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Morphological analysis of polymer electrolyte fuel cell electrode using high resolution X-ray computed tomography and subsequent performance analysis

Pratiti Mandal, Shawn Litster
Department of Mechanical Engineering
Carnegie Mellon University
Pittsburgh, PA 15213

The high loadings of expensive platinum catalyst in polymer electrolyte fuel cell (PEFC) cathodes contributes greatly to the high cost of PEFCs and thus limits their commercial competitiveness. Unfortunately, achieving the desired mass activity of new catalysts is hindered by the mass transport resistances imposed by the electrode structure. Detailed characterizations of electrode morphology with particular focus on transport properties will not only help improve the understanding of the complex transport phenomena, but will also assist in optimizing cost effective manufacturing of durable electrodes. Here we present a study on the morphology of the porous PEFC electrodes as well as the individual nanoparticle agglomerates that assemble into the electrode structure, and how those characteristics influence fuel cell performance.

In our work, the electrodes and agglomerates are three-dimensionally imaged using non-invasive and non-destructive nanoscale-resolution X-ray computed tomography (nano-CT). The nano-CT we use is a lab scale instrument that can achieve 50 nm resolution using X-ray optics (UltraXRM-L200, Xradia Inc., Pleasanton, CA, USA). The optics includes a single capillary condenser and a Fresnel zone plate. A Zernike phase contrast imaging mode is used to image the low atomic number (Z) carbon catalyst support. The imaging was done in the instrument's high resolution mode, which yields 50 nm resolution (16 nm voxels) with a 16.5 μm field of view.

Nano-CT has previously being used in imaging of PEFC catalyst layers to quantify porosity, pore and solid phase size distributions [1] and to evaluate the effects of an agglomerate size distribution on the predictions of the agglomerate model for the catalyst layer [2]. In the present work, we use nano-CT to obtain 3D reconstructions of the individual catalyst agglomerates found in the electrode ink. A diluted sample of catalyst ink was deposited on commercially available low-X-ray attenuating polyimide film (Kapton®) at low enough loadings that individual particles were spatially separated. From the nano-CT image of this particle covered film, we obtain statistical information about the Pt/C agglomerate morphologies simultaneously for a large number of agglomerates. Figure 1a shows a scanning electron microscope (SEM) image of the particle covered Kapton film (after a 20 nm sputtering of gold for SEM) and Figure 1b shows the nano-CT image of the same particle agglomerates prior to the gold coating. Figure 1c shows 3D images of the selected catalyst agglomerates. Comparing the nano-CT and SEM images, we identify that agglomerate features with 50 nm and larger radii are resolved by the nano-CT

Subsequently, the same catalyst ink (before dilution) is deposited onto gas diffusion layers to prepare PEFC gas diffusion electrodes that are then imaged by the nano-CT. Figure 2 shows a three-dimensional reconstruction of a prepared electrode. The same GDE samples are also electrochemically evaluated in a PEFC. By using the same ink batch in the two imaging steps, and in the fuel cell testing and then systematically varying ink composition and processing, we can delineate the complex relationships between catalyst ink, electrode structure, and fuel cell performance.

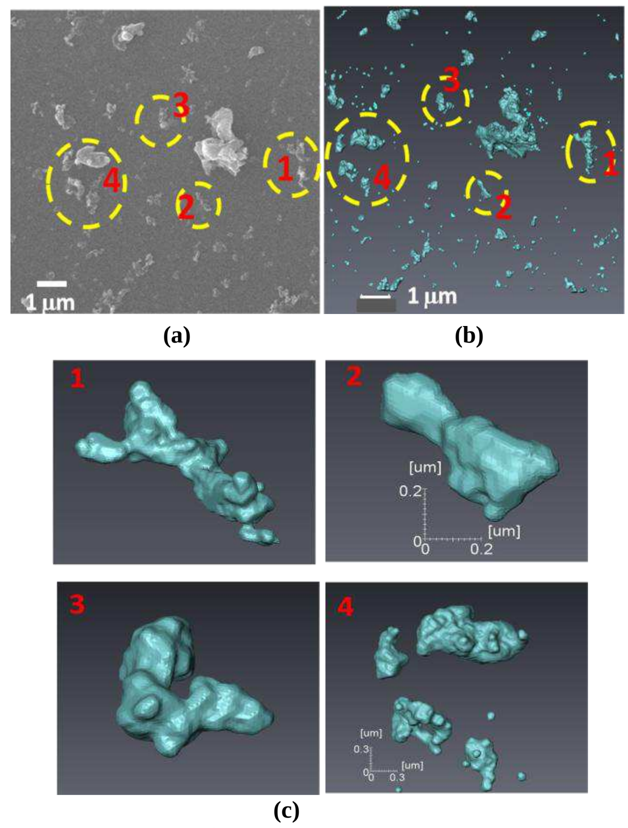

Figure 1: Images of dispersed catalyst (20% Pt/C) agglomerates. (a) Image obtained using SEM (b) 3D image of the same sample obtained using nano-CT (c) 3D nanostructures of a few individual agglomerates in the regions corresponding to the labels in (a) and (b)

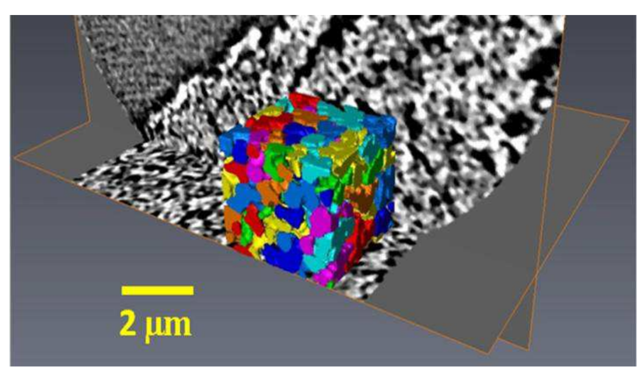

Figure 2: Solid agglomerate structure a PEFC electrode.

1. W. K. Epting, J. Gelb and S. Litster, Advanced Functional Materials, **22**, 555 (2012).
2. W. K. Epting and S. Litster, International Journal of Hydrogen Energy, **37**, 8505 (2012).