Measurement of Tortuosity of Anode Porosity by 3D Micro X-ray Computed Tomography

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Abstract

A series of samples taken from prebaked pilot anodes was analysed by micro X-ray computed tomography. The shortest path length through pores ascending from one side of the volume was calculated, giving a measurement of the tortuosity. Significant differences in the tortuosity were observed for the measured samples. In addition to the tortuosity calculations, the average pore diameter was calculated using a method from medical analysis of bone structure. The results are compared to the gas permeability, the coefficient of thermal expansion (CTE) and the electrical resistivity. The same model was applied to the solid carbon phase, providing tortuosity and inter-pore distances of the solid phase.

Keywords: Prebake Anode, Porosity, µCT, 3D, Image Analysis.

1. Background

In recent years, Micro X-ray Computed Tomography (µCT) has emerged as a strong supplement to microscopy based image analysis. SINTEF and Hydro Aluminium has collaborated in several projects, and has found µCT useful in studies of coke and carbon anodes, both structure, aggregate packing and for porosity distribution. Additionally, some work has been done on mapping of electrolyte bath at the anode working surface. A major advantage seen with µCT is that the technique can be used to image fairly large samples non-destructively at a good resolution within a reasonable time and cost. The data output is a recording of the whole 3D volume of the test piece, allowing for more complex investigation of the pore network and carbon structure. A variety of views can be generated post-measurement from the recorded raw data set. A previous paper presented on TMS Light Metals 2017 [1] provided details about our development of µCT as a characterization technique for carbon materials. The current paper goes further into the analysis of the pores as a 3D structure, rather than analysis of their 2D intersections in a plane.

Two main methods have been used for 3D analysis: Pore tortuosity and pore thickness. Since these terms are loosely defined, the chosen definitions and implementation will be described in detail in this paper.

2. Methods applied

The instrument used for the µCT analysis is an X-TEK XT H225 ST delivered by the UK company Nikon Metrology. It has a 225kV maximum acceleration voltage and employs a reflection source selectable between four different targets: Cu, Mo, Ag and W. The data used in the current paper was acquired using a W target at 135 kV acceleration voltage and 200µA current. The anode samples were drilled cores of 40mm diameter, and a sub-volume of each sample was exported as a 2024x2024x2024 volume at a voxel size of 10.8µm. The data was exported using the CT instrument software, and post-processed using ImageJ [3]. Further details can be found in [1].

2.1. Pore Tortuosity

Tortuosity is a concept that describes the difference between actual path-length (including twists and turns) and the shortest path-length (point-to-point distance). Tortuosity is commonly used to describe porous media in geo-science, such as soil and sandstones.

Figure 1 illustrates the concept of tortuosity in a pore network. The calculation was done from an X-ray tomography reconstruction of a porous sandstone; only the pores are shown [2]. The colour represents the shortest distance within the pore space from the left limit of the image to any point in the pores. Comparing this distance to the straight-line distance shows that the tortuosity is about 1.5 for this sample.

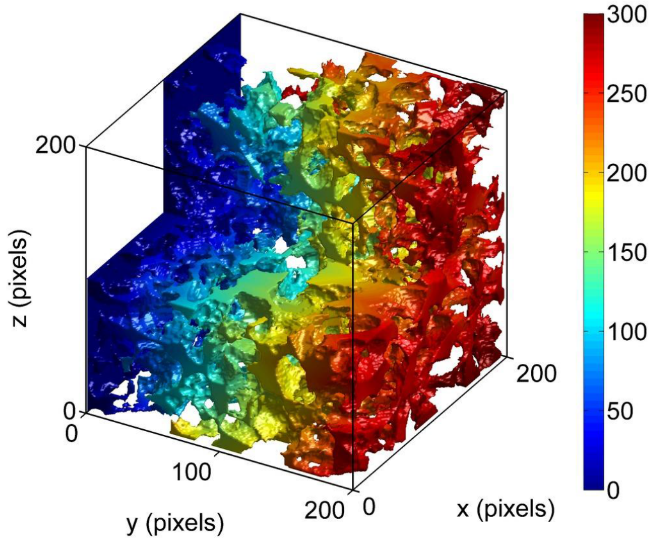

Figure 1. Illustration of the concept of tortuosity of a pore network. (Figure taken from the Wikipedia article on https://en.wikipedia.org/wiki/Tortuosity)

To simplify the calculations (since the datasets are large, up to 2048x2048x2048 voxels), the following algorithm for calculating a tortuous distance map was implemented.

1. A cubic volume is cropped from the inside of the original dataset (40mm core)
2. The calculations are done on a matrix of fractional numbers (32-bit)
3. All pore voxels in the volume are set to a value of -1.0, and background voxels to 0.0
4. The pores at the bottom slice are set to a value of 1.0
5. The slices are traversed from the bottom and up, and all adjacent pore voxels in each slice get added a value according to the smallest geometrical distance to a pore voxel in the previous slice:
    - Directly adjacent voxels (horizontally or vertically) get added a value of 1
    - Voxels adjacent diagonally in one direction (a connected edge) get added a value of $\sqrt{2}$
    - Voxels adjacent diagonally in two directions (a connected corner) get added a value of $\sqrt{3}$
6. Voxels that are not connected to voxels in the previous slice, retain their value of -1.0
7. When the top is reached, the dataset is traversed in the other direction using the same algorithm
8. The process is repeated from step 5 through 7 several times, until no more pixel values get changed, or a maximum number of iterations has been reached.

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6. References

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