3D-POROSITY STRUCTURAL ANALYSIS OF LONG - FIBER - RENFORCED CERAMIC MATRIX COMPOSITES USING X-RAY TOMOGRAPHY

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ABSTRACT

This study has been devoted to a volumetric exploration of the meso-structure of different fiber composite materials using x-ray tomography. A series of projection images have been used to reconstruct the three dimensional composites' architecture and a quantitative analysis of the morphology of the porosity network has been carried out in order to characterize the different fiber-tissue structures. The tomogram image's analysis provide with interesting parameters like the amount of porosity, the open porosity fraction, the connectivity of the porous network, the tortuosity and the thickness distribution of the open porosity. The composites studied were a $[0,+60,-60]_n$ C_f/C laminate and a 2D-SiC_f/SiC with cross-weave fibers. The X-ray tomography suitability for characterizing these structural materials at a meso-scale level is critically assessed.

1. INTRODUCTION

Continuous-fiber-reinforced ceramic matrix composites (CMCs) are promising thermostructural materials [1]. The fiber yarn's reinforcement enhances the ceramic mechanical response; in contrast, the material design and processing become more expensive and difficult. The fiber preforms are densified by the matrix, usually deposited from a gas precursor. The densification process is incomplete, leaving a residual network of porosity in the composite that have a strong effect on its mechanical behavior.

The X-ray computed tomography is a non-destructive characterization technique which allows for the 3D observation of the meso-structure of highly porous materials. This technique, based on X-ray attenuation, provides numerical images of the bulk of the materials. The 3D images obtained give information on the shape, the dimension and position of large voids, cracks or pores [2].

The main objective of this paper is to assess the feasibility of using x-ray tomography as a capable technique for an accurate characterization of volumetric porosity in CMCs. In order to achieve this objective, the pore's morphology of two composite materials with different fiber-woven structures is compared.

2. EXPERIMENTAL PROCEDURE

2.1 Material

Two different composites, a 2.5D laminated carbon-carbon composite and a 2D SiC_f-SiC, provided by SNECMA (France), were selected for this study. The C_f-C laminate consists of 0, 60 and −60º fiber plies sequence with a few percent of fiber bundles oriented through-thickness to improve the interlaminar properties. The 2D-Nicalon-fiber-reinforced SiC matrix composites were fabricated from preforms of 0/90º woven Nicalon NLM 202 fibers (from Nippon Carbon Co, Japan). Both composites were processed from a fiber yarn preform of 2.5D carbon or 2D SiC, respectively, by chemical vapor infiltration (CVI) densification. Details of these fiber-reinforced

composites fabrication are described elsewhere [3-4]. These two non-oxide CMCs have different yarn architectures with distinguishable pore structures.

2.2 X-ray tomography conditions

This study focuses on the applications of x-ray tomography for structural characterization of composite materials at micro- and meso-scales. The key point of this method is acquiring a series of x-ray radiographs of a sample that rotates step by step about an axis perpendicular to the incident beam. The acquisition system (Figure 1) involves a standard laboratory tomograph composed of an X-ray nanofocus tube and an amorphous silicon medical detector. A mathematical algorithm is used to reconstruct the distribution of absorption coefficients within the sample volume. The internal 3D structure of the sample can be determined via this x-ray absorption coefficient's mapping, measured for cubic elements of matter called voxels. A detailed description of this non destructive technique can be found in [5].

The samples were scanned at a magnification value on the detector of 28 (corresponding to pixel size of 4.5 μm). The beam energy was set to 85 kV and the tomogram images were reconstructed from 1200 projections.

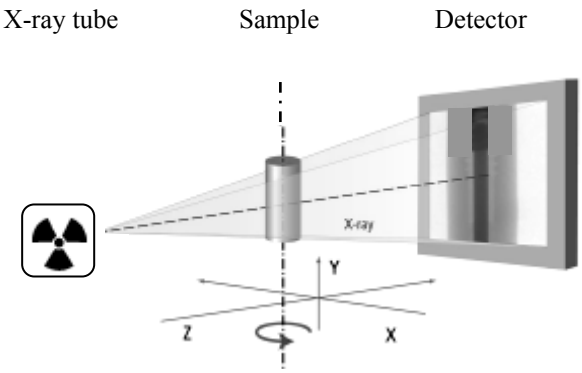

Figure 1: A schematic of the experimental device.

3. IMAGE PROCESSING AND MORPHOLOGIC PARAMETERS

The piled-up 2D reconstructed tomographic slices provide images of the inner structure of the sample allowing, in particular, the study of the porosity. These images were processed to reduce the amount of noise by 3D median filtering over a neighbouring of one voxel.

The porosity analysis is performed over these enhanced grey level images separating the voxels belonging to the fibre/matrix phase (black phase) from the cracks and pores (white phase). The binary images obtained have the topology of the samples' pores, which allows studying its connectivity, open porosity size distribution and tortuosity.

The porosity was measured by the white voxels ratio, calculating the number of white voxels and dividing by the total size of the block in voxels [6]. The profiles of density along different directions are calculated as well as the average porosity fraction over the volume.

The connectivity between pores has been measured in order to select the open porosity. More details about how to characterize the 3D pore connection can be found in [7]. The characterization of the interconnected-pore network has been made by measuring the channel size and tortuosity.

The thickness distribution was determined by a computational processing procedure that allows measuring the density distribution of the thickness of the pore phase in the case of inter-penetrating pore/solid networks [6].

Finally, the tortuosity characterizes the straight or winding shape of the open pore. It was defined as the ratio of the length covered between two points within the pore phase to the total distance in straight line. The tortuosity was measured along the three main perpendicular directions (x, y and z directions).

4. EXPERIMENTAL RESULTS AND DISCUSSION

4.1 X-ray tomography images

Figures 2 (A) and (B) show the tomogram images corresponding to two-dimensional reconstructed tomographic slices for each material. Both are reconstructions' views of the z-axis stacking.

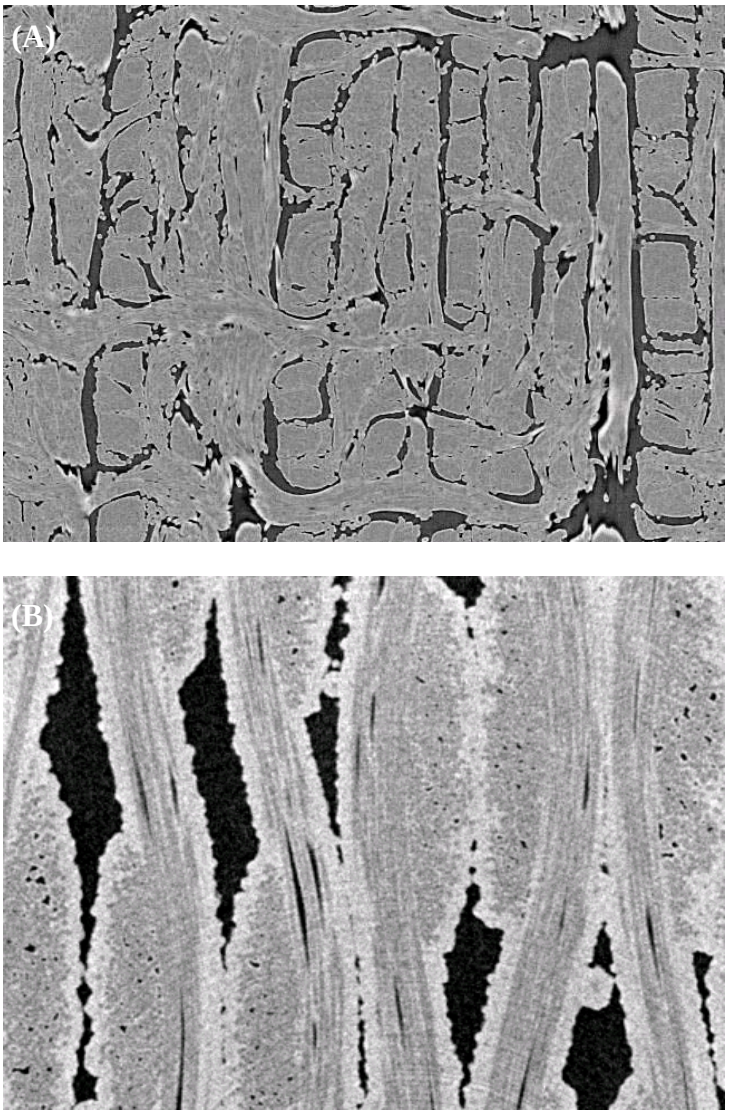

Figure 2: (A) Transversal slice of 2.5D-C_f/C and (B) 2D-SiC_f/SiC composites

The longitudinal and transversal plies are distinguished from the matrix in the case of SiC_f/SiC composite (Fig. 2 (B)). However, the selected magnification and the attenuation contrast between matrix and fibers are not enough to allow the separation of the carbon fibers in the C_f/C composite (Fig. 2 (A)). The different size of the carbon and the SiC fibers explains the lack of resolution at this magnification. Nevertheless, the inter- and intra-ply porosity is well observed for both materials.

Figure 3 shows a three-dimensional rendering of the porosity's structure (magenta color). These 3D images are obtained by drawing a surface between all the voxels exhibiting the same gray level (the voxels belonging to the solid phase being transparent). These images clearly show the 3D pore's architecture.

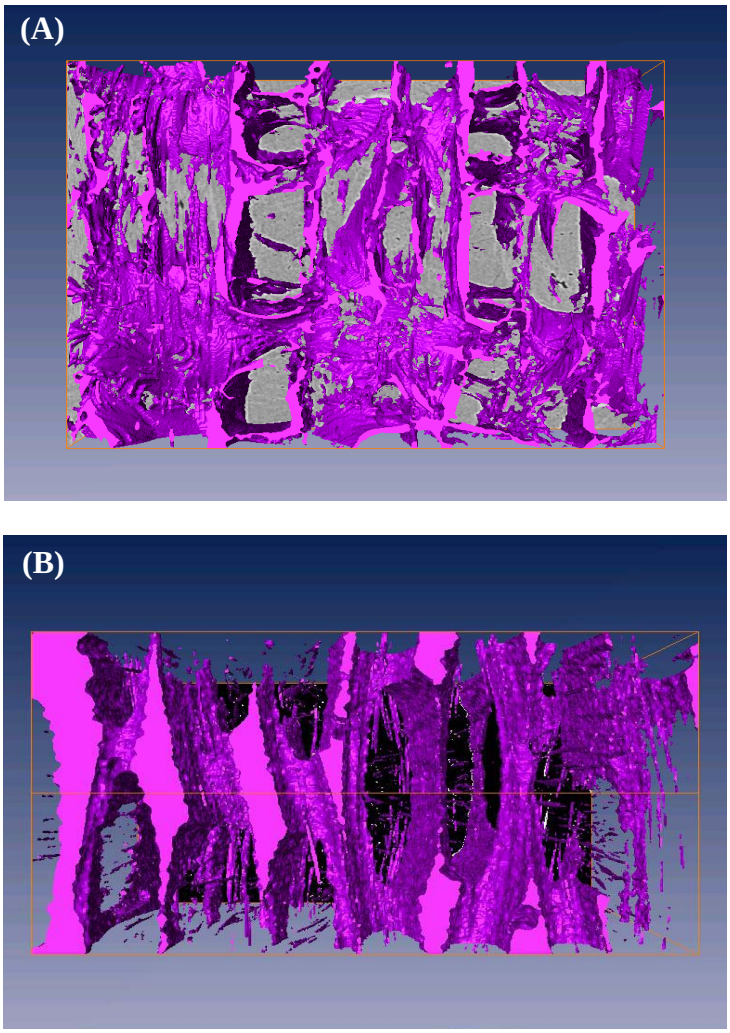

Figure 3: (A) 3D-volume rendering of the axial view of pores in 2.5D-C_f/C and (B) in 2D-SiC_f/SiC composites

From a qualitative viewpoint, the 3D images allow concluding that 2D-SiC_f/SiC is more porous and has higher pore channels than 2.5D-C_f/C composite.

4.2 Density measurements and size distributions

The tomogram images can also be used to characterize the structure of the porosity in a quantitative way. They have been processed to obtain parameters that quantify the connected-pore's thickness distribution and the tortuosity of the open pore.

Typical profiles of the porosity values measured in each slice as a function of the slice number have been plotted in Figure 4, showing that the surface porosity fractions are strongly fluctuating in the x-direction. The C_f/C surface fractions along the other directions are nearly homogeneous. The SiC_f/SiC composite also exhibits fluctuating surface fractions in the other piled-up directions, although less noticeable than along x-

direction. The sizes of the analyzed blocks are non isotropic which explains why there are different number of measurements according to the different directions and the material.

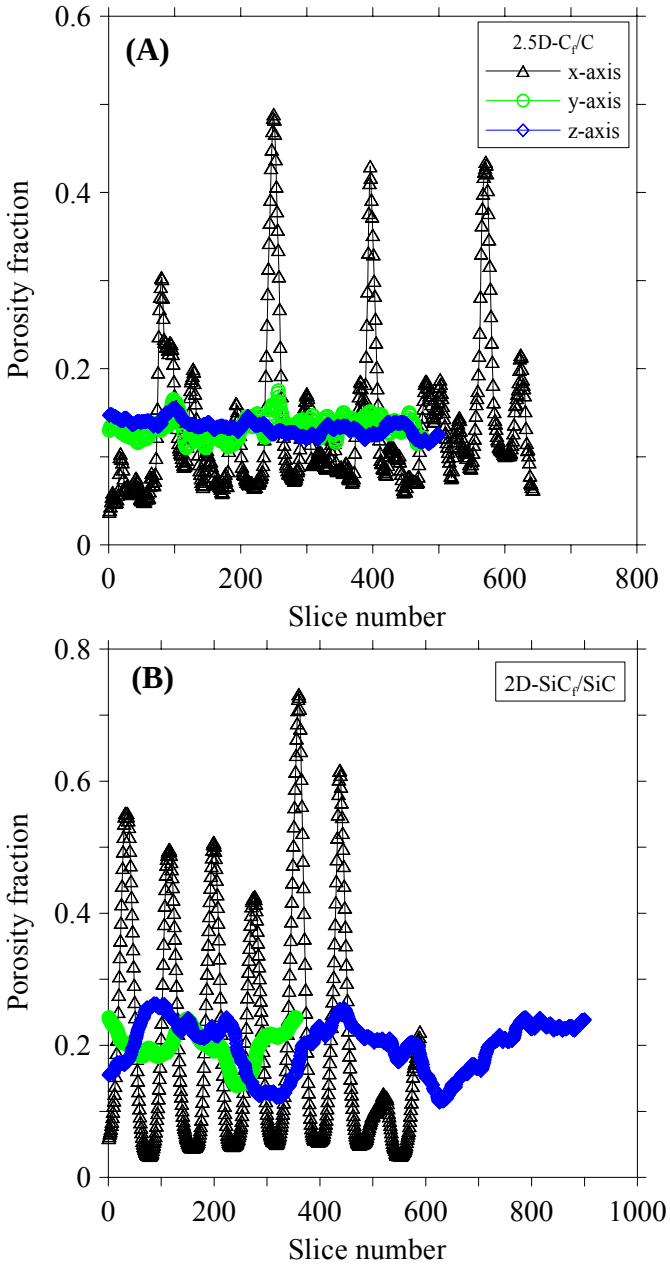

Figure 4: Volume fraction profiles as a function of the slice number in the three spatial directions for (A) 2.5D C_f/C and (B) 2D SiC_f/SiC.

The average porosity volume fraction obtained along the three main perpendicular directions (x, y and z) was 13% for the C_f/C composite and 20% for the SiC_f/SiC composite. These results are in good agreement with direct measurement by optical microscopy [8]. The open porosity fraction was 88 and 79 vol. % of the total porosity, respectively for C_f/C and SiC_f/SiC composites.
The average density values obtained along each perpendicular direction are the same in each case, but the relative standard deviations (RSD) are different (Table I).

The tortuosity values measured along x, y and z directions (Table I) give an average value of 1.216 ± 0.006 and 1.312 ± 0.005 for C_f/C and SiC_f/SiC composites, respectively.

Table I. Relative standard deviation of the porosity fraction (RSD) and tortuosity values along three perpendicular directions obtained from processing tomography data.

Sample	RSD_x (%)	RSD_y (%)	RSD_z (%)	Tortuosity (x)	Tortuosity (y)	Tortuosity (z)
2.5D-C_f/C	64	9	6	1.422 ± 0.009	1.136 ± 0.003	1.091 ± 0.007
2D-SiC_f/SiC	87	13	20	1.823 ± 0.011	1.078 ± 0.002	1.035 ± 0.003

The density distributions of the thickness of the open porous network are compared for the two materials studied (Figure 5).

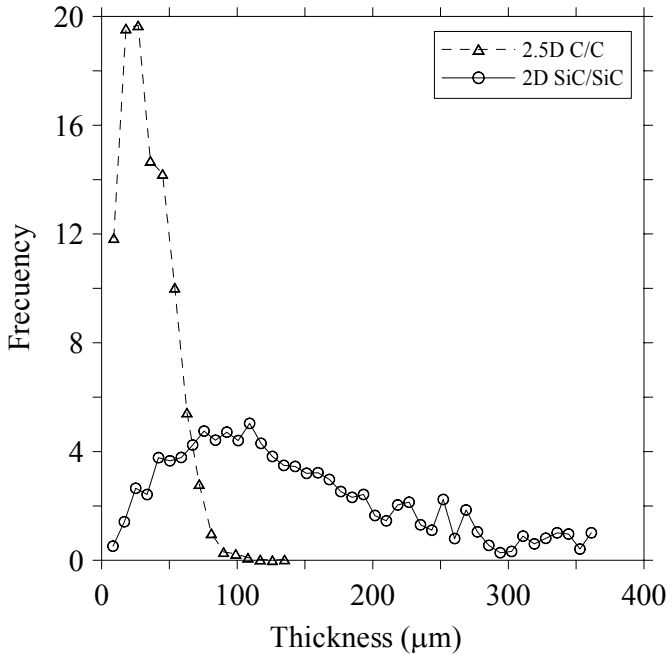

Figure 5: Thickness histograms of the open porosity measured on the two composites studied.

The histograms represent the proportion of the open pore exhibiting a given thickness. The SiC_f/SiC thickness distribution sweeps a wider size range. The average thickness values (and RSD) of the connected porosity were 34 μm (54%) and 139 μm (60%) in C_f/C and SiC_f/SiC composites, respectively.

5. CONCLUSIONS

- X-ray tomography images of two different long-fibre-reinforced ceramic matrix composites were collected. This technique provides 3D images of these heterogeneous microstructures in a non-contact, non-destructive and quick way.
- The volume porosity, the open porosity fraction, the thickness distribution of the connected pore's network and the tortuosity are interesting parameters that can be measured from tomographic images.

- The average values of these measurements have been compared for the different fibers stacks, showing that the porosity distribution along the three main directions selected, the size distribution of the connected pore and its tortuosity are adequate parameters to characterize the differences between the selected composites with different fiber-tissue preforms. Tomography analysis is very useful to measure these complex shapes.

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