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Focused Ion Beam Tomography of Diffusion Media for Fuel Cells

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Fuel cells efficiently convert chemical energy into electricity, producing a high power density with low emission of pollutants and, as a result, are a promising power technology for a variety of applications. In particular, polymer-electrolyte fuel cells (PEFCs) are emerging as a compact, low-temperature alternative to more common solid-oxide fuel cells. PEFCs consist of a polymer-electrolyte membrane (PEM) sandwiched between porous anode and cathode layers. At the anode, H_2 molecules are split by a catalyst (typically Pt) thereby releasing electrons. Resultant H^+ ions pass through the PEM and are combined with O_2 molecules at the cathode to produce H_2O that is flushed out of the system. PEFCs are constructed with conductive, macro-porous gas-diffusion layers that allow transport of H_2 and O_2 and hydrophobic microporous layers (MPLs) that inhibit the flow of H_2O. The macro-porous layer is typically a carbon-fiber paper or cloth, and the MPL is a coating of carbon black mixed with polytetrafluoroethylene. Detailed knowledge of the real microstructures of these layers is necessary to model and optimize their transport properties and maximize the utility and efficiency of PEFCs [1, 2].

We employed focused ion beam scanning electron microscope (FIB-SEM) tomography to quantify the three-dimensional pore structure of the MPL coating on a commercially available SIGRACET SGL 10BC gas-diffusion layer. A series of cross-sectional SEM images of the MPL was obtained using a FEI Strata DB235 FIB-SEM and FEI Slice and View software. To minimize contrast gradients across the SEM images of the cross sections, a peninsula-shaped volume of interest (Fig. 1a) was milled from the surrounding material. 20nm thick slices of MPL were removed from the volume using a 500pA/30kV ion beam, and high-resolution SEM images (Fig. 1b) of each section were captured using a through-the-lens secondary electron detector.

Following acquisition, the images were treated by a multistep image-processing and data-segmentation procedure using codes written in MATLAB. First, small displacements of the viewing area in successive images result from sample drift or beam shifts. These displacements were removed through a cross-correlation algorithm initially using the whole image and then, more precisely, using stationary features outside the area of interest. Because the milled surface is not orthogonal to the SEM beam, successive images were shifted by an amount determined by the slice thickness and the SEM angle of incidence. Also, each image was stretched to compensate for the vertical foreshortening caused by the angular projection. Contrast gradients in the images were determined by a biparabolic fit and removed. Finally the data set was segmented into a binary volume (Fig. 2a) identifying pores and MPL material, and isolated pores were removed.

Once the image processing and segmentation was complete, the data set was analyzed to determine porosity, internal surface area, pore-size distribution, tortuosity (Fig. 2b), and connectivity of the pore network. These unique datasets can be further used to accurately model the transport phenomena in the MPL and optimize the water-management capabilities of PEFCs [3].

[1] B. C. H. Steele & A. Heinzel, *Nature* 414 (2001) 345.
[2] R. P. Ramasamy, E. C. Kumbur & M. M. Mench, *Int. J. Hydrogen Energy* 33 (2008) 3351.
[3] We acknowledge the use of the FIB-SEM in the Centralized Research Facilities at Drexel University.

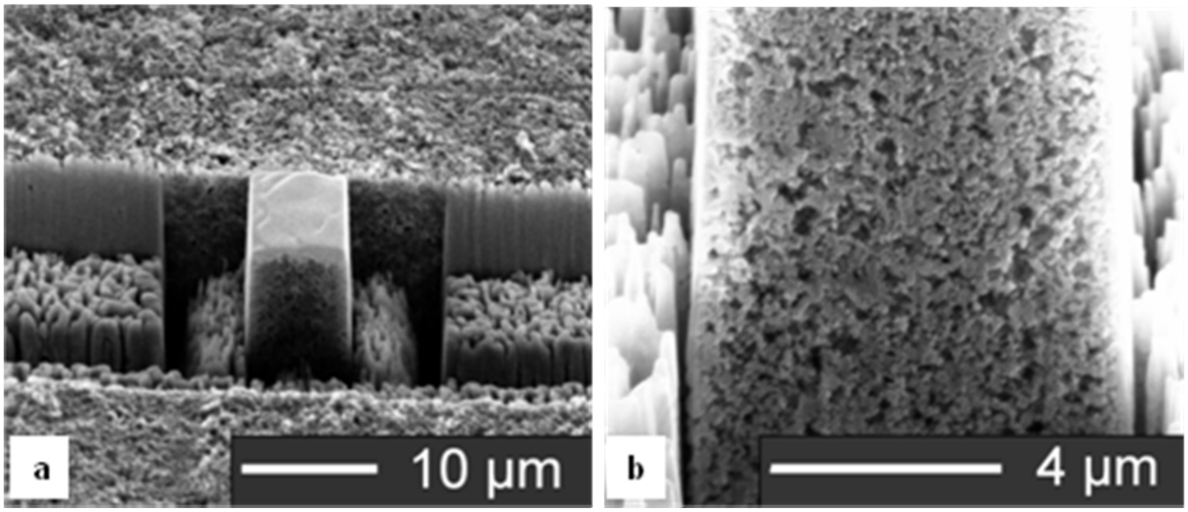

FIG. 1. Cross-section preparation and imaging. A peninsula-shaped volume (a) was prepared before sectioning to minimize contrast gradients across images. The FIB was used to remove about ~20 nm of material per slice through a total thickness of ~3µm and high-resolution SEM images (b) of the polished faces were obtained after each slice.

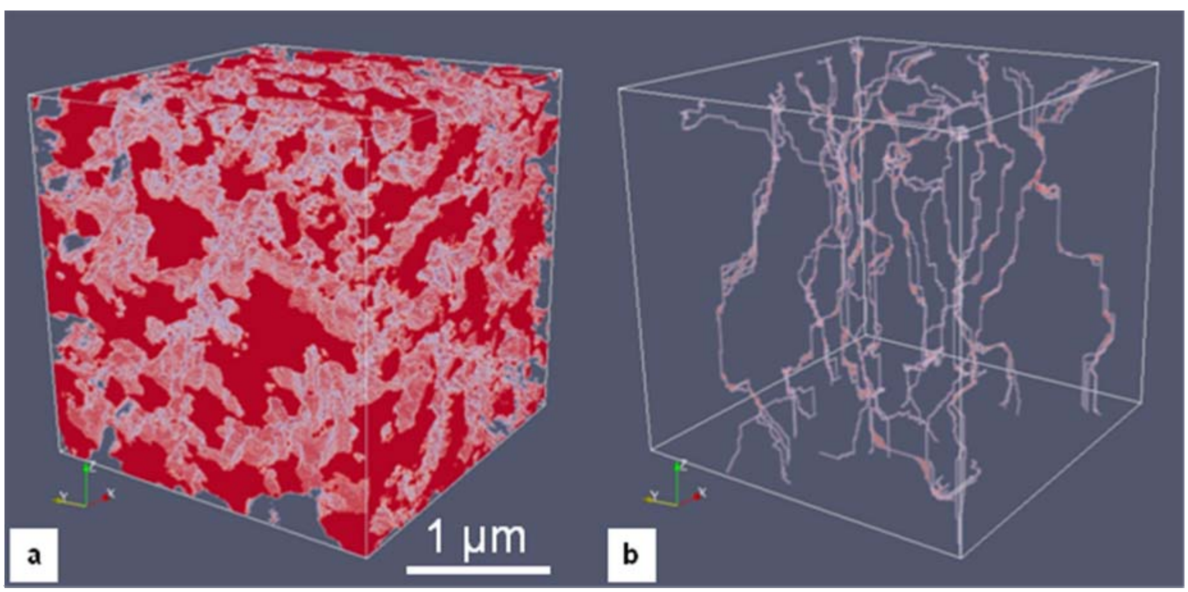

FIG. 2. Segmented 3D data set and quantification of tortuosity. The 3D data sets were segmented (a) to identify MPL material (red), networked pores and isolated pores. The data sets were used to determine several key properties of the MPL including porosity, internal surface area, pore-size distribution, tortuosity (b), and connectivity of the pore network.