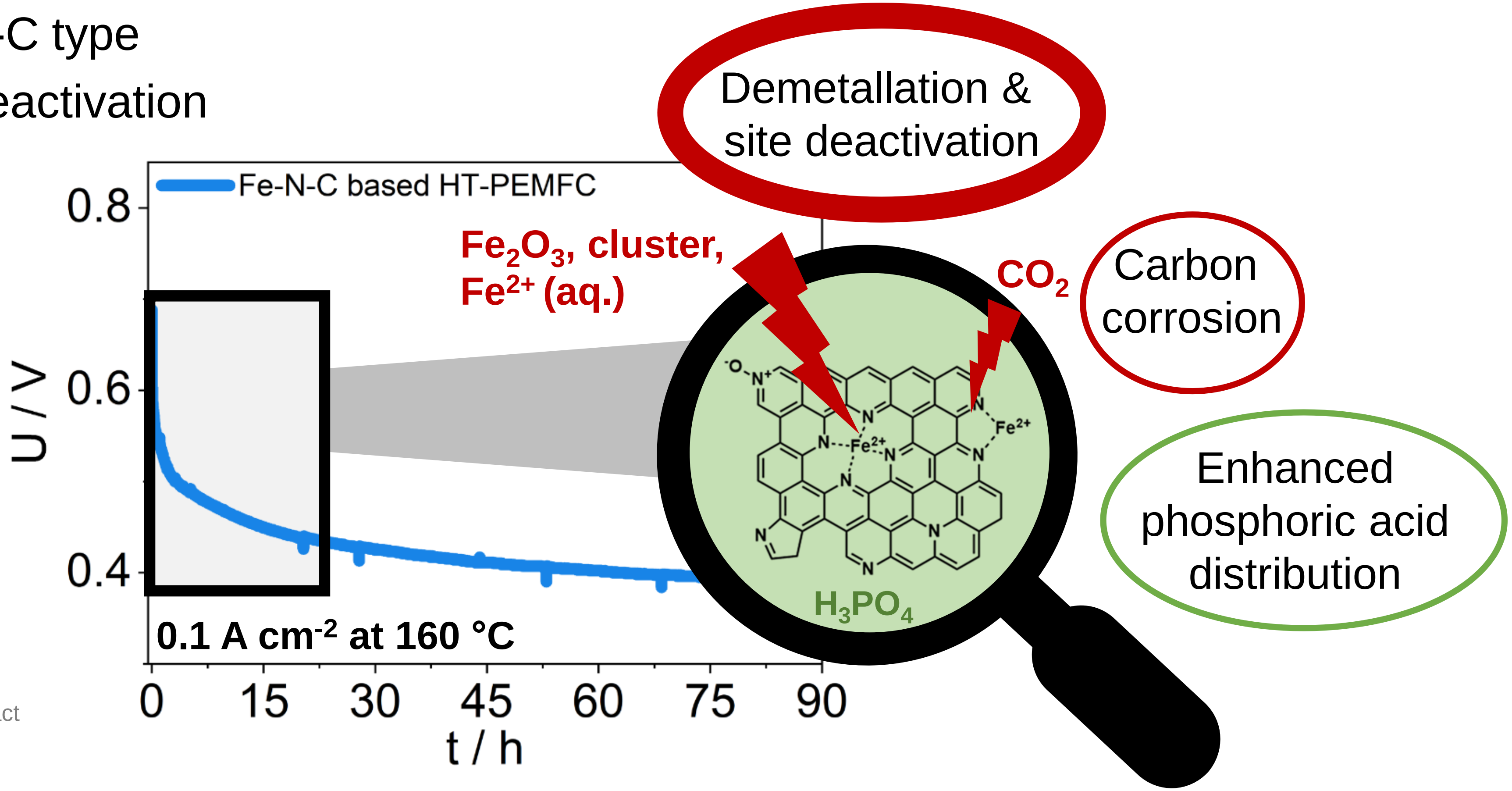
J. Müller-Hülstede et al., J. Power Sources **2022**, *Implementation of Different Fe-N-C Catalysts in High Temperature Proton Exchange Membrane Fuel Cells – Effect of Catalyst and Catalyst Layer on Performance*, 537, 231529.

J. Müller-Hülstede et al., Int. J. Hydrog. Energy **2023**, *What determines the stability of Fe-N-C catalysts in HT-PEMFCs?*, 50, Part C, 921.

Stability Limits of Fe-N-C-based HT-PEM FCs

HT-PEM Single Cell Study 160 °C

- Voltage decay in first 24 h of operation
  - Independent of Fe-N-C type
  - Attributed to Fe-N_x deactivation
- Fe-N-C stability independent of electrochemical analysis
- Cell activation in terms of acid distribution



Demetallation & site deactivation

Carbon corrosion

Enhanced phosphoric acid distribution

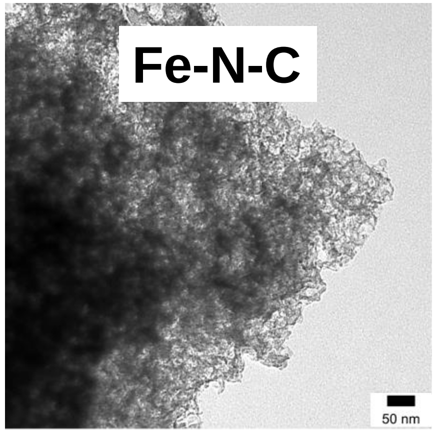
HT-PEM single cell testing: 160 °C, 0.1 A cm^{-2}, contact pressure 0.75 MPa, Dry H_2/Air (1.5/2.0).

J. Müller-Hülstede et al., Int. J. Hydrog. Energy **2023**, *What determines the stability of Fe-N-C catalysts in HT-PEMFCs?*, 50, Part C, 921.

Hybrid HT-PEM Electrodes

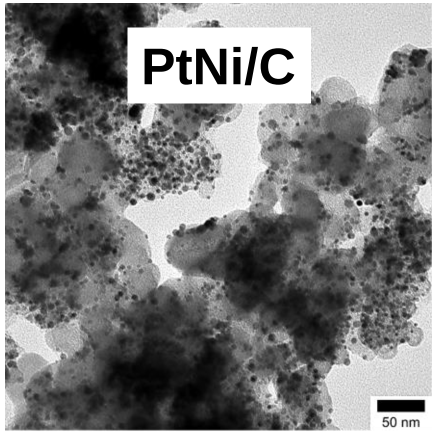
HT-PEM Single
Cell Study
160 °C

- Pt content reduction through incorporation of catalytic active filler
  → Studying the effect of Fe-N-Cs in Pt-alloy cathodes



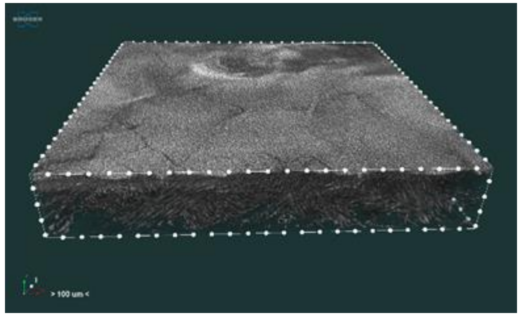
10 wt%

+ 40 wt% PTFE



50 wt%

Hybrid Gas Diffusion Electrode (GDE)



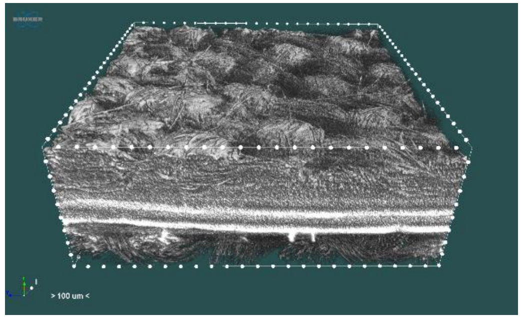
Ultrasonic spray coating of cathode on woven Celtec®-based gas diffusion layers followed by sintering at 350 °C for 10 minutes in N_2 atmosphere

GDE 1: 0.40 mg_{Pt} cm^{-2}

GDE 2: 0.65 mg_{Pt} cm^{-2}

Standard: 0.85 mg_{Pt} cm^{-2}

Membrane-Electrode-Assembly (MEA)

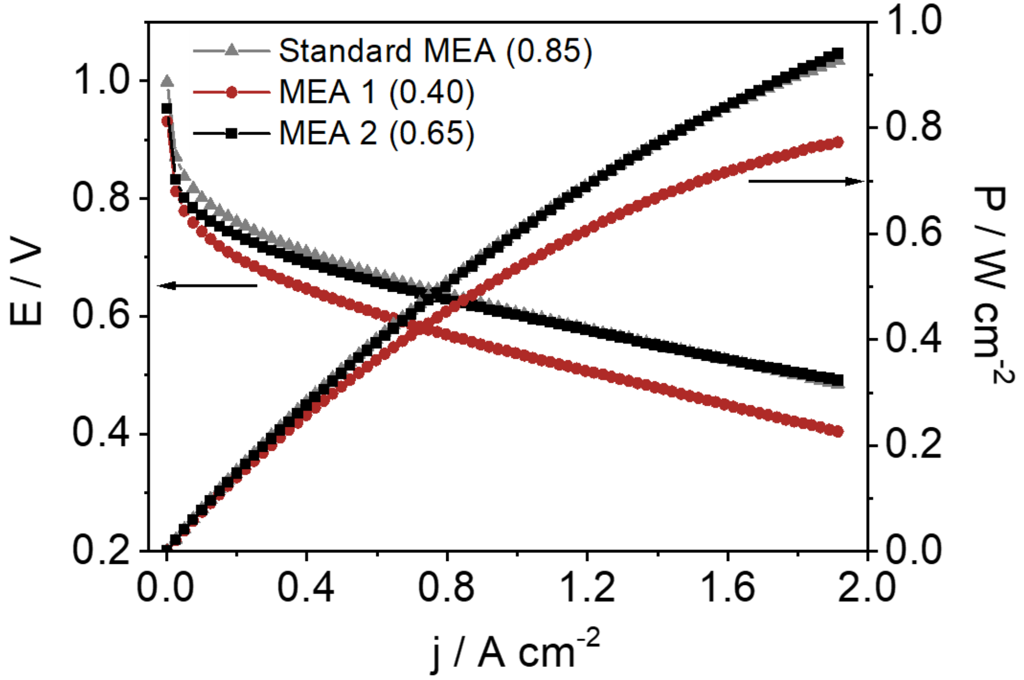


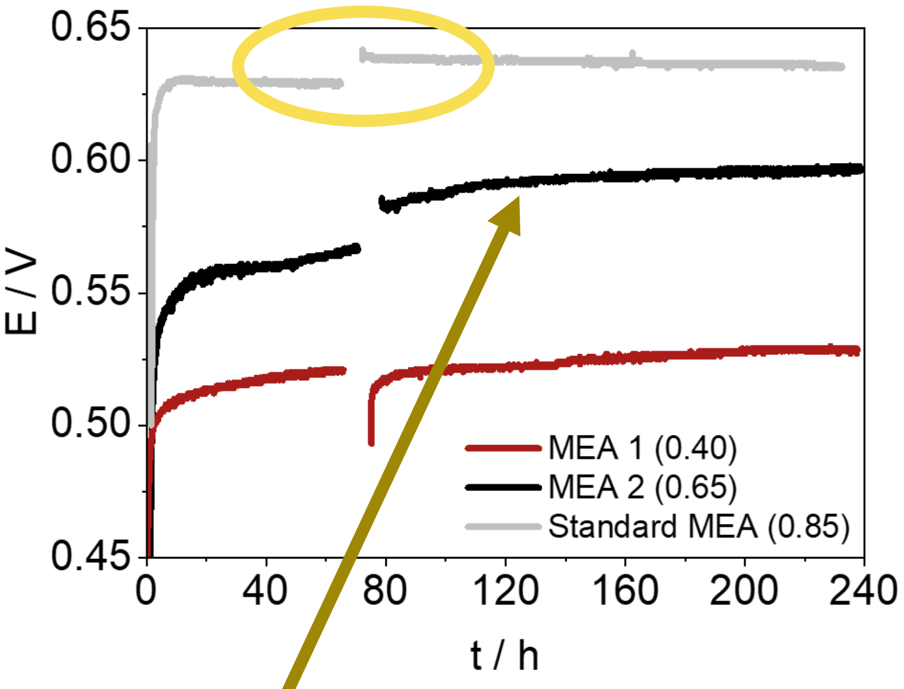
MEA-assembling analogous to Celtec®-technology with active area of 20.25 cm² and reduced Pt-content on cathode site

J. Müller-Hülstede et al., ChemSusChem **2023**, *Towards the Reduction of Pt Loading in High Temperature Proton Exchange Membrane Fuel Cells – Effect of Fe-N-Cs in Pt-alloy Cathodes*, e202202046.

Hybrid HT-PEM Electrodes

HT-PEM Single Cell Study 160 °C





HT-PEM single cell testing: 160 °C, contact pressure 0.75 MPa, Dry H_2/Air (1.5/2.0); polarization curves: H_2/O_2 (1.5/9.5)

Voltage increase in case of conventional Pt-based MEA immediately after electrochemical measurements

Slow voltage increase caused by electrolyte redistribution in presence of Fe-N-C

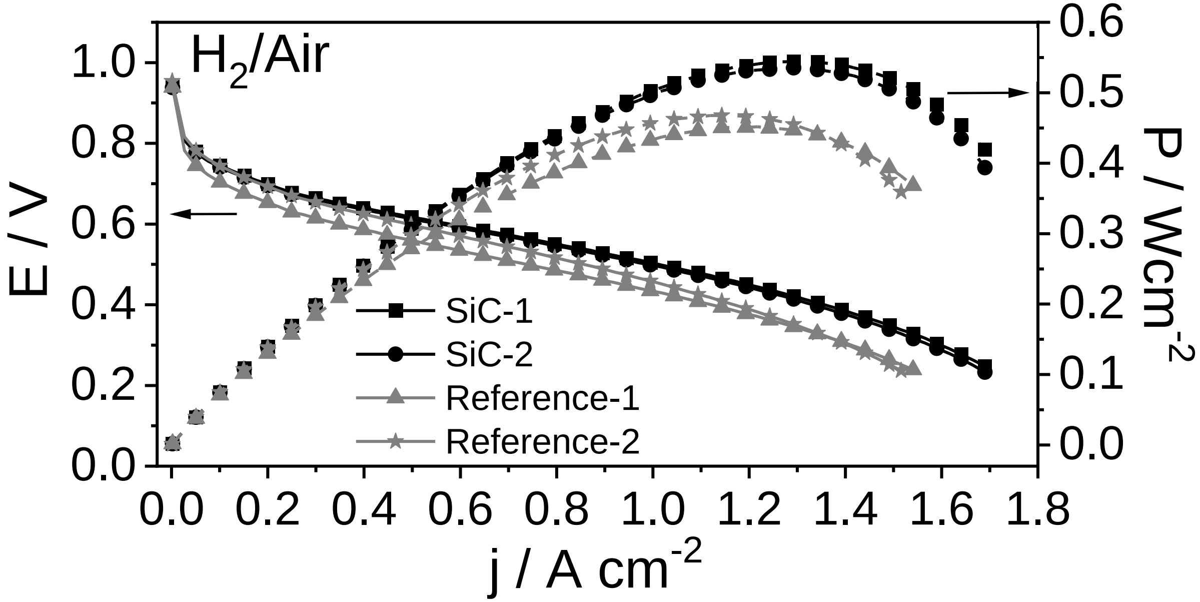
J. Müller-Hülstede et al., ChemSusChem **2023**, *Towards the Reduction of Pt Loading in High Temperature Proton Exchange Membrane Fuel Cells – Effect of Fe-N-Cs in Pt-alloy Cathodes*, e202202046.

HT-PEM FC – MEMBRANES

Silicon carbide based HT-PEM membranes
Initial Performance

HT-PEM Single Cell Study 160 °C

- Celtec®-based study with BASF
  - Standard Celtec® P1200 as reference
  - Addition of 2 wt% SiC to patented PPA based membrane fabrication
- SiC-based MEA better performance



HT-PEM single cell testing: 160 °C, contact pressure 0.75 MPa, Dry H_2/Air (1.5/2.0).

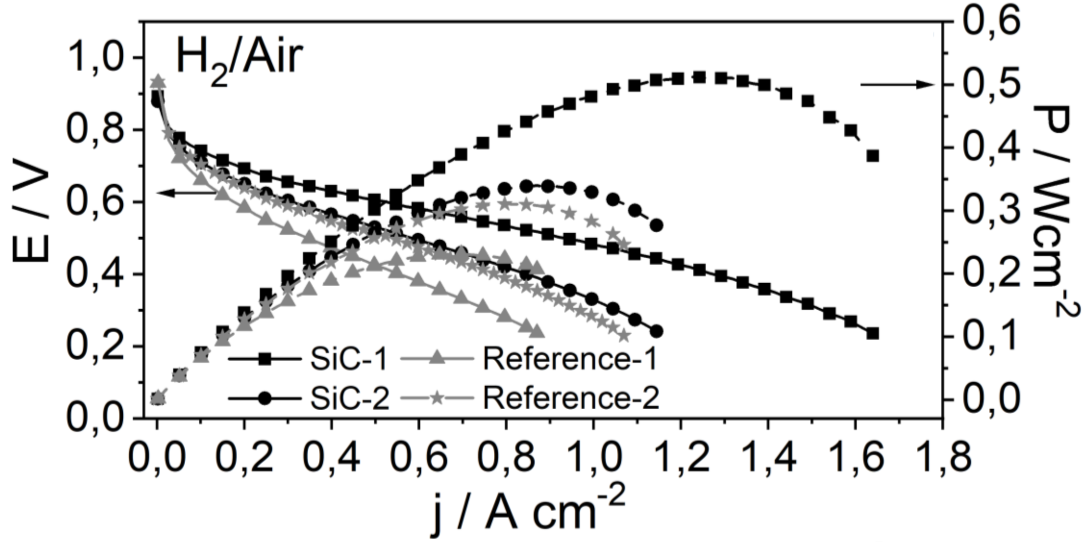
D. Schonvogel et al., J. Power Sources **2024**, *Performance and durability of high temperature proton exchange membrane fuel cells with silicon carbide filled polybenzimidazole composite membranes*, 591, 233835.

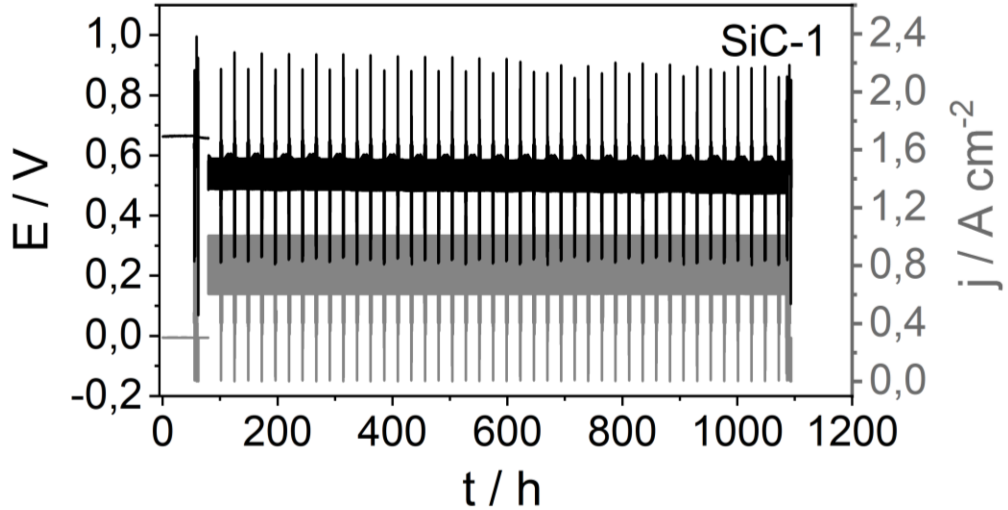
Silicon carbide based HT-PEM membranes
Degradation over Time

HT-PEM Single Cell Study 160 °C

- 1,000 h of load cycling
  - 4 min at 0.6 A/cm² & 16 min at 1.0 A/cm²
  - SiC-based lower degradation rates (<65 µV h^{-1} for SiC, >100 µV h^{-1} for Celtec)
  - But: Lower OCVs in case of SiC
- Lower acid losses in case of SiC

HT-PEM single cell testing: 160 °C, contact pressure 0.75 MPa, dry H_2/Air (1.5/2.0).



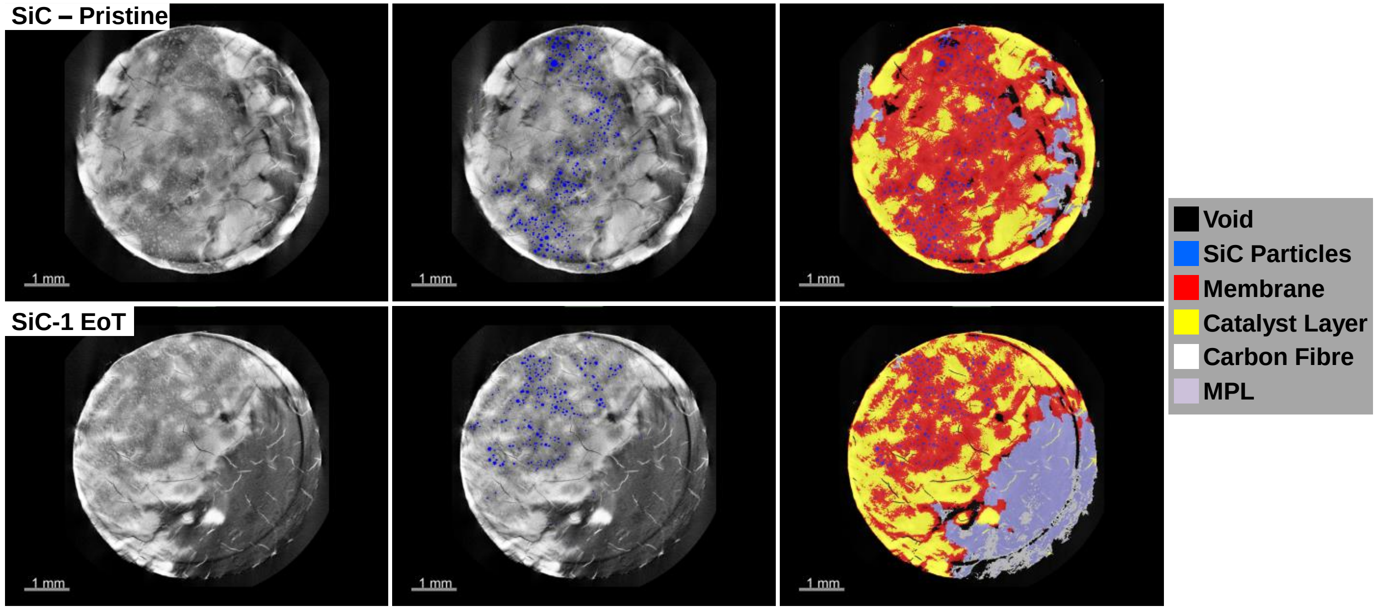


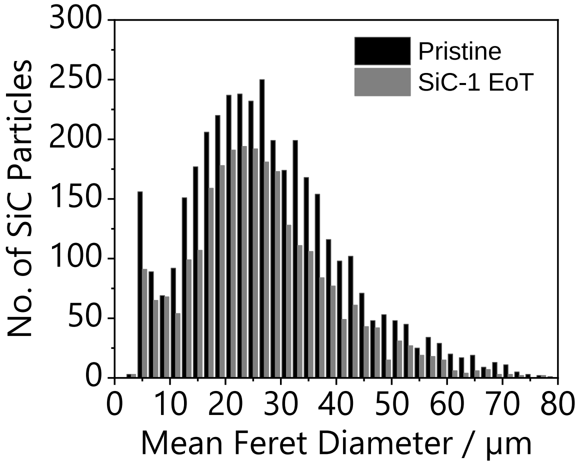
D. Schonvogel et al., J. Power Sources **2024**, *Performance and durability of high temperature proton exchange membrane fuel cells with silicon carbide filled polybenzimidazole composite membranes*, 591, 233835.

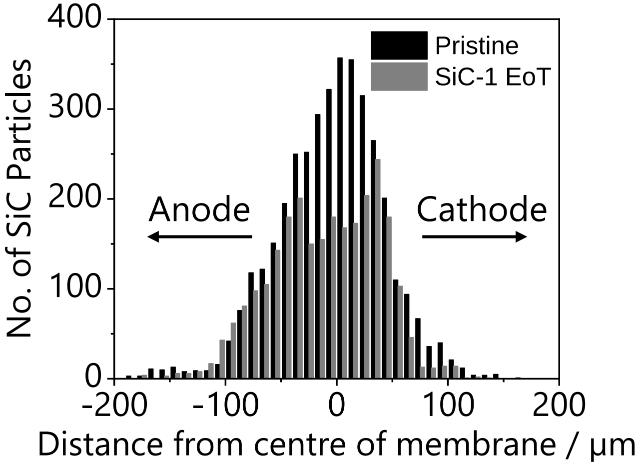
Silicon carbide based HT-PEM membranes
Computer Tomography with Machine Learning

HT-PEM Single Cell Study
160 °C

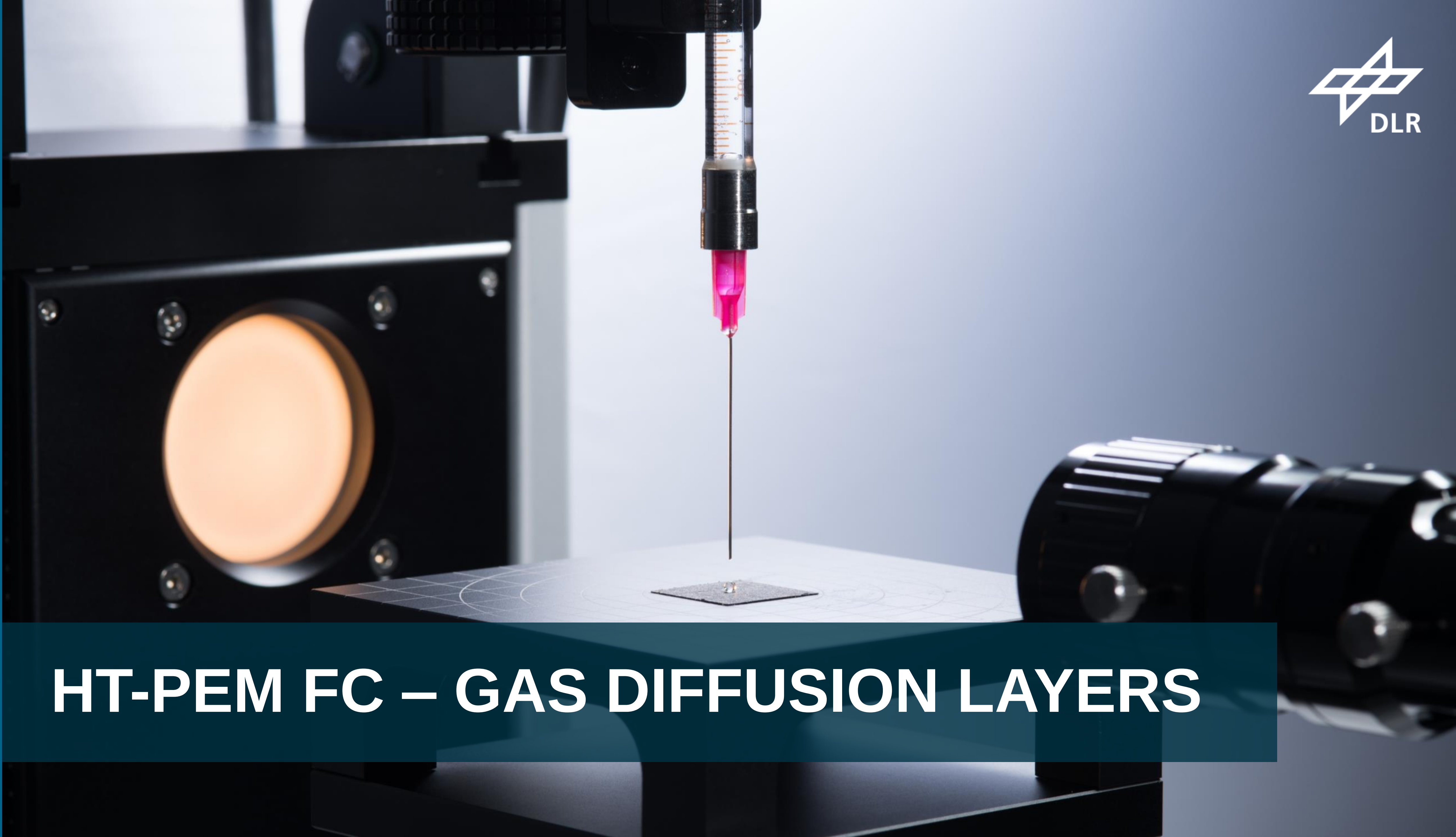
- Cooperation with UNSW Sidney
- Lower membrane thinning using SiC
- Evidence of mobility and redistribution of SiC particles







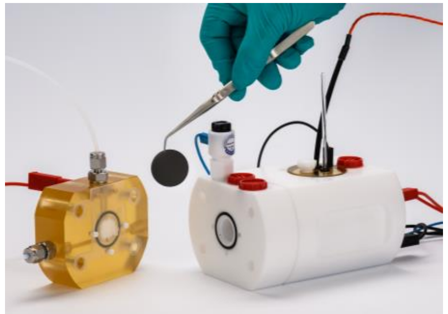
D. Schonvogel et al., J. Power Sources **2024**, *Performance and durability of high temperature proton exchange membrane fuel cells with silicon carbide filled polybenzimidazole composite membranes*, 591, 233835.



HT-PEM FC – GAS DIFFUSION LAYERS

Ex-situ GDL Ageing under HT-PEM Conditions
Method Development

GDL Study
Conc. H_3PO_4
160 °C



GDE half-cell test at 160 °C in conc. H_3PO_4 with O_2 flow of 1.5 L min^{-1}

CV before ageing
20 mV s^{-1},
N_2, 0 to 1,2 V

→

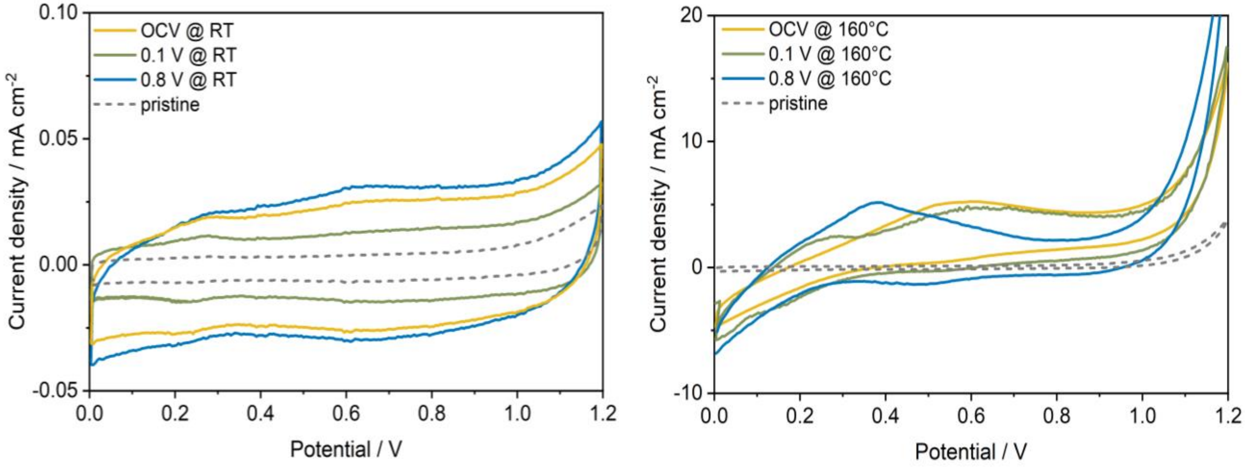
Ageing
OCV / 0,1 V / 0,8 V / 1,2 V
Cycl. 0,6-1,2 V, N_2/O_2

→

CV after ageing
20 mV s^{-1},
N_2, 0 to 1,2 V

Post mortem analysis: ICP-MS, confocal microscopy, contact angle, CT, C-AFM, SEM-EDS

- Effect of higher temperature (oxidation / higher hydrophilicity)
- Increasing corrosion with higher potential and /or cycling
- Independence of gas atmosphere

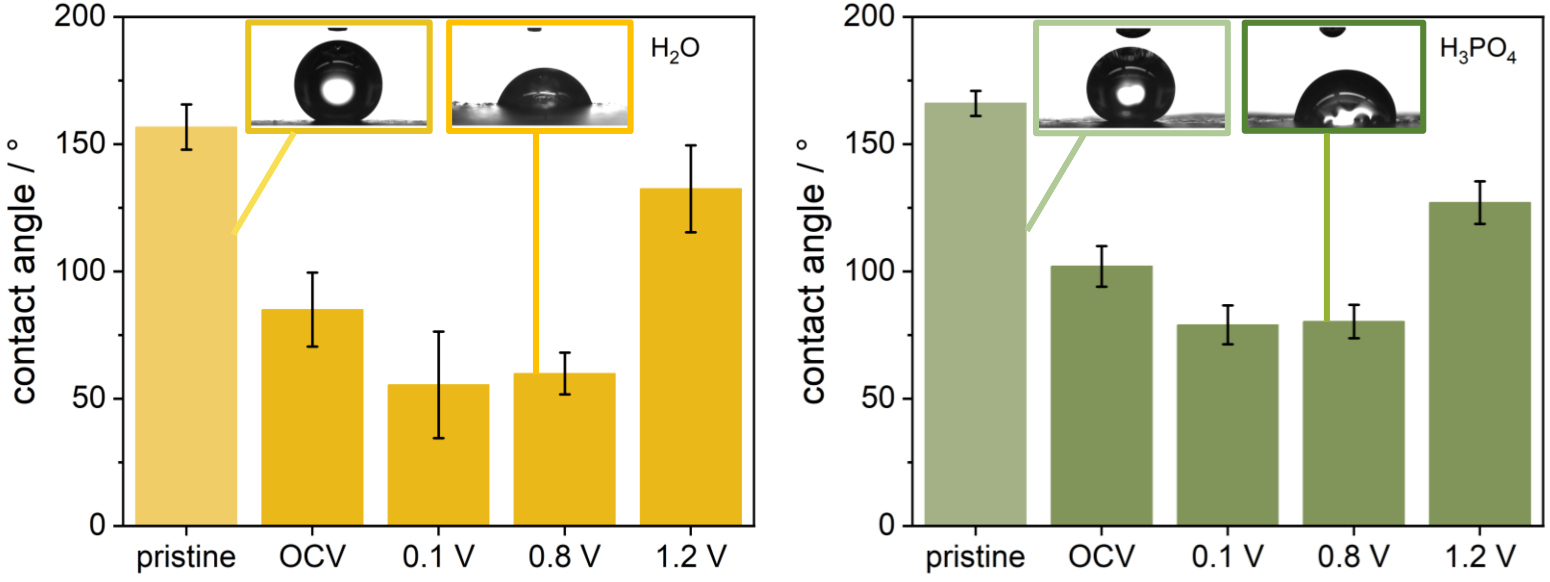


N. Pilinski et al., ECS Trans. **2023**, *Investigation of Ageing Methods on Gas Diffusion Layers for HT-PEMFC Application*, **112,** 257.

Ex-situ GDL Ageing under HT-PEM Conditions

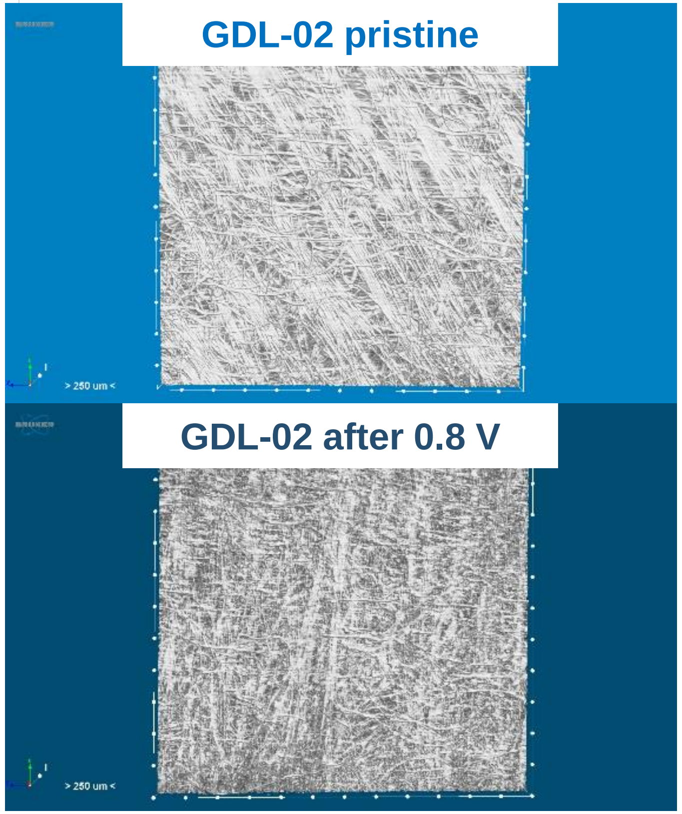
Surface Analysis after Aging

GDL Study
Conc. H_3PO_4
160 °C



→ Loss of PTFE as hydrophobic component **AND / OR** carbon corrosion

- Surface change after 0.8 V visible using CT



N. Pilinski et al., ECS Trans. **2023**, *Investigation of Ageing Methods on Gas Diffusion Layers for HT-PEMFC Application*, **112,** 257.

ACKNOWLEDGMENTS

Collaborators

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University of Bremen: Michaela Wilhelm

Leibniz University Hannover: René Lucka, Franz Renz

FZ Jülich: Marc Heggen

Projects

QM-GDL

HT-PEM 2.0

LaBreNA

DisCO2very

Federal Ministry
for Economic Affairs
and Climate Action

HT 2.0

Federal Ministry
for Digital
and Transport

NOW
NOW-GMBH.DE

Federal Ministry
of Education
and Research

DLR

THANK YOU FOR YOUR ATTENTION!