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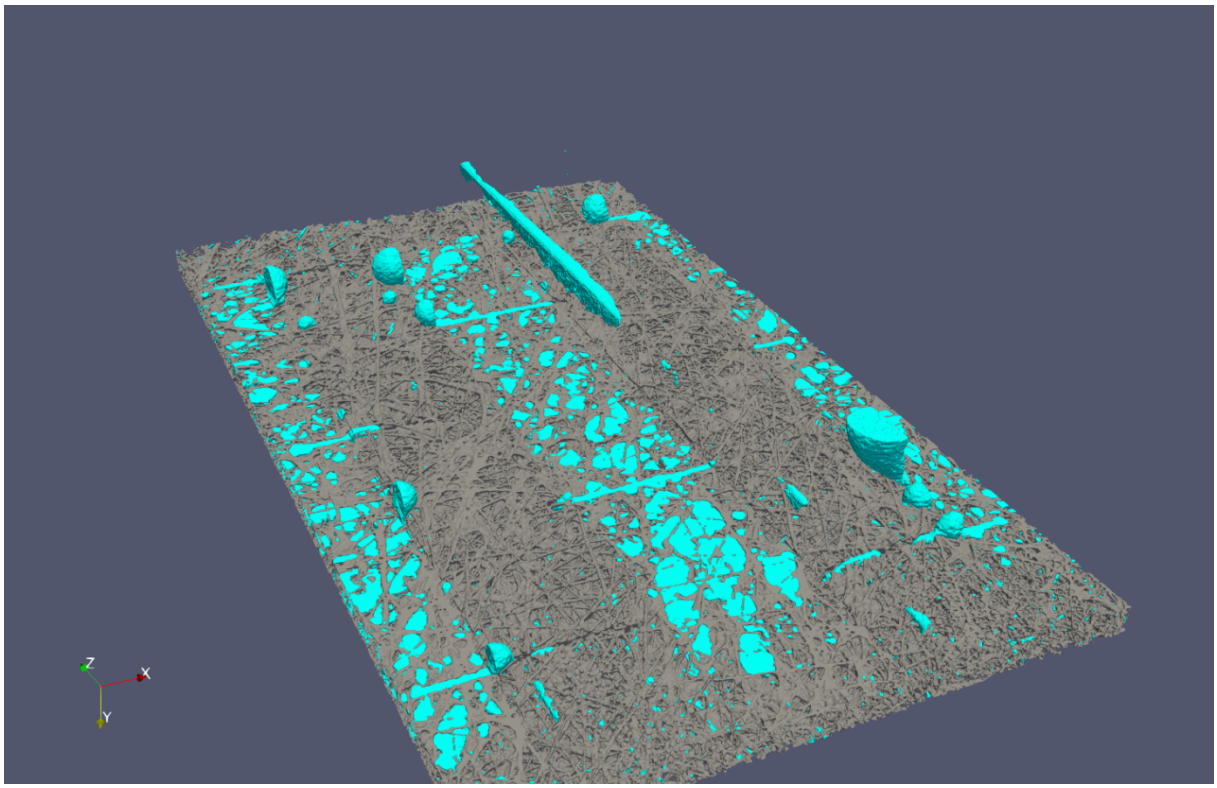
Preliminary final report dated 14.5.2021

Autostack Industry Switzlerland

Material Development for High Performance Polymer Electrolyte Fuel Cells



Source: © Paul Scherrer Institut, 2020; operando X-Ray tomographic image, water emerging from a laser-perforated gas diffusion layer.

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Subsidy recipients:
Paul Scherrer Institut
Forschungsstrasse 111, CH-5232 Villigen
www.psi.ch

Authors:
Christoph Csoklich, PSI, christoph.csoklich@psi.ch
Stefan Tschumi, PSI, stefan.tschumi@psi.ch
Dr. Felix Büchi, PSI, felix.buechi@psi.ch

SFOE project coordinators:
Dr. Stefan Oberholzer, stefan.oberholzer@bfe.admin.ch

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Zusammenfassung

Der wichtigste Weg um die Kosten für Brennstoffzellenstacks in Automobilanwendungen zu reduzieren zielt auf eine Erhöhung der Leistungsdichte der Systeme ab. In diesem Projekt tragt das PSI auf zwei Ebenen zu diesem Ziel bei.

Zum einen wurde ein Stack eines Generation 1, 11-Zellkurzstacks von und im Rahmen des *Autostack Industrie Deutschland* getestet.

Zum anderen wurden neue Gasdiffusionsschichten entwickelt, allen voran Materialien mit deterministischen Strukturen. Hierfür wurden Gewebe von *SEFAR* und *BOPP* untersucht. Im Hochstrombetrieb zeigen die *SEFAR*-Materialien in einer Brennstoffzelle eine verbesserte Leistung als Standardmaterialien. Um die Leistungssteigerungen und Effekte zu verstehen wurden verschiedene Charakterisierungsmethoden, unter anderem operando Röntgentomographie, angewendet. Ausserdem wurden kohlefaserbasierte Materialien (auf dem Stand der Technik) mit einem Laser modifiziert. Leistungssteigerungen mit diesen Materialien konnten auf den Wassertransporteigenschaften in diesen Strukturen zurückgeführt werden. Die Ergebnisse werden nun verwendet um Designempfehlungen zu formulieren.

Summary

Increasing power density is the most important development route to reduce cost for automotive fuel cell stack hardware. In this project PSI contributes on two levels

Sub-stack testing has been performed on a Generation 1, 11-cell sub-stack from the German Autostack partners.

For the GDL development, deterministic structures for gas diffusion layers are developed. Woven materials from SEFAR and BOPP have been investigated. Under high current density conditions, SEFAR materials show better performance in the fuel cell than the standard materials. In order to understand the performance effect in detail and direct the development, different characterization methods, including X-ray tomographic microscopy have been employed. Further state-of-the-art carbon fiber materials were laser modified to understand the effect of (added) deterministic structures. Improvement was shown to be through the guidance of liquid water in the added deterministic structures. Results will used to formulate design guidelines.

Main findings

1. The general concept of deterministic structures in GDL is fundamentally investigated and proven with laser modified standard GDL materials.

2. Introduction of deterministic structures improves transport of product water efficiently. Fuel cell performance is increased at regular operating conditions, but especially under high current oxygen operation.

Contents

Abbreviations

CCM	Catalyst Coated Membrane
EDX	Energy dispersive X-Ray spectroscopy
GDL	Gas Diffusion Layer
HFR	High Frequency Resistance
MPL	Micro Porous Layer
PEFC	Polymer electrolyte fuel cell
RH	Relative Humidity
SEM	Scanning Electron Microscopy
XTM	X-Ray Tomographic Microscopy

1 Introduction

1.1 Background information and current situation

Hydrogen is not only an important chemical, necessary for a wide range of syntheses from ammonia to synfuel production, but is also a versatile energy carrier.[1,2] Its potential of long time storage and coupling the energy sectors drive the current research to increase the efficiency of involved conversion processes.[3–5]

Polymer electrolyte fuel cells (PEFC) are efficient energy converters for hydrogen to power.[6] Hydrogen fuel cells are not only more efficient energy converter than internal combustion engines, but also a cleaner and CO_2 free (if green hydrogen is used).[6] Even though some fuel cell systems for both stationary and mobile application have been commercialized, market penetration has not been achieved due to a combination of their high price, durability issues and the necessary fueling infrastructure.[7]

Materials development for increasing the power density of PEFC is one of the mainstream development routes to reduce specific cost.

1.2 Purpose of the project

PSI performed work on two levels:

1. Test of *Autostack Industrie* Hardware on the sub-stack level.
2. Development of new GDLs having deterministic structures.

The first task is a service to the project and is financed by PSI. It provides knowhow about the state-of-the-art automotive hardware.

The second topic aims at further improving fuel cell technology and also incorporates activities with Swiss companies. This part is financed by BFE. This report focuses on the BFE financed part.

Current fuel cell designs use a sandwich assembly of flow field plates, gas transport layer, catalyst layer, at anode and cathode and a membrane as central component.

From the channel to the catalyst, feed gas diffuses through the tortuous pore network of the transport layer. It consists usually of a macro-porous gas diffusion layer (GDL) made from carbon fibers and a microporous layer (MPL), made from carbon nano-particles. Product water vapor can condense in this structure, where it blocks pores, thus decreasing the effective gas diffusivity, and preventing sufficient gas supply.

This project develops new approaches to GDL materials to improve water transport in these porous structures and minimize mass transport losses, thereby increasing power density. Which is an important contribution to future cost reduction.

This work takes places on two levels: i) testing of deterministic structures (based on woven materials of Sefar AG, Switzerland and G. Bopp & Co. AG, Switzerland) and ii) development of the fundamental understanding of deterministic structures for GDL, based on laser modified state-of-the-art GDLs.

1.3 Objectives

This project investigates fundamentally different new transport layers with deterministic structure and their impact on water management. The basic approach is to create direct water channels from catalyst layer or MPL to the gas channel.

Woven, non-carbon-based fabrics with are investigated as a fundamentally new class of GDLs. Fabrics made from or with conductive fibers promise cheap roll-to-roll manufacturing, processing, and flexibility of the material. Their deterministic structure allows fine-tuning of transport properties, compared to stochastic, carbon-fiber GDLs. In this part, separate collaborations have been formed with two Swiss companies, *Sefar AG* and *G. BOPP + CO. AG*, both experts in filtration material fabrication.

This work is complemented with fundamental investigations using laser modified conventional GDL materials, with a research focus on a systematic study of perforations. This is an easy applicable, comparably cheap, and tunable modification technique.

All materials are characterized with ex-situ imaging techniques (SEM/EDX, XTM) and their transport properties are simulated. Their performance is tested under a controlled set of operating conditions, similar to application conditions. The water transport in these modified structures is also characterized by operando X-Ray Computed Tomographic Microscopy (XTM) to deduce the underlying mechaniusms and provide design rules for the GDL design.

2 Procedures and methodology

2.1 Sub-stack testing

Tests are performed on PSI's ms2 test bench equipped for testing up to 30 kW.

Figure 1: Mounting the Autostack-sub-stack at PSIs ms2 testbench

2.2 Gas Diffusion Layer Development

2.2.1 Water Transport in Porous GDL

Usually, water transport in the small pored GDL (avg. pore sizes in the order of 10-100µm) is capillary driven and can be described by the Young Laplace equation (Equ. 1):

$$p_c = \frac{2\gamma \cos\theta}{r} \tag{1}$$

The pressure difference p_c between gas and liquid phase (capillary pressure) is required to push the liquid with surface tension γ through a pore with radius r, made of a material with the contact angle θ. In order to reduce the required pressure and facilitate water transport, one can now either decrease the contact angle or increase the pore radius, which leads in principal to a chemical and a mechanical modification approach.

Understanding of the underlying transport mechanisms of water and gas phases is necessary. In a first step, by the variation of laser induced perforation patterns and tuning of the contact angle, these phenomena were characterized and quantified.

2.2.2 Woven GDLs

Woven GDLs promise a cheap and simple production and an immense potential of tuning transport properties. As the characteristic length scale of common GDLs lies in the tens of micrometer range, fabrics with similarly fine fibres and pores are required. These considerations led to the collaboration with two Swiss companies that are experts in filtering applications and can provide materials with the desired structural properties.

1. **Sefar**

The Sefar bas material was a double layered, PET filtering fabric with a fiber diameter of 80 µm (carrier layer) and of roughly 25 µm (surface layer) and an original thickness of 180µm. To grant the material

electrical conductivity, gold sputtering was chosen to apply films of around 100-500 nm thickness onto the base material. Already the thin layer of Au grants a conductivity similar to conventional carbon based GDLs. Additional hydrophobic coating was tested but later discarded as the surface contact angle with the gold coating was already high enough (around 110 °) and additional used coating process only blocked the fine pores partially. The flexible materials integrates very well into the existing fuel cell designs, requiring no further modifications of the experimental setup.

2. Bopp

Bopp specializes in stainless steel fabrics for filtering applications. The use of stainless steel is promising for GDLs, as it is resistant to the electrochemical environment in the cell, does not poison the catalyst and could be sufficiently conductive to avoid a sputtering process. However, the thickness and stiffness of the materials challenge nowadays used fuel cell designs. The main challenge here was to find suitable material candidates and investigate their structures influence on fuel cell performance, rather than improving fuel cell performance in the first place.

a) b)

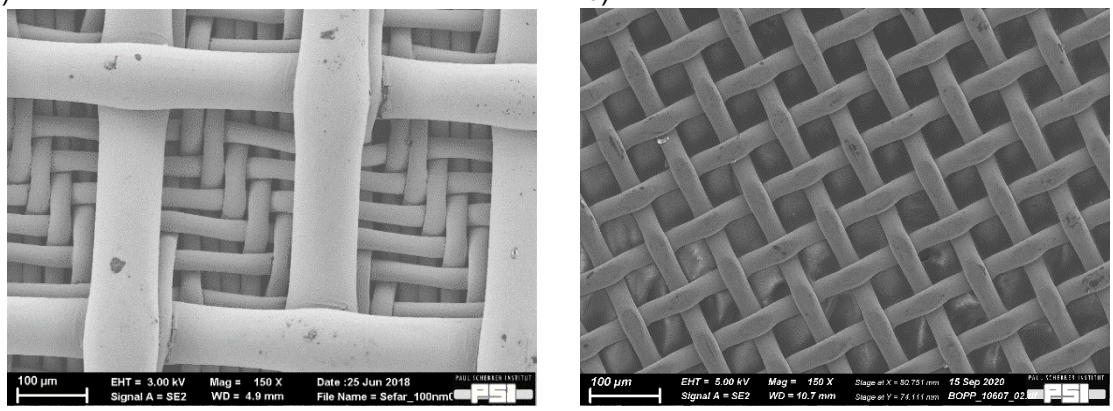

Figure 1: SEM images of Au-sputtered PET fabric (by Sefar) and a one-layered stainless steel fabric (Bopp).

2.3 Imaging Techniques

2.3.1 SEM/EDX

Scanning Electron Microscopy (SEM) in combination with Energy Dispersive X-Ray Spectroscopy (EDX) is a fast, powerful imaging technique to investigate surface properties of conductive samples. They capture not only the surface morphology down to a resolution of some tens of nanometers, but detect also the elemental surface composition. For GDLs, this is especially useful to examine the fluorine loading (originating from the hydrophobic coating) and distribution.

2.3.2 Ex-situ XTM

X-Ray Computed Tomographic Microscopy has a lower resolution than SEM, but can probe not only surface properties but also 3D structural information down to a micrometer resolution. The in-house available lab-CT can operate with a voxel size of below 2 µm, that is sufficient to measure GDL structures. The hereby-received information is used as input for bulk property calculations and diffusion or imbibition simulations. Metrics as porosity, pore/throat size distribution, effective diffusivity or breakthrough pressures allow a material comparison and help separating the structures influence on the several transport mechanisms in an operating fuel cell.

2.3.3 In-situ operando XTM

Fast (sub-second) operando XTM, developed for fuel cells at PSI in collaboration with the Tomcat Beamline of the Swiss Light Source (SLS), enables capturing fast water dynamics in the porous structures of a complete operating fuel cell. These experiments are an essential completion of ex-situ analysis, as they help validating ex-situ experiments and understanding the complex fluid transport mechanistic of full, operating cells. For the details of this complex experimental setup, following literature is suggested.[12]

2.4 Simulations & Modelling

For all bulk descriptor calculations and transport simulations, the GeoDict® software (Math2Market®, Germany) was used. It is a powerful but intuitive tool where any segmented material structure can be imported and used as pore network for fluids and gases.

To gain more insights from the experimental data, the polarization behavior was also modelled.[8],[11] The chosen modelling approach however has been only partially successful. Its voltage break down describes accurately the observed polarization curves and therefore delivers values for the mass transport overpotentials, but fails to deliver sensible results for the oxygen transport resistance. This was finally achieved with limiting current measurements at different oxygen in nitrogen concentrations.

2.5 PEFC operando Test – Differential Cell

Small-scale, differential single fuel cells allow for accurate and controllable comparison of different fuel cell components without the complexity of down the channel or cell-to-cell variations in a stack assembly. For all performance experiments, the GDLs were tested in an in-house designed, 5 cm^2 active area fuel cells with parallel channels, that was operated at differential conditions. Polarization curves, limiting current measurement and pulsed gas analysis were the chosen methods to quantify performance and mass transport overpotentials. The details of the experiments are described in the prepared publications.

3 Results and discussion

3.1 Stack Level Performance

Due to a large time-delay in the German Autostack Industry Project, the sub-stack testing could only be performed in 2020 with the following time-line

- Sub-stack recieved on June 23th, 2020
- Integrated on ms2 at PSI on August 4th, 2020
- BoT PolCurve under ASC reference operating conditions on August 21th to 24th, 2020
- Durability testing starting End. Nov. 20

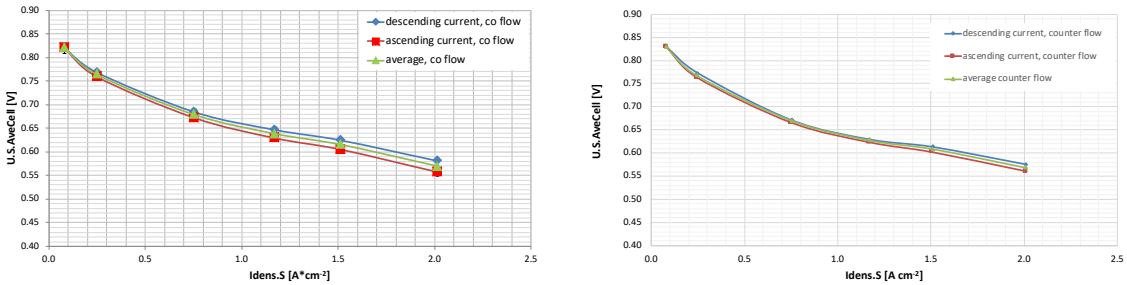

Figure 3: Current/voltage characteristics of 11-cell substack with co- and counter-flow of the gases under automotive conditions.

Results show that the stack performs similarly in both gas feed strategies, in co- and counter-flow of the anode and cathode gases up to a maximum current of 480 A.

The durability testing could only be performed over about 50 hours on the dynamic 30 min synthetic NEDC test protocol (see Figure 4). After 25 hours test time, a cell developed low voltages at high currents, which impeded the test to last the foreseen duration of 500 hours. Probably a pin-hole developed in the experimental membrane material.

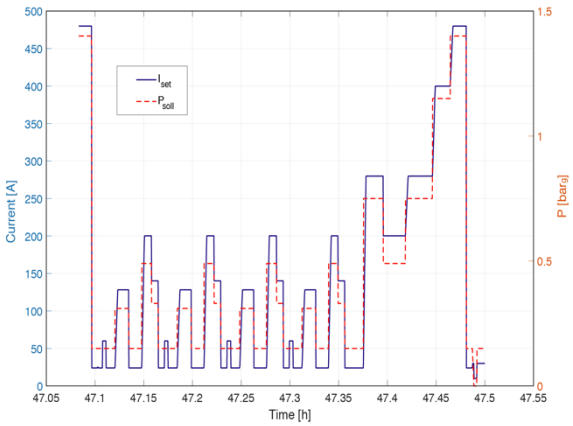

Figure 2: 30 min synthetic NEDC test cycle.

3.2 GDL Development: Material Properties

3.2.1 Woven GDLs

Based on the double-layer fabric by Sefar (referred to as S-PET), a parametric study of different thread sizes and densities (referred to as D-PET-1-4) was conducted and the structural influence on the transport properties analyzed. Figure summarizes the main findings and illustrates how different weavings and thread diameter directly influence the pore space and thereby transport properties. The simulation details and further discussion is found in the manuscript.

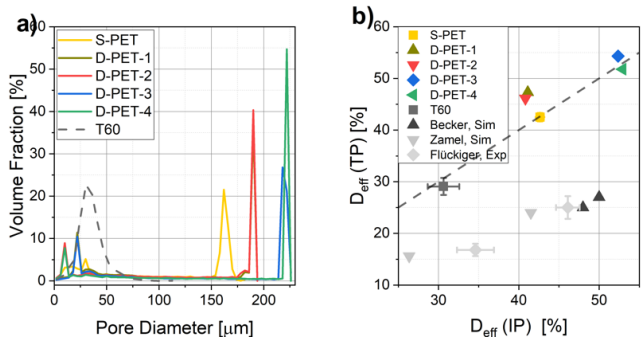

Figure 5: Simulation results for the real S-PET fabric, four digitally created woven fabrics, a real carbon GDL (T60) and literature values for the same structure; a) Pore size distribution of the 6 materials, b) effective diffusivity (in-plane and through-plane).

3.2.2 Perforated Carbon GDLs

Two main perforation types, slits and holes (Figure 6a) were chosen for the final fuel cell tests. The EDX data in 6b shows clearly how the order of laser treatment and hydrophobic coating changes the properties around the perforations. Perforating bare GDLs and wet proofing them in a second, later step renders also the perforations and their vicinity covered with FEP (cf. 6, where also the walls of the perforations show the fluorine signal).

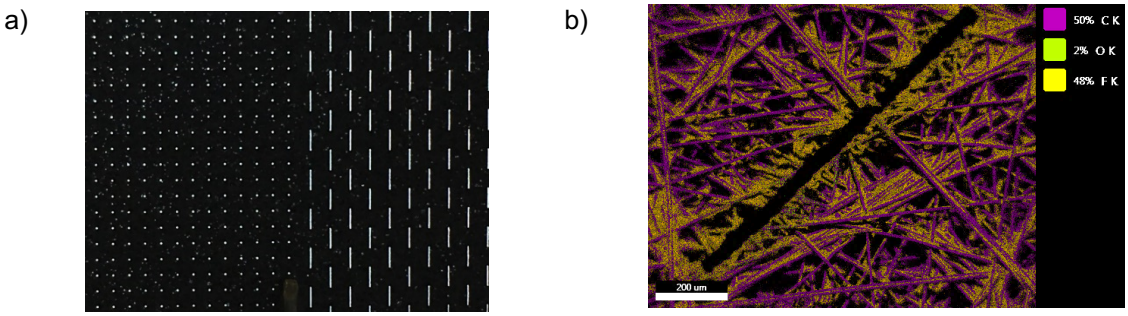

Figure 6: Laser perforated GDL with a) two types of perforated patterns on a plain TGP-H-060 GDL and b) the EDX mapping of the hydrophobized material, showing sufficient wet-proofing (fluorine signal in yellow) also around the perforated slit.

3.3 PEFC Performance

3.3.1 Woven GDLs

The performance of the S-PET-500 GDL was evaluated with a detailed operation parameter variation of temperature and humidity (cf. Table 1) for a standard TGP-H 060 carbon GDL with commercial wet proofing and woven GDLs with and without self-standing MPLs.

Table 1: Overview of the experimental conditions

T_{Cell}	50/60/70/75/80 °C
RH_C	60/80/100 %
GDLs	TGP-H-060 (w. MPL) Sefar500 (w. MPL)

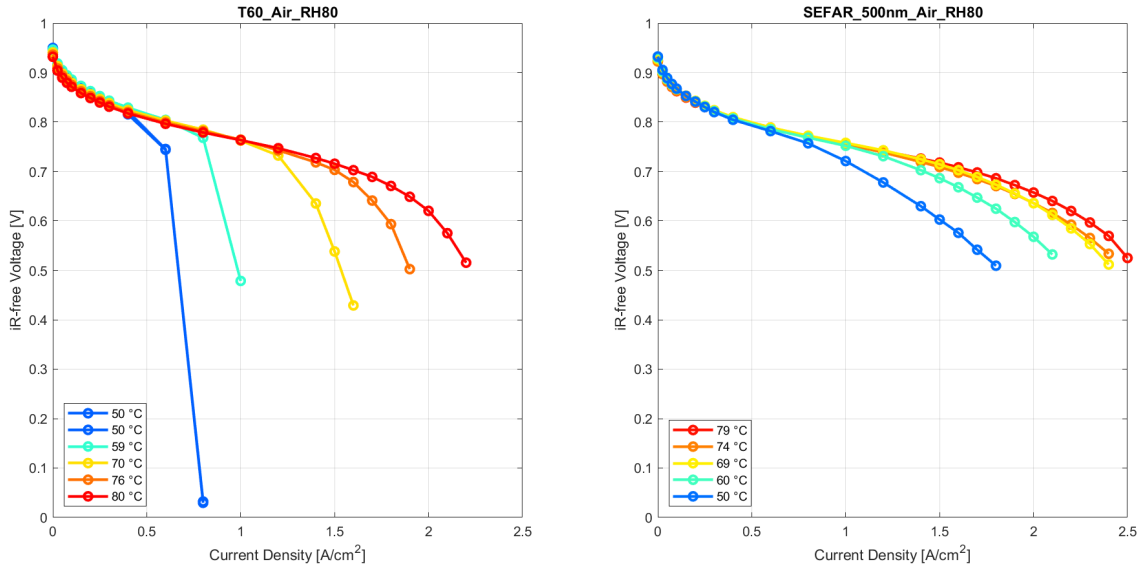

Figure 7: Influence of the operating temperature from 50°C-80°C for TGP-H-060 (left) and Sefar500 (right). (50-80°C, 1.5 bar H_2 vs. Air/O_2, 3 NL/min, RH 80/80, 5 cm^2 act. Area)

The material shows already at standard operating conditions an increased performance compared to the carbon paper (when comparing the iR-free voltage). At lower temperatures, the woven fabric excels the conventional GDL even more and can reach more than 1 A/cm^2 higher current densities. Overall, the Sefar fabric performs its electron, heat, and fuel transporting duties stable over the investigated temperature range and the tested time (60 hours). However, the high frequency resistance (HFR) of Sefar500 is around 20 % higher. This is most likely due to the lower contact area with the catalyst layer and the thin coating. With solid metal fibers in the structure, this effect can be potentially minimized.

Also under rising cathode humidity levels, the Sefar GDL performs by far more stable compared to the carbon GDL without major mass transport losses at high current densities and high RH_C (see Figure 7).

3.3.2 Perforated Carbon GDLs

The influence of laser perforations in GDLs (conventional Toray carbon paper TGP-H 060 as base material) on a fuel cells performance has been investigated with five perforation types (cf. Table 2):

Table 2: Perforation Parameters

ID/perf. Type	Perf. Dimensions	Perforated area
Holes	Ø 80 µm,	0.5 %
Slits1	50 µm x 1 mm (1 mm spacing)	2.5 %
Slits3	50 µm x 3 mm (1.5 mm spacing)	1.7 %
Slits4	50 µm x 1 mm (0.5 mm spacing)	5.0 %
Slits5	50 µm x 1 mm (0.3 mm spacing)	8.3 %

For all perforations the performance of the GDLs were tested by measuring polarization curves in air and pure oxygen at different set levels of the cathode relative humidity (RH_C 60/80/100 %) and comparing them to the unperforated reference GDL of the same material (cf. 8). The voltage difference follows the convention $\Delta U = U_{perf} - U_{unperf}$.

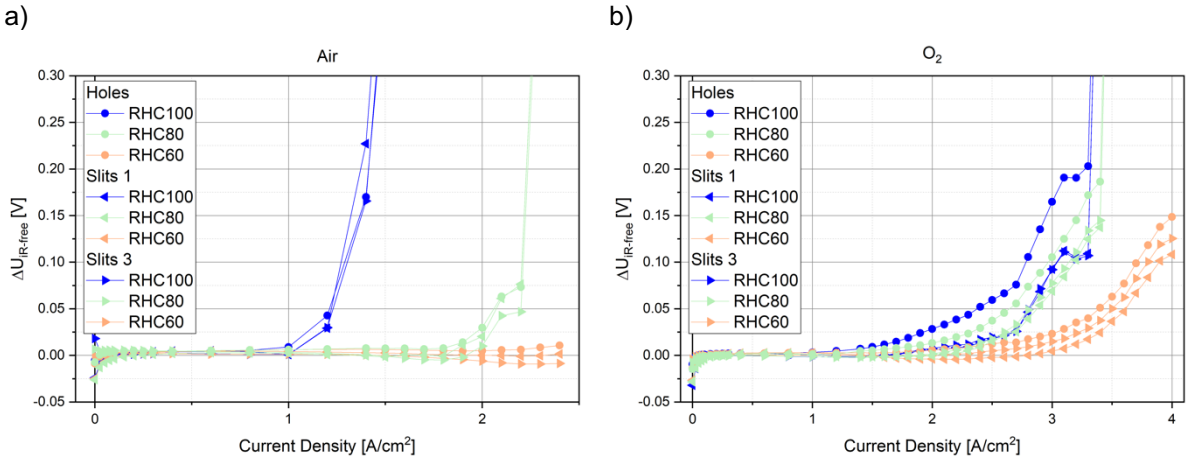

Figure 8: Voltage difference ΔU of three perforated and a comparison GDL at varying cathode humidity levels (RH_C) in air (left) and pure O_2 (right) on the cathode side. (80°C, 1.5 bar H_2 vs. Air/O_2, 3 NL/min, 5 cm^2 act. Area)

For air operation, following observations were made: at dry conditions, all GDLs perform similar. At elevated RH_C, perforated samples (excl. Slits 4/5) exhibit a significant voltage gain compared to the plain carbon GDL. This is both visible in an increase in the slope of ΔU as well as in the rapid overshoot, indicating the voltage loss at conditions close to limiting current. Here, the perforated GDLs can handle larger amounts of product water and postpone the mass transfer limits. For reasonable operating conditions, however, the voltage gain is in the range of around 50 mV compared to the base material.

In O_2 operation, the perforated GDLs perform already at dry conditions and high current densities much better: a voltage gain of more than 100 mV and an increase in current density of more than 0.5 A/cm2 seems achievable. The reason for this beneficial behavior is still under discussion.

When too much of the GDL area is perforated (slits4/5), the electrical conductivity drops, as too many fibers are interrupted and electrons have to hop more between fibers. This effect can be seen when the HFR behavior of these materials is observed.

3.4 Water Transport Mechanistics

In the end of August 2019, the fuel cell systems and diagnostics group had been granted a beam time at the Tomcat beamline of SLS. During the campaign, also some modified GDLs (T60 plain, T60 slits2, S-PET-500) were imaged under operating conditions (60 °C, H2/air, amb. Pressure, 0.1 NL/min, A_{act} 0.16 cm^2). All GDLs were used in combination with the same self-standing MPLs to ensure the same boundary conditions at the catalyst layer.

For the perforated carbon GDL it was found out, that the water saturation in the GDL indeed is drastically reduced by the perforations. Water quickly accumulates in the slits, but they constantly release the produced water to the channel as desired. Figure 9 summarizes the main findings: the direct comparison (Figure a) shows how the water saturation in the GDL pore space is decreased when the perforated GDL is used. The slits equilibrate the profile in this view along all GDL areas below ribs and below channels. Figure b depicts for the perforated GDL the saturation profile along the channel, which is orthogonal to the slits direction as the current density was increased with time during the polarization curve. The narrow, sharp peaks in the saturation profiles identify the position of the slits.

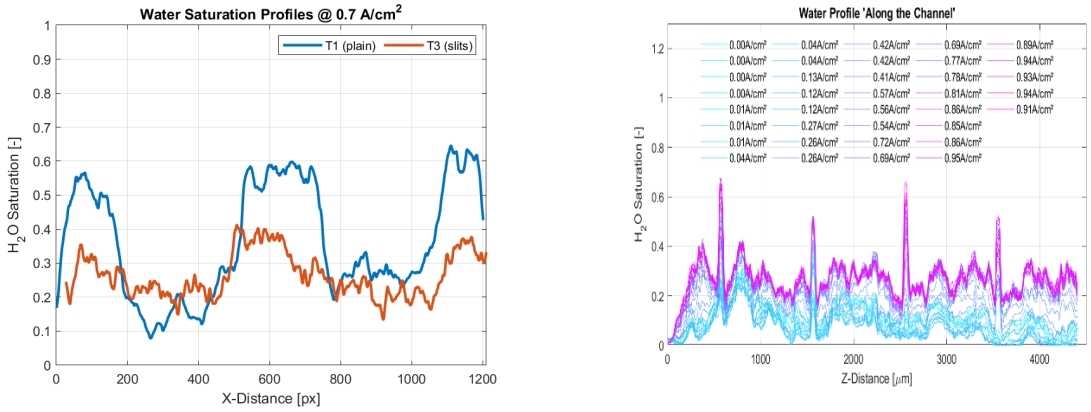

Figure 9: a) Water saturation profiles across the channels for the plain GDL (blue, standard Toray T1) and the perforated one (red, modified Toray T3). B) Water saturation profile along the channel of the perforated GDL (T3) as a function of current density, mirroring the passed time during the polarization curve.

For the S-PET-500 GDL a different water transport mode was observed during a polarization curve at lower RH: no water at all is present below the channels, as once emerged from through the throat at the bottom layer it can evaporate or moves away with the gas stream. Same observation is made for areas below the flow field ribs, that are connected to the channel. Only when fibers parallel to the gas channels block of some pores, water accumulates until close to full saturation. Figure captures this behaviour for a scan at a high current density of 1.5 A/cm^2.

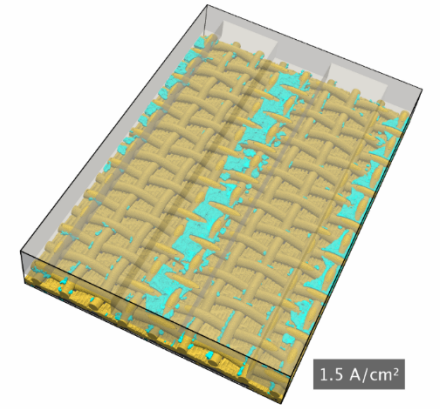

Figure 10: Volume Rendering of the gold-coated S-PET-500 cathode GDL (gold), the position below the flow fields (transparent gray, top) and water (light blue) during the experiment at 1.5 A/cm^2.

4 Conclusions

The stack testing experienced a long delay from the German project. The first stack was only received in mid 2020. With this stack only the begin-of-live performance characterization could be performed successfully. The durability test was ended after 50 hours due to mal-function of one of the cells. A durability test with a new stack will be performed after the ending of the current project.

For the GDL development it was observed that with deterministic structures enable direct water pathways in GDLs, which seem crucial for efficient product water removal. These structures lead to increased fuel cell power densities. Operando XTM showed that indeed the perforations transport the water fast away from catalyst layer to the channels. The perforations can be customized to the requirements of the cell or stack components and its operation profile.

The tests with woven GDLs from SEFAR (with intrinsic deterministic structure) were a big success and the performance under all operating conditions was improved, especially the power density at low operating temperatures and under high humidity. The simple, two-layered structure with only one throat seems to be the key to an improved water transport, as was observed also with operando XTM.

5 Outlook and next steps

Together with Sefar and Bopp, discussions and experiments are continuing for an optimized fabric design. The main tasks here are an optimized weaving to empty the GDL structure below the ribs, increasing the electrical conductivity at the catalyst/GDL interface, and new materials to guarantee sufficient electronic conductivity in general. A future project can be to vary the weaving or materials along the channel in a long cell, to tune GDL properties to the complex down the channel gradients in fuel cell stacks.

Both GDL modification paths turned out to be promising. The results are currently prepared for two publications. In the next experimental steps for conventional carbon GDLs, the base material will be changed to a newer type of carbon GDL. Furthermore, the large impact of the oxygen concentration on the performance will be further investigated with limiting current experiments.

6 National and international cooperation

6.1 National Cooperation

We gratefully acknowledge the cooperation with BOPP (esp. Reto Bärlocher) and Sefar (esp. Roland Steim) especially the provision of materials, the discussion and feedback.

Sefar AG, Hinterbissaustrasse 12, 9410 Heiden, CH

G. BOPP + CO. AG, Bachmannweg 21, 8046 Zürich, CH

6.2 International Cooperation

6.2.1 ASI Deutschland

The German Autostack Industry project provided the state-of-the-art sub-stack for testing and will provide further stacks in 2021.

7 Publications

C. Csoklich et al., Gas Diffusion Layers with Deterministic Structure for High Performance Polymer Electrolyte Fuel Cells, *ACS Applied Materials & Interfaces – Submitted, Manuscript attached*

Three more publication are work in progress and will be published in 2021.

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9 Appendix

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