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Advanced Characterization Methods for Solid Oxide Fuel Cell Materials

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Lanthanum strontium manganite (LSM)/yttria stabilized zirconia (YSZ) and YSZ/Ni materials are currently under investigation for the cathode and anode layers in solid oxide fuel cell (SOFC) devices, respectively. Optimizing the performance of these materials requires engineering of the composite microstructures at the sub-micron scale. We have found that a combination of secondary electron (SE) imaging and Auger electron spectroscopy (AES) elemental mapping provides a good understanding of porosity and phase distributions within these materials. Backscatter imaging in an SEM does not show contrast variations between the LSM and YSZ phases, as their average atomic weights are very similar. The improved spatial resolution of AES versus energy dispersive x-ray spectroscopy (EDS) makes it a better choice for determining the location of LSM/YSZ and YSZ/Ni phase interfaces.

Examination of the cathode and anode layer in SOFC device stacks requires cross sectioning. Traditional epoxy mounting and polishing is not an option for the combined SEM/AES characterizations, since this method is incompatible with the UHV environment required by AES. This paper will discuss two methods used to prepare SOFC device stacks, depending on the stack size and complexity. The first method is a modified transmission electron microscopy (TEM) sample preparation technique. This method is used for thin (<2mm), SOFC device stacks. Pieces (~5mm x 10mm) of the stack are sandwiched between similar sized pieces of silicon wafer and held together with a thin layer of epoxy. A diamond saw is used to cut cross section slices from the sandwich and a three step dimpling method is used to generate the final polished cross section. Figure 1 contains optical images showing examples of the sandwich cross sections before and after polishing. The second method is to use focused ion beam (FIB), localized cross sectioning, to generate the final polished region for the SEM/AES analysis. This technique has been used when the original sample is thicker than 2mm. The biggest advantage of FIB cross sectioning is its ability to produce smooth cut surfaces on non-homogeneous and porous materials. Another advantage of this technique is precise positioning of the cuts with respect to numerous interfaces (and therefore localized AES mapping) in the complex stacks under investigation. First, a rough cross section is made via hand polishing. The FIB milling is then used on the cross section to generate the smooth surface required for the SEM/AES analysis. An angled FIB box is generated to allow for argon ion sputter cleaning within the AES instrument. Figure 2 contains SE images showing an example of hand polished cross section and an angled FIB cut within the LSM/YSZ layer. Figure 3 contains an SE image and AES La MNN and Zr LMM elemental map overlay obtained from a FIB polished cross section.

The SE images and AES elemental maps can be used to determine the pore, LSM, and YSZ phase distributions within the cathode layer. Similar sample preparation and analyses have been performed on the YSZ/Ni anode materials. Determining the microstructure and phase distributions, at sub-micron scales, within these layers following various process and test conditions allows optimization of the SOFC manufacturing process and an understanding of performance.

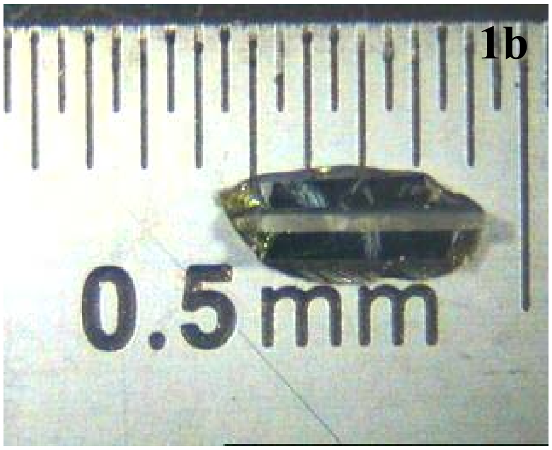
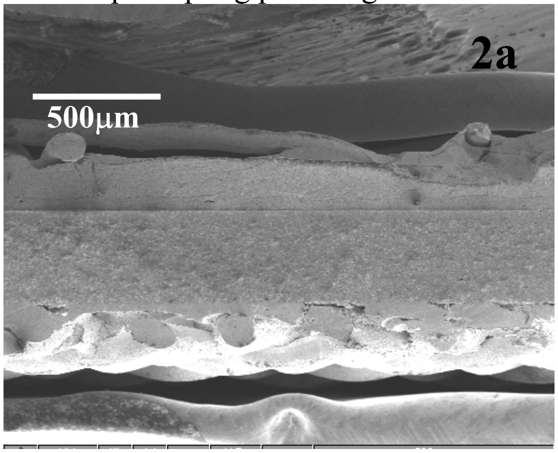


Fig. 1a. Optical image showing an example of a non-polished, Si-SOFC stack-Si sandwich. Fig. 1b. Optical image showing an example of a Si-SOFC stack-Si sandwich following the three step dimpling polishing method.



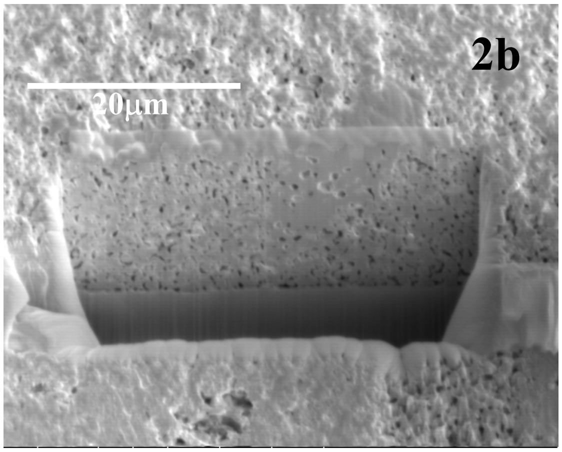
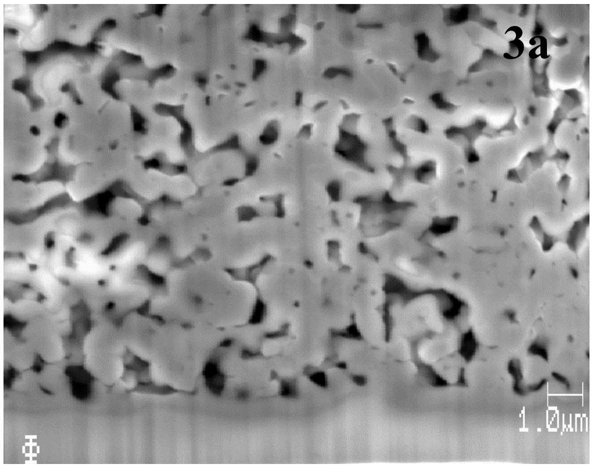


Fig. 2a. SE image showing an example of hand polished cross section prepared for FIB. Fig. 2b. SE image showing an example of an angled FIB box used for the SEM/AES analysis.



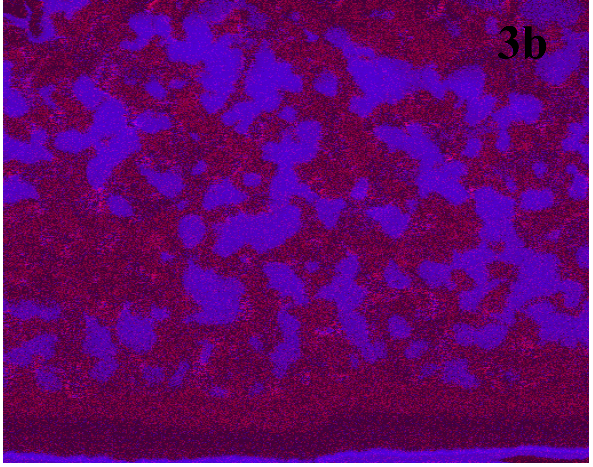


Fig.3a. SE image obtained from angled FIB box showing the porosity within the LSM/YSZ layer. Fig. 3b. Overlay of the AES La MNN (blue) and Zr LMM (red) elemental maps used to determine LSM and YSZ phase distribution within the region in Fig. 3a.