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Evaluation of crack structures in catalyst layers of dynamically operated HT-PEFCs from in situ synchrotron X-ray radiographs

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Outline

- Aims of project

- Preparation of gas diffusion electrodes
- *Through-plane* synchrotron X-ray radiography

- Image preprocessing for crack analyses
- Detection of cracks by radar method
- Distribution of crack widths
- Identification of anodic and cathodic catalyst layers

- Conclusion

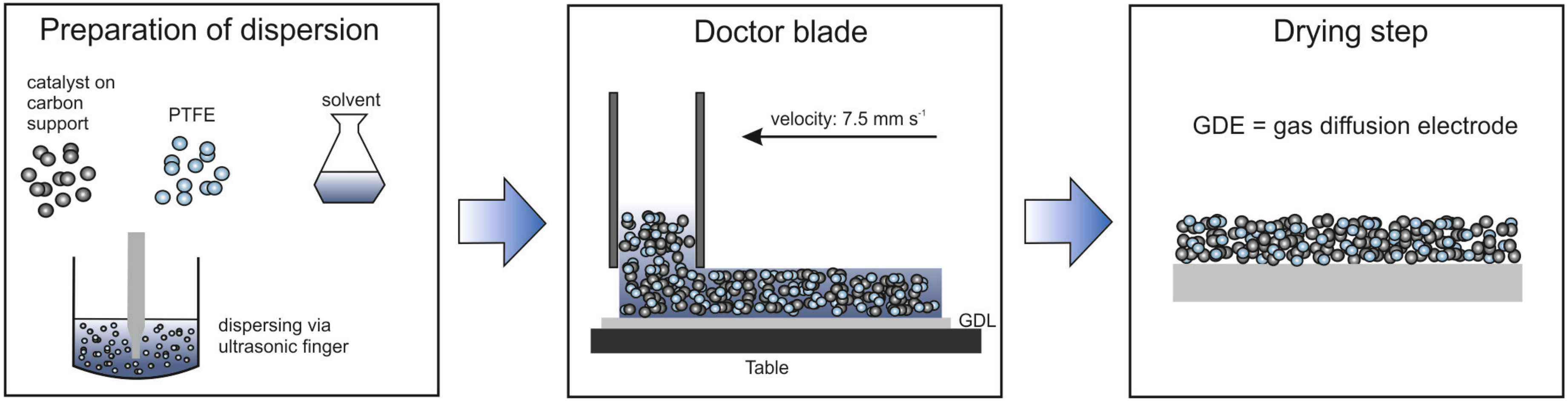
Aims of project

- Optimization of anodic and cathodic catalyst layers of high-temperature polymer electrolyte fuel cells
- Analyses of appearance of cracks within catalyst layers and their behavior during fuel cell operation

➢ Investigations of crack structures within catalyst layers of dynamically operated fuel cells with synchrotron X-ray radiography

➢ Analyses of crack width distribution from in situ synchrotron X-ray radiographs and tomograms with radar method

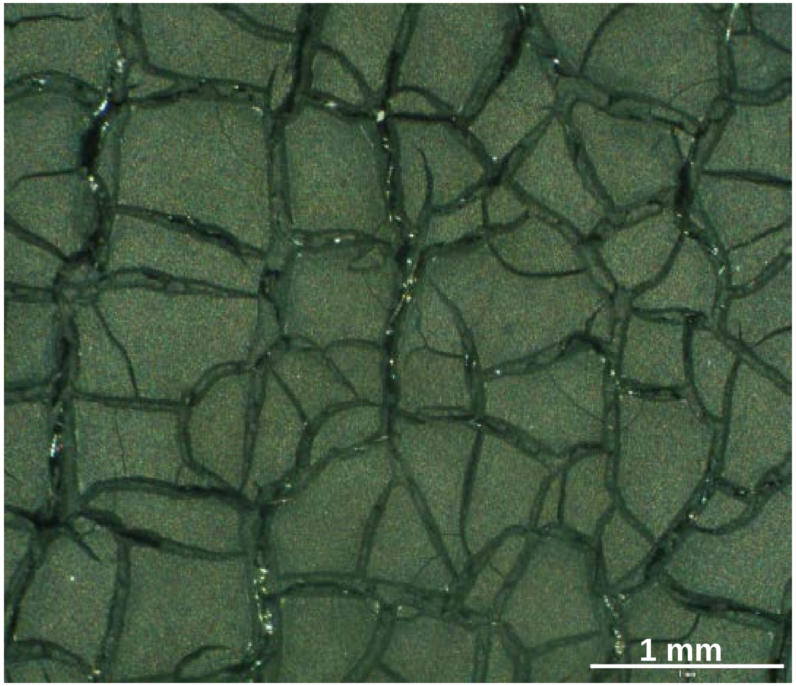
Preparation of gas diffusion electrodes



- Preparation of a homogeneous dispersion composed of platinum catalyst on carbon support, PTFE, and different solvents
- Coating of 1 mm wet electrode layer on a carbon non-woven gas diffusion layer (GDL) by doctor blade technique
- Drying of wet catalyst layer over night / evaporation of solvents

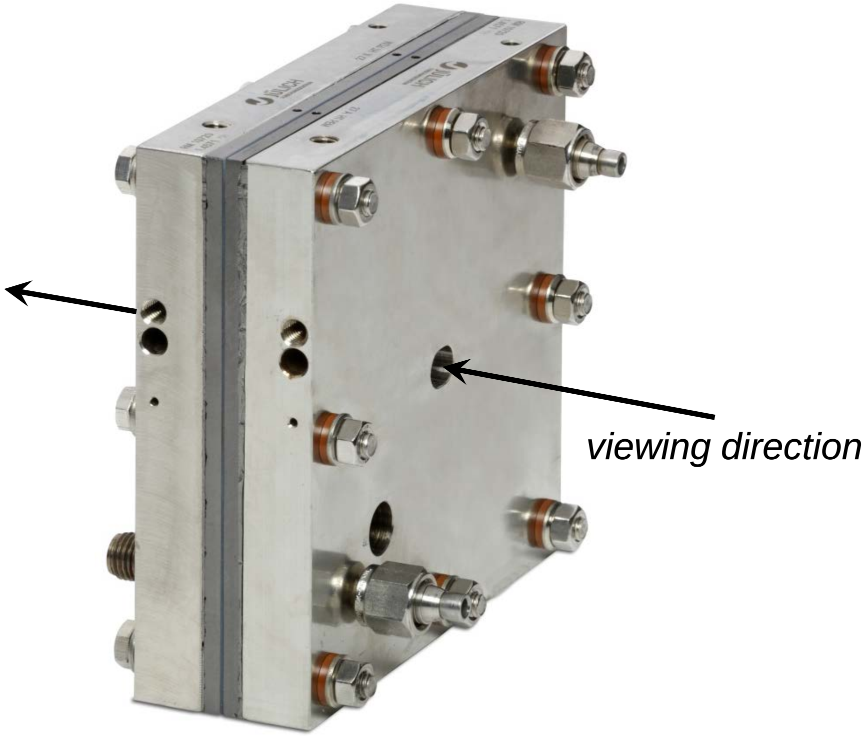
Preparation of gas diffusion electrodes

- After the drying step:
  - thickness of the catalyst layer: ~ 100 µm
  - platinum loading of catalyst layer: ~ 1 mg cm^{-2}
- Formation of crack structure within the catalyst layer due to solvent evaporation during the drying step
- No possible quantification of crack widths even under real operating conditions

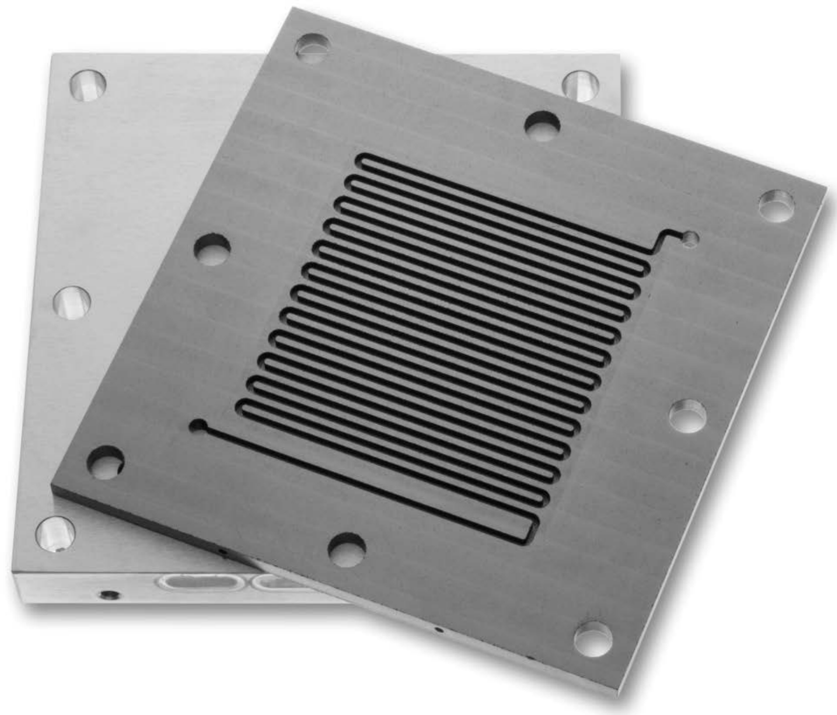


Microscope image of a gas diffusion electrode after the drying step over night.

Through-plane synchrotron X-ray radiography



Assembled measuring cell for through-plane measurements

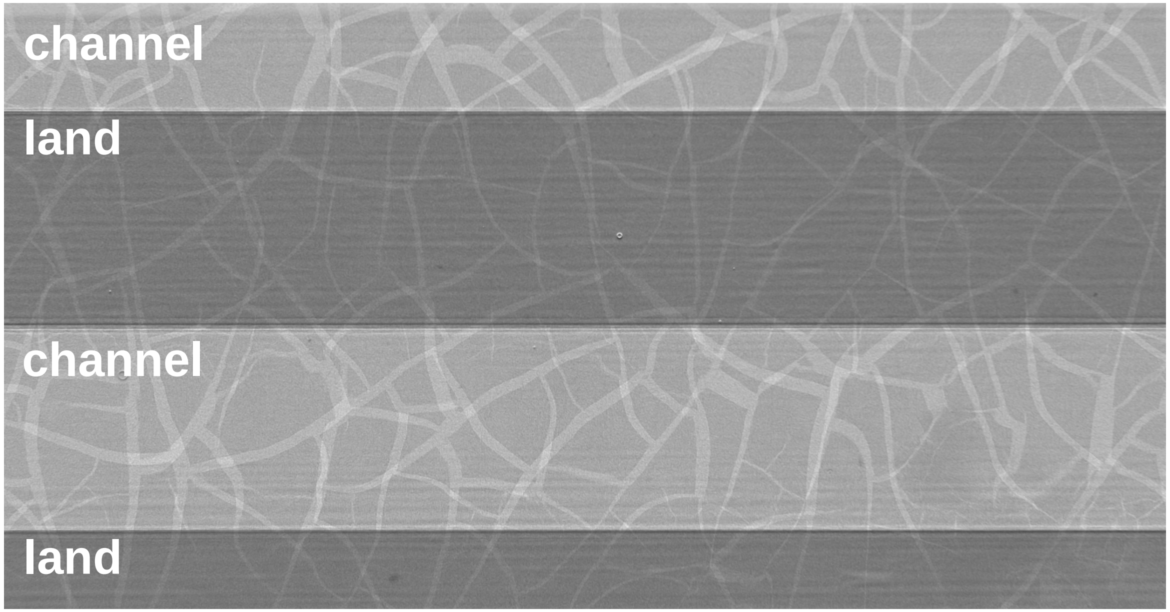


Single channel graphitic flowfield

Flowfield geometry

active cell area:	50.41 cm^2	channel length:	1.383 m
channel depth:	2.5 mm	channel width:	1.5 mm
land width:	1.5 mm		

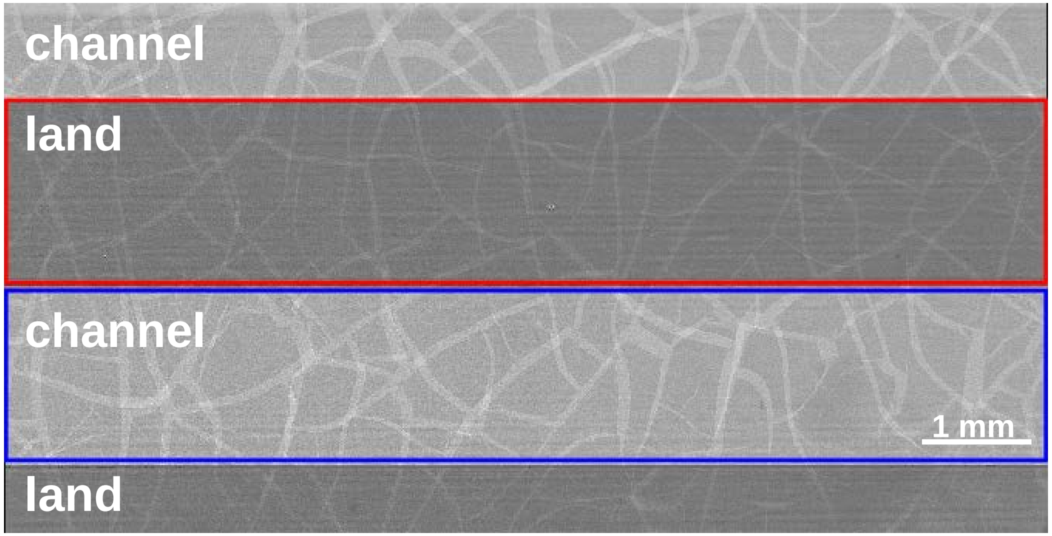
Through-plane synchrotron X-ray radiography



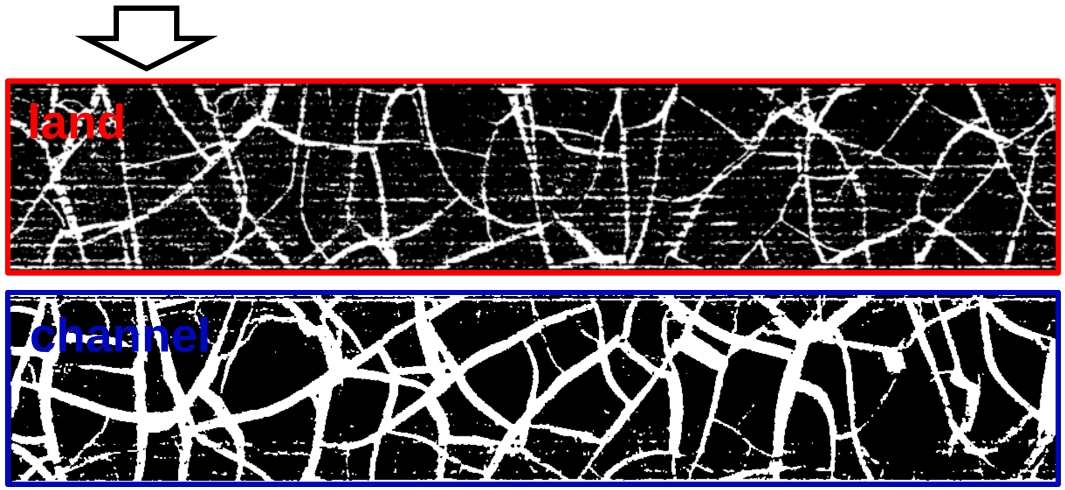
Operating conditions: 160 °C, I = 2/2, hydrogen /air, ambient pressure at gas outlet.

- Recording of synchrotron X-ray radiographs at different operating conditions:
  - 0 mA cm^{-2}
  - 140 mA cm^{-2}
  - 350 mA cm^{-2}
  - 600 mA cm^{-2}
  - 0 mA cm^{-2}
- Radiographs show overlaying crack structure of anodic and cathodic catalyst layers

Preparation of synchrotron X-ray radiographs

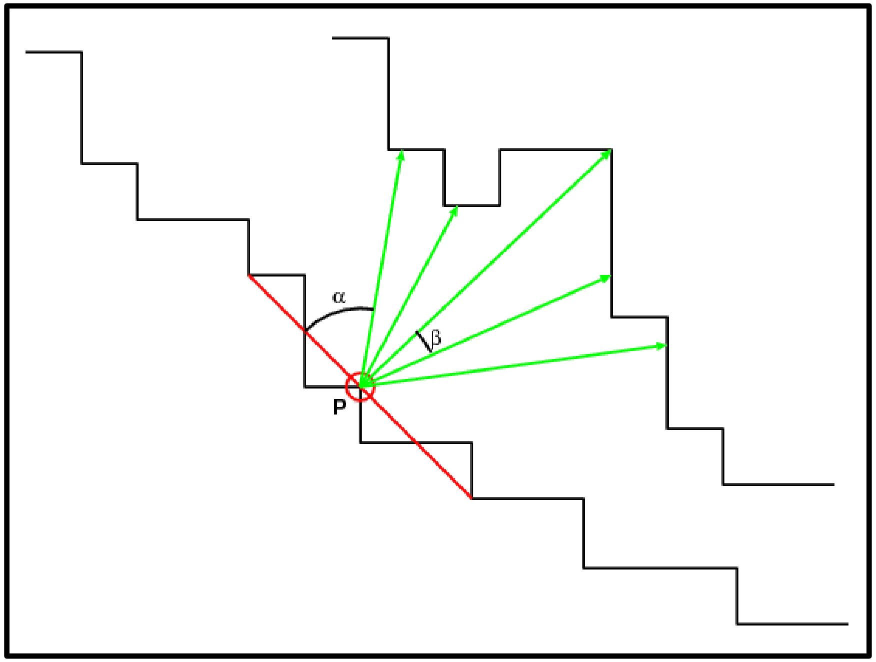


- Recording of synchrotron X-ray radiographs at different operating conditions
  - Clear distinction between channel and land regions
  - Different crack structure under channel and land

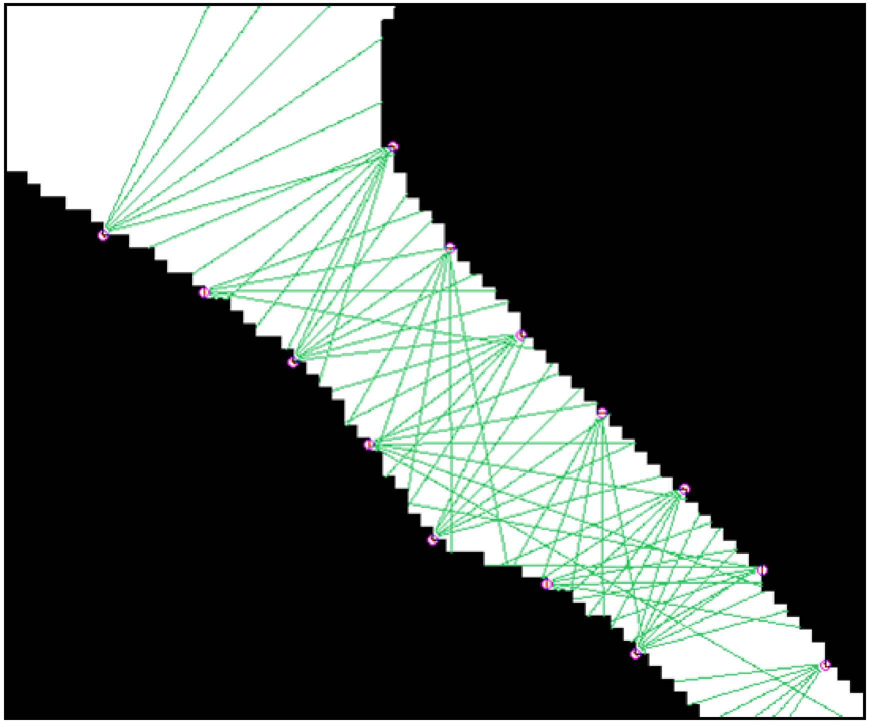


- Conversion of synchrotron X-ray radiographs into black-and-white images
- Analysis of the crack structure with the radar method

Radar method



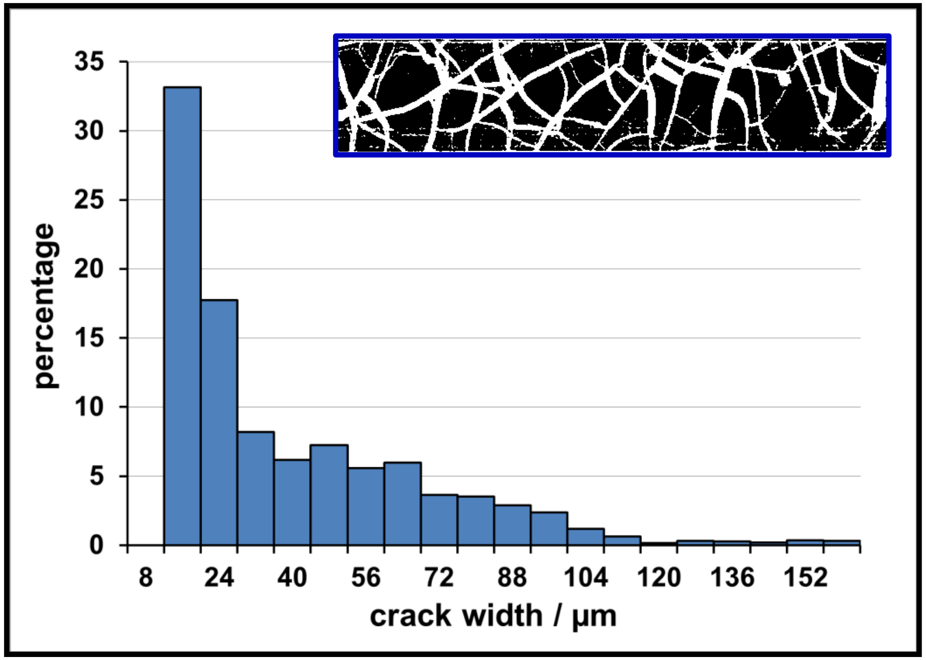
- Principle of the radar algorithm:
  - α = starting angle
  - β = rotation angle
  - P = starting point



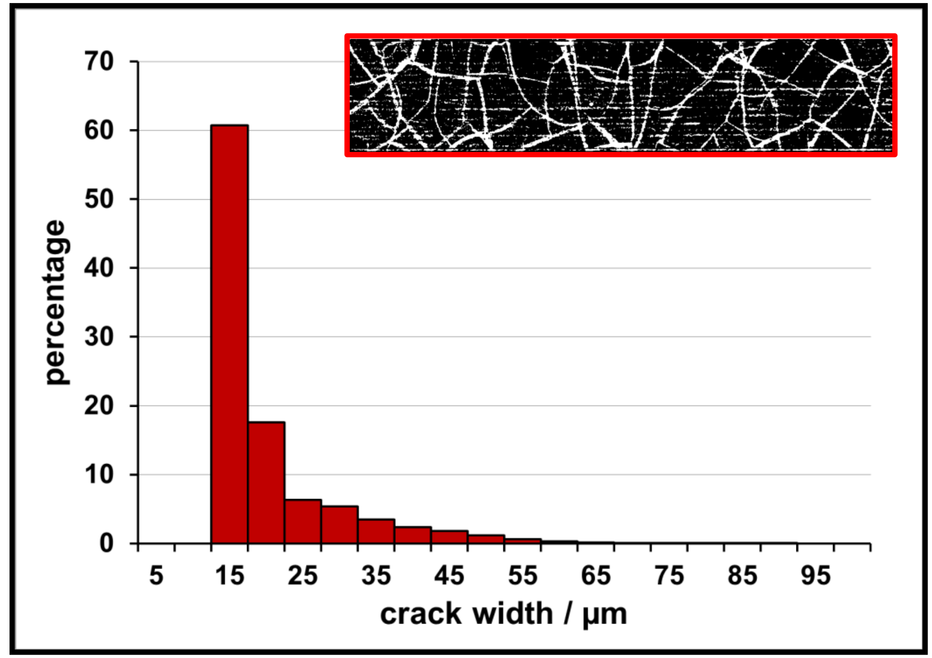
- Radar method applied to a section of a black-and-white image
- Estimation of the shortest way between the starting point to the opposite side of the crack

Distribution of crack widths

a) channel

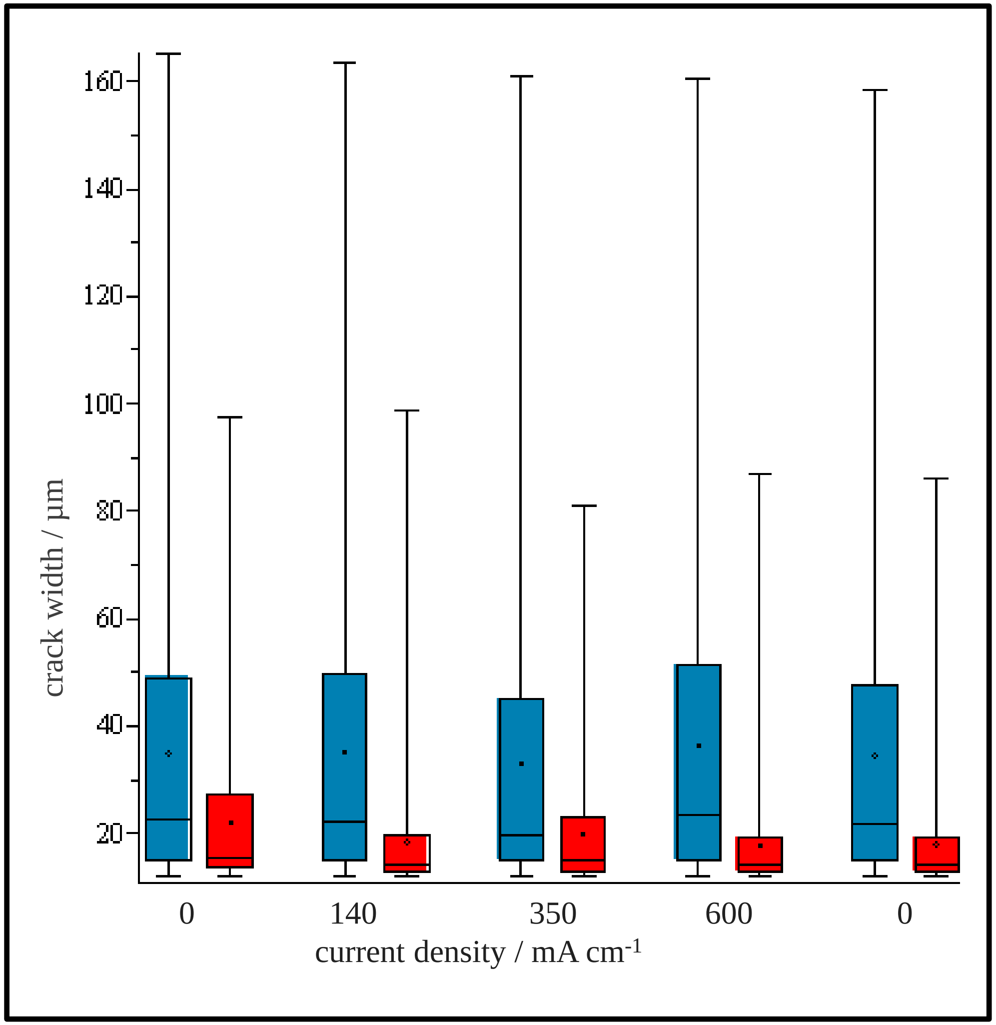


b) land



- Histograms show similar shape for the analysis of the crack structure under the channel and under the land regions
- From the histograms it is difficult to clearly identify the difference in crack widths under the channel and the land regions → display results in boxplots

Distribution of crack widths



- Crack width as a function of fuel cell operating conditions:
  - For all operating conditions cracks under the channel are twice as wide as cracks under the land
  - Proven by stochastic Mann-Whitney-U-test
  - No clear tendency between changes in crack width and operating conditions

Blue boxplots = channel
Red boxplots = land

Distribution of crack widths

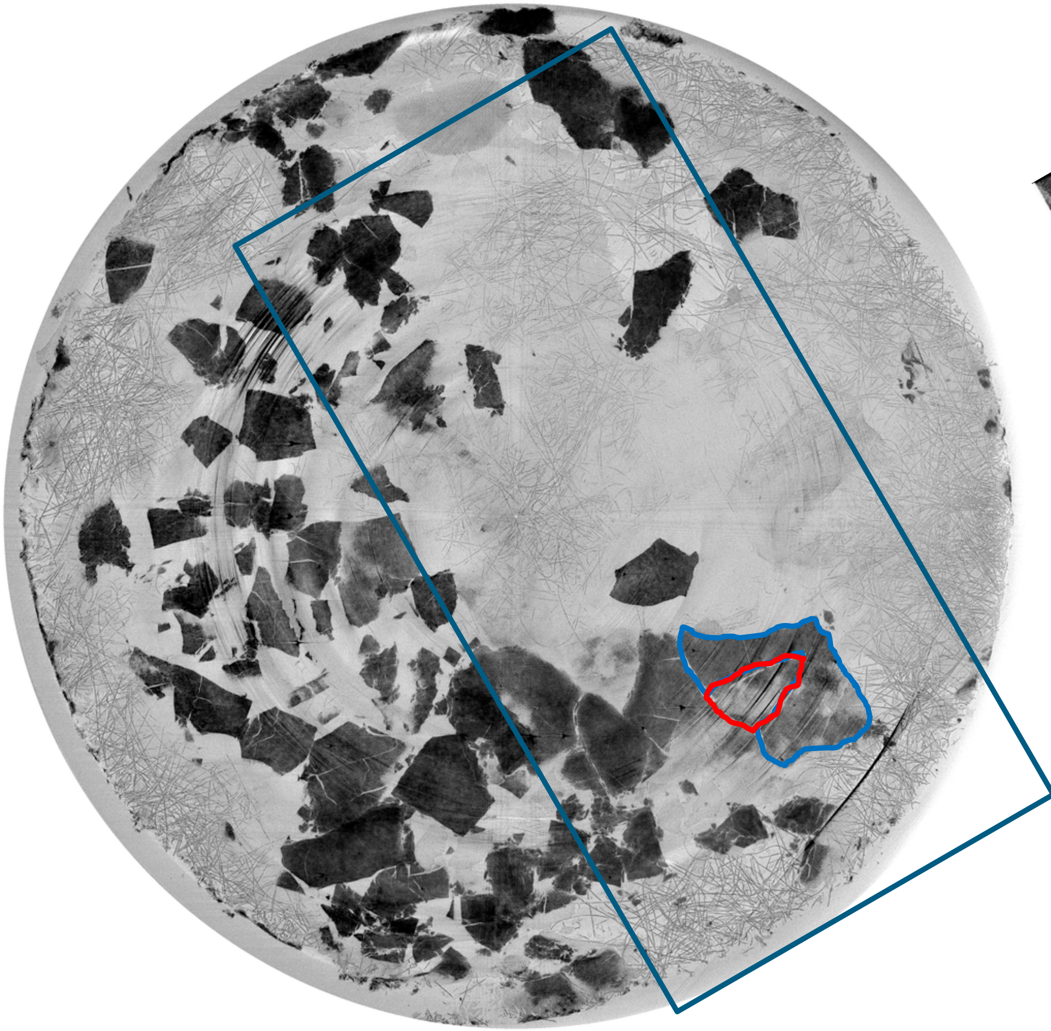
Problem:

- *Through-plane* synchrotron X-ray radiography measurements provide images which show an overlaying of the anodic and cathodic catalyst layers
- Separation of crack structures which belong to the anode or to the cathode not possible from synchrotron X-ray radiographs

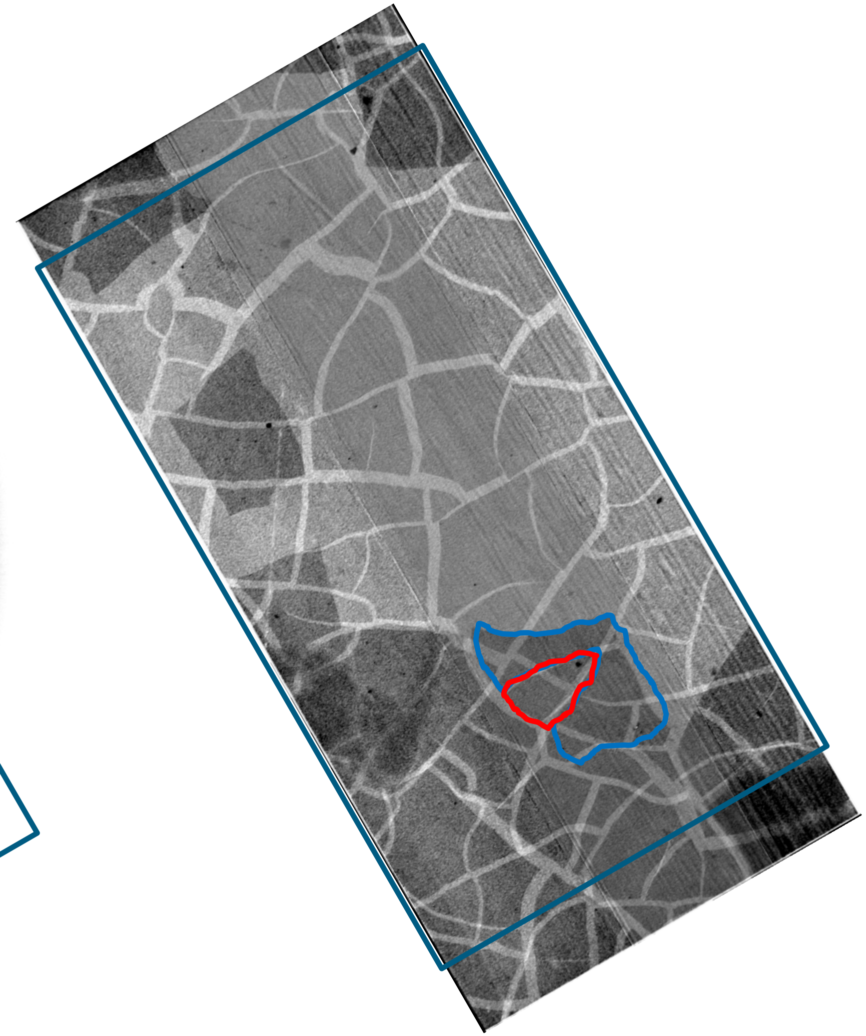
Solution:

- After synchrotron X-ray radiography measurements the analyzed area will be stamped out and analyzed by synchrotron X-ray tomography
- From the tomograms a clear identification of anodic and cathodic catalyst layers is possible by analyzing the crack structures

Identification of catalyst layers: Anode

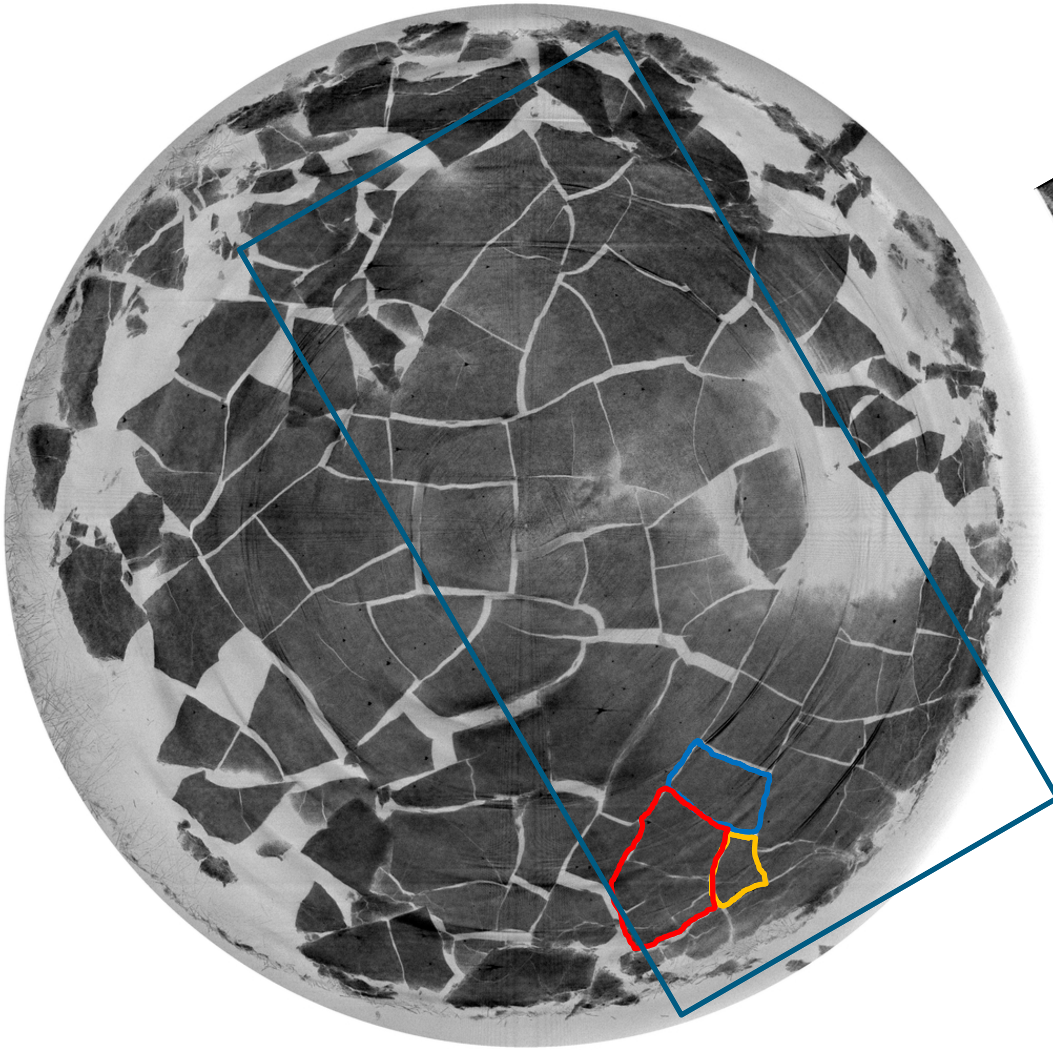


Synchrotron X-ray tomogram

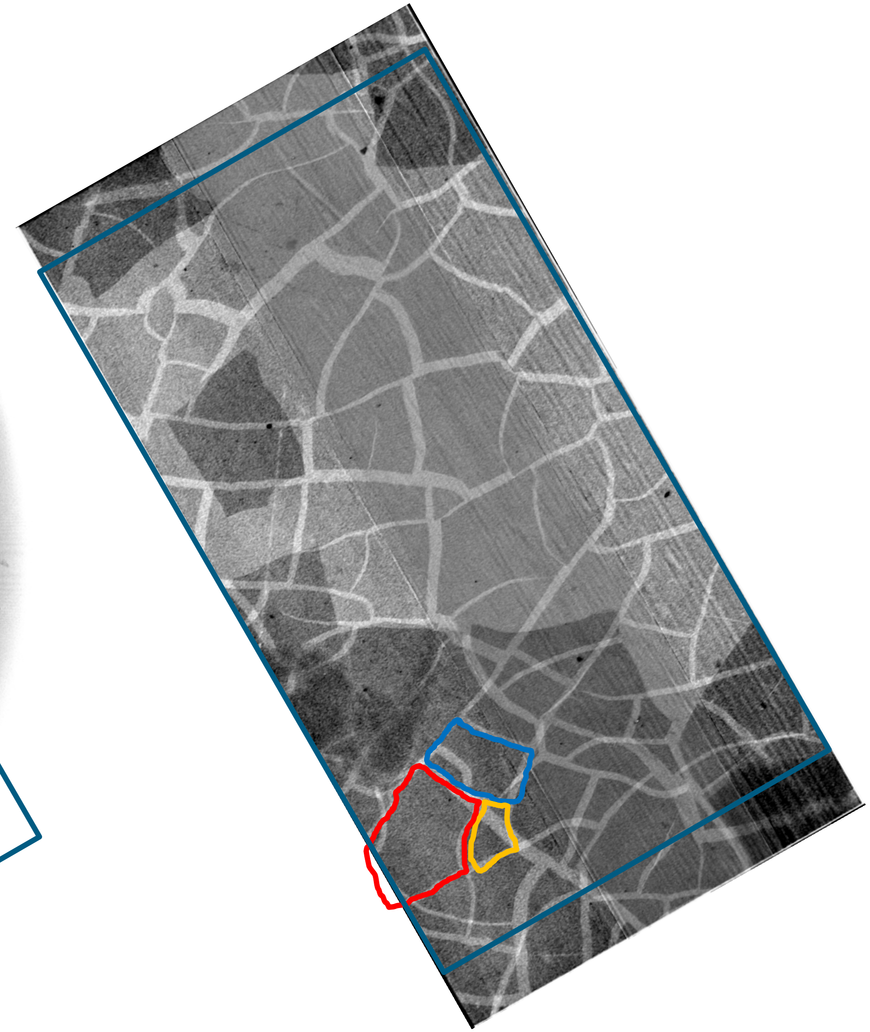


Synchrotron X-ray radiograph

Identification of catalyst layers: Cathode



Synchrotron X-ray tomogram



Synchrotron X-ray radiograph

Conclusion

- Synchrotron X-ray radiography and tomography are useful tools to analyze in situ the crack width distribution of dynamically operated fuel cells
- Analyses of the crack structure of catalyst layers from synchrotron X-ray radiographs with the radar method show that for all operating conditions the cracks under the channel are twice as wide as under the land
- With Synchrotron X-ray tomography anodic and cathodic catalyst layers can be separated