FRAUNHOFER INSTITUTE FOR SOLAR ENERGY SYSTEMS ISE
DIVISION »HYDROGEN TECHNOLOGIES«

Ex-situ characterisation tools for materials and components in PEM water electrolysis and fuel cells

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Ex situ analysis of materials and components

Overview

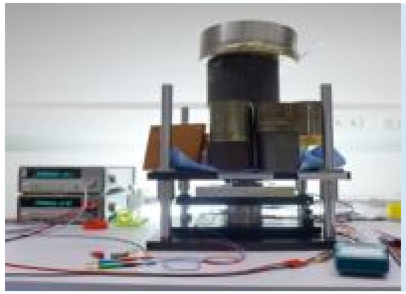
Capillary flow porometry (CFP) for through-plane gas permeability of porous media

Porosimetry for absolute and relative in-plane gas and liquid permeability

Environmental Scanning Electron Microscopy (ESEM) and Energy Dispersive X-ray Spectroscopy (EDS)

High temperature and Near ambient pressure X-ray photoelectron spectroscopy (HT NAP-XPS)

X-ray computed tomography (Micro CT)



Electrical conductivity and interfacial contact resistance (ICR) and force-displacement measurements

Inductively coupled plasma mass spectroscopy (ICP –MS) for element analysis of liquids

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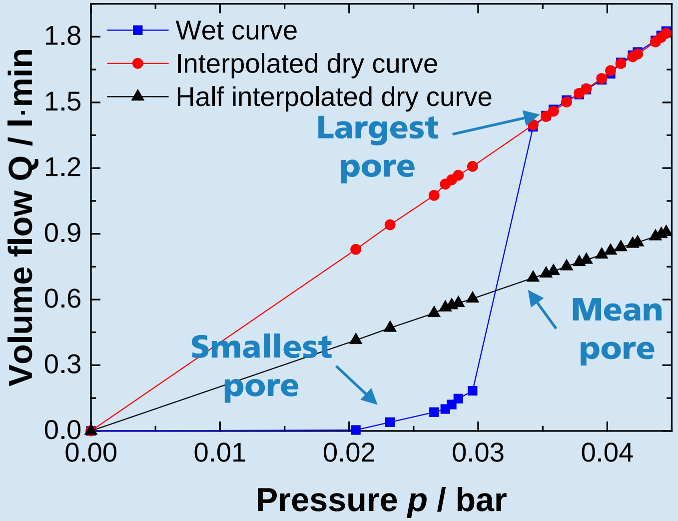
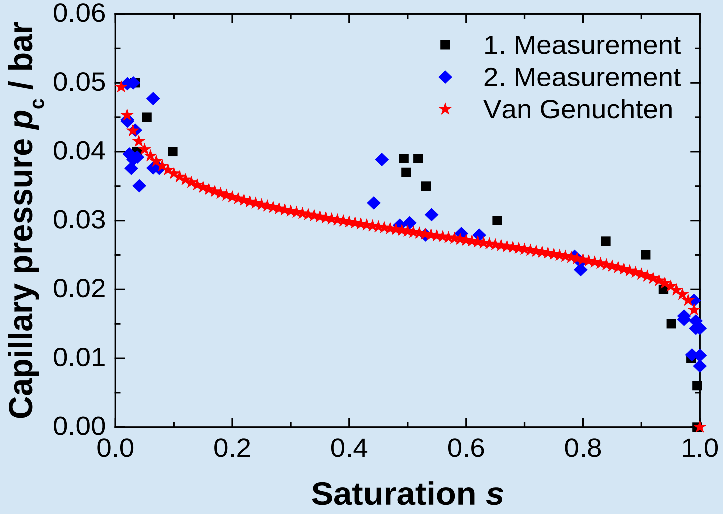
Capillary flow porometry for porous media

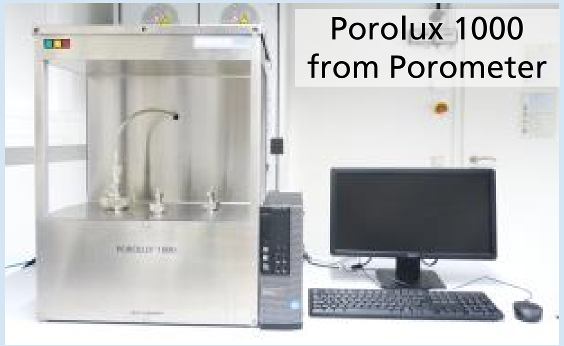
Gas-liquid displacement porometry with pressure step/stability method:

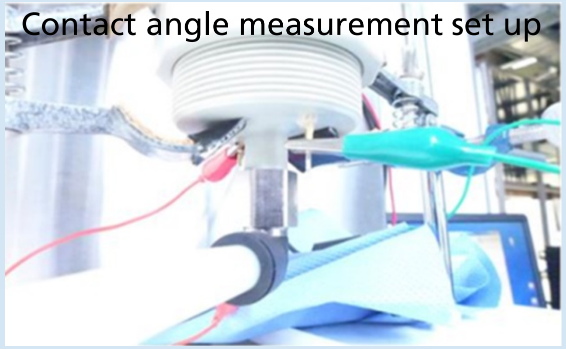
- Determination of pore diameter:
  - Largest / mean / smallest
- Through-plane gas permeability
- Relationship capillary pressure vs saturation
- Inner (mean) contact angle

➢ Important parameters for PTL design









Bromberger, K., Ghinaiya, J., Lickert, T., Fallisch, A., Smolinka, T.: Hydraulic ex situ through-plane characterization of porous transport layers in PEM water electrolysis cells, Int. J. Hydrogen Energy, Vol. 43, Issue 5, 2018, 2556-2569, https://doi.org/10.1016/j.ijhydene.2017.12.042.

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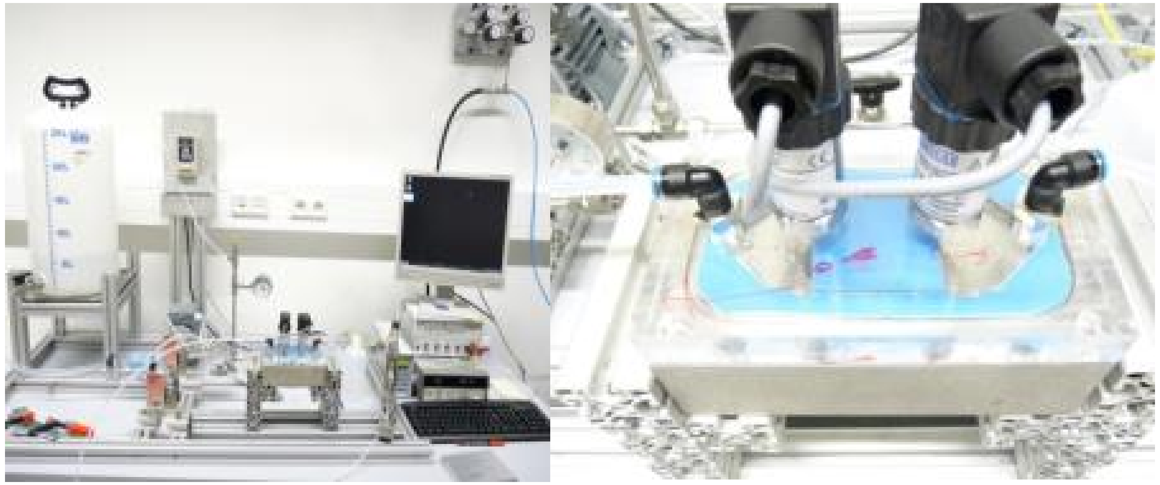
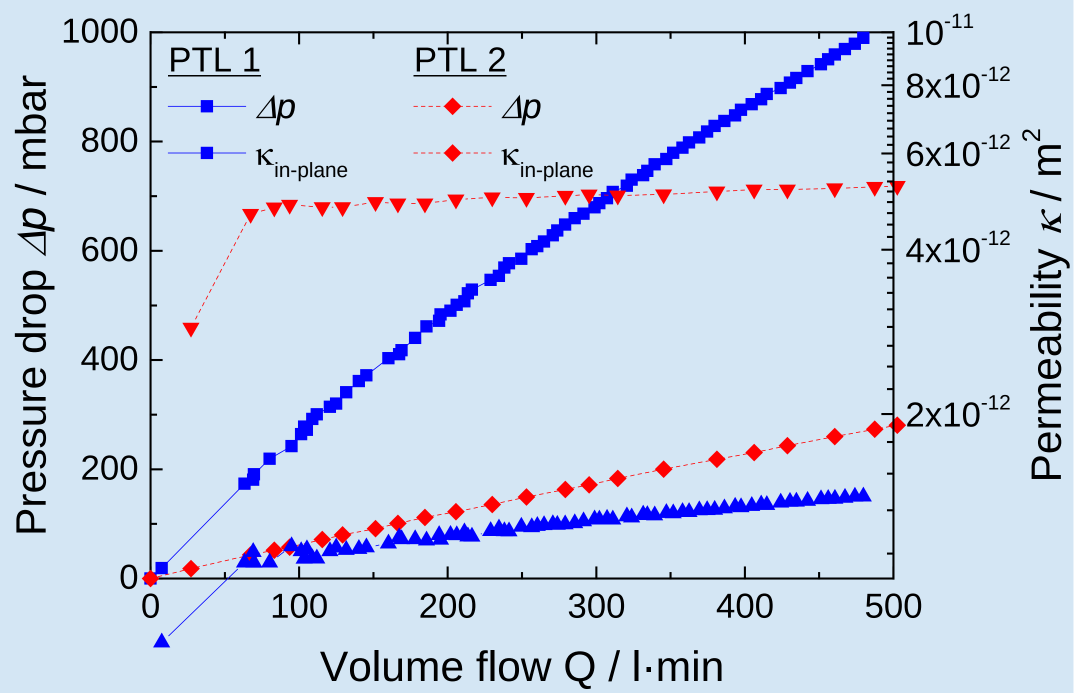
In plane permeability of porous media

- Absolute and relative in-plane gas and liquid permeability
- Test set up with a cell area of 25 cm²
- Method established for GDL / PTL etc. using Darcy or Darcy-Forchheimer law





In-plane pressure difference and gas permeability for different gas flow rates of sintered titanium based PTLs

Test set up for in-plane permeability measurements and detailed view on test cell

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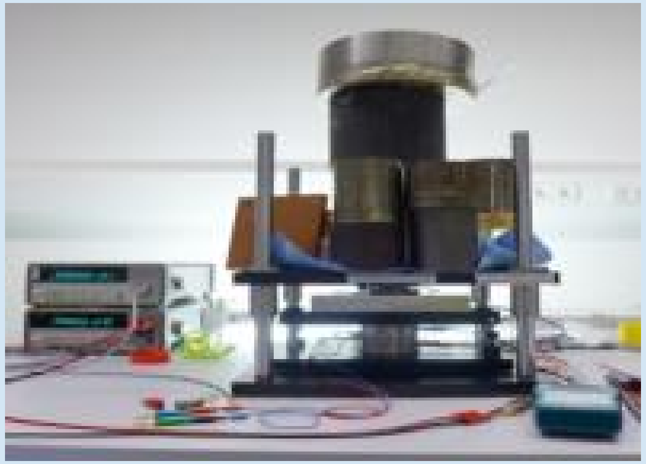
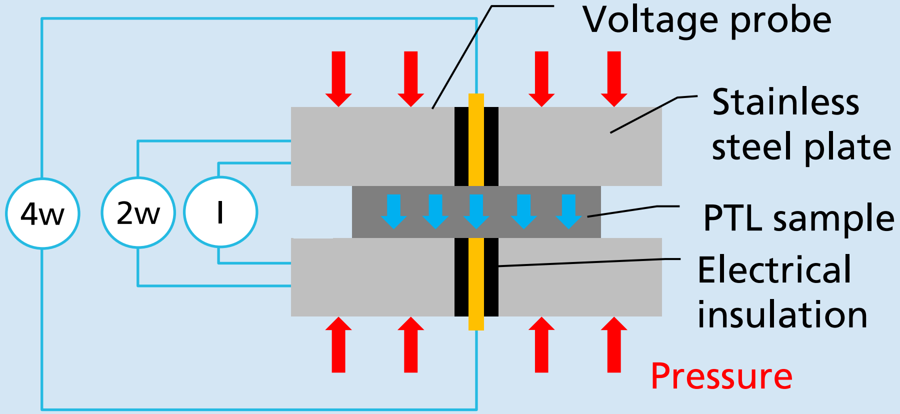
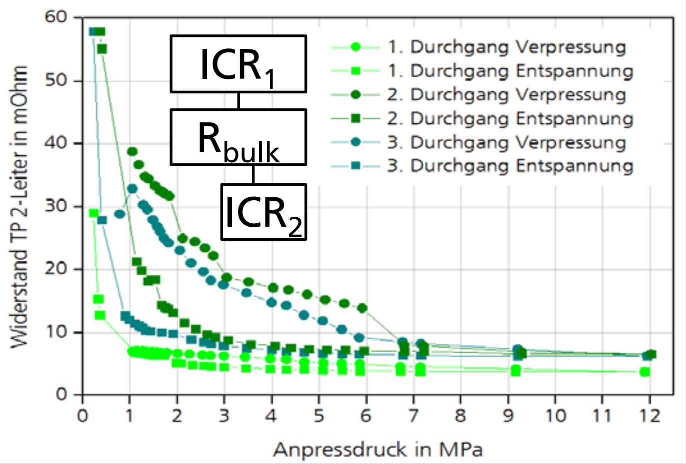
Electric conductivity and interfacial contact resistance

Understanding of internal cell resistance and individual contributions is crucial for stack design

- Measurement of in-plane and through-plane resistance/conductivity as function of compression
- Interfacial Contact Resistance (ICR) of passivation layers
- Thickness measurement by eddy current sensors (resolution 3 µm)
- Combination of clamping pressure and thickness measurement allows a force/displacement analysis







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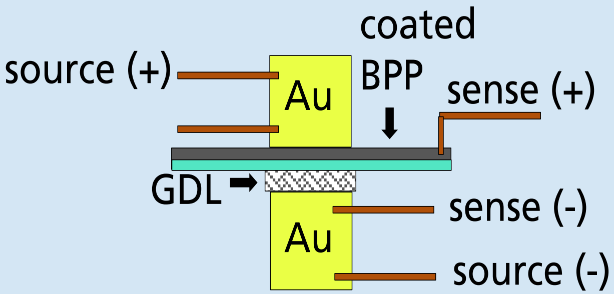
Through-plane resistance and interfacial contact resistance

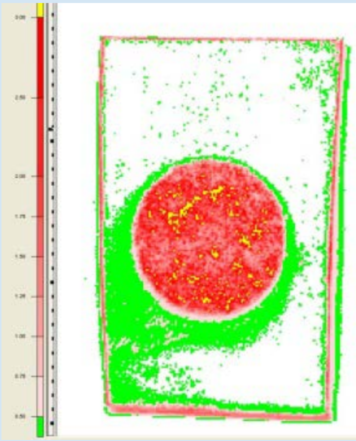
- Measurement of through-plane resistance of different materials
- Interfacial contact resistance (ICR) between bipolar plate and GDL
- Bulk resistance of GDL
- Thickness measurement

Measurement setup

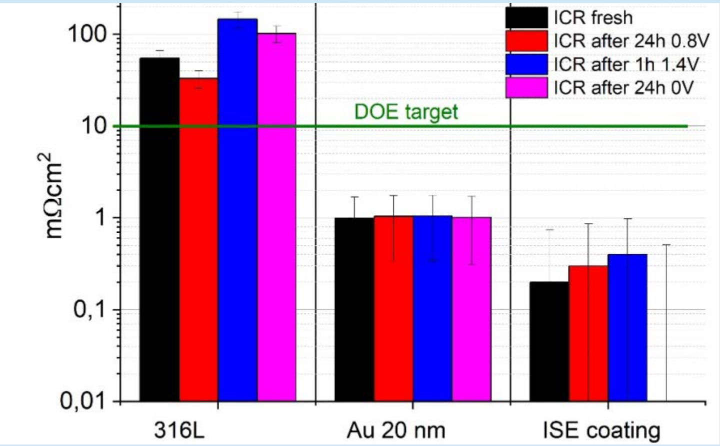
Measurement principle



Homogeneous pressure distribution



ICR of uncoated and coated SS 316 L before & afeter electrochemical testing



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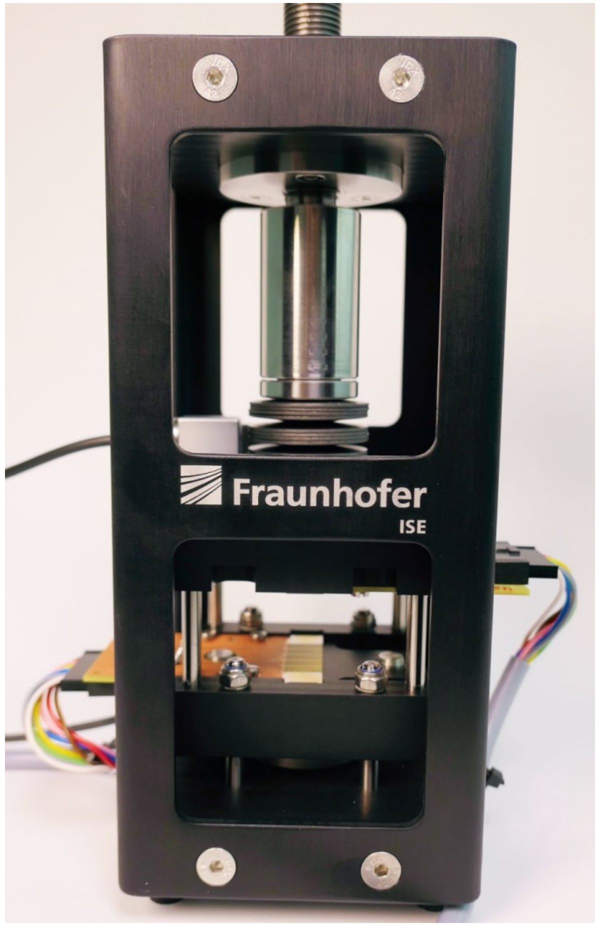
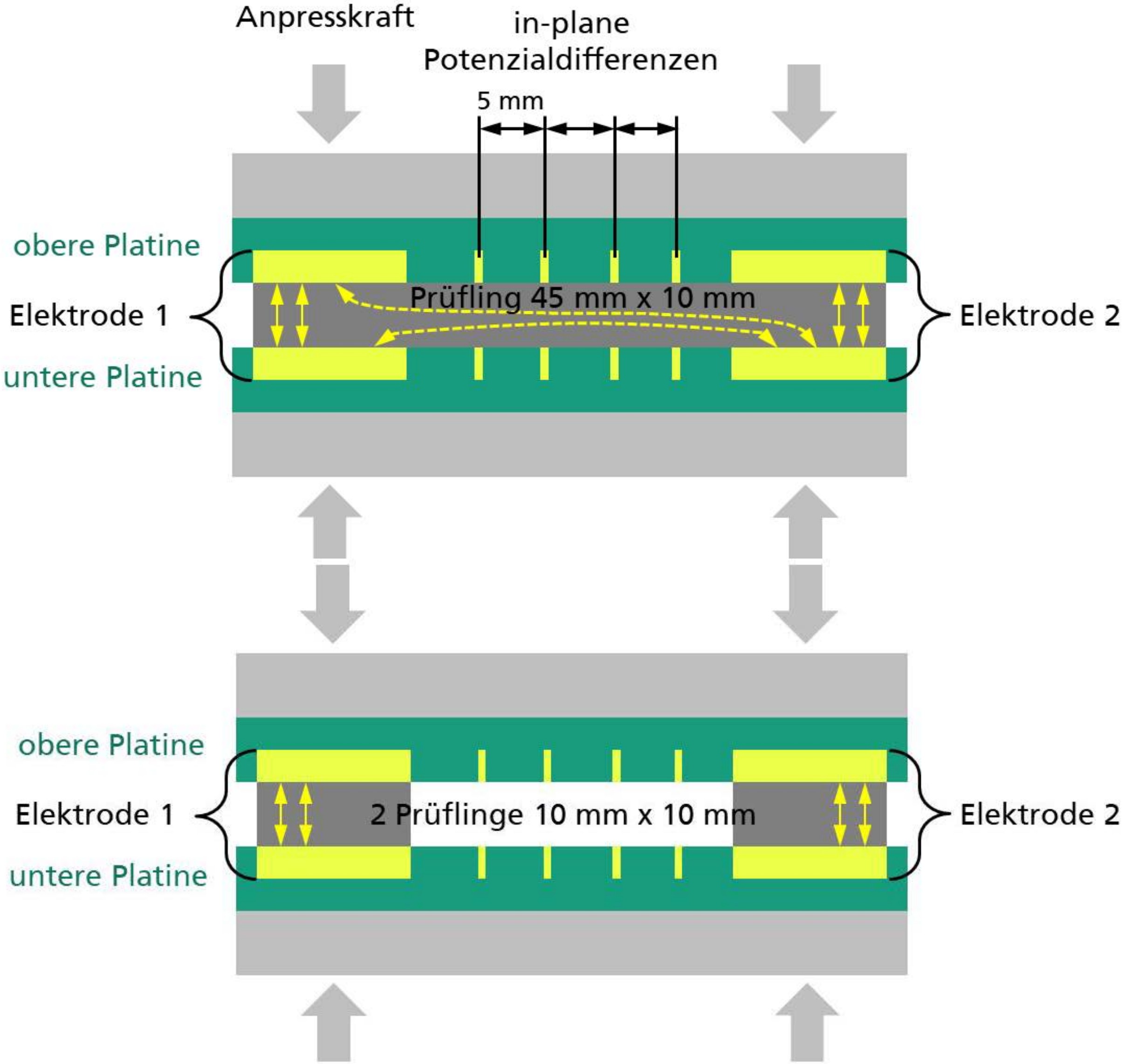
Inplane & through-plane resistance





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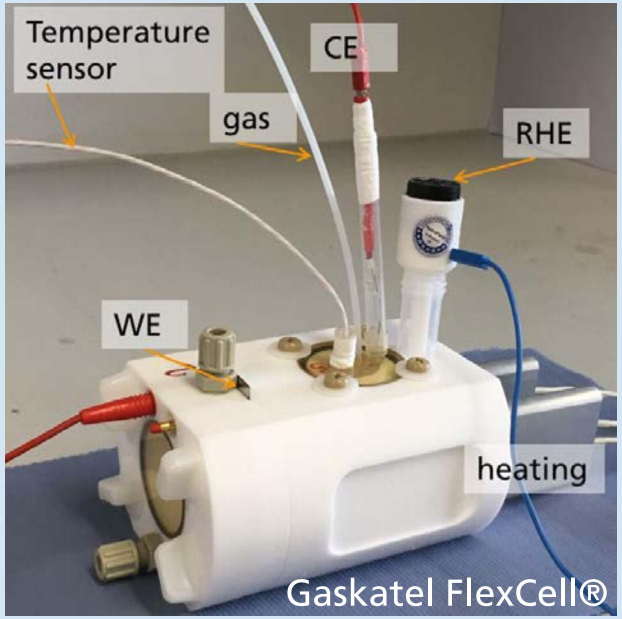
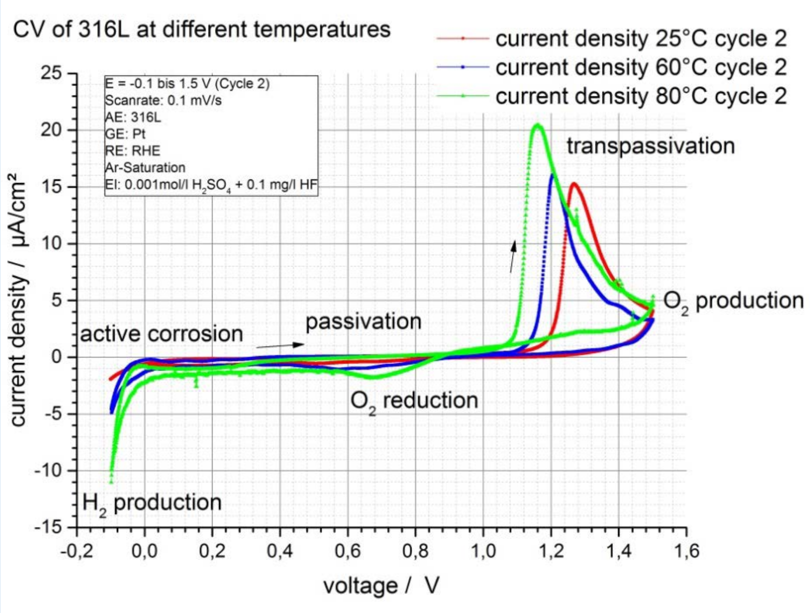
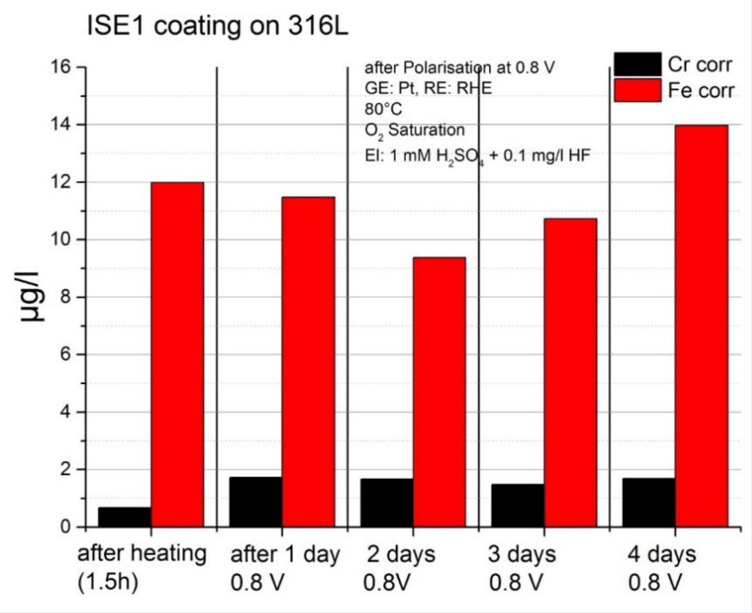
Electrochemical characterization of bipolar plate materials



Test cell made of PTFE, integrated heating, gas (Ar or O_2), working electrode (WE) (sample), reference electrode (RHE), counter electrode (CE).



Cyclic voltammogramm of stainless steel at different temperatures, electrolyte: 0.001 M H_2SO_4+0.1 mg HF



Electrochemical measurements is combined with elemental analysis of the electrolyte with ICP-MS (here during potentiostatic test at 0.8 V for 4 days), SEM/EDX analysis, and contact resistance measurement.

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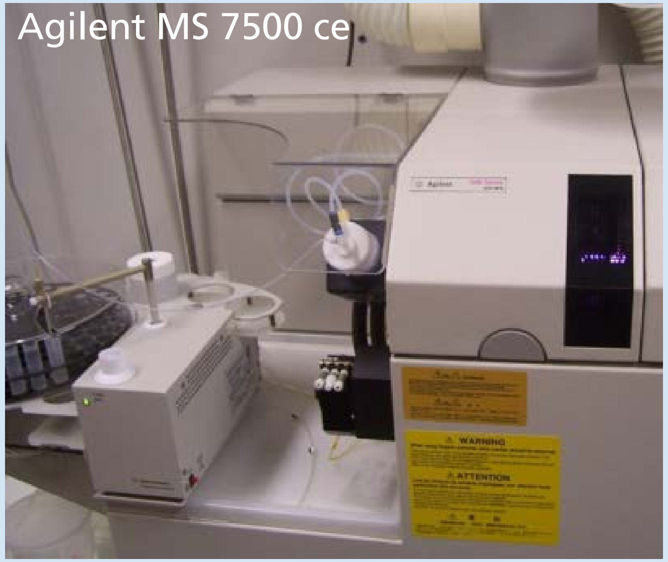
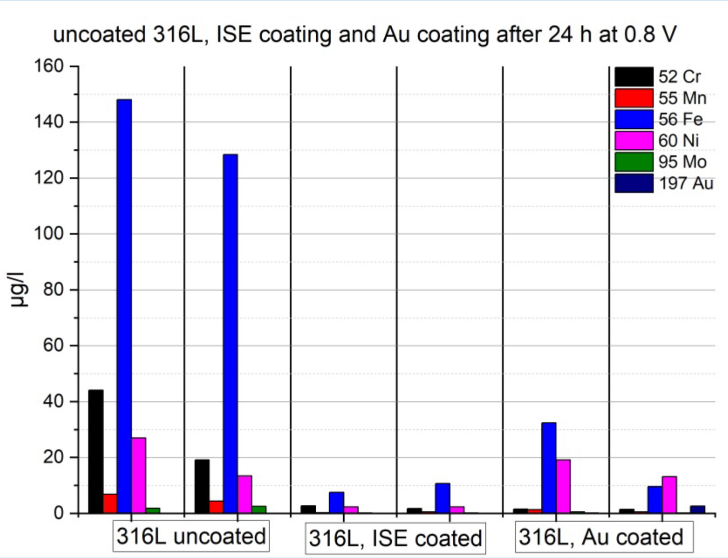
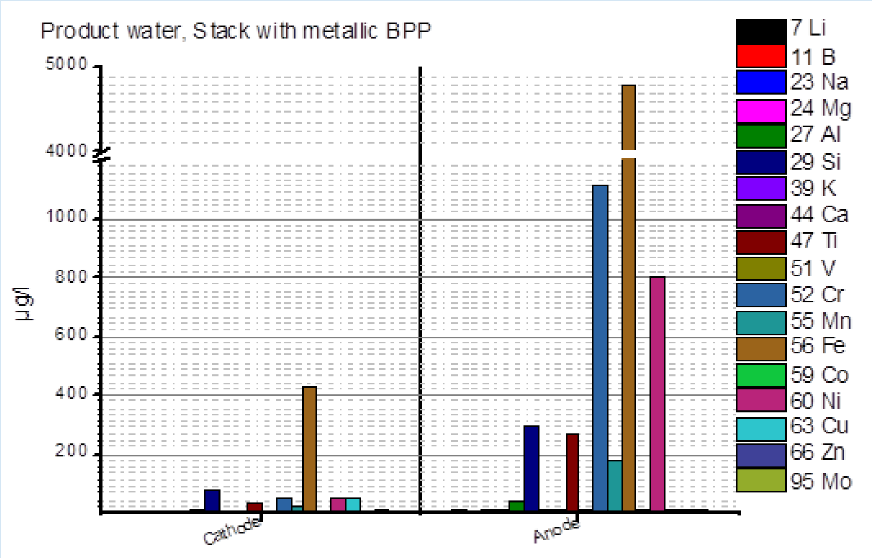
Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS)







Element analysis of liquids with ICP-MS. In Ar-plasma the molecules are destroyed and ionised. In the mass spectrometer the ions of every mass are counted.

ICP-MS analysis of electrolyte after ex-situ corrosion test (24 h at 0.8 V) of metallic bipolar plates. (ISE-coating compared with Au plated and uncoated stainless steel substrate)

ICP-MS product water analysis of a fuel cell stack with metallic bipolar plates (extreme example)

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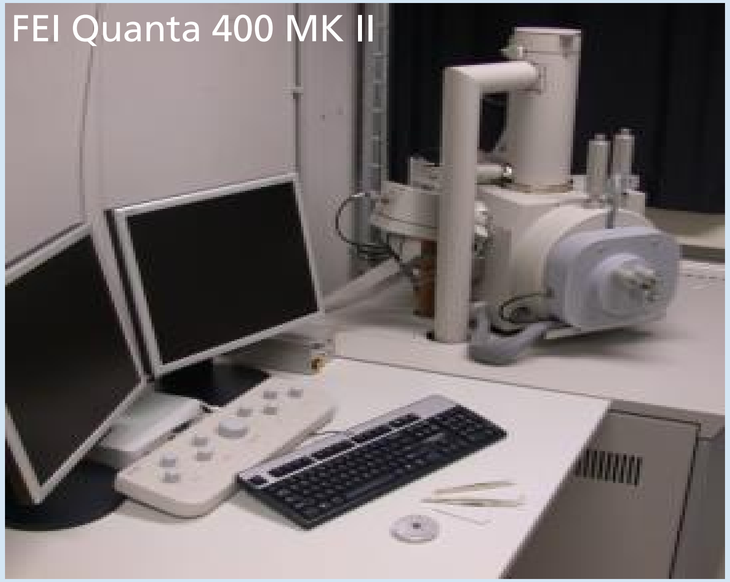
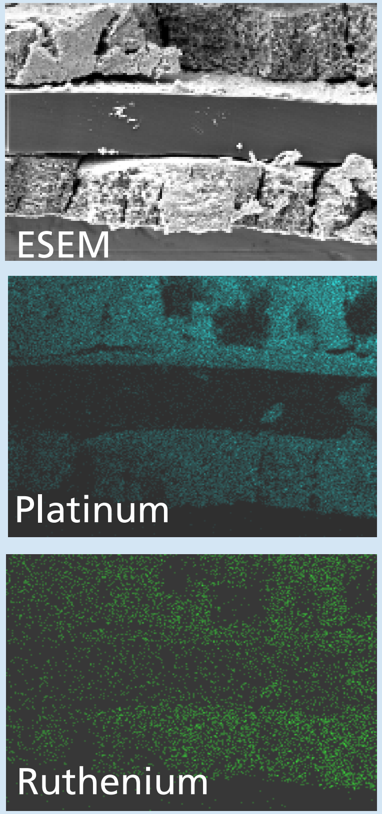
Scanning Electron Microscopy and Computed X-ray Tomography

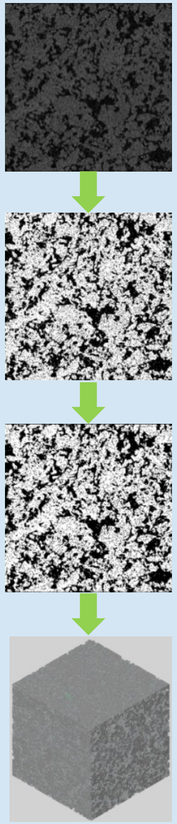




Environmental Scanning Electron Microscopy (ESEM) and Energy Dispersive X-ray Spectroscopy (EDS) for element analysis

Computed X-ray tomography with a nominal resolution of 600 nm

non-destructive scanning and 3D reconstruction of internal micro-structures incl. compression stage

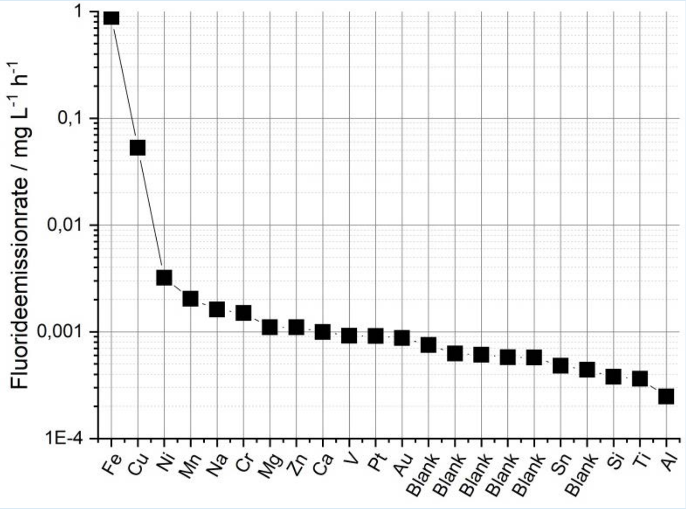


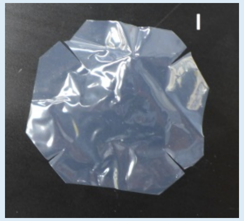
3D reconstruction of a PTL for PEM water electrolysis

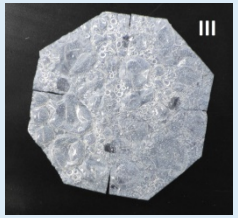
Zielke, L., Fallisch, A., Paust, N., Zengerle, R., Thiele S.: Tomography based screening of flow field/current collector combinations for PEM water electrolysis. RSC Adv (2014), 10.1039/c4ra12402b

Fenton testing

- Investigating the chemical stability of fuel cell or electrolysis membranes regarding radical attacs
- Membrane degradation: cations catalyze radical formation from H_2O_2 and radicals attac the membrane polymer
- Fenton test: Insertion of membrane in 30% H_2O_2 with metal salts (typically Fe^{2+}) and heat at 80°C
- Measurement of Fluoride-release over time as measure for membrane decomposition
- F- measurement with ion sensitive electrode
- Use of chemical inert polymer bottles (safer and less contamination than glass)
- Testing with different temperatures, cations and/or membranes; variation of cation concentration







membranes after Fenton-Test with: I: Al, III: Fe,

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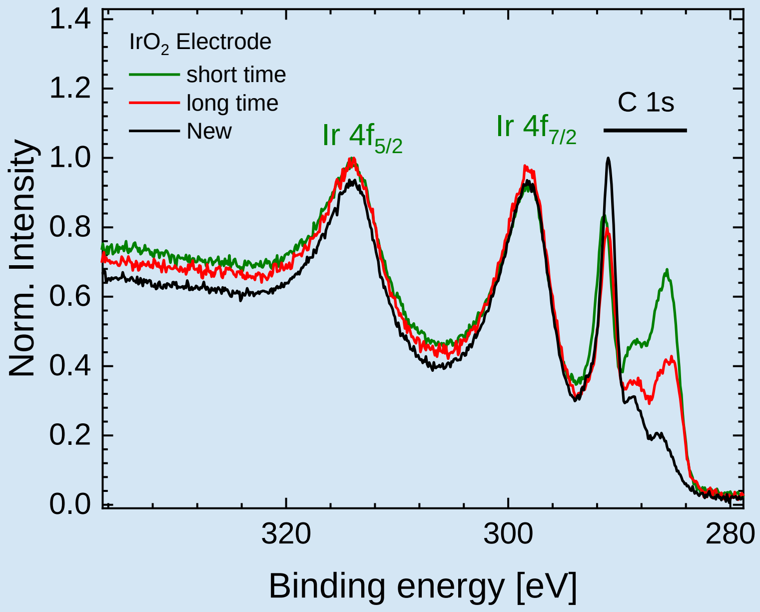
Near ambient pressure X-ray photoelectron spectroscopy (NAP-XPS)

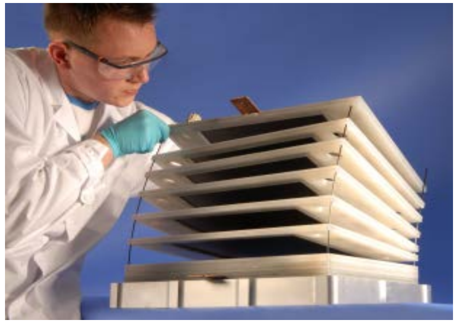
- X-ray source: AlKα (1.4 keV)
- Spot size 300 µm Ø
- Pressure: 10^{-8} mbar up to 20 mbar
- Sample temperature: 5 - 800 °C
- Sample size up to 120 mm Ø
- Plasma cleaning of the surface
- Fully automated vacuum and gas dosing system (N_2, Ar, H_2, O_2)

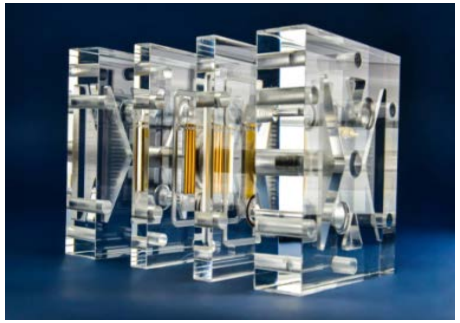


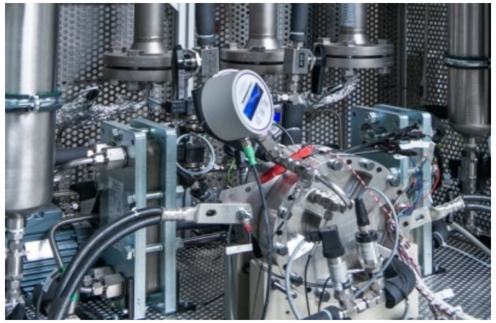
Surface characterisation of an IrO_2 based anode for PEM water electrolysis as received, after short-term and long-term operation

EnvironESCA: Electron spectroscopy for chemical analysis under environmental conditions at near ambient pressure of catalysts, liquids and liquid-solid interfaces.

Thanks a lot for your kind attention!







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