Advanced three-dimensional microstructure description of porous nano-sized Ni-SDC cermets using FIB-SEM nanotomography

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Solid oxide fuel cells (SOFCs) have the potential to become one of the most important energy conversion systems because of their high conversion efficiency and fuel flexibility. A SOFC system consists of two porous electrodes which are separated by dense solid ceramic electrolyte. Most commonly anode material is based on two phase cermets. To maintain sufficient ionic conductivity at reduced operating temperatures in contemporary SOFC devices, conventional yttrium-stabilized zirconia (YSZ) based cermets, is typically replaced with samarium-doped ceria (SDC) cermets (Ni-SDC). The electro-catalytic activity of cermet anodes towards fuel oxidation is directly related to the length of a so called triple-phase boundary (TPB) where fuel, electronic- (Ni), and ionic-conductor (SDC) meet. High density TPB cermet microstructure can be achieved when fine Ni and SDC powders are homogenously mixed. However, TPB's are electro-catalytically active only if three phases are continuously connected throughout the anode and accessible to gaseous fuel stream. Therefore, cosintering of nano-scale powders during Ni-SDC cermet fabrication represents one of the most important steps to achieve highly efficient and reliable electrodes. For this purpose, it is crucial to establish quantitative relationship between primary microstructural parameters, which can be adjusted during material processing, with the higher order topological features that control the electrochemical performance of the anode.

In porous cermets critical topological features such as connectivity, TPB density and the tortuosity of transport pathways within the pores can only be established based on a detailed 3D microstructural information. In this work we present quantitative three-dimensional microstructure description of nano-sized Ni-SDC cermets, fabricated at various sintering conditions, using FIB-SEM nanotomography. The samples were serial sectioned using a fully automated slicing procedure with active drift correction algorithms and auto focusing routine to obtain a series of low-loss BSE images with 5 nm reproducible step. Advanced image processing algorithms were developed and applied directly to image data volume. In the next step, individual phases within highly dense voxel matrix were separated using python assisted 3D watershed segmentation algorithm. After phase separation, the entire probed volume was reconstructed and 3D quantification algorithms were established. Volume fractions and porosity as well as feature sizes, specific surface areas of individual phases were calculated directly from 3D reconstructed microstructure. Segmented 3D data cube was used for grain connectivity analysis, TPB density calculations and tortuosity simulations. Active and inactive TPB's were identified and quantitatively evaluated using "in house" programing algorithm based on the centroid method. In final stage, gas transport streamlines and absolute gas permeabilities were simulated through porous network.

The presented analytical approach will serve as a tool for detailed quantitative microstructural description of various porous nano-sized SOFC anode cermets. The detailed three-dimensional microstructural data will be used to optimize their fabrication process in direction to achieve highly-efficient and durable electrodes.

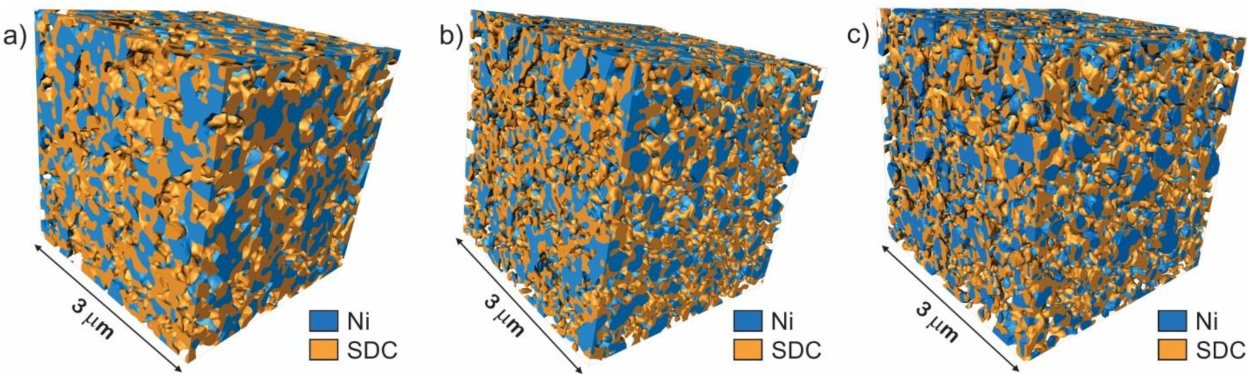

Figure 1: 3D reconstructions of porous Ni-SDC cermets sintered at a) 1300 °C, b) 1200°C and c) 1100°C.

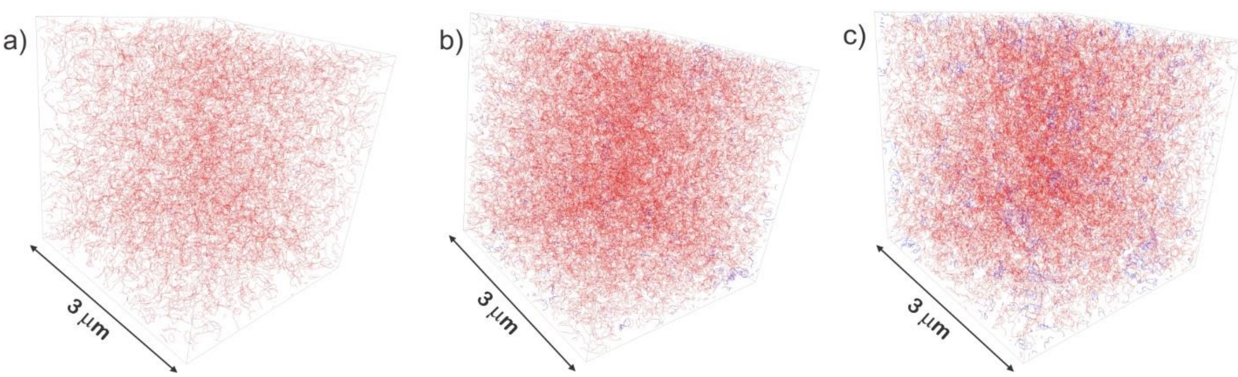

Figure 2: 3D distributions of active TPB's (red) and inactive TPB's (blue) for the case of Ni-SDC cermets sintered at a) 1300 °C, b) 1200°C and c) 1100°C.

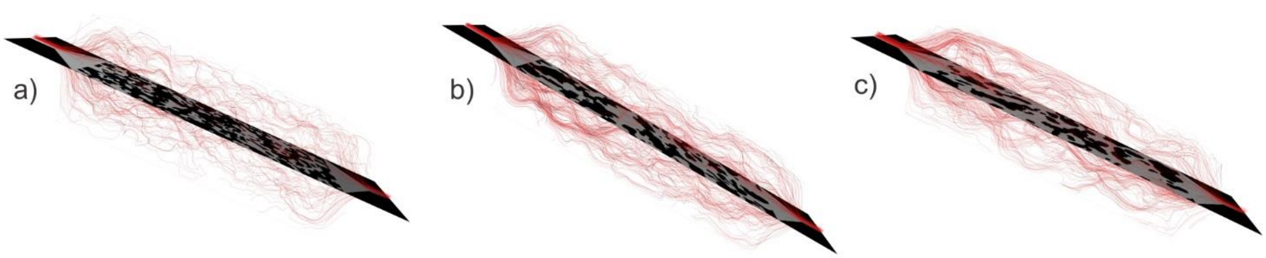

Figure 3: Simulations of absolute permeability in Z direction through the complex porous structure with visualization of gas stream lines and pressure profile for the case of Ni-SDC cermets sintered at a) 1300 °C, b) 1200°C and c) 1100°C.