Temperature dependent water transport mechanism in gas diffusion layers revealed by subsecond operando X-ray tomographic microscopy

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**HIGHLIGHTS**

- Subsecond and submicron operando X-ray tomography of liquid water in PEFCs
- Quantification of the water transport modes in GDL during cell startup
- Capillary-fingering dominates the water transport mode at 40 °C
- Phase-change-induced flow contributes significantly to the water transport at 80 °C

**A R T I C L E I N F O**

**Keywords:**
Polymer electrolyte fuel cell
Gas diffusion layer
Water transport
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**A B S T R A C T**

The product water of polymer electrolyte fuel cells (PEFCs) has to pervade the gas diffusion layer (GDL) and can lead to shortages of educt gas diffusion pathways to the catalyst layer hence to significant performance losses. Here we report on efforts made to enable subsecond and submicron operando X-ray tomographic microscopy (XTM) over a wide range of cell operating temperatures. The short XTM scan times allow to quantify the contributions of capillary-fingering and phase-change-induced flow on the overall water transport inside the cathode GDL at two different cell operating temperatures of 40 °C and 80 °C during a current ramp-up process. The results suggest that phase-change-induced water transport dominates the initial increase of saturation levels at typical automotive PEFC operating temperatures of about 80 °C, whereas capillary-fingering driven transport dominates the development of saturation from the beginning to stagnating stage at 40 °C.

1. Introduction

Polymer electrolyte fuel cells (PEFCs) are electrochemical devices that transform the energy chemically stored in hydrogen into electrical power, producing water and heat solely. Sophisticated water management is required to achieve high PEFC performance [1], considering the multiple water transport processes that take place inside the different cell components while keeping the membrane well humidified for good proton conductivity. At the same time, the accumulation of excessive product water has to be avoided as it is prone to saturate the catalyst and gas diffusion layer at the cathode, leading to unstable power output [2] and significant performance losses [3]. Thus efficient water removal through the cathode GDL is demanded, especially at high-current-density operations.

Two major water transport mechanisms for water emerging from the catalyst layer (CL) towards the flow fields (FF) are commonly considered, namely capillary-fingering of liquid water through the pores of the microporous layer (MPL) and the gas diffusion layer, and gas phase transport of water vapor as illustrated in Fig. 1. The capillary-fingering mechanism in the GDL has been described by Litster et al. [4] using confocal microscopy. The relationship between the capillary pressure that is necessary to invade the pores and the resulting saturation in the GDL was evaluated by different groups [5–7]. Temperature gradients typically present between the CL and FF and enhance the vapor phase transport by phase-change-induced (PCI) flow where water evaporates at the warmer CL and condenses near the colder FF side of the GDL as postulated by Owejan et al. [8] and quantified by Hatzell et al. [9] using high resolution

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neutron imaging. PCI flow is even able to transport all product water of the electrochemical reaction [10] and Weber and Hickner [11] calculated the temperature gradient needed to remove water at a given current density for a variety of temperatures. Carrere et al. [12] estimated the PCI flow dominated operation conditions by pore network modeling. Besides the broad application of neutron [13–15] and X-ray imaging [16–25] to localize liquid water in polymer electrolyte fuel cells, imaging based studies on PCI flow are rare and limited to ex situ conditions [9,26].

Both experimental observations and modeling works suggest that, apart from capillary pressure, phase change could be a key factor influencing the liquid water transport hence impacting cell performance. Detailed operando experimental validation is necessary to identify and quantify the water transport mechanism at different temperatures in the GDL. In this work, subsecond and submicron operando X-ray tomographic microscopy is employed to reveal the dominating water transport mechanism at two operating temperatures (40 °C and 80 °C) under fully humidified conditions during cell startup, mimicking a small area of a larger technical cell in a differential cell approach. The water distribution during the current ramp up process is investigated and analyzed for several locations in the GDL domain, including MPL cracks, GDL and GDL binder pores. Time-series data of the water distribution is recorded and analyzed, and individual water clusters are identified and classified in the GDL domain according to their connectivity to the microporous layer (MPL) or flow field to understand the water transport processes. The water cluster analysis is then used to conclude about the contribution of phase-change-induced and capillary-fingering driven water transport in the cathode GDL at two operating temperatures.

2. Experimental

2.1. PEFC components & setup

An X-ray tomographic imaging compatible PEFC (Fig. 2a) with an active area of 0.16 cm² (4.5 mm × 3.6 mm), which is beyond typical GDL representative equivalent area (REA) values [27–30] to ensure reproducible electrochemical performance, was employed [31]. The cell was assembled with two graphitic flow fields (Sigracet BMA5, SGL Carbon, Germany), two gaskets (FEP 129/75 μm for anode/cathode, Angst Pfister AG, Switzerland), two differential gas diffusion layers (anode/cathode GDL of SGL 34BC/24BC; 315/235 μm initial thickness; SGL Carbon, Germany) and a catalyst coated membrane (CCM, Gore® Primea® A510.1/M815.15/C510.4; 15 μm thick membrane and anode/cathode Pt loadings of 0.1/0.4 mg/cm²; active area shaped by laser...
ablation). The cathode GDL was thinner than anode GDL to reduce oxygen diffusion resistance and improve power generation efficiency. Compression units made of polyether ether ketone (PEEK) material were used to compress the sandwiched components resulting in nearly 25% compression rate for both anode and cathode GDLs.

2.2. Cell performance

Pristine cells with the same GDL types were pre-conditioned in the lab at 60 °C following a 24 h break-in protocol [22]. Prior to the beamtime experiments with X-ray radiation, the cells were operated and characterized by polarization curve measurements with a current density sweep rate of 0.05 A cm⁻² s⁻¹ at the same operating conditions used during the XTM imaging as provided in Table 1. Both anode and cathode gases were humidified using a Nafion tube humidifier immersed in a water bath with temperature regulated by a high thermal-precision

![Fig. 2. a) Schematic of the PEFC components; b) subsecond operando XTM setup at the TOMCAT beamline with a slip ring device used for continuous rotation during imaging; c) illustration of the selected field of view (yellow box) of the 26-fold magnification focusing on the GDL rib region; d) image processing pipeline including image filtering, segmentation and water cluster labelling. The water cluster categories are: BC (bottom-connected; red), TC (top-connected; green), FC (fully-connected; blue) and NC (not-connected/isolated; purple). The dashed yellow lines represent the position of the labeling criteria for FC (top) and BC (bottom) clusters. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)](image)

Table 1

| Parameter                          | Value         |
|------------------------------------|---------------|
| Pressure [atm]                     | 1             |
| Cell name [-]                      | T1            |
| Cell temperature [°C]              | 40            |
| Cell temperature [°C]              | 80            |
| Dry air speed [m s⁻¹]              | 7.6           |
| Dry air flow rate [mml·min⁻¹]      | 219           |
| RH of air [%]                      | 100           |
| Dry H₂ speed [m s⁻¹]               | 5.3           |
| Dry H₂ flow rate [mml·min⁻¹]       | 153           |
| RH of H₂ [%]                       | 105           |
| Current density range [A cm⁻²]     | 0–1           |
2.3. Subsecond operando XTM imaging

The subsecond operando X-ray tomographic microscopy (XTM) experiments were performed at the TOMCAT beamline of the Swiss Light Source at Paul Scherrer Institut (PSI). In order to capture the liquid water evolution and transport phenomena during cell start at the different temperatures under fully humidified conditions (see Table 1 for operating condition details), the current density was ramped-up linearly from 0 to 1.0 A/cm² within 20 s and kept constant thereafter for 280 s. During the 300 s of cell operation a total of 30 XTM scans were acquired, inhomogeneously distributed over time (Table 2). The total exposure time for the 30 XTM scans is about 9s, which is below the limit 12 s for 100 mV cell voltage bias [32]. Since performance degradation happens at lower dose than severe case that the dew point of the feed gas was higher than cell temperature (anode RH = 105% at 40 °C) it is presumed that minor amounts of water vapor condense into liquid form within the cell. High frequency resistance (HFR) data was collected by a Tsuruga E3566 AC milliohm meter (Tsuruga Electric Co., Japan) at 1 kHz.

2.4. Image processing pipeline

2.4.1. Phase contrast reconstruction

All collected radiograms were processed with a phase-retrieval method known as Paganin’s method [35] before reconstructing the 3D volume. The key parameters as inputs for the Paganin algorithm are beam energy, pixel size, sample-to-detector (STD) distance, δ and β, where δ and β are the real and imaginary parts of the refractive index n (n = 1 - δ - iβ) [36], respectively. For various purposes, like liquid water or GDL fiber segmentation, different ratios of parameter δ and β can be used to achieve bulk-smoother and/or edge-enhanced reconstructed images [37]. In this work, a set of δ and β value of 7.5E-8 and 1E-10 was used for all liquid water segmentation, and an additional value set of 5E-9 and 1E-10 was used for GDL fiber extraction purpose. For all reconstructions, a sample-to-detector distance of 0.02 m was used.

2.4.2. Image segmentation

In order to enable a quantitative analysis of water and pore volume in the GDL, all XTM datasets were segmented to classify water, void and fiber voxels following the image processing pipeline shown in Fig. 2d. Firstly, the reconstructed wet volume (operando) was aligned to the reference high quality dry volume, filtered with a 3D mean filter (kernel size of 4 × 4 × 4 voxels), and then subtracted with the dry volume to create a difference image showing only the water signal and noise. Secondly, the difference image filtered with an anisotropic diffusion filter [38] (5 iterations) was binarized with a threshold at 50% between the gray scale value of water and the background signal. After further hole filling and cluster clearing (<100 voxels) morphological operations, the liquid water structures were finally extracted. The solid GDL structures were segmented with both fiber and binder as one solid phase utilizing the above mentioned 2 sets of values. Finally, porosity can be obtained from the amount ratio of void voxels in the segmented GDL structures to the analyzed GDL volume. The water saturation is defined by the ratio of voxels classified as liquid water to the void voxels. Both porosity and water saturation analysis were applied for each in-plane slice resulting porosity and saturation profiles. The porosity determined by XTM will be lower than the values determined by e.g. mercury intrusion porosimetry because MPL and GDL binder are treated as completely solid in the segmentation since their sub-micrometer pores in the MPL and binder cannot be resolved with the used imaging setup. The analyzed domain for porosity and water saturation was 780 × 780 × 58 μm³ located in the cathode GDL area under the rib (Fig. 2c).

2.4.3. Pore size distribution

The continuous pore size distribution (PSD) method was applied to the segmented GDL solid utilizing the PSD function in ImageJ plugin developed by Münch et al. [39]. An in-plane 2D PSD analysis was used to label each pore space voxel with the largest radii of a circle it could belong to and fit completely in the pore domain. The continuous PSD
results can be obtained then from the histogram of radii.

2.4.4. Water cluster labelling

In order to distinguish the liquid water accumulation near to the flow field (under the rib) or near to the Catalyst/MPL position, the segmented water structures of each operando XTM scan were classified into 4 types in terms of 3D cluster connectivity [21], namely bottom-connected (BC), top-connected (TC), fully-connected (FC) and not-connected (NC) as illustrated in Fig. 2d. Water clusters with voxels located at or below the bottom yellow dashed line at the MPL/GDL interface or above the top yellow dashed line close to the GDL-flow field interface are considered as BC or TC type, respectively. If a cluster fulfills both BC and TC criteria, it is labelled FC. Clusters not being BC, TC or FC type are labelled NC. The classification of the different water cluster types enables to differentiate the origin of the liquid water clusters within the initial period of the time series XTM data and hence to conclude on the water transport mechanism driving the evolution of the water distribution.

3. Results

3.1. Electrochemical characteristics

Prior to the X-ray radiation exposure, the performance of the two PEFCs used for XTM imaging was characterized by polarization curve measurements at the conditions described in Table 1. The IR-free polarization curves are presented in Fig. 3a. Cell T1 and T2 show very similar electrochemical behavior achieving a current density of 1.0 A/cm$^2$ at about 0.6 V. The development of the current density and the IR-free cell voltage during the 300 s operando XTM experiment of cell T1 and T2 are plotted in Fig. 3b-c indicating the distribution of the 30 XTM scans as dotted lines. After the current density ramp-up finished at 20 s, the current density was kept constant at 1.0 A/cm$^2$ for the remaining 280 s. After the ramp-up period, the cell voltage remains constant with some fluctuations in a range of 20–40 mV and no significant radiation damage was observed during the 30 XTM scans (Fig. 3b and c).

3.2. Submicron XTM resolved structures

The SGL 24BC GDL substrate at the cathode has a complex solid structure consisting of carbon fibers, porous binder domains and a microporous layer (3D renderings in Fig. 4a and b). The pores defined by the GDL solid structure have a very broad size distribution with a peak at 5 μm and diameters of up to 100 μm (Fig. 4c and d). A significant amount of void can be found in pores smaller than 10 μm (Fig. 4c and d) which is difficult to be identified precisely with lower magnification XTM scans. Though the two assembled cells do not have identical GDL structures, their pore size distributions show very similar statistics within the analyzed GDL subvolumes, enabling a valid comparison of the different temperature conditions.

Exploiting the high spatial resolution of the 26-fold microscope, operando XTM allows the detection of liquid water in small and critical pores beyond the large GDL pores (Fig. 5g). Partially saturated MPL cracks (SGL 24BC type) can be easily observed in the difference (Fig. 5c) and segmented image (Fig. 5d). Liquid water in the binder pores (Fig. 5k) shows also a strong signal in the difference image and contributes to the total saturation. In addition, in the binder solid, where the nano-pore structures cannot be resolved with the present XTM setup, various liquid water saturation levels, appearing as gray scale gradients, can be observed (Fig. 5m–p) though precise quantification of this type of liquid water accumulation needs further development of advanced segmentation methods. The present high spatial resolution XTM setup is able to properly detect the great majority of the liquid water accumulations in a GDL containing complex structures [32].

3.3. Quantification of the water distribution

Already the qualitative evaluation of the XTM data can provide relevant insights into the water transport processes. Evidence of capillary-fingering and phase-change-induced water transport has been observed in the XTM data at the GDL-rib interface for both studied temperatures. At the beginning of the current ramp-up (5 s) merely any water clusters were found at both temperatures (see Fig. 6).

Liquid invasion through the GDL pores close to the MPL can be seen at 40 °C after 15 s (Fig. 6a) followed by liquid breakthrough to the next GDL pore 10 s later. After 35 s, the droplet forms a continuous liquid path and touches the surface of the flow field rib, which can be seen also in the IP slice. Then at 45 s, the liquid water expands further and saturates a larger domain of the pore, however, the previous path is captured in a disconnected status. In addition, small condensation clusters can be found at the time step of 25 s (IP slice).

At 80 °C, many condensed discrete water clusters can be identified near the flow field after 15 s in Fig. 6b. As the condensed liquid droplets continue growing and merging, after 45 s a high liquid saturation is...
Fig. 4. 3D surface rendering of the GDL solid structure of a) cell T1 and b) cell T2 and the corresponding pore diameters c) and d) with the inserts showing exemplarily 2D cuts of the circular pore diameter fitting.

Fig. 5. Liquid water clusters observed in Cell T1: a-d) MPL cracks, e-h) fiber pores, i-l) binder pores and m-p) binder solid areas; the circles presented in m-p) indicate a binder solid area partially filled with water, appearing as gray scale gradients.
observed at the GDL-flow field interface, but it remains difficult to conclude on the water connectivity based on the tomographic slices. Animations of the time-series tomographic slices (Fig. 6a and b) can be accessed as mmc1.mp4 and mmc2.mp4 in the supplemental information.

For more detailed insights into the water distribution, the through-plane water saturation profiles were analyzed for both temperatures at selected XTM time steps as shown in Fig. 7a and b. The corresponding water volume fraction profiles (Fig. 7c and d) and the GDL porosity...
profiles (Fig. 7) for both cells are also provided as references. At the operation temperature of 40 °C, the water saturation profiles between 0 s and 15 s portrait a “downhill-like” continuous descending trend from the MPL domain towards the flow field with very low saturation near to flow field (Fig. 7a). Only after breakthrough happened at around 25 s, the water saturation at the FF-GDL interface starts to increase and the saturation profile starts to level out and stabilize after one to two minutes. Similar saturation profile patterns were observed during ex situ liquid injection XTM experiments [6,16,40]. At 80 °C the water saturation profiles between 0 s and 25 s show a different more “valley-like” shape with high saturation values at both end positions, but very low saturation values in the middle position of the GDL (Fig. 7b). Between 35 s and 55 s, the saturation in the middle position of the GDL increases rapidly and the water saturation profile stagnates slowly after 55 s. The differences in the stabilized water profiles between 40 °C and 80 °C are likely governed by the local porosity profiles with slight differences between the imaged sections of cell T1 and T2 (eg. Fig. 7e and f). The porosity difference in the bulk GDL suggests that the imaged GDL section of 0.6 mm² does not yet resemble a representative equivalent area for SGL 24BC GDL. The fact that the primary MPL and binder pores could not be resolved with the used setup and are treated as solid makes the porosity profiles unrealistically approach zero towards the CL and level out around 0.6 close to the flow fields and consequently results in higher saturation values than considering the actual porosity distribution. The water volume fraction profiles shown in Fig. 7c and d are free from this bias and better comparable to other radiography imaging data.

3.4. Water cluster connectivity

The evolution of the liquid water volume of the 4 types of water clusters (classified by their connectivity) captured by the 30 XTM scans is presented in Fig. 8, with the corresponding 3D renderings of cluster connectivity labelled water accumulations presented in Fig. 6c and d. At 40 °C, in the first 25 s, there is a similar share of bottom-connected (BC), top-connected (TC) and non-connected (NC) type of water clusters with slightly higher contribution of up to 2 nL for BC and TC from 25 s on (see Fig. 8a). After 35 s a first full-connected (FC) type water cluster shows up (see also Fig. 6c) and thereafter the FC type becomes dominating.

Opposite to that, at 80 °C TC type water clusters are dominating in the first 30 s. A wide spread coverage of the flow field rib by individual and disconnected TC type clusters can be observed already after 15 s (see Fig. 6d), merging afterwards and increasing their total volume to 5 nL after 35 s (see Fig. 8c). FC type water clusters start to drive the water volume increase after 30 s, while the volume of TC type clusters quickly decreases from thereon. BC and NC type clusters remain at relatively low volumes of 1–2 nL each. For both cells, the liquid volume stabilizes about 50–60 s after the current density ramp-up started. During constant current operation some fluctuations of 2–4 nL of TC and FC type water clusters can be observed and have been correlated to the fluctuating existence of small water bridges between water clusters in that size range. Because many of these water clusters are already connected to the lateral boundaries of the limited field of view of the high magnification microscope, these effects should not be over-interpreted since the existing
percolation paths to water clusters beyond the field of view would very likely result in stable high values of FC type but low TC type water clusters if a larger field of view would be considered. Animations of the time-series volume renderings (Fig. 6 c-d) can be accessed as mmc3.mp4 and mmc4.mp4 in the supplemental information.

4. Conclusions

Subsecond and submicron operando X-ray tomographic microscopy (XTM) was, for the first time, applied to reveal the dominating water transport mechanism inside the cathode GDL at two different temperatures during a current ramp-up process. With voxel sizes of 0.4 μm, fine structural details such as pores in SGL binder can be resolved and liquid water was detected therein as well.

At both temperatures of 40 °C and 80 °C, capillary-fingering as well as phase-change-induced water transport was observed, though with different contributions. At 40 °C the through-plane water distribution was "downhill-like" from the MPL to the flow field similar to observations in ex situ liquid injection experiments. The water distribution was initially driven by bottom-connected water clusters located at the GDL-MPL interface, while already after 50 s capillary fingering growth made fully-connected clusters that span from the GDL-MPL interface to the GDL-flow field to dominate the water distribution. At a typical automotive PEFC operating temperature of about 80 °C, the through-plane water distribution was more "valley-like" with the saturation increasing mainly at the GDL-MPL and GDL-flow field interface in the first 25 s. Top-connected water clusters located at the GDL-flow field interface dominated the water distribution in the first 30 s. These clusters were initially small and disconnected, but grew and merged over time. As soon as the top-connected phase-change-induced water clusters merged with the capillary-fingering driven bottom-connected clusters, fully-connected water clusters dominated the water distribution after only about 40 s.

The presented work demonstrates the possibility of investigating water dynamic phenomena with both high spatial and temporal resolution utilizing synchrotron XTM facilities. This enables a broad field of possible studies of liquid water dynamics in porous transport layers in PEFCs. Fundamental understanding of water transport at different temperatures addressing the contribution of capillary-fingering and phase-change-induced water transport is vitally important to improve the GDL materials design, dynamic two-phase water transport models, as well as operation strategies for advancing water management in PEFCs.

Fig. 8. Liquid volume evolution for 2 cells operated at 40 °C and 80 °C: a) and c) are the zoomed profiles for initial 65 s; b) and d) are the full profiles for overall 300 s. Color labelling is for different type of clusters. BC: bottom-connected, FC: fully-connected; TC: top-connected; NC: not-connected/isolated clusters. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)
Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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Author contributions

Hong Xu: Conceptualization, Methodology, Software, Formal analysis, Data Curation, Investigation, Writing – Original Draft, Writing – Review & Editing, Visualization. Shinya Nagashima: Project administration, Conceptualization, Writing – Review & Editing. Hai P. Nguyen: Project administration, Conceptualization, Writing – Review & Editing; Keisuke Kishita: Project administration, Conceptualization, Writing – Review & Editing; Federica Marone: Investigation, Writing – Review & Editing, Resources, Funding acquisition. Felix N. Büchi: Conceptualization, Resources, Writing – Review & Editing, Supervision, Project administration, Funding acquisition. Jens Eller: Conceptualization, Supervision, Project administration, Investigation, Methodology, Data Curation, Writing – Review & Editing.

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