Multiscale tomography on all-ceramic solid oxide fuel cells

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Solid oxide fuel cells (SOFC) are expected to play an important role in future energy conversion technology. Design concepts like all-ceramic SOFC components co-sintered on inert substrates are highly attractive in meeting the demands of cost efficiency [1]. To improve correlation of fabrication parameters and cell performance and consequently improve identification of aging mechanisms, sophisticated 3D chemical and microstructural analysis of all components is necessary. This analysis task covers many orders of magnitude: (i) large scale parameters like layer uniformity, porosity or crack formation [2], (ii) ionic/electronic transportation pathways [3,4] down to (iii) formation of nanoscale secondary phases [5]. Therefore, correlative tomographic techniques are required for a comprehensive understanding of the cell.

Within this study, a combination of X-ray computed tomography (micro-CT), focused ion beam - scanning electron microscopy (FIB-SEM) tomography and energy dispersive X-ray spectroscopy - scanning transmission electron microscopy (EDXS-STEM) tomography is employed on all-ceramic SOFCs. Special emphasis is put on imaging in FIB-SEM tomography using a state of the art FEI Helios G4 FX instrument equipped with numerous detectors. The simultaneous readout of up to four detectors yields complementary image information which can be used to improve the accuracy in the segmentation process. It is even possible to overcome the poor material contrast in all-ceramic composite cathodes composed of the electronic conducting electrocatalyst phase $(La,Sr)MnO_{3-d}$ (LSM) and the ionic conducting phase Y_2O_3 doped ZrO_2 (YSZ). However, the choice of detector is crucial as the LSM phase is affected by contrast inversion in the secondary electron SEM images as exemplified in Fig. 1a for two consecutive slices. This is caused by charging when individual LSM grains are isolated (cf. arrow in Fig. 1a) from the electron conducting and percolating LSM network. In contrast, the mirror detector (in-column backscattered electron detector) provides a unique assignment of the two phases without any contrast inversion (cf. Fig. 1b). One other advantage of the mirror detector is the uniform intensity across the whole image which reduces the need of contrast gradient correction. EDXS-STEM revealed the presence of Mn-rich secondary phases which could be assigned to specific contrast features in backscattered electron SEM images and is used in the FIB-SEM reconstruction. One of the reconstructions of a LSM/YSZ cathode is depicted in Fig. 1c showing an enrichment of Mn-rich phases at the cathode - electrolyte interface as well as cathode current collection layer.

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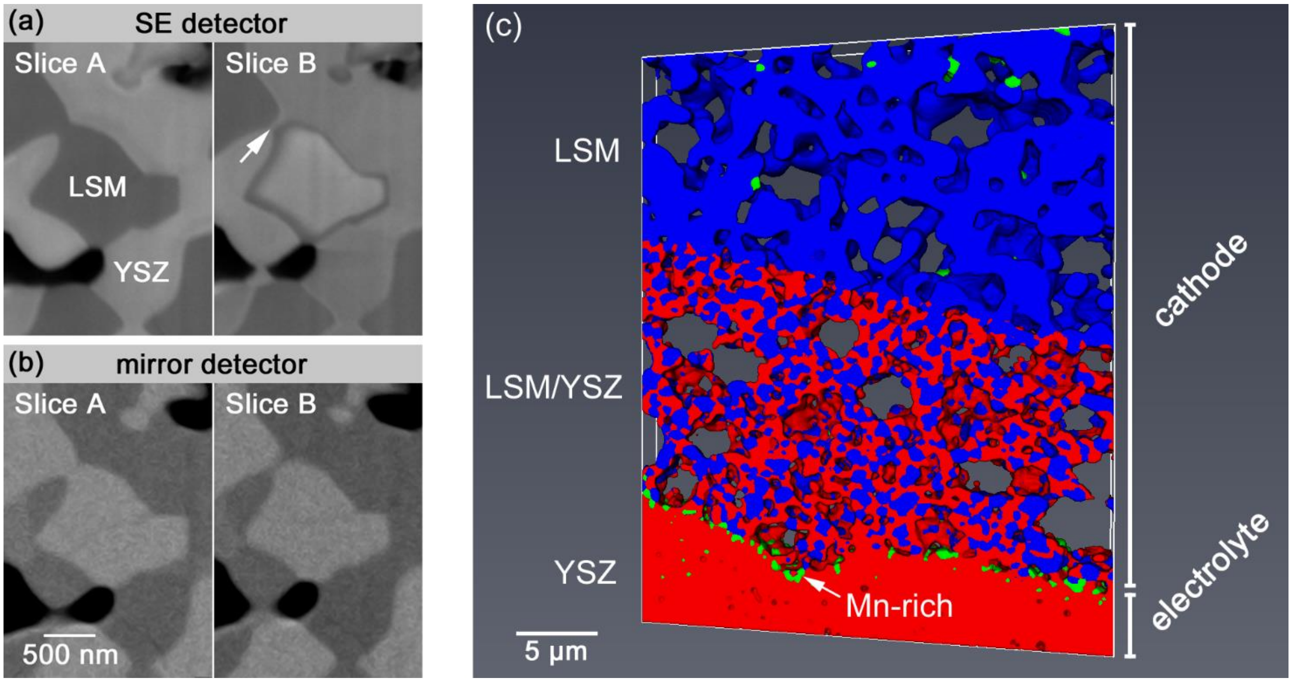

Fig. 1 SEM images of consecutive FIB slices taken simultaneously with (a) in-lens secondary electron and (b) mirror detector at 5 kV. (c) Resulting FIB-SEM reconstruction of a LSM/YSZ composite cathode.