



# Microscale X-ray tomographic investigation of the interfacial morphology between the catalyst and micro porous layers in proton exchange membrane fuel cells



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## HIGHLIGHTS

- Visualization and segmentation of gaps at the CL-MPL interface.
- Projected gap area studied as function of CL surface structure and compression.
- Gap area higher for CL-MPL interface with large difference in surface roughness.
- Large gaps in proximity of MPL cracks, which form due to large GDL pores.
- Small gaps induced by surface roughness features throughout the interface.

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## ABSTRACT

The interfacial morphology between the catalyst layer (CL) and micro porous layer (MPL) influences the performance of proton exchange membrane fuel cells (PEMFCs). Here we report a direct method to investigate the CL-MPL interfacial morphology of stacked and compressed gas diffusion layer (GDL with MPL)-catalyst coated membrane (CCM) assemblies. The area, origin and dimensions of interfacial gaps are studied with high-resolution X-ray micro computed tomography (X- $\mu$ CT). The projected gap area (fraction of the CL-MPL interface separated by gaps) is higher for GDL-CCM assemblies with large differences in the surface roughness between CL and MPL but reduces with increasing compression and similarity in roughness. Relatively large continuous gaps are found in proximity to cracks in the MPL. These are hypothesized to form due to the presence of large pores on the surface of the GDL. Smaller gaps are induced by the surface roughness features throughout the CL-MPL interface. By modification of the pore sizes on the GDL surface serving as substrate for the MPL, the number and dimension of MPL crack induced gaps can be manipulated. Moreover, adjusting the CL and MPL surface roughness parameters to achieve similar orders of roughness can improve the surface mating characteristics of these two components.

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## 1. Introduction

Proton exchange membrane fuel cells (PEMFCs) directly convert the chemical energy of hydrogen into electrical energy without combustion and only heat and water as reaction products [1]. A

single PEMFC consists of a proton exchange membrane (PEM) between pairs of catalyst layers (CLs) forming the catalyst coated membrane (CCM), gas diffusion layers (GDLs) and bipolar plates [2]. The membrane directly conducts protons from the anode to the cathode, while electrons are conducted through both the carbon backbone of the CL and GDL and the external load. The reactants (hydrogen and oxygen) and the product water are transported through pores in the GDLs and CLs to and from catalytic sites, where the reactions occur. Heat is conducted through the solid

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components (carbon backbone and ionomer matrix) as well as transported by the reactants and product water [3]. Each component and interface through which energy or mass is transported affects the PEMFC performance by introducing electronic, thermal, diffusional and ionic resistances.

In order to reduce the resistances arising from the interface between the CL (4–20  $\mu\text{m}$  thickness) and the GDL (100–300  $\mu\text{m}$ ), a micro porous layer (MPL, 50–100  $\mu\text{m}$ ) is conventionally added as an intermediate layer [4–7]. The MPL, which consists of a porous structure of carbon particles with PTFE as the binder and hydrophobic agent [8], enhances the PEMFC performance by reducing electric and thermal contact resistances and improving the water management in the electrode [4,9,10]. The CL consists of catalyst nanoparticles with sizes between 2 and 5 nm deposited on carbon nanoparticles sized 20–50 nm in diameter, which are dispersed in an ionomer matrix [11]. Fig. 1 presents a SEM image (in back scattered electron mode) of the assembly consisting of the CCM and GDL/MPL materials. The bright section labeled CCM represent both CLs attached to the membrane and enclosed by the MPL and partially visible GDL fibres on either side.

Imperfections at the CL-MPL interface deteriorate the PEMFC performance, as suggested by several modelling studies [12–14]. Gaps between the CL and MPL reduce the number of conductive (electronic and thermal) pathways across the interface and hence increase electric and thermal resistances [15]. Moreover, these gaps as well as cracks in the MPL have been identified as water pooling regions, which affect the reactant and product mass transport [16–18]. When the surface profiles of the CL and MPL are superimposed, the mean separation between the surfaces was found to be as large as 5–10  $\mu\text{m}$ , which depicts a significant void volume that can accumulate 6–18% of the total liquid water in a PEMFC [19]. This accumulating water at the interface can reduce the limiting current density up to 20% [20], which in turn can lead to the delamination of the CL from the MPL during freeze-thaw cycles [14,21]. The reduction in surface roughness of the CL and MPL is expected to reduce this separation and hence reduce the amount of accumulating water [17]. All of these modelling studies used surface profile data obtained by optical profilometry, to model the interfacial morphology between the CL and MPL. However, when the layers are stacked and compressed, the surfaces deform since the carbon, PTFE and ionomer particles and agglomerates penetrate into voids and interfacial gaps [22,23]. Hence, the direct experimental evaluation of the interfacial morphology and the deformation of gaps under compression is needed.

X-ray micro computed tomography (X- $\mu\text{CT}$ ) is a non-destructive

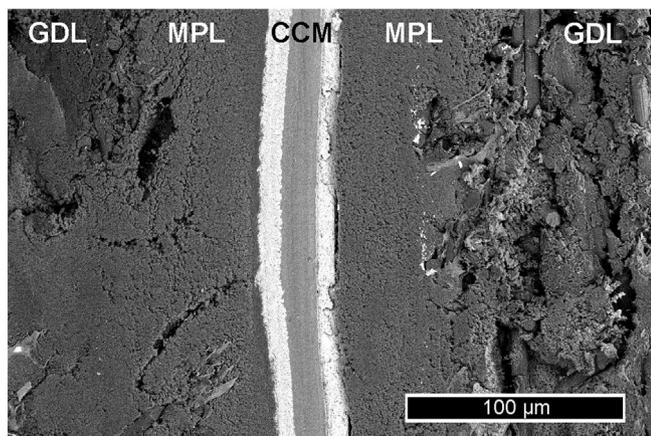


Fig. 1. Cross-section of a freeze fractured GDL-CCM assembly after single cell testing.

technique that characterizes the internal 3D microstructure of materials. In PEMFC technology, this technique has been widely used to assess the porosity distributions within GDLs [7,24,25], the effect of MPL cracks on the porosity [26,27] and bulk porosity measurement of the GDLs [28] and CLs [29,30]. Further, X- $\mu\text{CT}$  has been used to investigate the effect of non-uniform compression on the GDL morphology [31], to compare the morphology of spray-coated and doctor-bladed CLs [32] as part of GDL-MPL-CL electrodes, and to assess the relationship between compression and electrochemical activity within a fuel cell [33].

In this research, we present the results of the application of the X- $\mu\text{CT}$  technique to compressed GDL-CCM assemblies in order to study the interfacial morphology between the CL and MPL. Specifically, we investigated the formation of interfacial gaps between the layers as a function of compression and surface roughness of the CL. Based on the 3D reconstructions, we then quantified the origin and dimensions of gaps as well as the projected gap area (fraction of the interface separated by gaps) between the CL and MPL. Scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and optical profilometry were also used to characterize these materials for interpretation of the observed interfacial structure based on the material characteristics.

## 2. Experimental methods

### 2.1. Materials and sample preparation

This study was conducted by analyzing six compressed GDL-CCM assemblies. Each assembly consisted of an in-house fabricated pseudo-CL (without catalyst particles) on a membrane, compressed against a GDL in a clamping setup made from Ultem PEI (McMasterCarr). The CL inks were prepared using commercially available Cabot Vulcan<sup>®</sup> XC72R carbon powder (Fuel Cell Store), LIQIOn<sup>®</sup> solution LQ-1105 containing 5% NAFION<sup>®</sup> (1100 EW) by weight (Ion Power) with isopropanol (Fisher Scientific) and deionized water as solvents. The CLs were fabricated without any catalyst particles mimicking similar studies reported in the literature [34–36]. Like these previous works, we assumed that the effect of the interactions between the components with the catalyst on the macroscopic CL structures is negligible [37,38]. Sigracet 25BC (Ion Power) with MPL served as the GDL material, while a Nafion 117 membrane (Fuel Cell Store) was used as the substrate for the CL deposition.

A CCM is commonly fabricated by depositing layers of colloidal CL ink onto a proton exchange membrane using techniques such as (electro-) spray-coating [39,40], decal-transfer method [41,42] and screen printing [6,43]. The coating process is followed by heat treatment and eventually hot-pressing, during which the solvent is evaporated and the adhesion of the CL to the membrane is improved [42]. The CLs used in the present study were directly spray-coated simultaneously onto the Nafion membrane without a subsequent hot-pressing step. Three different CL inks were prepared by mixing specified amounts of each ingredient with different ionomer/carbon (I/C) ratios (0.3, 0.6 and 0.9 named CL 1, CL 2 and CL 3) with the solvents. The inks were ultrasonicated for 3 h prior to the spray-coating process to ensure the uniform dispersion of the components. The membrane was fixed on a heating plate at a temperature of 80  $^{\circ}\text{C}$ , while the ink was deposited in multiple layers to facilitate the solvent evaporation.

The GDL-CCM assemblies used for the X- $\mu\text{CT}$  scans consisted of small sections, 2  $\times$  4 mm, of the GDL and the CCM, which were compressed in a custom clamping setup. The assemblies were enclosed in a pair of clamping pieces made from Ultem PEI and held together by two nylon screws. Either 3 or 4 layers of 76.2  $\mu\text{m}$  (3 mil) thick Kapton were used as spacers to achieve thicknesses of 228.6

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