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Different Methods for Determining Porosity of Gas Diffusion Layer using X-ray Microtomography

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ABSTRACT

Gas diffusion layer (GDL) is a crucial component in polymer electrode membrane fuel cells. Being highly porous, this layer facilitates transport of species from the flow field to the reaction sites and vice versa. One of the main characteristics of GDLs is porosity, which has been measured using a number of different methods including 3D X-ray microtomography (μ XCT). Despite the extensive use of this technique in investigating the properties of GDLs, there are variations in the results since the surface of the three dimensional volume used to obtain the bulk porosity of GDLs is difficult to quantify. In this paper, a robust surface identification method, referred to as "Rolling Ball", is introduced to identify systematically the surface and hence porosity of GDLs from μ XCT datasets. In this method, the diameter of the GDL carbon fiber is used as the characteristic length in combination with a Distance Transform (DT) to robustly identify the surface topology. This method is then used to estimate porosity of four different samples of a highly porous GDL, SGL 25BA. The results between different samples show great consistency. A comparison with other methods is also performed, and variations in the bulk and in-plane porosity are observed. The main advantage of the proposed Rolling Ball method compared to prior methods used in the literature is that it uses the carbon fiber diameter to identify the surface results in a systematic fashion. This methodology can be easily applied to other highly porous media.

1. Introduction

The proton exchange membrane fuel cell (PEMFC) is a promising technology that has a great potential to create electrical power and replace fossil fueled energy conversion devices. Due to its high efficiency, zero emission, and low working temperature, PEMFC is a predominant choice for the automotive industry [1,2]. In essence, PEMFC creates the electrical power through an electrochemical reaction during which hydrogen (i.e., fuel) is reacting with oxygen (i.e., oxidant) producing heat, water and electrical power. The PEMFC generally consists of a catalyst coated membrane (CCM) across which the protons are transferred from the anode (where the fuel is oxidized, producing protons and electrons) to the cathode (where the protons, oxygen and electrons react, producing water). The CCM is supported on both sides by a porous gas diffusion layer (GDL) and a bipolar plate, providing passages for transport of reactants, water, and electrons to or from the CCM [3]. In some applications [3], the catalyst layer (CL) is directly deposited on the GDL, forming a gas diffusion electrode (GDE) layer supporting the solid membrane. The GDL also provides mechanical support for the membrane as well as removing heat generated during reaction [2,4]. The GDL has been thoroughly characterized using different methods [2], resulting in identifying important factors affecting the performance of PEMFC. One of the most important properties of the GDL is its porosity, since it provides a qualitative measure of the layer's effectiveness in transporting different species. Porosity also plays an important role in other transport properties of the GDL including permeability, diffusivity and tortuosity [5].

There are two categories of techniques used for determination of porosity of GDLs: destructive and non-destructive. Mercury Intrusion Porosimetery (MIP) [6] is recognized as the most commonly used destructive methods, while techniques like X-ray microtomography (μ XCT) fall into the non-destructive category. μ XCT is an imaging technique that provides the 3D internal structure of material with very high resolution [7]. Sinha et al. [8] were the first group using μ XCT (with a 10-μm resolution) for imaging GDLs. Their interest was to visualize the water distribution inside a GDL, which resulted in estimating an optimum purging time of water from the samples. The use of μ XCT to determine porosity in GDLs was first proposed by Buchi et al. [9], who measured porosity distributions along the thickness of GDLs.

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Bazylak and coworkers, in a series of comprehensive studies [7,10–13], employed μ XCT to categorize different GDLs. First, they showed that porosity distributions of felt–based GDLs are more homogenous than those of paper-based [10]. In their next study [11], they investigated the effect of polytetrafluroethylene (PTFE) on the porosity distribution through the thickness of GDLs, showing that PTFE is distributed non-homogenously across the thickness of the GDL. They also studied the effect of cracks on the effective porosity of the micro porous layer (MPL) using μ XCT. They found that, within the MPL, the percentage porosity related to cracks steadily decreases toward catalyst layer. However, as the voxel size of the tomographic imaging was 2.44 μm, smaller porosity including the pores within the MPL were not captured [12]. In another study, Bazylak's group [13] conducted imaging of both paper and felt -based GDLs compressed to 1.2 MPa, and compared the measured porosity distributions to uncompressed GDLs. Their results showed that the effect of compression is not uniform through the thickness of samples, and the felt-based GDL is more sensitive to compression in comparison with paper-based GDLs. James et al. [14] also examined the effects of compression on the transport properties of GDLs by creating a sample holder mimicking the channels and ribs of the bipolar plate. Using μ XCT for imaging the resulting structure, they showed that the area under compression has significantly less porosity. By developing a computational fluid dynamics (CFD) model based on the μ XCT-derived geometry they found that the transport properties in the region under compression differ by a factor of 2 in comparison with the un-compressed regions.

The analysis of μ XCT datasets to observe the 3D structure of materials involves several steps, including filtering, binarization and volume rendering. Among them, the most challenging step is binarization, which is used to differentiate the material from background. This step requires a threshold value. The most common method for thresholding is Otsu's method [15], which has been used to analysis different GDLs [10,14]. In one study, Ostadi et al. [16] investigated the effect of different threshold values on the properties of the GDLs. This was accomplished by first visually selecting a global threshold value, and then varying the threshold value in increments of 1 grayscale units, calculating porosity at each increment. The results indicated that porosity changes by 2% by changing the threshold value by 3%. Following this, Ostadi et al. [17] compared the 3D images of GDL obtained using μ XCT with the SEM images. The fiber diameter obtained from the SEM image was used to identify the right threshold value for processing of μ XCT images. Odaya et al. [18] used a global thresholding method (instead of the Otsu method [15]) to extract the GDL phase from the background, and compared porosity in single and dual-layer GDLs. Although global thresholding is considered unsophisticated, the results showed that for some cases the use of a global threshold is equivalent to and sometimes even better than those obtained from the Otsu method [15].

Regardless of the image processing steps employed for determining porosity of the sample, the identification of the surface of the sample remains an open question. Assuming that porosity is calculated by $\varepsilon = (1 - V_{GDL})/V_{total}$, where V_{GDL} is the volume of the GDL material and V_{total} is the total volume, surface identification is key since it affects V_{total}. The problem in highly porous materials with porosity more than 80%, like GDLs, is that the surface is not clear and/or quite rough. In most studies mentioned above, subjective assumptions were made to identify the surface of the GDL. Fishman et al. [10] assumed the surface of GDL to be where the GDL fiber is present in a 2D image in amounts greater than 1%. This method, which is referred to as the 99% Porosity method here, requires that the 3D μ XCT dataset be perfectly aligned with the global coordinate system. Ostadi et al. [16] assumed a cubic bounding box to measure GDL porosity, resulting in porosity being a qualitative measure only – dependent on the bounding box size. Another method is to take the mean surface height, which is the standard definition for quantifying surface roughness of bulk materials. In this study, a robust method inspired from paper physics [19], which we name the Rolling Ball method, is proposed to define the surface of GDLs and consequently the GDL bulk porosity. Unlike the previously proposed arbitrary methods, the proposed Rolling Ball method uses the actual dimensions of the fibers to identify the surface. Thus, it provides a systematic method of determining the porosity of GDLs and comparing between materials and manufacturing methods.

2. Methodology

In this section, the description of the GDL material is provided along with the procedures used to acquire the μ XCT images and to binarize the resulting 3D datasets.

2.1. Materials

One sheet of the GDL material SGL25BA was used for this investigation. SGL25BA is a single layer GDL (with no MPL) with 5 wt% PTFE loading and specified thickness of 190 μm [21]. Four samples, from different parts of the sheet, were used in order to show consistency of the methods and results.

2.2. Scanning procedure

The tomographic imaging was performed using a Zeiss μXCT-400 computed tomography microscope following the procedure outlined in Odaya et al. [18]. Briefly, the SGL25BA samples were cut to a size of 4 mm^2 in cross-section and firmly secured to the sample holder to minimize sample flutter during image acquisition. Approximately an area of 1 mm^2 was imaged at a voxel size of 1.167 mm, resulting in a volume consisting of approximately $900 \times 900 \times 200$ voxels. This size of the sample is large enough for investigating the porosity distribution [10].

2.3. Image processing

Each 3D data set was post-processed to reduce noise and to convert to the binary form (black and white) using the ImageJ, MATLAB and Avizo software packages. First, a median filter was applied on the images to reduce noise. Since the purpose of this study is to identify the GDL surface, no effort was made to separate the binder/PTFE and carbon fiber. Second, a manual threshold value [18,20] was applied to segment the GDL from the background. As approximately 5% of the volume (at the top and bottom) was quite noisy, it was cropped from the dataset.

3. Porosity quantification

3.1. Rolling Ball method

Fig. 1-a shows the binary image of a cross section of a 2D image. The black (white) part represents void (GDL material). Fig. 1-b shows the corresponding 3D view of all the 2D images stacked on top of each other. As it can be seen, the carbon fiber strands are randomly distributed. This intrinsic feature of the GDL makes porosity highly variable from one position to another. Some parts consist of very large pore sizes and other areas have very small pores. The high surface roughness present on GDLs can be clearly seen in Fig. 1, especially in the cross sectional view. In order to accurately and robustly quantify porosity in GDLs, the surface of this highly porous material must be identified.