

Three-Phase Segmentation of Solid Oxide Fuel Cell Anode Materials Using Lab Based X-ray Nano-Computed Tomography

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Abstract

Triple-phase boundaries are an important microstructural metric to assess the performance and durability of solid oxide fuel cell electrodes and are known to significantly influence the performance at cell level. In recent years many advancements have been made in the quantification of TPBs including the use of focused ion beam scanning electron microscopes and synchrotron X-ray tomography although neither technique comes without limitation; the former being destructive and the latter having limited availability. This work demonstrates the first example of the application of lab-based X-ray nano-CT for non-destructive, microstructural characterisation of a SOFC electrode, where three-phase segmentation has been achieved. A SOFC anode cermet consisting of nickel and yttria-stabilised zirconia was imaged under X-ray using two fields of view: $64 \mu\text{m} \times 64 \mu\text{m}$ and $16 \mu\text{m} \times 16 \mu\text{m}$, with compositional data displayed for several samples at the two resolutions. This work highlights the possibility of three-phase segmentation using lab-based equipment allowing non-destructive quantification and mapping of triple-phase boundaries without the need for synchrotron radiation.

Keywords

Electrode; Microstructure; Multi-length Scale; Solid Oxide Fuel Cell (SOFC); Triple-phase Boundary; X-Ray CT

1. Introduction

In widely used composite solid oxide fuel cell (SOFC) anodes, the electron, ion and gas transport occurs through the nickel, yttria-stabilised zirconia (YSZ) and pore networks respectively. Locations where these three networks meet are named triple-phase boundaries (TPBs) and are known to greatly influence the electrochemical performance of the cell [1 - 3]. The abundance of TPBs has emerged as a key metric in the evaluation of SOFC electrodes, often presented as a TPB density for direct comparisons, and it is therefore desirable to locate and map these points linking microstructural and electrochemical characterisation. As well as maximising TPB density, the addition of electrolyte material (often YSZ) to the anode has been largely adopted for its benefits in reducing thermal expansion mismatch [4] between the anode and electrolyte layers, although other issues have been observed in such cermet materials when in the presence of non-uniform thermal environments [5, 6].

To date, there have been two favoured methods for the quantification of TPBs within SOFC anodes: focused ion beam scanning electron microscopes (FIB-SEM) and synchrotron X-ray computed tomography. FIB-SEM requires the milling of a sample face using an ion beam with sequential two dimensional imaging via an electron microscope [7]. This technique can achieve three-phase segmentation [8 - 12] via the high quality, lab-based imaging of electron microscopes; however, FIB-SEM also results in the destruction of the sample, removing the possibility of future analysis. X-ray computed tomography (CT) is a widely applied, non-destructive characterisation technique: radiographs are taken at many projections achieved via the rotation of either the source or sample, which are reconstructed into a virtual 3D volume. It is then possible to segment different materials according to their greyscale value, which is dependent on the X-ray attenuation of each constituent material [7].

In previous work, two-phase segmentation of SOFC anodes has been achieved with the use of lab-based equipment [13 - 15]. Whilst two phase data cannot provide TPB information, the microstructural data can be used to investigate metrics such as pore size distribution, tortuosity and diffusive flux [15, 16]. In order to map the TPBs within a microstructure three phases must be segmented, however, to date this has relied on the application of specialist synchrotron facilities which provide high brilliance, tuneable monochromatic radiation [16, 17]. In synchrotrons, high spatial resolutions are obtainable through the use of focusing and magnifying optics, although comparable resolutions are now obtainable using lab-based equipment which can achieve voxel sizes down to $16 \times 16 \times 16$ nm³.

Here, for the first time, we report the non-destructive three-phase segmentation of Ni-YSZ electrodes using X-ray nano-CT in a laboratory setting. The results demonstrate the possibility for the identification and quantification of not only metrics derived from two-phase information such as pore size distribution, but also solid phase composition, distribution and triple-phase boundaries. The sample volumes and voxel sizes demonstrated here are comparable to those currently available from synchrotron sources [16 – 18]. Due to the greater availability of lab-based techniques in comparison to the limited accessibility of synchrotron facilities, lab-based X-ray CT offers the potential to accelerate microstructural analyses and the design of improved electrodes.

2. Experimental

Model SOFC anode samples of varying microstructure were prepared using 12 tonnes of iso-static pressing of NiO-YSZ powder (Fuel Cell Materials, OH, USA) to form cylindrical pellets which were subsequently sintered at 1200 °C for 2.5 hours in air. The pellets were then reduced to Ni-YSZ in forming gas (4% H₂ in 96 % N₂) for 2 hours at 800 °C. Reduction

was confirmed by energy dispersive X-ray (EDX) chemical analysis and scanning electron microscope (SEM) imaging. The expected heterogeneity within the samples prepared here provides model microstructures of known composition ideal for technique development.

To conduct three dimensional microstructural analyses of the samples, fragments were removed from the reduced pellet bulk using a sharp razor producing sample diameters smaller than the largest X-ray field of view (FOV), *ca.* 50 μm . Each fragment was then attached to a pin head with the use of an epoxy to secure the sample; minimising drift during the scan and thus artefacts in the reconstruction. Tomographies were conducted with the use of a lab-based X-ray nano-CT instrument (Zeiss Ultra 810, Carl Zeiss., CA, USA). Whilst this sample preparation technique has acknowledged limitations [13], it has been successfully applied to prepare SOFC materials [4] and is deemed adequate for the provision of model samples required here.

X-rays are generated from a Cr source operating at 35kV_p giving a quasi-monochromatic X-ray beam for a Cr K-edge of 5.4 keV. The X-rays are focused using a capillary condenser, and provide full field illumination of the sample. Post-sample, the transmitted beam is focused onto the scintillator detector using a Fresnel zone plate, a full description of the system can be found elsewhere [19]. This process produces a radiograph projection displayed as a 2D greyscale image; in order to collect 3D information the sample is rotated through 180 degrees collecting a projection at each angular increment. By altering the zone plate the user is able to collect information at two length scales, namely: large field of view (LFOV) and high resolution (HRes) corresponding to FOVs of 64 \times 64 and 16 \times 16 μm respectively. Table 1 summarises the scanning parameters and pixel sizes for the two length scales presented in this work. Once collected the radiographs were reconstructed into tomograms using a filtered-back projection algorithm and segmented into three phases (Ni, YSZ, and pore) using a grey-scale threshold method after non-local means filtering

conducted in Avizo Fire software (FEI, France). Two threshold values were chosen at grey-scale frequency minima between the constituent peaks for each phase, triple-phase boundaries were then located by scanning the sample volume comparing neighbouring voxels; segmentation and TPB quantification techniques are described thoroughly elsewhere [1].

3. Results and Discussion

3.1. Lab-based Three-Phase Segmentation of Ni-YSZ Samples

The ability to map and quantify triple-phase boundaries is of great importance when analysing the influence of microstructure on the electrochemical performance of the electrode, but electrode microstructures can vary greatly between samples and within the same sample over time; heterogeneous microstructures can limit TPB density. Therefore heterogeneous samples have been chosen for this study to demonstrate the versatility and application of such techniques.

Four samples were imaged using the lab-based CT technique at both HRes and LFOV resolutions resulting in a total of eight tomographies. Once segmented compositional data was obtained via the summation of all voxels containing Ni, YSZ or pore, presented as a volume percentage for each phase. This was repeated for each tomogram, 2D slices for each of the four samples can be seen in both LFOV and HRes in Figures 1 b and c, respectively, with accompanying compositional data in Figure 1 d.

3.2. Comparing Microstructural Data Obtained at Two Resolutions

It is widely acknowledged that, when imaging the complex heterogeneous microstructures characteristic of electrochemical devices, the resolution requirement will depend on which microstructural property is under investigation [20]. For example, when determining pore or particles sizes, the effective spatial resolution must be sufficient to capture the volumetric

features in their entirety. Similarly, when assessing 2D properties such as surface area, the resolution should be sufficient to capture the materials surface roughness.

To allow direct comparison between the two imaging modes adopted here (LFOV and HRes) the same region of interest has been analysed at the two length scales for each of the four samples to determine whether resolution significantly affects the TPB measurement. Slices from the two segmented volumes are displayed in Figure 2. A comparison of the two modes has been conducted by examination of the volumetric composition, solid volume composition and triple-phase boundary density (ρ_{TPB}), all displayed in Table 2.

Microstructural data is comparable across the two length scales for volume and solid volume percentage compositions and the average solid volume percentages broadly match with that expected after reduction according to the manufacturer's data: 56 % YSZ to 44 % Ni [21]. As with all image-based quantification techniques, it is only possible to capture particles that are larger than the effective spatial resolution whilst the inspected volume element should be sufficient to ensure statistical significance of these solid volume fractions. It is noteworthy that the largest departure from the manufacturer's stated volume fraction occurs for a HRes scan with a small FOV. This further highlights the benefits of a multi-scale imaging approach.

TPB data is compared across the two length scales (Table 2) resulting in values similar to that presented previously for comparable microstructures [16, 22]. Small deviation is seen resulting in a higher TPB value recorded at HRes due to the higher resolution detecting an increased number of small particles, as described above.

3.3. Correlating Greyscale Value to Attenuation Coefficients

Microstructural investigations into SOFC materials often require repeated imaging to understand the development of structural changes over time. It is therefore of great

importance to understand the repeatability of this technique, particularly as some changes in microstructure are known to be subtle [23].

Although the greyscale histograms may differ from sample to sample under the same scanning conditions, if the material compositions are consistent within the FOV then the greyscale histograms should remain similar due to the attenuation properties of the materials. The intensity of the incident X-ray beam decays exponentially with distance through the material; this is described by the X-ray attenuation coefficient (μ_i), which characterises the amount of beam penetration through the material i and has units of length^{-1} [24 – 26]. The attenuation coefficient can be calculated from the mass attenuation coefficient (μ_{im}) [27].

The greyscale observed in the reconstructed tomogram scales proportionally with the beam intensity, hence the local attenuation coefficient of the materials, therefore the attenuation of two or more materials can be visualised by the greyscale histogram of the reconstructed tomogram: materials which are highly attenuating appear with high greyscale value (bright white) while low attenuation regions, such as a pores, appear with low greyscale value (dark grey or black). Therefore, if two materials are imaged under the same accelerating voltage and exposure time or within the same tomography, the difference in their greyscale values can be compared directly to their attenuation coefficients.

Describing generally for a two component system (A and B) the histogram plot produced from an X-ray tomogram using beam energy, E , will have a peak for each material which corresponds to greyscale value, GS_A and GS_B . At beam energy E , materials A and B will also have attenuation coefficients, μ_A and μ_B . A ratio taken of the two greyscale values (GS_{Ratio}) can be considered approximately equal to the ratio of the attenuation coefficients (μ_{Ratio}), demonstrated in Figure 3 a and b.

This method has been applied to the four samples analysed at both length scales (HRes and LFOV) resulting in a GS_{Ratio} for each of the eight tomograms calculated from the

greyscale histograms of the Ni and YSZ particles. In order to obtain the grey scale value three particles were chosen for Ni from each of the eight tomograms, with a central cube removed from each particle to determine the local grey-scale value, the three grey scale values were then averaged. This was then repeated for YSZ and a GS_{Ratio} was calculated for each tomogram. One GS_{Ratio} calculation is demonstrated in Figure 3 d displaying the local grey scale histogram for each particle. At 5.4 keV and using mass densities of 5.9 g cm^{-3} and 8.9 g cm^{-3} [28] for 8YSZ and Ni respectively the attenuation coefficients for μ_{8YSZ} and μ_{Ni} are approximately 1762 cm^{-1} and 1346 cm^{-1} [27], producing a μ_{Ratio} of *ca.* 1.31. Table 4 compares the calculated μ_{Ratio} to the average GS_{Ratio} obtained from the eight tomographies and Figure 3 e displays the GS_{Ratio} for each of the eight tomograms in comparison to the calculated μ_{Ratio} . It is seen that a the GS_{Ratio} obtained for each sample is consistent with the μ_{Ratio} .

3.4. Representative Volume Element Analysis

The sample volume investigated should be maximised in order to ensure that the tomogram is statistically representative of the material under investigation; a representative volume should be achieved [16, 18, 20].

In order to compare the statistical significance of a sample, a representative volume element (RVE) analysis can be conducted for a given parameter (TPB, VSSA, vol % etc) typically utilising a region growing algorithm considering sub-samples of successively larger volumes and extracting the metric of interest. At volumes below the RVE, oscillations in the considered metric will be observed, which are expected to dampen and disappear as the sample volume approaches the RVE. Typically the RVE will be quoted as the sample volume where oscillations between successive regions are small enough to keep within a defined

tolerance, say <2%, however for samples where there are heterogeneities on more than one length scale, this approach may not be sufficiently robust [29].

For example, it is conceivable that in a given sample, oscillations for a ‘nano-scale’ heterogeneity would disappear at a much smaller RVE than for a ‘micro-scale’ heterogeneity in the same sample. Noting the heterogeneities in these microstructures, we propose the following approach: once the oscillation is noted to fall below a tolerance of 2.5 % between successive regions, we continue the region growing algorithm for a pre-defined additional volume, monitoring the parameter of interest for the onset of any additional oscillations. This additional ‘stability’ window provides increased confidence in the RVE analysis where multi-scale heterogeneity is expected. The size of the stability window can be defined by the user, here we have chosen $50 \mu\text{m}^3$ for HRES and $100 \mu\text{m}^3$ for LFOV corresponding to ca. 10% of the overall scan volumes, with TPB as the metric of interest, the results of which are displayed in Figure 4 [30].

Eight RVE analyses were conducted producing triple-phase boundary density variation with volume analysed, seen in blue for LFOV and green for HRes for all four samples in Figure 4. The LFOV data for samples one and three show convergence around $700 \mu\text{m}^3$ while LFOV samples two and four reach a convergence but above $1000 \mu\text{m}^3$, an anticipated variation due to the increased heterogeneity within samples two and four. All samples within the HRes appear to stabilise within ca. $400 - 500 \mu\text{m}^3$ although this may not be representative of the macroscopic heterogeneities.

When investigating TPBs, the LFOV imaging technique provides sufficient statistical relevancy for the samples displayed here, through robust repeatability within large sample volumes. However, this may present lower accuracy than the HRes technique due to the

fractal-type nature of the TPB quantity, indeed this resolution dependence is a common problem throughout materials characterisation [31]. Therefore, although HRes offers potentially higher accuracy in TPB quantification, additional volume may be required for confidence in statistical relevancy, this can be achieved via the vertical stitching of two or more data sets or through the multi-scale approach adopted here.

4. Conclusion

Three-phase segmentation of a Ni-YSZ solid oxide fuel cell electrode has been achieved for the first time using multi-scale, non-destructive, lab-based X-ray tomography. This technique allows for the analysis of both large sample volumes and high resolutions, comparable to that which is currently achievable at synchrotron facilities.

Solid phase segmentation has been compared to values expected upon reduction of the NiO to Ni, showing similarity and consistency through a variety of model microstructures. The greyscale histograms for the 3D data sets have been analysed at both length scales and compared to the expected attenuation coefficients of the two solid materials, Ni and YSZ, showing small variation but close correlation on average at both length scales. Demonstration of the application of such three-phase data has been shown via the mapping and quantification of the triple-phase boundaries including an analysis of the volume required for the measured triple-phase boundary densities to be representative of the material bulk.

This technique has shown robustness in the quantification and mapping of triple-phase boundaries across various volumes and microstructures. By preserving the integrity of the sample it is possible, to perform repeat analysis enabling the quantification of TPB maps without the need for specialist facilities, such as synchrotrons, and will therefore prove valuable in future development of SOFC materials.

Acknowledgements

The authors would like to acknowledge the EPSRC (EP/M014045/1), the Centre for Doctoral training and the Royal Academy for Engineering for financial support, access to the Ultra 810 instrument was supported by the EPSRC (EP/K005030/1) and UC The authors would also like to thank Jeff Gelb and the team at Carl Zeiss for their valuable discussions.

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Table 1 Imaging parameters for the large field of view and high resolution tomographies collected using the lab-based X-ray CT system for Ni-YSZ samples

	Large Field of View (LFOV)	High Resolution (HRes)
Binning / no units	2	2
FOV / μm	64	16
Pixel size / nm	127	33
Exposure / s	45	65
Projections / no units	1101	1501

Table 2 Microstructural information for Ni-YSZ SOFC anodes obtained using lab-based X-ray CT at two length scales: high resolution (HRes) and large field of view (LFOV).

Sample	Technique	Vol.-%			Solid Vol.- %		ρ_{TPB} / μm^{-1}
		Ni	YSZ	Pore	Ni	YSZ	
1	LFOV	33	32	35	51	49	2.1
	HRes	36	31	33	54	46	2.6
2	LFOV	25	38	37	40	60	1.7
	HRes	23	32	45	42	58	2.8
3	LFOV	47	41	12	53	47	4.0
	HRes	44	38	18	54	46	5.6
4	LFOV	23	34	43	40	60	2.4
	HRes	30	31	39	49	51	3.3

Table 3 Greyscale information obtained from each of the four samples at both high resolution and large field of view displaying the greyscale values for Ni (GS_{Ni}) and YSZ (GS_{YSZ}) with accompanying GS_{Ratio} ($= GS_{YSZ}/GS_{Ni}$) for each sample at both LFOV and HRes.

Sample	Technique	Greyscale Value / no units		GS_{Ratio} /no units
		GS_{Ni}	GS_{YSZ}	
1	LFOV	114	148	1.30
	HRes	129	168	1.30
2	LFOV	129	211	1.64
	HRes	150	227	1.52
3	LFOV	121	156	1.29
	HRes	98	121	1.24
4	LFOV	143	195	1.36
	HRes	129	164	1.27
Average	-	127	174	1.36

Table 4 Comparing the calculated attenuation coefficient ratio μ_{Ratio} to the average greyscale ratio GS_{Ratio} obtained from the eight tomograms for Ni-YSZ materials imaged at 5.4 keV.

	Ni at 5.4 keV	YSZ at 5.4 keV
Greyscale Values, GS_i / no units	<i>126.6</i>	<i>173.8</i>
Attenuation Coefficient, μ_i / cm^{-1}	<i>1346</i>	<i>1762</i>
GS_{Ratio} / no units		<i>1.36</i>
μ_{Ratio} / no units		<i>1.31</i>

Figure 1 Microstructural information obtained from four Ni-YSZ SOFC anode samples using lab-based X-ray computed tomography at two length scales: high resolution and large field of view. Four samples (a) each with two tomograms of large field of view (b) and high resolution (c), with accompanying compositional information for LFOV –blue and HRes – green (d).

Figure 2 Demonstration of three-phase segmentation in the same region of interest at two resolutions LFOV (a, c, e and g) and HRes (b, d, f and h). Smoothed grey scale images (a, b), segmentation of the YSZ (c, d), Ni (e, f) and pore (g, h). Pore phase reconstruction with accompanying pore size distribution for LFOV – blue (i) and HRes – green (j).

Figure 3 Correlating the difference in attenuation coefficient to the separation of grey scale peaks for two materials: Ni and YSZ. A general example of the difference in attenuation coefficient (a) and grey scale (b) for materials A and B. A grey scale slice and extraction of two particles: grey Ni and white YSZ (c). Grey scale peaks for the LFOV sample 4 data-set (e). Grey scale ratios for all four samples at both length scales, HRes and LFOV with comparison to the attenuation coefficient ratio (f).

Figure 4 Triple-phase boundary maps with accompanying representative volume element analysis conducted via an expanding cube algorithm: a) samples 1 – 4, b) LFOV triple-phase boundary maps, c) HRes triple phase boundary maps and d) RVE analysis with LFOV presented in blue and HRes presented in green.