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Physical degradation of membrane electrode assemblies undergoing freeze/thaw cycling: Diffusion media effects

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Abstract

In this work, the effects of properties of diffusion media (DM) (stiffness, thickness and micro-porous layer (MPL)) on the physical damage of membrane electrode assembly (MEA) subjected to freeze/thaw cycling were studied. Pressure uniformity of the diffusion media onto the catalyst layer (CL) was determined to be a key parameter to mitigate freeze-induced physical damage. Stiffer diffusion media, enabling more uniform compression under the channels and lands, can mitigate surface cracks, but flexible cloth diffusion media experienced severe catalyst layer surface damage. The thickness of the diffusion media and existence of a micro-porous layer were not observed to be major factors to mitigate freeze-damage when the catalyst layer is in contact with liquid.

Interfacial delamination between diffusion media and catalyst layers, but not between the catalyst layer and membrane, was observed. This permanent deformation of the stiff diffusion media in the channel locations as well as fractures of carbon fibers increased electrical resistance, and may increase water flooding, resulting in reduced longevity and operational losses.

Although use of a freeze-tolerable MEA design (negligible virgin cracked catalyst layers with thinner reinforced membrane) [S. Kim, M.M. Mench, J. Power Sources, in press] with stiff diffusion media can reduce the freeze-damage in the worst case scenario test condition of direct liquid contact, extensive irreversible damage (diffusion media/catalyst layer interfacial delamination) was not completely prevented. In addition to proper material selection, liquid water contact with the catalyst layer should be removed prior to shutdown to a frozen state to permit long-term cycling damage and facilitate frozen start.

Keywords: Polymer electrolyte fuel cell; Degradation; Freeze/thaw; Diffusion media; Membrane

1. Introduction

One of the remaining technical challenges for polymer electrolyte fuel cell (PEFC) commercialization is to achieve shutdown to a frozen state and rapid start from frozen conditions without damage. PEFCs generate water as reaction product, so when PEFCs are subjected to sub-freezing environments without removal of residual water, they can experience irreversible damage. There exist conflicts in the literature regarding freeze-damage [1–11]. One important result shows that fuel cells dried to remove all liquid water during the shutdown experience neither observable physical damage nor electrochemical losses by freezing [1–4]. However, there are conflicting results in case of a cell with no significant purge. Some reported physical damage, performance loss, and electrochemical loss (electrochemical surface area, interfacial and charge transfer resistance increase) [2,5–8]. Physical damage includes membrane failure (holes and cracks), catalyst cracks and delamination, pore distribution change, and gas diffusion media (DM) fracture. Others observed no significant performance loss even without dry purge during the shutdown [9–11]. Freeze-damage can result from coupled factors including fuel cell design (flow field plate and stack structure), operation (water redistribution on shutdown) and materials (membrane electrode assembly (MEA) and diffu-

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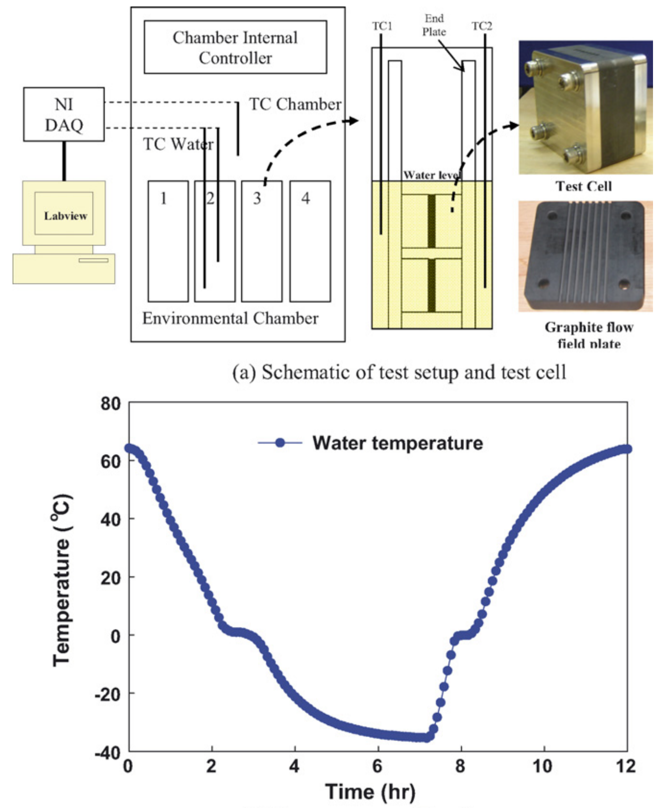
sion media). Thus, fundamental understanding of several factors on freeze-damage is required.

Kim and Mench [12] performed extensive an ex situ study of micro-structure effects of MEA on freeze-damage. This investigation of MEAs and diffusion media subjected to a submerged freezing environment has revealed very strong direction for the material choices in the PEFC and helped to conceptually validate the previous computational model performed by Mench and coworkers [13–15]. Specifically, the membrane was found to be a source of water that can damage the MEA upon thermal cycling to −40 °C. Damage was found to occur almost exclusively under the channel, and not under the land, due to the high overburden pressure of the land. From the fact that there was no damage under the land in the worst case scenario of Ref. [12] (direct liquid water contact with no diffusion media protection), the channel/land ratio and the channel width are also important factors. Although the larger land can be better for freeze/thaw (F/T), at a certain point it would cause dead zones in the center of the land and worsen flooding in high relative humidity conditions. The most robust material found to reduce F/T damage was determined to be a crack free virgin catalyst layer (CL) and a dimensionally stable membrane (e.g. reinforced) with low water content (e.g. thinner membrane), with diffusion media protection.

In the previous study [12], the role of the DM was not yet fully explored. That is, while it was obvious that the diffusion media play a critical role in damage mitigation, the influence of DM stiffness, thickness, and the presence of a micro-porous layer (MPL) was not explored. Thus, effects of these DM properties on freeze-damage of MEA were studied and are reported here.



(a) Schematic of test setup and test cell

(b) Temperature profile of water

Fig. 1. Schematic and thermal profiles of test setup used for the ex situ freeze/thaw thermal cycling tests. (a) Schematic of test setup and pictures of a test cell and a graphite flow field plate and (b) thermal profile of water in the test vessel per one freeze/thaw cycle.

2. Method of approach

Test cells were thermally 30 cycled between −40 and 70 °C in the water-submerged conditions. Fig. 1(a) shows a schematic of the ex situ test setup consisting of four test vessels, an environmental chamber (Thermal Product Solutions, Model: T10S-1.5), and a National Instruments Labview^{TM}-based data acquisition system. As shown in Fig. 1(b), one freeze/thaw thermal cycle took 12 h, and test cells were maintained more than 5 h below the water freezing point (0 °C): (1) 7.25 h for cooling, and (2) 4.75 h for heating. Thermal boundary conditions of two test cells in the same test vessel were identical, which enabled comparison.

The MEA was sandwiched between diffusion media, and then compressed by graphite flow field plates and stainless end plates with the same compression pressure as a typical fuel cell (approximately 1.0–1.5 MPa). Note that the MEAs in the test cells were fully hydrated even though hydrophobic diffusion media with a micro-porous layer was used. The detailed method for these ex situ tests was described in a previous paper [12].

Four kinds of diffusion media were investigated: (1) one flexible cloth type (CARBEL-CL^{TM}, W.L. Gore and Associates, USA), and (2) three rigid non-woven types (SGL series, SGL Carbon Group, USA). The CARBEL-CL^{TM} consists of a macro-gas diffusion layer and a MPL. The non-woven types of DM consist of carbon felt type (SGL 10BA and 10BB) and carbon paper type (SGL 25BC). SGL 10BA has no micro-porous layer, but a micro-porous layer is coated on the one side of the 10BB and 25BC. Macro-gas diffusion layers of SGL 10BB and 25BC, and SGL 10BA DM have the same PTFE content (5 wt%). The micro-porous layer on the SGL 10BB and 25BC DM has 23 wt% of PTFE content. The thickness of SGL 25BC and 10BB DM is 235 and 415 μm, respectively. The material properties supplied by manufacturers are summarized in Table 1. In addition to liquid submerged F/T cycling tests (worst scenario), vapor exposed F/T cycling tests were also performed using the SGL 10BB material and virgin non-cracked catalyst layer with a 35 μm reinforced membrane. Additionally, submerged tests with non-freezing conditions (5–70 °C) were also performed to assure the observed damage was a direct result of the freeze/thaw process.

The most tolerable MEA (a virgin non-cracked CL with 18 μm reinforced membrane) determined from the previous study [12] was used for a majority of testing. In some tests to

Table 1
Material properties

DM type	Thickness (μm)	PTFE content (wt%)	Existence of MPL
CARBEL-CL^{TM}	400	N/A	MPL
SGL 10BB	415	5	MPL
SGL 25BC	235	5	MPL
SGL 10BA	390	5	None

determine the effect of DM thickness, a virgin non-cracked CL with 35 μm reinforced membrane was used. Note that composition and micro-structures of the catalyst layer and the membrane are identical for all MEAs used.

F/T cycled samples were observed by scanning electron microscope (SEM) to investigate physical damage. To observe surface and cross-sectional images of cycled MEAs with DM, the DM should be removed without damage. The cycled DM-attached MEA was cut across the channel dimension with 2–3 mm width. Then, the DM sample was cut from the surface side intended for observation, enabling the DM to be removed with negligible damage to the surface. A new razor blade was used to slice MEA surface, providing sectional samples. The cutting direction of the blade can be important, because the sectioning itself may cause delamination. During sectioning, the membrane/electrode interface under the membrane (bottom part in the direction of cutting) may be delaminated in this manner, but delamination of any interface of the top part as a result of sectioning should be negligible. Figures in this work are shown as cut from top to bottom, so that observed delamination on the top surfaces is relevant. A more detailed description of the sample preparation for SEM was described in the previous study [12].

To investigate the effect of F/T cycle number, tests with 30 and 100 F/T cycles were conducted with both SGL 10BB and SGL 25BC DM. In addition to SEM observation, high frequency resistance (HFR) of the tested samples was also measured at 1 kHz by an Agilent 4338B milliohmmeter to correlate physical damage to interfacial resistance change. The F/T cycled samples, as well as non-cycled virgin samples were evaluated for comparison in the fully hydrated state at the ambient temperature and under identical compression.

3. Results and discussion

3.1. Effect of DM stiffness

As shown in Fig. 2, after 30 F/T cycles from −40 to 70 °C, the catalyst layer under the channel experienced severe surface cracks when a flexible cloth DM with MPL was used, but surface cracks were prevented using stiff carbon paper type DM with MPL (SGL 10BB). Note that the virgin surface crack density of the tested MEAs was negligible, and catalyst layers under the land were not damaged in all cases. From the previous study [12], the catalyst layers under the land were not damaged regardless of MEA micro-structures. Additionally, no damage was observed for cycling from 5 to 70 °C, indicating all damage observed is a unique result of freezing. This result agrees with computational model results [13–15] that indicate the higher compression pressure under the land can suppress the ice lens growth at the interfaces (CL/membrane, CL/DM).

Interfacial delamination can occur when a shear force induced by ice expansion or ice lens formation overcomes compressive forces from the material and overburden pressure (assembly compressive force). In a fuel cell with multi-layered structures, stiffness of diffusion media, assembly compression, and channel width and channel/land ratio are therefore key parameters in suppressing damaging delamination force. Stiffness is a material property that is a measure of resistance to deformation from an applied force, and is defined as ratio of applied force to deformation [16]. In the interfacial delamination due to F/T cycles, shear stiffness is an important parameter defined as the ratio of applied shear force to shear deformation [16]. Additionally, the material stiffness is a key parameter to transmit compressive forces under the lands to the diffusion media under the channels: the higher stiffness enables the more uniform compressive force under the channels. An infinitely stiff DM would have no variation in compression load across the CL, which is not the case for a fuel cell. The channel width and channel/land ratio are also important factors to determine the compressive force distribution on the catalyst layers under the channels: the smaller channel width or the smaller channel/land ratio, the more uniform compressive force distribution is under the channels.

The thickness of the flexible cloth and stiff non-woven (felt or paper type) DM used in the tests was almost the same and the MEAs were identical. Also, the channel width and channel/land ratio were identical. Therefore, the resisting force for the interfacial delamination was determined by material stiffness alone. The stiffness of the non-woven type DM (SGL 10BB) is significantly higher than that of the cloth type DM, which

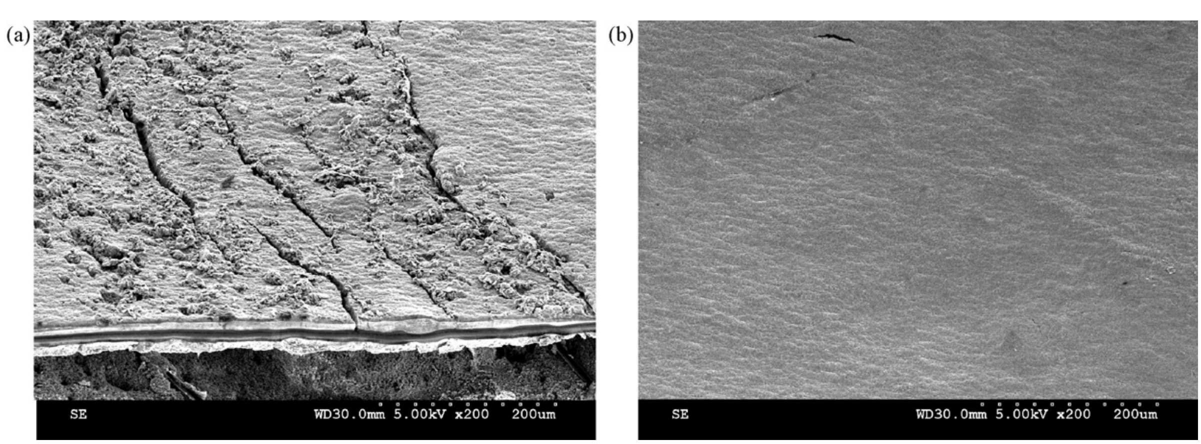


Fig. 2. Surface images of MEAs cycled 30 times between −40 and 70 °C with negligible virgin cracks in the catalyst layer and 18 μm reinforced membrane. Images shown correspond to locations under the channel: (a) CARBEL-CL^{TM} (cloth type) DM and (b) SGL 10BB (non-woven felt type) DM.

can be supported by observations that the cloth type DM has around twice the compressibility, and higher intrusion into the flow field channel than non-woven type DM [17]. Thus we can conclude that the flexible cloth type DM has lower compressive force under the channels, and lower deformation resistance due to lower stiffness for the same ice lens pressure, compared to the non-woven type stiff DM (SGL 10BB). This is a factor in the excessive damage observed in Fig. 2(a).

When the flexible diffusion media (cloth type) is used, the lower resisting forces cannot suppress the ice lens formation between catalyst layers and diffusion media with repeated F/T cycles, resulting in sag and the observed damage. During above freezing operation, liquid water can accumulate in this interfacial gap because of reduced capillary pressure in the large local pore, causing bending stress on the catalyst layers upon ice formation and expansion, resulting in the surface cracks.

The channel width is also an important parameter, and a smaller channel width can mitigate damage. The channel width used in testing here was 2 mm, which is larger than typically used in fuel cell design, but selected to show the damage that can occur from cycling in an accelerated manner.

3.2. *Effect of DM thickness*

From Fig. 3, physical damage (surface cracks and interfacial delamination (CL/membrane)) was not observed in both thin (SGL 25BC, 235 μm) and thick (SGL 10BB, 415 μm) diffusion media when a non-cracked CL with 18 μm reinforced membrane was used. On the contrary, when a non-cracked CL with 35 μm reinforced membrane was used, severe interfacial delamination was observed in both diffusion media. The MEA with a 35 μm membrane was used especially because it is known that a thicker membrane is much more susceptible to delamination damage under freezing [12]. We can conclude that stiff diffusion media having thickness between 235 and 415 μm does not significantly affect freeze-damage on catalyst layers. Note that a gap between diffusion media and catalyst layers was not observed for the observed number of cycles in both cases.

Uniform compression force under the channels and lands is a key factor to prevent interfacial delamination. Compression force distribution under the channel is dependent on DM material properties, the land width, and assembly pressure. For the fixed land width and channel/land ratio with typical fuel cell assembly pressure, material property (stiffness) affects compressive force distribution under the channel; the higher stiffness, the more uniform compression force. Stiffness is determined by micro-structure and thickness. Non-woven felt type DM (SGL 10 series) has higher stiffness than that of non-woven paper type DM (SGL 30 series and 20 series) because the felt type DM has more three-dimensional structure compared to two-dimensional structure of non-woven paper type DM (SGL 25BC). The higher stiffness of SGL 10BB DM is also attributed to higher thickness than SGL 30 and 20 series DM. The transverse and longitudinal (machine direction) bending stiffness of SGL 10AA (0.4-mm

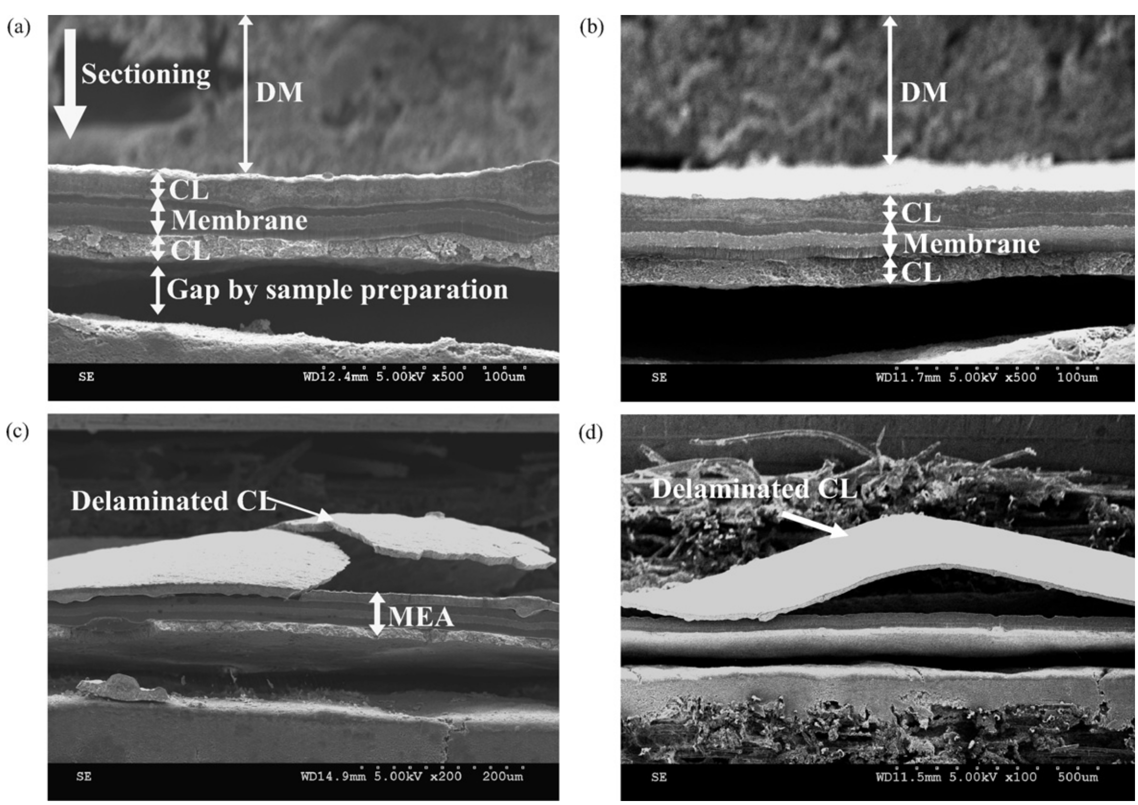


Fig. 3. Cross-sectional images of MEAs with negligible virgin cracked catalyst layers, F/T cycled 30 times: (a) 18 μm reinforced membrane with SGL 25BC DM (thickness 235 μm); (b) 18 μm reinforced membrane with SGL 10BB DM (thickness 415 μm); (c) 35 μm reinforced membrane with SGL 25BC DM; (d) 35 μm reinforced membrane with SGL 10BB DM.

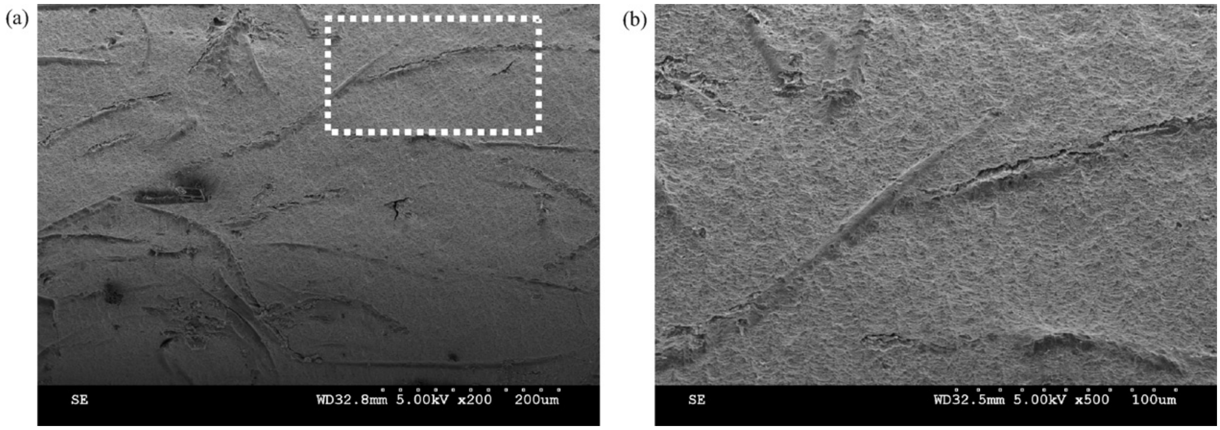


Fig. 4. Surface image of MEAs with negligible virgin cracked catalyst layers with 18 μm reinforced membrane on a 10BA DM, F/T cycled 30 times: (b) is a magnified image of a dotted box in (a).

thick) are 10 and 2.5 N mm, respectively and those of SGL 20AA (0.2-mm thick) are 2.5 and 1.0 N mm, respectively [18]. SGL 25BC DM has significantly lower stiffness than SGL 10BB DM, which may imply less uniform compression forces under the channel compared to SGL 10BB DM. However, for the SGL non-woven type DM ranging thickness from 235 to 415 μm, no significant difference in freeze-damage on the MEA was observed in 30 cycles. In Section 3.4, both DM showed significant permanent damage, but higher deformation and more carbon fiber fracture were observed in the thinner DM (SGL 25BC), which may be attributed to a lower uniformity in compression forces. However, the damage extent was very similar, so we can conclude that the thickness of stiff non-woven diffusion media is not a major factor for freeze-damage.

3.3. Effect of micro-porous layer

In the case of SGL 10BA (without MPL) DM, interfacial delamination (CL/membrane) was not observed, similar to that of SGL 10BB with MPL (cross-sectional images not shown here for brevity). From Fig. 4, small surface cracks in the catalyst layers were observed in case of SGL 10BA. The surface cracks were also observed under the land locations. Surface cracks were not attributed to F/T cycling, but intrusion of the carbon fibers

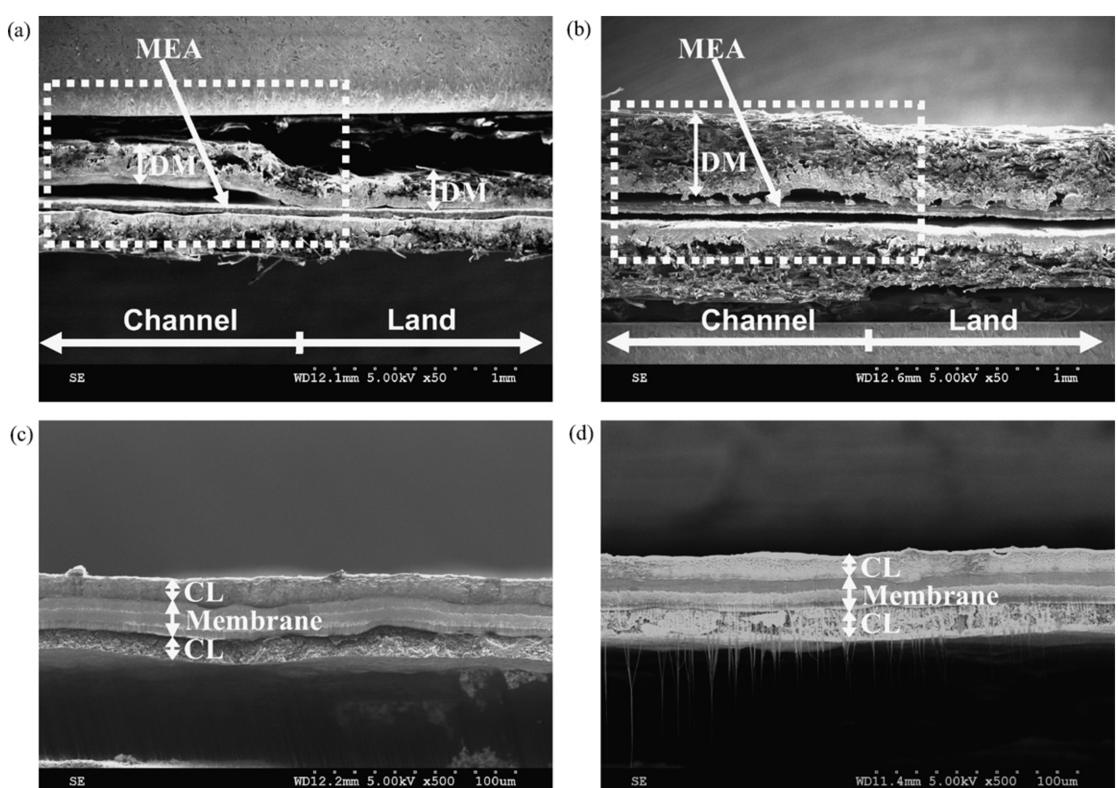


Fig. 5. Cross-sectional images of MEAs with negligible virgin cracked catalyst layers with 18 μm reinforced membrane, F/T cycled 100 times: (a) and (c) SGL 25BC DM; (b) and (d) SGL 10BB DM.

from the DM, which apparently are prevented by an MPL. These cracks can cause electrical short problems or localized water pooling, which can induce flooding and voltage instability in operation. From these results, the MPL was not a major factor for freeze-damage in this ex situ testing, where liquid water exists in contact with the catalyst layers due to the submerged condition. However, in an operating fuel cell, the MPL does play a role in maintaining water balance and reducing flooding in the catalyst layer, which itself can mitigate freeze-damage since the ultimate cause of damage is residual liquid water in contact with the catalyst layer. Stiff diffusion media with micro-porous media is more preferable to prevent electrical shorts, diffusion media sag, and delamination.

3.4. Effects of thermal cycles

In the tests with only 30 F/T cycles, interfacial delamination between diffusion media and catalyst layers was not observed in both thin (SGL 25BC) and thick (SGL 10BB) diffusion media. As shown in Fig. 5, after 100 F/T cycles, a different damage mode, interfacial delamination between catalyst layers and diffusion media was observed in both thick and thin DM. This was attributed to permanent deformation of the stiff diffusion media. The gap in the thin DM (SGL 25BC) seems higher than that in the thick DM (SGL 10BB) as shown in Fig. 5. A permanent gap increases electrical resistance, resulting in higher polarization loss. Additionally, the gap created results in a higher heat transfer resistance that can accelerate membrane degradation, or serve as a location of water pooling that can increase flooding, reducing longevity and causing voltage stability at high-current density.

Fracture of carbon fibers along the channel/land interface was also observed, as shown in Fig. 5(a) and (b). The extent of damage in the SGL 25BC seems larger than that in the SGL 10BB, but this is difficult to quantify. From Fig. 5(c) and (d), catalyst layer damage (interfacial delamination of CL from membrane and surface cracks) was not observed for either thick or thin diffusion media, when using the more freeze-tolerable MEA (non-cracked catalyst layers with 18 μm reinforced membrane) from the previous study [12].

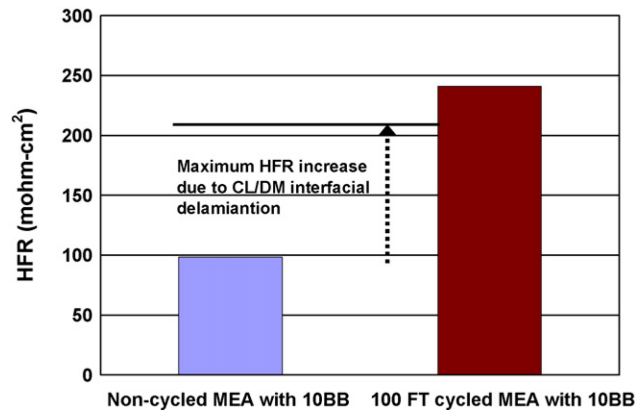


Fig. 6. High frequency resistance at ambient temperature of non-cycled and cycled MEAs (100 times) with SGL 10BB DM.

Fig. 6 shows the measured high frequency resistance of the test cell cycled 100 times with SGL 10BB DM. The HFR of the cycled cell was 2.5 times higher than that of non-cycled test cell (using virgin MEA and SGL 10BB DM) at ambient temperature, a fully hydrated state. High frequency resistance of the test cell cycled 100 times with SGL 25BC DM was measured to be 2.6 times higher than that of non-cycled test cell (using virgin MEA and SGL 25 BC DM). The slightly higher HFR increase in the SGL 25BC may be attributed to more damage compared to that of SGL 10BB because of its significantly lower stiffness. The significant increase in the HFR was attributed to the permanent gap observed between diffusion media and the catalyst layer, and pore-level damage in the catalyst layer. It is also possible that the membrane suffered some ionic contamination from corrosion of stainless steel 316 L test vessel. By assuming all interfacial delamination between CL and DM and neglecting distorted current passage through catalyst layers and membrane under the delaminated parts because of higher aspect ratio (channel width to MEA thickness), the maximum effects of interfacial delamination on HFR increase can be estimated. The maximum HFR increase was estimated to be 2.15 times that of the virgin material, but the actual HFR increase due to interfacial delamination may be much lower. The difference between the

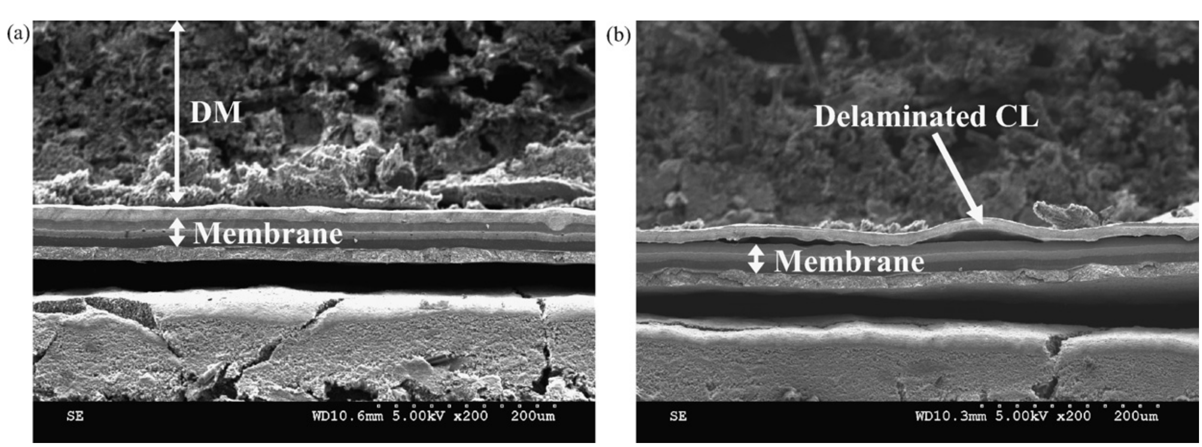


Fig. 7. Cross-sectional images of MEAs (negligible virgin cracks in catalyst layers and 35 μm reinforced membrane) with SGL 10BB DM, F/T cycled 30 times: (a) vapor exposed and (b) liquid submerged condition.

measured HFR increase of around 2.5 times and the maximum area effect of 2.15 times may be attributed to pore-level damage or membrane contamination.

3.5. Effect of vapor exposure

As shown in Fig. 7(a), even a much less freeze-tolerable MEA with 35 μm reinforced membrane did not experience any observable physical damage (interfacial delamination or surface cracks) when the MEA with SGL 10BB was exposed to only fully humidified water vapor. On the contrary, the same MEA in contact with liquid water experienced severe interfacial delamination shown in Fig. 7(b). It is obvious that liquid water contact with the catalyst layer is responsible for all observed damage, and should be removed from the catalyst layer at shutdown to mitigate freeze-damage.

4. Conclusions

Uniform compression pressure from the diffusion media to the catalyst layer surface is a key parameter for selection of diffusion media and design of the flow field to mitigate freeze-induced physical damage of the membrane electrode assembly. Stiffer diffusion media enable more uniform compression pressure on the catalyst layers under flow field lands and channels, mitigating damage. On the contrary, flexible diffusion media which transmit less compression to the catalyst layer surface cannot significantly suppress ice lens growth under the channel, and experience significant observable surface damage of catalyst layers. In the case of stiff diffusion media, the thickness and MPL were not major factors for freeze-damage in the tests conducted, although the MPL assisting in limiting residual water at shutdown is a key factor. To mitigate freeze-damage, stiff diffusion media and more freeze-tolerable MEA are required.

As another damage mode, permanent interfacial delamination between diffusion media and catalyst layers was observed after 100 freeze/thaw cycles, even with the best material combination. As F/T cycles are repeated, water in the catalyst layers and/or diffusion media may increase ice lens thickness between DM and CL, resulting in permanent deformation of DM, as predicted by [15]. This permanent deformation of stiff diffusion media as well as fractures of carbon fibers increased the measured electrical resistance significantly, and may increase water flooding and mass transfer resistance, resulting in reduced longevity and operational stability. An MEA with stiff diffusion media was not damaged in vapor exposed F/T cycling, but severely damaged in liquid water-submerged testing.

Freeze-tolerable MEA design (negligible virgin cracked catalyst layers with thinner reinforced membrane) with stiff diffusion media can reduce the freeze-damage in the worst scenario test condition, but irreversible damage (DM/CL interfacial delamination) can still occur as the worst conditions are repeated. Therefore, in addition to use of the most robust materials described here and in Ref. [12], liquid water should be removed from the catalyst layer at shutdown to mitigate freeze-damage, and the channel/land design should be optimized to reduce cycle-to-cycle damage accumulation.

Acknowledgements

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