Nondestructive Nanoscale 3D X-ray Imaging of Solid Oxide Fuel Cells in the Laboratory

Stephen T. Kelly*[1], Sandrine Ricote†[2], Peter Weddle[2], Alexis Dubois[2], Benjamin Kee[2], Will Harris[1], John Berger[2], Robert J. Kee[2]

[1]Carl Zeiss X-ray Microscopy, Inc., Pleasanton, CA, USA
[2]Department of Mechanical Engineering, Colorado School of Mines, Golden, CO, USA

Keywords: fuel cells, x-ray nanotomography, porous materials, energy materials

Summary: We used nanoscale x-ray tomography to image and analyze the complex 3D microstructure of several varieties of solid oxide fuel cell. Quantitative analysis of the pore space, material distributions, and material boundaries are shown. We discuss how this 3D understanding contributes to enhanced simulation capabilities and opens the door to experiments not accessible by other means.

Fuel cells and similar energy devices rely heavily on complex 3D microstructures for efficient operation. While traditional imaging techniques such as light microscopy or scanning electron microscopy provide detailed views with sufficient resolution, they do not capture the intricacies of these 3D structures. As such, 3D imaging techniques must be employed to fully capture the structures and provide geometric input to advanced performance models that rely on experimental observations for validation. In addition, ideal experimental observations in this framework can track the evolution of sample parameters (microstructure, chemistry, loads, etc) non-destructively over several stages of processing or operation and across multiple length scales.

As an example of this approach, we present here results from non-destructively imaging and analyzing the microstructures of two different fuel cell architectures using x-ray nanotomography. All samples were imaged using a ZEISS Xradia 810 Ultra x-ray microscope after being prepared using a combination of laser ablation and focused ion beam milling to create samples of the correct size and shape (cylinders ~20 μm in diameter) for the tomography measurements.

First, we examined a H+ -solid oxide fuel cell (or protonic ceramic fuel cell) anode made of $BaCe_{0.2}Zr_{0.7}Y_{0.1}O_{3-d}$ (BCZY)/Ni. The sample was prepared by solid-state reactive sintering as described in [1]. The material contrast and spatial resolution (down to 50 nm) of the technique allows for discrimination of discrete Ni and BCZY phases, and identification of gas-phase features such as pores and cracks. Quantitative analyses include measuring and visualizing the triple phase boundary, plotting the trajectory of cracks in 3D, and can be used to build a 3D structural model of the sample for further computational input such as transport or mechanical simulations. Results showing a variety of material properties computed from the imaging results will be presented.

Second, we present an analysis on a Ni + ZrO_2:8%Y_2O_3 (8YSZ) / 8YSZ / $La_{1-x}Sr_xMnO_3$ (LSM) solid oxide fuel cell stack exposed to air to promote nickel oxidation. The detailed 3D tomographic reconstructions allow for several important material parameters to be quantified. We analyzed the pore size distribution in the cathode material, examined the connectivity of pores in the electrolyte layer, and were able to identify the different phases and plot the triple phase boundary in the anode.

The 3-dimensional nature of the results presented here reflects the complexity of the 3D electrodes, and enables more advanced analyses inaccessible through 2D imaging alone. Furthermore, the non-destructive nature of the technique opens the door to a wide array of experimental possibilities such as additional imaging or analysis with

*e-mail: steve.kelly@zeiss.com
†e-mail: sricote@mines.edu

complementary techniques (such as SEM or FIB-SEM), or imaging the sample after a processing treatment. In sum, these techniques provide valuable data for validation and refinement of the 3D structural models used to predict the performance and failure mechanisms in these devices, and help close the loop in this "materials by design" approach.

References

[1] S.M. Babiniec, S. Ricote, N.P. Sullivan, Characterization of ionic transport through $BaCe_{0.2}Zr_{0.7}Y_{0.1}O_{3-\delta}$ membranes in galvanic and electrolytic operation, Int. J. Hydrogen Energy **40** (2015) 9278

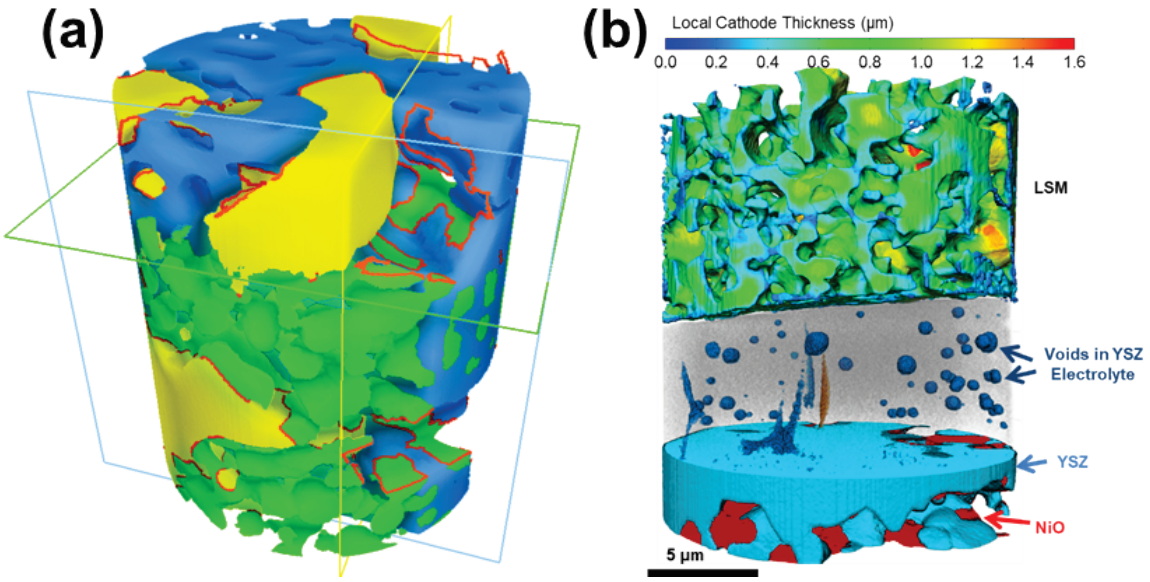

Figure 1: (a) Segmented image of the H+ solid oxide fuel cell anode imaged in this study. Green indicates pore space, blue indicates nickel, yellow indicates the BCZY ceramic phase, and orange indicates the triple phase boundary. The diameter of the analyzed cylinder shown is 16 μm. (b) Segmented image of the Ni + 8YSZ / 8YSZ / LSM fuel cell stack imaged and analyzed in this study. Local cathode thickness is shown at the top of the image, corresponding to the color scale above.