



Investigation of Hot Pressed Polymer Electrolyte Fuel Cell Assemblies via X-ray Computed Tomography



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ABSTRACT

The hot pressing process for fabricating polymer electrolyte fuel cells membrane electrode assemblies (MEAs) has been widely adopted, yet little is known of its effects on the microstructural properties of the different components of the MEA. In particular, the interaction of the electrolyte, electrode and gas diffusion layer (GDL) due to lamination is difficult to probe as conventional imaging techniques cannot access the internal structure of the MEA.

Here, a novel approach is used, which combines characterisation of hot-pressed membrane electrode assemblies using X-ray computed tomography, thermogravimetric analysis, differential scanning calorimetry and atomic force microscopy, with electrochemical performance measurements from polarisation curves and high-frequency impedance spectroscopy. Membrane electrode assemblies hot pressed at 100 °C, 130 °C and 170 °C reveal significant differences in microstructure, which has a consequence for the performance. When hot pressed at 100 °C, which is lower than the glass transition temperature of Nafion (123 °C), the catalyst only partially bonds with the Nafion membrane, leading to increased Ohmic resistance. At 170 °C, the Nafion membrane intrudes into the electrode, forming pinholes, degrading the catalyst layer and filling pores in the GDL. Finally, at 130 °C, the interfacial contact is optimum, with similar roughness factor between the catalyst and Nafion membrane surface, indicating effective lamination of layers.

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1. Introduction

Polymer electrolyte fuel cells (PEFC) fuelled with hydrogen are among the most promising energy conversion technologies for a broad range of applications, including portable, stationary and automotive power delivery.

In order to maximise performance and durability, it is crucial to have an optimised process for the manufacture of membrane electrode assemblies (MEAs). The hot pressing process [1] bonds together the gas diffusion medium (gas diffusion layer (GDL)/micro-porous layer), the catalyst layer and the electrolyte membrane (usually Nafion) to form the MEA. Compression pressure, time and temperature are the key parameters that determine the product of hot pressing: of these, the temperature is most commonly used to optimise the process [2], typically by a trial and error approach. In

spite of the ubiquity of the hot pressing process, little is understood regarding its influence on the resulting MEA structure. With an increasing array of materials available to fabricate MEAs, there is a need for more robust methods of analysis to relate hot pressing conditions to the structure of the MEA and the consequent electrochemical performance and durability.

Here, high resolution 3-dimensional imaging of MEAs has been achieved using X-ray computed tomography (X-ray CT). Hot pressing temperatures of 100 °C, 130 °C and 170 °C (at 2757 kPa for 3 minutes) were studied, representing the range typically employed in practice for Nafion-based MEAs [1,3–16]. The approach allows for the segmentation of the electrolyte membrane, micro-porous layer/catalyst (MPLC) and gas diffusion layer, so as to study the interpenetration of each phase and the beneficial/detrimental structural properties that relate to electrochemical performance.

1.1. Membrane electrode assembly

The MEA is composed of a symmetrical structure with the electrolyte membrane in the centre, and the catalyst medium, gas

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diffusion layer and micro-porous layers on both sides of the membrane (Fig. 1).

The electrically conducting, porous gas diffusion layer [17] provides reactant transport and acts as an effective current collector, whilst also improving water management by controlling the water flow [18]. The structure of the GDL, fibre geometry and porosity [19], as well as the effects of applied compression [20] have been widely investigated to understand how to optimise gas, water and electron transport. Engineering of GDL materials has been of particular focus, as its structure and PTFE content directly affect water management and fuel cell performance [21–24]. The GDL is usually composed of carbon fibres around 5–10 μm in diameter, coated with a PTFE/carbon-based ‘micro-porous’ layer [25].

The catalyst layer, in direct contact with the membrane and the GDL, is typically composed of a thin film (5–20 μm) [26,27] of highly dispersed platinum nano-particles ($\sim 3\text{--}5\text{ nm}$) deposited on carbon particles ($\sim 30\text{--}50\text{ nm}$) [28,29], with a Nafion ionomer additive to enhance the triple phase boundaries between the electrolyte, catalyst and the gaseous fuel [30,31]. It is either coated onto the microporous medium of the GDL or onto the Nafion membrane, via hand-painting, air-brushing, screen printing or sputtering [6,11,32–34]. The fabrication method used imparts different advantages; for example, coating the catalyst layer directly onto the Nafion membrane can improve the ionic contact at this interface, potentially creating a larger electrochemical surface area and lower contact resistance [6]. In contrast, coating the catalyst layer onto the carbon fibres of the GDL may make handling large amounts of materials easier, and reduce manufacturing cost. Furthermore, coating large amounts of gas diffusion material is easier than coating Nafion, due to the dimensional instability of the membrane. In addition, GDEs are easier to store than Nafion. Coating the catalyst onto a porous media may lead to material loss, penetrating too far from the electrode surface into the carbon fibre network. On the other hand, a controlled penetration depth may create a better electrode with lower charge transfer resistance, instead of a surface electrode in the case of the catalyst coated onto the membrane.

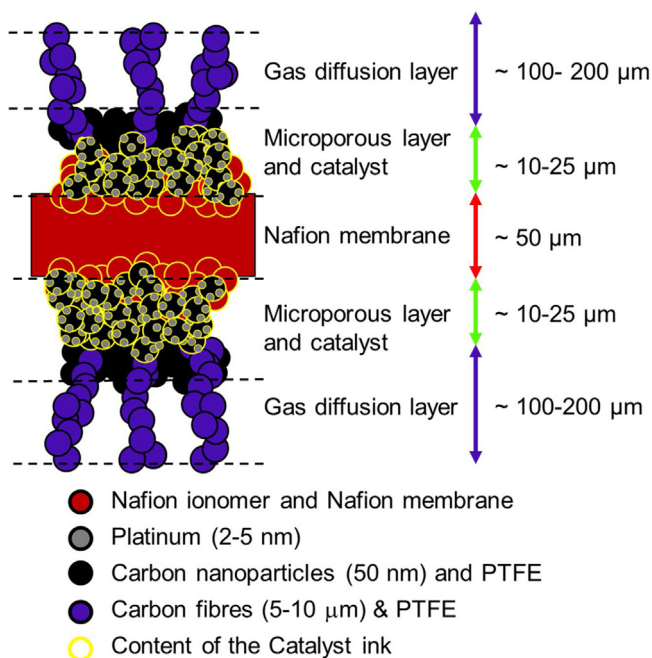


Fig. 1. Schematic of the MEA showing the distributed interface between different components and their typical thicknesses.

The hot pressing process will directly affect the quality of the MEA and impact on the electrode structure, catalyst utilization, migration and coalescence, consequently influencing the transport of ions, electrons, water and reactants in the system [16].

A wide range of process conditions are reported in the literature with hot press compression pressure ranging from 1380 kPa to 49,000 kPa, temperature from 90 $^{\circ}\text{C}$ to 170 $^{\circ}\text{C}$, and time of compression from 90 s to 360 s [1–16,35–41]. Depending on the materials used, a large number of internal parameters affect the hot-pressing process, which explains why such a wide range of conditions are reported.

1.2. Internal parameters affecting hot-pressing

Fig. 1 illustrates the different components of the MEA. Considering the catalyst layer as an example, the following parameters have an impact on the quality of the MEA obtained after hot pressing: layer initial thickness, crack size and distribution [30,42,43], extent of intrusion into the carbon fibres and micro-porous layer [44], the catalyst deposition method [32,45], micro-porous layer porosity [46]. Collectively, these will affect the bonding process and different hot pressing conditions will be required to optimise the system.

Hot pressing temperature has a critical effect on the electrolyte membrane as it undergoes macro-structural changes before and after its glass transition (typ. Nafion ionomer 117–127 $^{\circ}\text{C}$) [8,36,47,48] which affects its ability to flow and bond. The ionomer content within the catalyst layer will also affect the quality of the bonding process between the catalyst and the electrolyte membrane, as the Nafion ionomer with the catalyst will bond with the Nafion membrane [39,49–51].

Attempts to optimise the hot-pressing process include design-of-experiments [4,36] correlated with structural (e.g. SEM) and electrochemical analysis (electrochemical surface area and impedance spectroscopy) [52]. These studies have shown that the pressing temperature has the most important influence on the bonding process between electrode and electrolyte [2,36,52].

1.3. High resolution imaging of the membrane electrode assembly

Until recently, the main technique used to determine the structure of components within an MEA was scanning electron microscopy (SEM), frequently coupled with energy dispersive X-ray spectroscopy (EDS) analysis. SEM is useful in obtaining surface structural data, layer thickness, ‘smoothness’ of different interfaces, the orientation of carbon fibres and the PTFE content [18,19,44,46,53–60], catalyst surface [43], MEA structure [4,6,36] and degradation [54], but fails to reveal the porosity, connectivity and most importantly, the cross-sectional or surface view does not allow the internal structure to be quantified. Focused Ion Beam (FIB) SEM has also been used to study the structure of the micro-porous layer and catalyser layer [46,61–63], revealing nano-porosity, effective diffusivity, permeability and capillary radius, yet this method is destructive.

Micro- and nano-X-ray CT are non-destructive methods that can achieve sufficiently high resolution for imaging carbon fibres, which typically have diameters between 5 and 10 μm and have been increasingly used to characterise GDLs [20,46,64–67]. Recent work from the authors has linked GDL structure and PTFE content to water distribution using neutron imaging and X-ray CT [44]. Furthermore, X-ray CT has also been used to characterise different catalyst deposition methods [32], degradation mechanisms, crack length and catalyst distribution [30].

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