Solid Oxide Fuel Cells (SOFC) offer electrochemically generated sources of electricity using oxygen ion transport at elevated temperatures. Analysis of materials used in SOFC using electron microscopy provides insights of foreseeable chemical reactions that govern the performance of the fuel cell. Materials used in SOFC can be divided into four categories; anode, cathode, electrolyte and interconnects/sealants. Materials at anode satisfy the requirement of either pure electronic conduction or appropriate balance of mixed electronic/ionic conduction. The electrolyte is an oxide ceramic material with fluorite structure and a typical combination of group 4 transition metal and oxygen. The oxygen vacancies in fluorite oxide structure can be increased by replacing group 4 element with a transition metal (with matching ionic radii) either from group 3 or lanthanoids category. Cathode is composed of perovskite oxide ceramic structure with $\text{ABO}_3$ configuration. Oxygen vacancies can be created by doping group 2 element with $2^+$ valence state (and matching ionic radii) at A or B site. The oxygen vacancies at cathode promote oxygen intake and mobility at high temperatures. To avoid direct gas mixing of hydrogen and oxygen at high temperatures, anode and cathode are isolated during active fuel cell operation.

From the last ten years, one of the authors had been involved in studying pure and doped ceria/zirconia as SOFC anode [1-9], Ni-YSZ as anode for direct hydrocarbon fuel feed SOFC [7, 8], YSZ, ceria micro/nanostructures as SOFC electrolyte [1, 2], and Ni-alloys for SOFC interconnects and seals [10]. During SOFC growth, dense electrolyte, porous anode and cathode are preferred to avoid gas mixing and to get high Triple Phase Boundary (TPB) area along the interfaces. As shown in the Figure 1, we have studied SOFC stack composed of NiO-GDC / NiO-YSZ as anode, (Y:Sc)ZrO$_2$ as electrolyte and LSM/LSM-GDC as cathode. The anode/electrolyte and cathode/electrolyte interfaces are the main focus of this study as efficiency of the SOFC depends upon grain boundary structure at these interfaces. As shown in Figure 1, energy dispersive X-ray spectroscopy (EDX) mapping using CeLα line revealed the presence of ceria rich material at both interfaces. Indexing using Electron BackScatter Diffraction (EBSD) along these interfaces produced zero solutions. This suggests presence of nano-crystalline or amorphous ceria. We have also used Mill & Monitor™ process to understand the nature of the bulk interface in the third dimension. The process was carried out using Hitachi NB5000 nano DUE™T double beam electron and ion microscope. Ga ions were used to create thin sections (20-50 nm) along both SOFC interfaces. Reconstruction from hundreds of slices illustrates the nature of surface morphology within the bulk. The presence of 40 kV Ga ion beam allows finer beam focusing and enhanced control over the slicing distance between two successive runs.

Creating structures with graded porosity could dramatically improve SOFC performance due to activity improvement in catalyst and higher TPB. Typically active catalyst like nano-phase Ni or NiO can lead to faster hydrocarbon dissociation. Even though electrochemical data confirms actual SOFC performance, our goals in the present study are mainly aligned with effective usage of electron/ion microscopy based techniques to realize high surface area and distribution of phases as a function of...
change in operating temperatures. Using 3-D analysis techniques, we aim to plot the diffusion profile for these interfacial materials. Such diffusivities can modify the oxygen ion transport properties at the interface. It is known that grain boundary diffusivities are orders of magnitude higher than bulk diffusivities. Therefore, systematic studies of diffusion length, grain distribution, porosity and phase transformations using electron/ion microscopy based techniques can provide valuable information about long term stability of SOFC.

References:

[11] The authors acknowledge usage of electron microscopy resources at Clemson University Electron Microscopy Laboratory and at Hitachi High Technologies America Inc.

Figure 1. Schematic representation of solid oxide fuel cell (SOFC) stack studied in this work. The EDX mapping using CeLα line indicated segregation of ceria along both interfaces. Inverse pole figure data shows zero solution at the interface indicating nano-crystalline or amorphous cerium based phase.

Figure 2. Partial reconstruction of anode/electrolyte interface. Serial sections spaced 100nm apart were assembled into a three dimensional model to examine grain structure and porosity. Grain coloration was based on contrast of the backscattered electron images. Model is approximately 20um wide.