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Water management in novel direct membrane deposition fuel cells under low humidification

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A B S T R A C T

Polymer electrolyte membrane fuel cells (PEMFCs) fabricated by direct membrane deposition (DMD) were shown to work even at dry conditions without significant deterioration of the membrane resistance. Here, in situ neutron radiography is used to investigate the water management in those fuel cells to uncover the phenomena that lead to the robust operation under low humidification. A constant level of humidification within the membrane electrode assembly (MEA) of a DMD fuel cell is observed even under dry anode operation and 15% relative humidity on the cathode side. This proves a pronounced back diffusion of generated water from the cathode side to the anode side through the thin deposited membrane layer. Over the entire range of polarization curves a very high similarity of the water evolution in anode and cathode flow fields is found in spite of different humidification levels. It is shown that the power density of directly deposited membranes in contrast to a 50 µm thick N-112 membrane is only marginally affected by dry operation conditions. Water profiles in through-plane direction of the MEA reveal that the water content in the DMD fuel cell remains steady even at high current densities. This is in contrast to the N-112 reference fuel cell which shows a strong increase in membrane resistance and a reduced MEA water content with raising current densities. Thus this new MEA fabrication technique has a promising perspective, since dry operation conditions are highly requested in order to reduce fuel cell system costs.

Keywords:

PEMFCs
Direct membrane deposition
Neutron radiography
Water management
Back diffusion

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Introduction

Direct membrane deposition (DMD) is a novel MEA fabrication method, where the membrane layer of a PEMFC is directly deposited onto its anode and cathode gas diffusion electrodes [1]. This MEA fabrication method enables very low membrane resistances below $13 \text{ m}\Omega \text{ cm}^2$ and therefore high fuel cell power densities beyond 4.0 W/cm^2 with H_2/O_2 as feed gases at $300 \text{ kPa}_{\text{abs}}$ and beyond 1.3 W/cm^2 under stoichiometric $1.2/2.0 \text{ H}_2/\text{air}$ operation at $300 \text{ kPa}_{\text{abs}}$. The high performance was linked to a very low membrane thickness of about $12 \text{ }\mu\text{m}$ and an improved interface between catalyst layer and the membrane itself [1,2]. Despite the high power densities another significant difference was identified compared to conventionally cast membranes: The cell polarization measurements in the work of Klingele et al. [1] reveal that the ionic resistance increased only slightly at low humidification. Even when the humidification of the reactant gases was reduced to zero, the maximum cell power dropped only by about 10%. This finding diverged from the known characteristics of fuel cells with conventional Nafion[®] membranes [3–5] and is therefore of high interest for further investigation. Even though dry operation of fuel cells with Nafion[®] membranes was reported in literature before, a more significant drop in cell power density between 20 and 40% was usually observed [3]. To enable dry fuel cell operation by self-humidification, complex composite structures such as thin Nafion/Pt/SiO₂ [6], Nafion/Pt-clay [7] or sulfonated poly(ether ether ketone) (SPEEK)/Pt-Zirconia [8] composite membranes have been proposed. Klingele et al. demonstrated the operation of a directly deposited pure Nafion membrane without any external humidification. This finding was attributed to a strongly facilitated back diffusion of water from the cathode to the anode side through the about $12 \text{ }\mu\text{m}$ thin directly deposited membrane. This back diffusion was believed to humidify the membrane sufficiently even under dry conditions and at high current densities. To prove this hypothesis, this work presents in situ neutron imaging of fuel cell operation with dry anode feed gas to analyse the amount of water in the MEA under low humidification.

Neutron imaging is well suited to investigate water evolution and has been widely employed in in situ fuel cell experiments [9–14]. The strong attenuation of neutrons in water enables imaging liquid water evolution within a fuel cell in operation. The neutron-based analysis of various fuel cell operation conditions [15], flow field designs [16] or gas diffusion materials [17] was reported in literature previously using conventionally cast membranes. Water back diffusion, even in a thick Nafion[®] N-117 membrane, was recently investigated by Iranzo et al. [18]. These studies motivate the use of neutron imaging for the characterization of the water distribution in DMD fuel cells.

In situ neutron radiography

In situ neutron radiography experiments were performed at the neutron beamline “ANTARES” at Heinz-Maier-Leibnitz Zentrum (MLZ) at the Technische Universität München

(TUM). The setup was described in detail by Calzada et al. [19] and is shown schematically in Fig. 1a). The neutron beam coming through a pinhole from the reactor can be used for imaging purposes in the two separate beam chambers. The fuel cell, shown in Fig. 1b), was located in the first measurement chamber (blue area in Fig. 1a). We used a specifically designed aluminium fuel cell fixture (see Fig. 1b). This ensured sufficient transparency of the PEMFC for the beam. The fuel cell was operated from outside the beam chamber via 5 m long heated gas lines. This ensured that the sensitive electronics of the fuel cell tester could be placed outside the beam chamber and thus were not damaged during the experiments. For the neutron experiments, the fuel cell was placed in-plane with the beam direction to observe the dynamic water evolution inside its anode and cathode flow channels separately (Fig. 1b). A 18 mm pinhole was used, resulting in an L/D ratio of 500 at the sample position. The effective pixel size was about 27 μm and was determined experimentally from the known geometry of the micro-milled gas flow channels of 500 μm width. For the neutron images during polarization measurements shown in Fig. 3, an integration time of 60 s was chosen to enhance water sensitivity. To track the water evolution as dynamically as possible during a steady potential, an integration time of 5 s was chosen. Due to this short integration time, the recorded radiographies at steady state conditions were rebinned to a resolution of 137 μm in order to achieve a sufficient signal-to-noise ratio as described elsewhere [20].

To extract the actual water generation in the flow channels, the neutron absorption of the dry fuel cell was taken as reference. The water profiles shown in Figs. 2 and 3 were generated by summarizing the water signal in the fuel cell. The offset of the CCD detector (Andor, Ikon L, 2048 $\times$ 2048 pixels) was eliminated by subtraction of a dark image with closed beam shutter. Gamma spots were corrected in the post-processing. Before each in situ experiment a neutron image under OCV conditions was recorded to exclude a falsification by residual water from previous operation.

Sample preparation and fuel cell operation

To fabricate the DMD fuel cell, one layer of Nafion[®] dispersion (1 part Nafion[®] D2020 mixed with two parts 1-propanol) was inkjet-printed onto 5 cm² anode and cathode gas diffusion electrodes (GDEs) purchased from Paxitech SAS. The GDEs were loaded with 0.5 mg_{Pt}/cm² at 70% Pt/C and contained Nafion[®] as ionomer. The thickness of each catalyst layer was about 10 μm (measured by scanning microscope imaging on MEA cross-sections), the thickness of each gas diffusion layer (in the compressed state) was approximately 200 μm , according to the manufacturer. The fuel cell was assembled by placing the two GDEs covered by the printed membrane layers face to face. A 50 μm thin PTFE foil was used as subgasket to prevent parasitic currents and gas crossover occurring at the edges of the GDEs. The subgasket reduced the active fuel cell area to 4 cm². The fuel cell (shown in Fig. 1b) was tightened with a torque of 1 Nm. A detailed description of the sample preparation including the printing process was published previously [1,2]. As reference, we used an N-112 Nafion[®] membrane, coated with catalyst layers of identical Pt loading

Fig. 1 – Schematic of the ANTARES setup at FRM-II (a). The neutron beam exits the reactor and passes through two measurement chambers. The fuel cell (b) is located in the first measurement chamber (blue shaded area in a). The fuel cell is operated with opposing feed gas flows from outside the beam chamber. The fuel cell is placed in-plane in the beam direction to analyse water evolution separately in anode and cathode flow channels. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 2 – In a), the evolution of current density (left) and high frequency resistance (right) over time is plotted for the 35%/35% DMD fuel cell (green), the dry/15% DMD fuel cell (blue) and the 35%/35% N-112 reference cell (yellow) at a fixed cell voltage of 0.5 V. The vertical lines indicate the different points in time ($t_1 = 20$ s, $t_2 = 140$ s, $t_3 = 560$ s) of the image acquisition. The orientation of the fuel cell is indicated in the neutron radiography of the dry cell in b). The scale bar corresponds to 1 mm. c) Shows the corresponding water profiles as a function of the position within the MEA between anode (left side) and cathode flow field (right side) at different times for each cell. The operation conditions were: 70 °C, 1.5/2.5 H₂/air, atmospheric pressure. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

and stacked between the same gas diffusion media (Paxitech SAS).

For the characterization of the DMD and the reference fuel cell, a Scribner 850e fuel cell testing system was used. Polarization data was recorded by sweeping the current from high to low voltages. The data for steady state measurement was obtained by applying a fixed potential of 0.5 V for 10 min. The high frequency resistance (HFR) was measured in situ at a fixed frequency of 3.2 kHz by a frequency response analyser

which is integrated into the Scribner 850e system. The HFR corresponds to the series resistance of the fuel cell and its main contributions are the ionic membrane resistance and the interface resistance between membrane and catalyst layer ionomer [13]. The fuel cell was heated to 70 °C whereas the relative humidity (RH) was adjusted to dry/15% (anode/cathode) and 35%/35%. Each state of humidification was held for at least 3 h in order to avoid any artifacts due to remaining humidity from prior measurements and to fully equilibrate the

Fig. 3 – In a) polarization data and HFR measurements of the 35%/35% DMD fuel cell (green), the dry/15% DMD fuel cell (blue), and the 35%/35% N-112 reference cell (yellow) are shown. In b) the averaged corresponding water profiles are shown at different current densities of 125 mA/cm^2 , 500 mA/cm^2 and 1250 mA/cm^2 . The operation conditions were: 70 °C, 1.5/2.5 H_2 /air, atmospheric pressure. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

fuel cells. Prior to the dry/15% RH measurement the fuel cell was kept at 70 °C and was purged with dry N₂ gas for 15 h to exclude falsifications due to remaining anode humidification. In Ref. [1], the DMD fuel cell was operated under entirely dry conditions (anode and cathode side) at 300/300 kPa_{abs}. In this work, we operate a DMD fuel cell with dry anode and a minor level of cathode humidification of 15% RH even in ambient conditions without artificial pressurization. As depicted in Fig. 1b), an opposing stoichiometric reactant flow of 1.5/2.5 H₂/air was applied to the fuel cell.

Results and discussion

Two experiments were performed to investigate the diverging water management in a DMD fuel cell compared to a commercial Nafion® N-112 CCM fuel cell:

In the first experiment the dynamics of water evolution from application of a voltage step to steady state condition was investigated. A fixed potential of 0.5 V was applied to the fuel cell for 10 min and water evolution over time was examined (Fig. 2). The generally long time until the fuel cell converges towards steady-state condition after the high voltage step to 0.5 V was attributed to the configuration of the test setup: Due to the 5 m long gas lines and moderate stoichiometric gas flow the stabilization of the current density took several minutes. In situ neutron radiographies with 5 s integration time were recorded continuously during the current density measurement. This enabled dynamic observation of water formation in the fuel cells. Since current density and HFR was logged with a precision of 1s during the 10 min steady state potential, water management and fuel cell performance and resistance could be correlated.

Fig. 2a) shows the temporal evolution of the current density and the HFR. A significantly higher current density for the DMD fuel cell was found compared to the N-112 reference sample. The current densities of the DMD fuel cell steadily

converged to stable values (1300 mA/cm^2 at 35%/35% RH, 1100 mA/cm^2 at dry/15% RH). In contrast, the current density of the N-112 sample showed non-monotonic behaviour. It raised first and then dropped to a constant value. As can be seen in Fig. 2a) this was linked to an increasing HFR value over time for the N-112 sample. The increase of the HFR was a hint for a drying-out of the N-112 membrane. For the DMD sample a very constant evolution of the HFR was observed which was a sign for a continuous and sufficient humidification of the thin membrane. This was confirmed by the measured water profiles across the active fuel cell area which are shown in Fig. 2c) for three times ($t_1 = 20 \text{ s}$, $t_2 = 140 \text{ s}$, $t_3 = 560 \text{ s}$). The position of the MEAs was depicted with bold lines in the range of about -0.25 mm and 0.25 mm . The water content in the DMD fuel cell MEA for the two different states of humidification (dry/15% RH and 35%/35% RH) is comparable over the entire time range, which is in good agreement with the similar stable current density and HFR evolution. Additionally a huge amount of water was found on the anode side in both cases, proving an expressed back diffusion of water from the cathode to the anode side. For the reference sample with N-112 membrane we found a different water formation pattern. In the start-up phase the N-112 fuel cell MEA was sufficiently humidified, as shown for the time t_1 in Fig. 2c). At t_2 the water content in the membrane was lowered over time during the operation at 0.5 V which was well reflected in the starting HFR increase of the N-112 fuel cell (Fig. 2a). At t_3 the water content was lower than in the DMD sample, emphasizing the advantageous water management in the DMD fuel cell. Again the HFR evolution was well reflected in the measured water distribution: The measured increase of the HFR from $100 \text{ m}\Omega \text{ cm}^2$ at t_2 to about $150 \text{ m}\Omega \text{ cm}^2$ at t_3 in the N-112 fuel cell shown in Fig. 2a) came along with a reduction of the water content in the MEA as can be observed in Fig. 2c). The observed water patterns were in good accordance to a neutron imaging study of a fuel cell with identical membrane (Nafion N-112) [21]. The observed accumulation of water on the anode side at $t_2 = 140 \text{ s}$

and its decrease at $t_3 = 560$ s for all three samples was likely due to the stoichiometric gas flow (1.5/2.5) which correlates with increasing current density. With raising current density an increasing gas flow facilitates the purging of the flow channels. Furthermore the anode side was fed with pure hydrogen, whereas the cathode side was fed with air (21% pure oxygen fraction). This led to a significantly higher total gas flow on the cathode side, which also favoured a faster water removal in the cathode flow channels.

In a second experiment polarization data was recorded at two different levels of humidification. For each current density step a neutron image was recorded. The polarization curves for the N-112 reference sample under 35%/35% RH and the DMD cell under 35%/35% RH and dry/15% RH are shown in Fig. 3a). At a level of humidification lower than 35%/35% RH it was impossible to operate the N-112 reference fuel cell. Even under slight loads a too high HFR led to an immediate breakdown of the N-112 fuel cell voltage. For the DMD fuel cell the maximum power density (not shown) only drops by about 14% from 650 mW/cm² (at 35%/35% RH) to 560 mW/cm² (at dry/15% RH). In comparison, only 200 mW/cm² are obtained for the N-112 reference cell at 35%/35% RH. An even more significant difference was found in the evolution of the HFR. While the HFR showed only a diminutive increase for the DMD fuel cell when the humidification was reduced from 35%/35% RH to dry/15% RH, the HFR of the N-112 fuel cell reached already at 35%/35% RH values of up to 250 mΩ cm². This was about five times higher than the reported resistance values for this Nafion® membrane under fully humidified conditions [5]. The increase of the HFR with increasing current density was a hint for the overcompensation of the water back diffusion by the electroosmotic drag of water molecules from the anode to the cathode side, drying out the N-112 membrane [5,22]. This was in accordance with the results shown in Fig. 2 and previous publications, where the electroosmotic drag was found to dominate the water transport at high current densities [23–25]. This strongly increased HFR was a hint for a reduced proton conductivity of the N-112 membrane due to poor humidification, which was not observed for the thin DMD fuel cell under identical operation conditions.

In Fig. 3b), the water profiles in the DMD fuel cell at dry/15% RH and 35%/35% RH and the N-112 reference fuel cell were compared at different current densities to link the diverging HFRs to differences in the water management. Regarding the DMD fuel cell only minor differences in the water profile were detected between the two states of humidification over the entire current density range from 0 up to 1500 mA/cm². In the supplementary section a video sequence is provided showing the evolution of the neutron images of the DMD fuel cells in both states of humidification with raising current density. The high similarity in spite of different levels of humidification is in excellent agreement with the HFR measurements in which also no significant increase of the ionic resistance was observed. Even though the anode humidification was reduced to zero, we found a nearly identical water evolution over the entire range of the current density. Thus the water on the anode side in Fig. 3b) observed at the current density j_2 likely originated from back diffusion of generated water from the cathode side. Regarding the N-112 reference cell the increase of the HFR with raising current density was confirmed by a

reduction of the total water content in the MEA over time. The water content in the MEA was reduced from j_1 to j_2 where a lower water content was found compared to the DMD fuel cell MEA.

Supplementary video related to this article can be found at <http://dx.doi.org/10.1016/j.ijhydene.2016.05.018>.

The results for the DMD sample with 12 μm thin membrane are in good agreement with results from industry, where fuel cells with extremely thin membranes (down to 5 μm thickness) also showed very low membrane resistances and stable operation under low RH [26,27]. However, the membrane resistance is not the only reason for the improved power of DMD fuel cells. As reported in the work of Klingele et al., moreover a reduced charge transfer resistance was found for the DMD fuel cell [1]. This became in particular evident using electrodes with low Pt-loading, where at high current densities a strong deviation was observed between a CCM and DMD sample in the iR-free representation. In recent literature new concepts with patterned membranes revealed that in spite of identical membrane thickness a modified catalyst–membrane interface can strongly improve the cell performance [28,29]. These observations are in good agreement with the observed characteristics of DMD fuel cells.

Conclusion

A fuel cell with directly deposited membrane was operated at low levels of humidification to investigate its liquid water transport behaviour in the MEA. In situ neutron imaging was performed in order to unravel the robust operation of DMD fuel cells with 12 μm thin membranes under dry conditions. The presence of water on the non-humidified anode side proved the facilitated back diffusion of reaction water from the cathode to the anode side. The continuous membrane humidification via back diffusion explained the significantly improved fuel cell performance under dry conditions: Even though the humidification was reduced from 35%/35% RH to dry/15% RH only a marginal increase of the HFR was measured. In consequence a very similar water distribution was found in the MEA and in the anode and cathode flow channels comparing the two states of humidification. These nearly identical water patterns and similar HFR values despite of two different humidification levels confirmed that in the DMD fuel cell back diffusion compensated the electroosmotic drag even at higher current densities beyond 1200 mA/cm^2 resulting a very stable operation at high current densities. Thus the neutron measurements in this work contributed to a better understanding of the advantageous characteristics of DMD fuel cells.

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