On the use of autocorrelation functions, permeability tensors, and computed tomography to characterize the anisotropy of diesel particulate filter materials

D. Bernard*[1,2], F. Léonard[3], E. Plougonven[4], G. Bruno[3,5]

[1]CNRS, ICMCB, UMR 5026, F-33600 Pessac, France
[2]Univ. Bordeaux, ICMCB, UMR 5026, F-33600 Pessac, France
* Dominique.Bernard@icmcb.cnrs.fr
[3]BAM, Bundesanstalt für Materialforschung und -prüfung, 12205 Berlin, Germany
[4]University of Liege, Belgium
[5]University of Potsdam, Institute of Physics and Astronomy, 14476 Potsdam, Germany

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Summary: We show how the combination of autocorrelation function and permeability calculations, applied to 3D X-ray computed tomography data, allows yielding quantitative information on the anisotropy of both microstructure and fluid flow in Diesel Particulate Filter (DPF) materials. Several aspects about the meaning of the outputs of the autocorrelation function are discussed.

1. INTRODUCTION

3D images such as those produces by X-ray tomography can provide a wealth of information on the internal structure of materials, but quantification of specific geometrical or topological characteristics linked to some bulk physical property is far from being straightforward. This study focuses on methods to quantify the differences in physical properties as a function of direction, *i.e.* their anisotropy, and how it can be linked to measures of anisotropy of the internal structure of the material.
The auto-correlation function gives a similarity measure in the volume as a function of distance and direction. This is a cross-correlation of the image with itself fast to compute and relatively insensitive to noise. It is why we focus on this method to compare with the physical property of our DPF material.
Diesel Particulate Filter (DPF) materials are porous ceramics that; a) can be used at very high temperatures; b) have very good thermal shock resistance; c) are inert; d) can be manufactured with tailored porosity. Their usual way of production consists of the extrusion of a slurry into the desired filter shape, with successive ceramming at high temperature. This process causes anisotropy at both microscopic and macroscopic levels.

2. CHARACTERIZATION METHODS

X-ray computed tomography acquisitions were performed using two different systems: 1) a GE v|tome|x L 300/180 equipped with a 180 kV source, a tungsten transmission target, and a GE 2000 × 2000 pixel DXR-250 detector. The source was operated at a voltage of 60 kV and a current of 170 µA. The sample projections were taken at 1500 angular positions per 360° rotation, and an exposure time of 3 seconds. The samples position, and a binning of 2×2 pixels resulted in voxel size of 4.0 µm. 2) the beamline BAMline at the synchrotron source BESSY II of the Helmholtz-Zentrum Berlin für Materialien und Energie (HZB), Berlin, Germany. The beam energy was set to 15 keV, and a PCO4000 CCD camera (4008 × 2672 pixels) was used in combination with an Optique Peter microscope equipped with a $CdWO_4$ scintillator and a 10-fold magnifying objective, resulting in a pixel size of 0.434 µm. A series of 3200 projections were acquired per 180° rotation, with a 4 s exposure.
The **normalized autocorrelation function of the centred image** $(I(\mathbf{u}) - \langle I \rangle)$, NACFC, noted $C_I(\mathbf{v})$, has the following expression: $C_I(\mathbf{v}) = \frac{ACF_{I-\langle I \rangle}(\mathbf{v})}{Var(I)} = \frac{\langle [I(\mathbf{u})-\langle I \rangle].[I(\mathbf{u}+\mathbf{v})-\langle I \rangle] \rangle}{\langle [I(\mathbf{u})-\langle I \rangle]^2 \rangle}$, where $I(\mathbf{u})$ is the value of the image at the position specified by the vector **u**, $I(\mathbf{u}+\mathbf{v})$ the value of the image at the position **u** plus the lag vector **v**, $< >$ is the averaging operator over the support of the image, and Var(I) is the variance of the image I. For anisotropy estimate, the volume $V_I(c)$ constituted by all the voxels for which NACFC is larger than the given threshold c is considered. The tensor of inertia of this volume gives the three directions of anisotropy (principal directions) and the degree of anisotropy (ratio of the larger to the smaller eigenvalues).
The **permeability tensor** is obtained by averaging the solution of a partial differential problem, the closure problem, derived by applying the volume averaging method to Stokes equations. For many materials, direct measurement of permeability in a single direction is arduous, and estimating the full permeability tensor is generally impossible. Although in practical applications solving the closure problem on a grid defined from 3D images is always computationally demanding, this approach produces a good approximation of the complete permeability tensor.

3. DPF MATERIALS

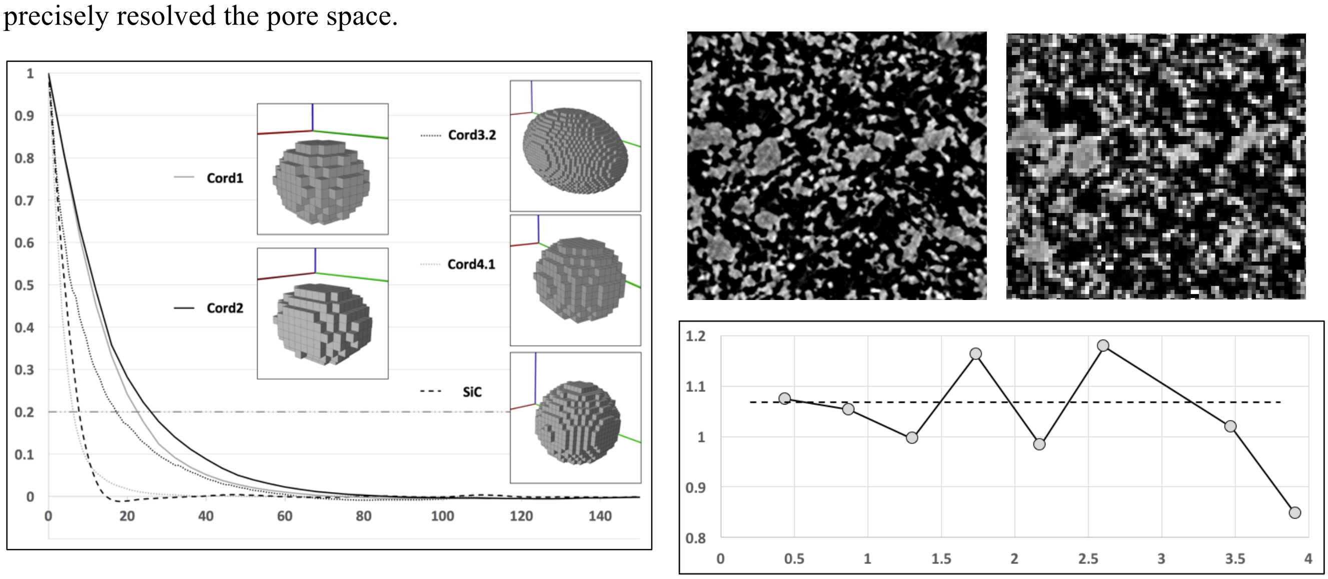
A total of 5 materials have been investigated, four are cordierites (named Cord1 to Cord4) and one is SiC. All of them are extracted from larger filters produced by extrusion. Cordierite specimens Cord1 and Cord2 were scanned on a laboratory CT system at BAM, and samples Cord3, Cord4, and SiC were scanned on the synchrotron beamline BAM*line*. Qualitatively, Cord1 to Cord3 are similar materials showing sharp-edged pores with a large size distribution; Cord4 possesses much smoother pore/material interfaces and a larger porosity fraction. For SiC, footsteps of the initial powder particles are visible producing rounded pores.

4. RESULTS

The five 3D grey-level images were first realigned with the reference axes A (along the wall), B (extrusion direction), and C (normal to the wall), then cuboid sub-volumes were extracted from the porous zones; one for Cord1, Cord2 and SiC, two for Cord3 and Cord4. After filtering (3D median and in-house developed anisotropic diffusion), seven binary sub-volumes were obtained. For each of them the permeability tensor and the NACFC were computed. The extrusion process induced a higher permeability coefficient in the B direction except for SiC. To quantify this anisotropy, the degree of anisotropy DA_k was computed as the ratio of the larger to the smaller eigenvalues of the tensor (Table).
From the NACFC, the different volumes $V_I(c)$ were built and their tensors of inertia calculated, giving, as functions of the threshold c, the directions of anisotropy and a new estimate of the degree anisotropy, DA_Inertia(c), defined as the ratio of the larger to the smaller eigenvalues (Table).

Sample	Cord1	Cord2	Cord3.1	Cord3.2	Cord4.1	Cord4.2	SiC
k_{AA}	6.01	2.45	0.73	0.85	1.91	1.92	0.70
k_{BB}	6.70	3.31	0.84	1.04	2.12	2.13	0.65
k_{CC}	5.93	2.64	0.64	0.76	1.80	1.81	0.63
DA_k	1.14	1.36	1.42	1.44	1.18	1.18	1.12
DA_Inertia(0.20)	1.18	1.37	1.56	1.43	1.26	1.26	1.08

To quantify the correlation between the structural anisotropy (from NACFC here) and the functional anisotropy (from permeability here), the evolution with c of $\overline{\mathrm{DA}} = \mathrm{DA\_Inertia}(c)/\mathrm{DA\_k}$ is examined. It appears that the level of correlation is very high and that c=0.2 is the best compromise for the considered materials. Figure 1.a is presenting, for five of the cuboid binary sub-volumes, the evolutions of NACFC with the lag v in one direction of anisotropy. 3D rendering of the associated volumes $V_I(0.2)$ are also presented.
The directions of anisotropy were also compared with a very good correlation.
All the results presented above were obtained from binary images, a requirement for permeability computation. Segmentation is always a problematic process because of its influence on the final results. Knowing that it is as easy to compute NACFC from a binary or a grey-level image, we performed a test summarized Figure 1.b. A reasonable estimate of the degree of anisotropy can be obtained from 3D images with pixel size too large to precisely resolved the pore space.



a) b)

Figure 1: (a) NACFC in one direction as a function of the lag (in µm) for five sub-samples. Inserts: 3D rendering of the volumes $V_I(0.2)$ for the same sub-samples. (b-top) The same section of Cord4 with pixels of 0.434 µm and 3.47 µm. (b-bottom) Evolution of $\overline{\mathrm{DA}}(0.2)$ with the pixel size. The dashed line indicates the value obtained for the B&W image.