3D-Orientation Mapping of Nano-grains in Solid Oxide Electrochemical Cells using TEM

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The performance of functional materials such as solid oxide fuel cells (SOFC) is closely related to the nano- and micro granular structure due to the large fraction of grain boundaries [1]. To make SOFCs efficient they must have high ionic conductivity [2]. Orientation effects on conductivity in SOFCs have previously been studied using impedance spectroscopy [1][3][4][5]. This showed that by aligning the particles, the ionic conductivity can be enhanced by up to two orders of magnitude in 2D thin films. However, 3D bulk samples appear to have different electrical properties and the experimental results point in different directions. Researchers discuss the validity of the experimental results since cracks and other 3D structural defects can affect the measurement. Impedance measurements should therefore be accompanied with a visualization of the 3D structure. The materials have previously been studied using 3D Electron Backscatter Diffraction [3]. However, this technique is highly destructive and is therefore not suitable for making in-situ or post operation imaging. Furthermore, the resolution could be enhanced using Transmission Electron Microscopy (TEM). In order to successfully understand the relationship between the performance and structure of SOFCs, it is thus necessary to visualize them nondestructively in 3D.

We propose 3D Orientation Mapping in TEM (3D-OMiTEM) [6], a promising technique for non-destructive visualization in 3D. A feasibility study on aluminum showed that 3D-OMiTEM can be done on approximately 100 nm thick samples with grains smaller than 20-30 nm with a resolution of a few nm. A simulated map of a tessellated nanostructure is shown in figure 1. Even though a tessellated nano-structure does not have realistic morphologies, the figure shows how colors can indicate grain orientations measured with 3D-OMiTEM. 3D-OMiTEM utilizes dark-field conical scanning in combination with sample tilts to obtain combined reciprocal and direct space information. The conical scanning uses beam tilt to choose diffraction rings from which dark field images are formed. Therefore, the reciprocal information is given by the beam tilt and sample rotation. Each measurement thus constitute a 2D-projection of the 3D grain shapes observed for a point in the reciprocal space. The subsequent reconstruction procedure provides orientation determination locally in the interior of the sample. Typically, each of the 6-10 inner rings in the diffraction pattern are used for every sample rotation angle. This results in up to 10.000 images that are reconstructed into a 3D grain orientation map.

Here we apply the 3D-OMiTEM method on 8% Yttria-stabilized Zirconia (8YSZ) which is a state of the art material for SOFCs. Electro spun and calcined 8YSZ nanofibers with the high ion conducting cubic phase were chosen since they have constant thickness when rotated in 3D-OMiTEM. Bright field, selected area electron diffraction pattern and dark field TEM images of a fiber is shown in figure 2. The diffraction analysis indicates that there is a minor fraction of tetragonal phase in the sample. The talk will give an overview of 3D-OMiTEM studies of YSZ.

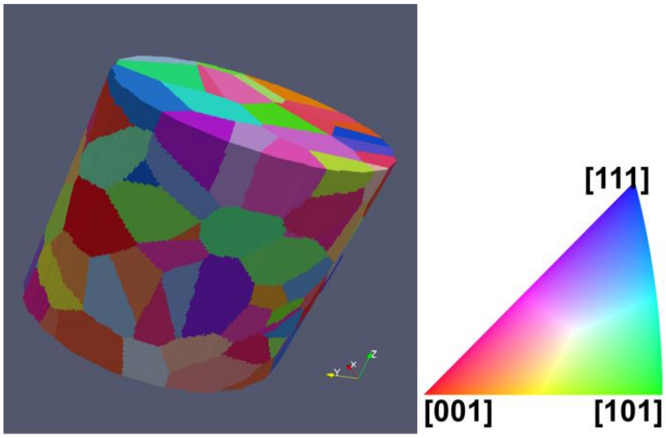

***Figure 1**: Simulated 3D-OMiTEM data of tessellated nano-structure after reconstruction in Dream3D. The colors indicate the grain orientation.*

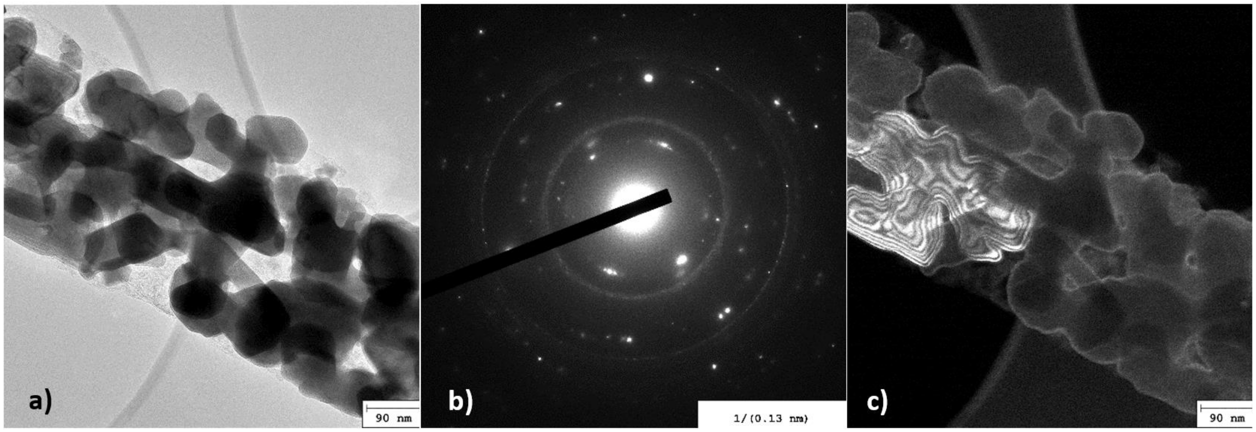

***Figure 2:** a) Bright-field TEM image of an 8YSZ nanofiber b) selected area electron diffraction pattern from the fiber c) Dark-field TEM image of the fiber.*

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