Metallic foams characterization using X-ray microtomography

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Abstact

Due to their multi properties, metallic foams have become of major interest for both, basic research as well as industrial applications. In order to implement a thin modelling approach of fluid flows in such porous media, a three dimensional investigation of their internal structure is fundamental. An open-cell nickel foam is imaged using X-ray microtomography. The morphology of the foam is studied in detail using 3D image analysis. Parameters as the strut length and orientation distributions, the connectivity and the specific surface are determined from the 3D rendering volume of the foam to characterize the metallic structure. To qualify the porous space, we propose a 3D segmentation method based on the alpha-shapes approach. Pore size and orientation distributions are determined. This approach provides a tool to study the relationships between the foam microstructure and the physical properties, that should allow to determine optimal foams for given applications.

Keywords: X-ray microtomography, metallic foam, 3D image analysis

1 Introduction

Nickel foams as a classical open cell porous medias are already produced on a large scale. Due to their high surface to volume ratio thay are used in many field of applications such as batteries, fuel cells, catalysts and filters. However, the water management in fuel cell remains a key point for their development. We focuse our study in the optimization of the drainage of water in PEMFC (Proton Exchange Membrane Fuel Cell) to improve their efficiency. The aim is to simplify the global system and thus to have an effect on the cost, the bulk and the weight, three main criteria for PEMFC development. In this way, it is necessary to propose descriptive behaviour laws of the fluid flows in this porous medias. Simulation models rely on the real geometry of the components, in particular of the porous media used for the gas distribution. A suitable investigation of the 3D structure of these porous media is thus necessary to provide a thin geometric characterization of them. In this context, our work is focused on collecting basic knowledge on two-phase fluid flows in metallic foams used as gas distributor in fuel cells. It consists of an experimental part that provides geometrical data. These data are then used to implement simulation models.

This paper concerns the experimental part of the study and the 3D analysis of the data. We propose to describe the microstructure of open-cell nickel foams by determining geometric parameters of the metallic structure and characterizing the porous space. The foam analysis is carried out from computed X-ray microtomography (μCT) data. This non-destructive imaging technique provides 3D images at a high spatial resolution that enables a thin description of the studied samples. After a brief description of the imaging system, we describe the 3D image analysis procedures used to extract structural parameters quantifying the internal structure of the foam, and the image processing tools developed to close and segment the open cells in order to extract morphometric parameters describing the porous space. First results obtained from a small sample of foam are presented.

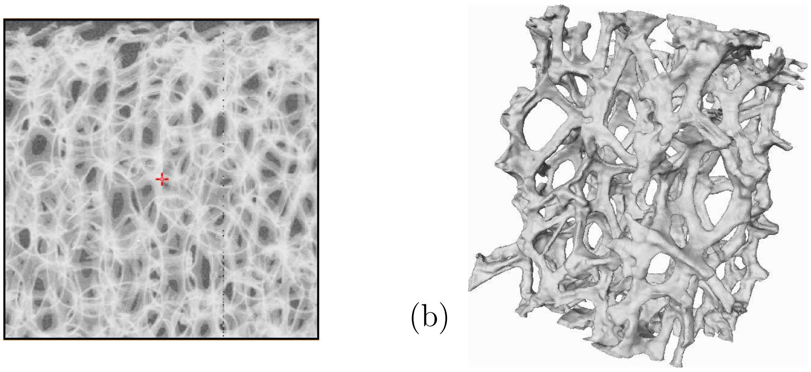
2 Materials and methods

The studied foam is a standard product from INCOFOAM^{TM}. It is a 1.6 mm thickness and has a specific density of 420 g/cm^2. The pore size is around 500 μm and the porosity from 95 to 99 %. A 1 mm^2 sub-sample was cut out from a foam sheet and imaged using an X-ray microfocus generator coupled with an imaging detector. Tomographic experiments have been performed using a lab experimental set-up at CEA-List (Commissariat à l'Energie Atomique, Saclay).

2.1 *Experimental set-up*

The sample is fixed on a rotation stage between the X-ray source and the detector that acquires the transmitted X-ray images. The X-ray generator is a microfocus; it is a commercial product from Feinfocus firm. It allows to work from 20 to 160 kV, presents a maximum power of 14 W, and has a focus spot size around 1 μm for low energy functioning. The target material is 5 μm tungsten deposited on a diamond substrate for thermal dissipation. The detector (Medipix 2, [1, 2, 3, 4]) is a photon counting technology developed through a collaboration of 16 European partners around Cern. It is very low noiseless and presents a high detection efficiency in our energy range (<25 keV). During acquisitions, the sample is disposed near the source to have a magnification of a factor 7 (pixel size: 8 μm), and is progressively rotated over a total angular range to provide a set of 360 radiographs (exposure time: 5 s ; angular step: 1 °). Each radiograph is pre-processed to get the projection data used in the reconstruction algorithm. The correction removes inhomogeneities from the X-ray field and detector response. Figure 1.a shows an example of a corrected projection.

The classical algebraic OSEM (Ordered Subset Expectation Maximization) algorithm was implemented and used for reconstruction [5, 6]. This iterative approach gives reliable results but presents the disadvantage of a slow convergence. In order



(a) (b)

Figure 1: (a) Example of a radiographic projection of the foam, (b) 3D volume rendering of the reconstructed sample.

to speed up the reconstruction step, a parallel version of the OSEM algorithm has been implemented using the MPI (Message Passing Interface) library [7]. The benefit of using 2 quadri-processors AMD Opteron 2.4 GHz vs one processor is a factor equal to 5.6. By using 12 processors instead of one, the factor is greater than 7.

2.2 *3D Image analysis*

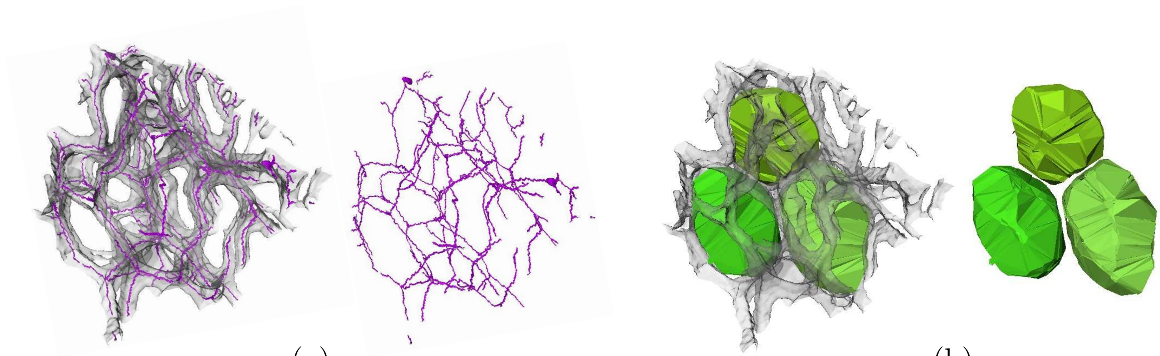
The 3D initial volume is a set of 2D gray level slices derived from reconstruction. The first step of quantification is image segmentation for separating the solid phase to the background. Due to a large difference between the linear attenuation coefficient of air and nickel, the distribution of the images appears bimodal. So, a simple global threshold is applied to generate binary images, and hollow struts are filled in. A 3D isosurface is then generated from the binary slices (fig. 1.b), and the specific surface is estimated from it as the sum of the surfaces of all the triangles constituting the 3D surface.

The morphologic analysis of the metallic structure is based on the 3D rendering volume showed on the figure 1.b. The 3D skeleton of the structure is computed using the Pandore library [8] ; the homotopic skeleton operator allows to obtain the homotopic core of a 3D image by a sequential deletion of simple points (a simple point being a point whose deletion does not modify the image topology). Structural parameters as the strut length and orientation distributions, the curvature angles and the connectivity are estimated from this skeleton to characterize the morphology and the organization of the foam architecture. Figure 2.a shows the result of the 3D skeleton of a part of the metallic structure (just a region of the total volume is showed for a better clarity of the figure).

The 3D analysis of the porous space of open-cell foams is a more complicate task because of the 3D segmentation of the cells. To compute morphometric parameters, the cells of the foam have to be isolated first. A simple threshold as used for closed cells is not possible [9] and the watershed method poses the problem of an over segmentation. So, we propose in this work to use the concept of the alpha shapes [10] to segment the open cells. The alpha shape is a concrete geometric concept which is mathematically well defined: it is a generalization of the convex hull and a subgraph of the Delaunay triangulation. Given a finite point set S, a family of shapes can be derived from the Delaunay triangulation of S. A real parameter called "alpha", $\alpha \in [0, \infty[$, controls the desired level of details. The alpha shape of S is a polytope which is neither necessarily convex nor necessarily connected. For sufficiently large alpha, the alpha shape is identical to the convex hull of S. As alpha decreases, the shape shrinks and gradually describes cavities. These cavities may join to form tunnels and voids. For sufficiently small alpha, the alpha shape is empty. An alpha shape is a concrete geometric object that is uniquely defined for a particular point set. In our case, the value of α is fixed so that each interface between open-cells can be closed and the pores isolated. Figure 2.b shows the result of three segmented cells. Using a fixed α supposes a homogeneous size of the interfaces between cells.

3 Results

To characterize the architecture of the metallic struture of the foam, quantitative parameters have been extracted from the 3D rendering volume and its skeleton. The specific surface, or exchange surface, is estimated by summing the surfaces of all the triangles defining the surface of the 3D volume rendering. It is about $10\,\mathrm{mm}^2/\mathrm{mm}^3$. The morphologic analysis of the foam is studied from the 3D skeleton of the structure. After having located all the nodes of the skeleton and associated a node connectivity list, the number of strut per node and mutual angles distributions



(a) (b)

Figure 2: *(a) A part of the 3D rendering volume of the foam superimposed with its skeleton shown alone on the right (b) 3D segmented open-cells superimposed with the same part of the 3D structure shown alone on the right.*

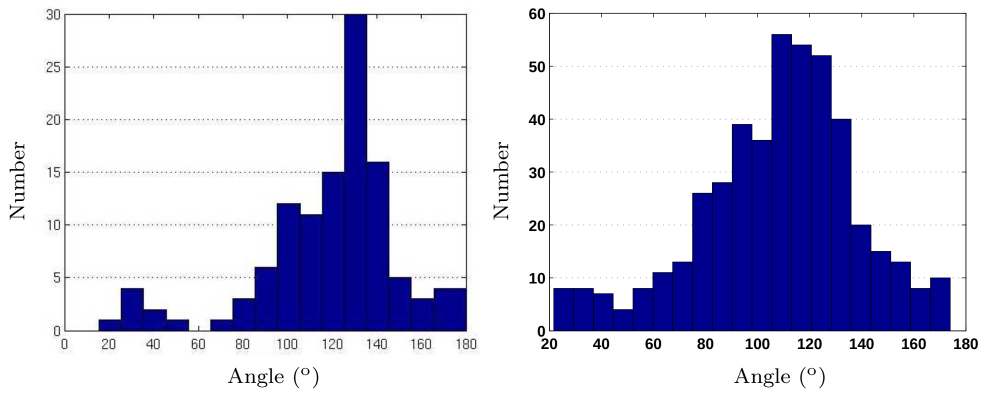


Figure 3: *Mutual angle distribution for the nodes connecting (a) 3 struts or (b) 4 struts. (c) Strut length distribution of the studied foam sample.*

are determined. There are four struts per node for more than 53 % of cases, three struts per node for 27 % and five for 18 %. The other 2 % relate to the nodes connecting more than five struts. The mutual angles distributions for the nodes connecting three or four struts are respectively presented on the figure 3.a and 3.b. It shows that the mutual angles values are centered around 120° what corresponds to the angle described by the first laws of foam geometry described by Plateau.
For the analysis of the open-cell morphology, incomplete cells at the boundary are excluded. Figure. 2.b shows an example of isolated cells obtained by the alpha-shape method. On the left image, we easily observe that the alpha-shape surface well delimits the porous space inside the metallic structure. On the right one, the 3D volume rendering of the isolated cells allows to observe that the pores are non-spherical. Considering the whole of the segmented closed cells, the mean of the longest diameter of the cells is 486 μm with a standard deviation of 4 μm that well corresponds to the value of 500 μm given by INCOFOAM^{TM} for the pore size. The estimated volumes of the isolated cells are however very heterogeneous: the maximal volume is 0.41 mm^3 and the minimal one 0.02 mm^3.

4 Conclusion and perspectives

As mentioned by previous studies [9, 11, 12, 13] the computed X-ray microtomography technique is suitable for the investigation of the 3D structure of foams. Moreover, we have just showed that quantitative analysis of foam materials can be done directly in a lab with a microfocus generator and a low noise image detector. It opens encouraging perspectives to propose systematic studies on porous media and define with simulation models optimal foams for given applications. A larger sample had to be now analysed to obtain more representative results, and others parameters as the radius curvature of struts and nodes remain to be estimated to configure

behavior models. The segmentation of the open-cells remains a tricky step because of the α value adjustement. In order to be sur to close all the open-cells it would be judicious to compute the alpha-shape using a distribution of values instead of a unique one.
Previous experimental study had first been done to show the feasibility of imaging the drying of a foam and the water distribution over time [14, 15]. Microtomographic experiments are now in progress to study two-phase flows in such metallic foams. In parallel, 3D structures as presented in this paper are injected in CFD models to simulate two-phase flows. Such developements should allow to understand and predict the phase transport properties to mass and thermal transfer processes.

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