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1 Microstructure reconstruction of fibrous C/C 2 composites from X-ray microtomography

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7 Abstract

8 The precise characterization of the microstructure of fibrous composites is essential
9 for an accurate determination of their properties and behaviour. However, fabrica-
10 tion processes usually introduce serious deviations from simple ideal spatial arrange-
11 ments of preform fibres and matrix. The characterization of composites has been
12 performed using destructive and non destructive testing techniques, many of them
13 based on image analysis. Carbon fibres in carbon matrix composites, however, pose
14 a difficult challenge to image analysis systems due to the poor contrast between
15 the different phases. We describe a procedure for the reconstruction of the true
16 microstructure of carbon/carbon composites using phase contrast X-ray microto-
17 mography, show some results, and discuss its performance.

18 *Key words:* Carbon/carbon composites, Chemical vapor infiltration, Image
19 analysis, Microstructure

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20 **1 Introduction**

21 The special properties of fibre reinforced composites responsible for their suc-
22 cess are based on their inherent anisotropy, a characteristic dictated by the spa-
23 tial arrangement of the constituent phases. Indeed, the microscopic arrange-
24 ment of the complex fibrous structure and matrix plays a major role in the
25 determination of the macroscopic properties of the material, such as their elas-
26 ticity, strength, permeability, or thermal conductivity. As such, there is a whole
27 range of numerical methods designed to estimate bulk properties assuming the
28 characteristics of the phases and their spatial arrangement are known, such as
29 those based on Finite Elements Models. Among the structural characteristics
30 that influence the properties of the material are those regarding the fibres,
31 such as the distribution of lengths and orientations, waviness, curvature, and
32 volume fraction, and similar quantities for the matrix and void phases. Man-
33 ufacturing techniques are fine tuned to control these characteristics, but the
34 tuning process usually requires repeated cycles of fabrication and comparison
35 with characterized samples. The recent advances in computer aided modelling
36 and simulation have not eliminated the necessity of experimental work, but
37 rather have increased the demand of accurate experimental data for training,
38 testing, and validation, once the early models based on ideal, regular, or uni-
39 form structures have been superseded. Moreover, no matter how much fine
40 tuning, there is no such a thing as the perfect manufacturing process and,
41 therefore, different factors in the fabrication chain introduce in the real mi-
42 crostructure of the product deviations from the theoretical design which affect
43 the predicted performance of the material.

44 Costly, limited, error-prone manual methods [1, 2] for the characterization

45 of samples of fibrous composites have been outdated by automatic methods,
46 such as those in [3–20], using modern imaging systems [21] and digital image
47 processing [22]. Most of them are based on optical microscopy, and therefore
48 limited to the study of a 2D cross-section, serial sectioning, or, at most, limited-
49 depth volumes of translucent materials using optical confocal microscopy [23].
50 However, only some aspects of the 3D microstructure can be reconstructed
51 from a 2D section, such as fiber orientation [20], subject to a series of assump-
52 tions [24]. Producing and analyzing series of close, thin parallel slices of the
53 samples [25], such as in [7, 9, 11, 19], is labour intensive and adds the problem
54 of matching among slices.

55 X-ray microtomography [26, 27] is a non destructive technique that produces
56 accurate images of 3D volumes by reconstruction from multiple X-ray pro-
57 jections [28], allowing the direct characterization of the 3D microstructure of
58 samples with significant thickness. The huge amount of data produced by this
59 kind of imaging system for large enough samples, even at moderate resolu-
60 tions, precludes any manual processing such as fibre identification. However,
61 2D image processing techniques usually do not have a direct translation into
62 the 3D domain, and processing a 3D microtomograph just as a series of 2D
63 sections is clearly a waste of valuable information. Therefore, specific 3D tech-
64 niques are needed [29, 30], but the combined effect of the increased amount of
65 data to process and the added dimension (most processing algorithms scale
66 geometrically with the dimension of the dataset) calls for extreme care in the
67 selection of the methods and their optimization.

68 Carbon/carbon (C/C) fibre reinforced composites [31] are made of carbon fi-
69 bres embedded in a continuous matrix of carbon. The fact of both matrix
70 and fibres being fabricated from carbon produces a unique combination of

71 properties, including very low thermal expansion coefficients and high ther-
72 mal conductivity, retaining their mechanical properties at high temperatures
73 ($> 2000^{\circ}\text{C}$ in nonoxidizing atmospheres), high specific strength, excellent re-
74 sistance to abrasion and ablation, high resistance to thermal shock, very high
75 elastic modulus, low density, high electrical conductivity, low hygroscopicity,
76 nonbrittle failure, low biological rejection, resistance to chemical corrosion,
77 and reasonable machinability [32].

78 However, C/C composites pose a significant challenge to X-ray imaging sys-
79 tems due to the close densities of inclusions and matrix, yielding very poor
80 contrast among the constituent phases. Sensitivity can be increased for com-
81 posites made up of materials with neighbouring densities such as C/C compos-
82 ites by using coherent X-rays (e.g. from a synchrotron source) and varying the
83 distance from sample to detector [27]. Yet this technique, called holotomogra-
84 phy, applies only to very small C/C samples for which all fibres show the same
85 orientation. Indeed, in larger samples, or in samples where fibres perpendic-
86 ular to the tomography occur, these fibres cause very high cumulated phase
87 lag values which saturate the detector. The resulting image is blurred, render-
88 ing the separation of fibres and matrix impossible [33]. The alternative, also
89 requiring a coherent X-ray source, is phase contrast tomography, also called
90 edge-detection mode CMT, where interfaces between constituent phases are
91 detected through the interference patterns arising from the out of phase waves
92 propagated through materials with different refraction indices [34].

93 Previous attempts have been made to characterise the microstructure of a
94 C/C composite using X-ray microtomography [18, 34]. However, the carbon
95 matrix proved to be very difficult to separate from the carbon fibres, and only
96 the separation of the porosity from the solid phases was achieved. Later, the

97 method was successfully applied to the separation of the three phases in an
98 Al-SiC_p composite [35, 36]. On the other hand, the X-ray microtomography
99 segmentation procedures in [29, 30] deal with easily separable phases, such as
100 glass fibres in a polypropylene matrix, therefore the constituents are clearly
101 resolved in the microtomographs and the segmentation phase is straightfor-
102 wardly addressed by simple thresholding, ineffective in the case of a C/C
103 composite synchrotron X-ray microtomography.

104 In the following, we will show a procedure for the reconstruction of the 3D
105 microstructure of a C/C fibrous composite from a phase contrast X-ray mi-
106 crotomography, based on advanced image processing techniques, allowing the
107 separation of fibres, porosity, and matrix, and the subsequent analysis, char-
108 acterization, and even visualization by means of enhanced reality techniques
109 as an aid to design and analysis. Section 2 describes a sample we use as study
110 case to illustrate the method, described in section 3. The description is ac-
111 companied by some results obtained with the study sample. Finally, section 4
112 is a brief discussion and section 5 closes with a short summary.

113 **2 A C/C fibre reinforced composite microtomographic image**

114 The image in Figure 1 was obtained at the European Synchrotron Radia-
115 tion Facility (ESRF) ID19 High Resolution Diffraction Topography Beamline,
116 dedicated to radiography, microtomography, and diffraction imaging experi-
117 ments [27, 34]. The X-ray beams produced at third generation synchrotron
118 radiation facilities such as the ESRF at Grenoble (France) have a high de-
119 gree of coherence. This results from the small source size, σ , about 50 μm ,
120 and the large distance from source to sample, L , in the 100 m range, so that

121 the transverse coherence length, $dc = \lambda L/2\sigma$, is in the $100 \mu\text{m}$ range. Phase
122 changes occur at the edges of a particle or porosity embedded in a matrix
123 having a different index of refraction, and out of phase transmitted radiations
124 produce an interference fringe marking the location of the interface. The im-
125 age shown was obtained using phase contrast with fixed distance from sample
126 to detector [34].

127 The sample comes from the French aeronautics industrial company Snecma
128 Propulsion Solide (SPS). SPS has decades of experience in isothermal chemical
129 vapour infiltration (CVI) [32,37,38] for the development of proprietary C/C,
130 C/SiC, and SiC/SiC composites for industries as varied as heat treatment,
131 silicon, electronics, glass, nuclear power, and space. Figure 1 shows a $200 \times$
132 200×200 portion of a synchrotron microtomograph of a sample of one of SPS's
133 C/C composites at an incomplete densification stage (sample "CC2" in [34]).
134 Figure 2 shows a 200×200 slice of the microtomograph in Figure 1.



Fig. 1. A synchrotron microtomograph of a 0.0033 mm^3 (side length $< 0.15 \text{ mm}$) cubic sample of a C/C composite. Voxel size is $0.745 \mu\text{m}$.

135 Given that we are dealing with a C/C composite, there is no doubt that the

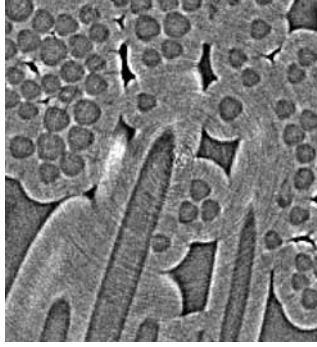


Fig. 2. A slice (0.0222 mm^2) of the microtomograph in Figure 1.

136 image in Figure 1 provides a very good discrimination between fibres and
137 matrix to the human observer. The porosity typical of the CVI densification
138 process [39–41] can also be clearly seen in the image. However, Figures 1 and
139 2 also show a high level of noise (greylevel variations not due to differences in
140 the material but to added noise, such that the same greylevels appear mixed
141 in different phases), poor use of the dynamic range (few different meaningful
142 greylevels, see below), and moderate resolution. Resolution is expected to
143 improve in the near future. X-ray detection is performed through film or visible
144 light scintillators, and, therefore, the system is diffraction limited. X-ray lenses
145 to magnify the image before the detector, such as Kirkpatrick-Baez focusing
146 devices, may overcome this limitation. Nevertheless, resolution is also limited
147 by data bandwidth and storage capabilities.

148 However, limited resolution is not the worst characteristic of the image. Noise
149 levels and poor quantization are. In Figure 2, apart from random noise, rings of
150 varying intensity can be clearly seen around the tomography axis, an artefact
151 due to variations in the efficiency of neighbouring pixels in the detector. Figure
152 3 shows the histogram of the slice in Figure 2. Out of 256 possible levels in the
153 8 bit quantization, only 53 have a pixel count different from zero. Moreover, 51
154 of the 53 levels are grouped in 25 groups, thus reducing the effective dynamic

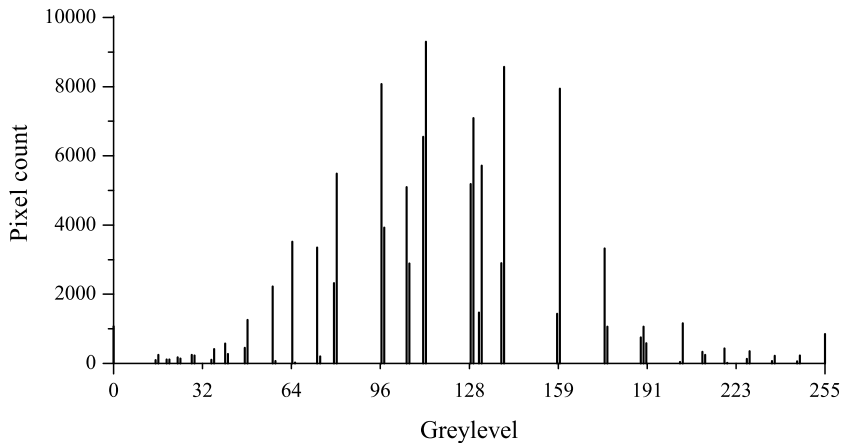


Fig. 3. Histogram of the image in Figure 2. There are 53 levels out of 256 possible levels in the dynamic range, grouped in 27 groups, thus in practice reducing the effective dynamic range to 27 levels.

155 range to 27 levels, i.e. the equivalent to less than 5 bit quantization.

156 The effect of the noise and the reduced dynamic range can be seen in the
 157 magnified details in Figure 4. Close scrutiny reveals that, in spite of what could
 158 be assumed from Figure 1, greyscale based per-pixel segmentation of fibres
 159 from matrix by simple thresholding is not possible. The same grey levels are
 160 found in all phases of the material. The human visual system is able to segment
 161 the image because it performs high level processing involving edge detection
 162 and pattern matching with circular/elliptical primitives [42–44] corresponding
 163 to the expected fibre cross-sections (see the small windows at the lower right
 164 corners). Note for instance the effect that just a sparse couple of brighter pixels
 165 have on the recognition of the fibre in the lower right corner of the upper part
 166 of Figure 4.

167 Within this context, our aim is to use image processing techniques for the
 168 automated extraction of fibres and porosity from the matrix, to allow the
 169 detailed characterization of the material.

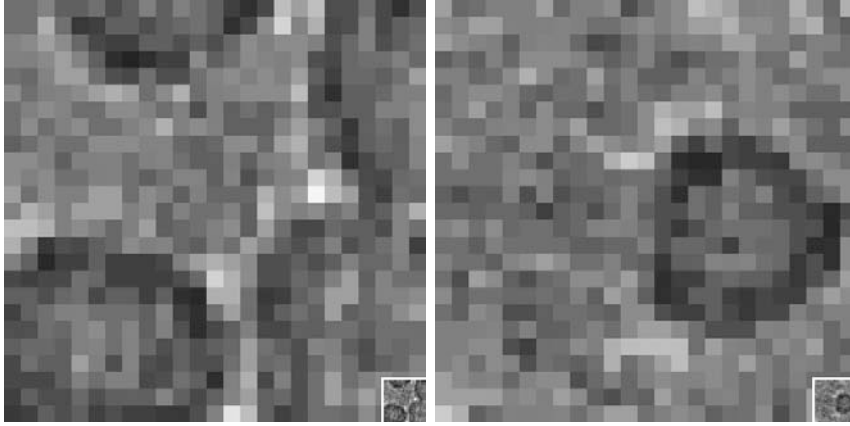


Fig. 4. Details of fibres/matrix in the image in Figure 2. The small windows at the lower right corners show the entire detail at normal scale.

170 **3 Method**

171 *3.1 Preprocessing*

172 As we have just seen, the grey levels of individual voxels are not enough to
173 distinguish fibre voxels from matrix voxels. It is the edges —contrast among
174 adjacent regions in the image— that determine the boundaries of the fibres.
175 Fibres are restituted by our visual system from the edge information. Thus, we
176 need the edges to define the fibres in the image. We have to reduce the noise
177 level, but any noise filtering must respect the edges. Otherwise, we would be
178 losing the information we are seeking, the basis of the segmentation procedure.
179 The noise has a high spatial frequency, but low pass filtering would also blur
180 the edges. Median filtering could preserve to some extent the edges, but the
181 resolution is too poor: The edges are too narrow as to not be wiped away
182 by any but the smallest of filters, which would be useless. A noise reduction
183 method capable of efficiently removing high frequency noise while preserving
184 edges is anisotropic diffusion [45, 46].

185 Perona and Malik [45] noted that the convolution of an image $I_0(x, y)$ with a
 186 Gaussian kernel,

$$187 \quad K_\sigma(x, y) = \frac{1}{2\pi\sigma^2} \exp\left(-\frac{|x|^2 + |y|^2}{2\sigma^2}\right) \quad (1)$$

188 with standard deviation σ yields the same result as the solution of the isotropic
 189 diffusion (heat) partial differential equation,

$$190 \quad \frac{\partial I(x, y, t)}{\partial t} = \text{div}(\nabla I(x, y, t)), \quad (2)$$

191 where $I(x, y, t)$ is the image $I(x, y)$ at time $t = 0.5\sigma^2$, with initial conditions
 192 $I(x, y, 0) = I_0(x, y)$, and ∇I is the image gradient.

193 Introducing in (2) as *diffusion conductance* or *diffusivity*, $g(s)$, a rapidly de-
 194 creasing function of a greyscale edge detector such as the gradient magnitude,
 195 $s = |\nabla I|$,

$$196 \quad \frac{\partial I(x, y, t)}{\partial t} = \text{div}(g(|\nabla I(x, y, t)|)\nabla I(x, y, t)), \quad (3)$$

197 smoothing on both sides of edges becomes much stronger than across them. A
 198 diffusivity constant with time but varying with location (x, y) would make (2)
 199 a linear nonhomogeneous diffusion equation. However, if g is made a function
 200 of time, as in (3), the diffusion equation becomes nonlinear and nonhomo-
 201 geneous, referred to as *anisotropic* in the image processing literature, even
 202 when conventional PDE terminology reserves the term for the case where the
 203 diffusivity is a tensor, varying both with location and direction. The conse-
 204 quence of this “pseudoanisotropy” is that only the magnitude but not the
 205 direction of the diffusion flux can be controlled. Noise close to edges remains
 206 unchanged due to the small flux in the vicinity of edges. To enable smoothing

207 parallel to edges, (3) must be generalized with a diffusivity matrix G with
 208 nonzero off diagonal elements (see, for instance, [46–50]), thus rendering it
 209 truly anisotropic.

210 Perona and Malik suggested

$$211 \quad g(s) = \frac{1}{1 + s^2/\kappa^2} \quad (4)$$

212 and

$$213 \quad g(s) = \exp(-s^2/\kappa^2) \quad (5)$$

214 as diffusivity functions. Since then, many other diffusivity functions have been
 215 proposed [46, 51]. In fact, Black *et al.* [51] demonstrated that anisotropic dif-
 216 fusion in the sense of (3) is the gradient descent of an estimation problem
 217 with a robust error norm induced by $g(s)$, thus providing a sound theoretical
 218 foundation to choose adequate diffusivity functions.

219 As several authors have revealed [52–55], (3) is an ill-posed problem, in the
 220 sense that images close to each other are likely to diverge during the process [54],
 221 but it can be stabilized by regularization. One common approach [53] is to
 222 smooth the variable of the diffusivity, i.e. to use a smoothed version of the
 223 image for the gradient in each step, as in

$$224 \quad \frac{\partial I(x, y, t)}{\partial t} = \operatorname{div}(g(|\nabla I_\sigma(x, y, t)|)\nabla I(x, y, t)), \quad (6)$$

225 where $I_\sigma = K_\sigma * I$, with K_σ a suitable local convolution kernel of width σ , for
 226 instance a Gaussian kernel. However, Weickert and Benhamouda [56] proved
 227 that a standard spatial finite difference discretization is sufficient to turn (3)
 228 into a well posed system of nonlinear ordinary differential equations. Therefore,

229 direct implementations of the Perona-Malik filter tend to work reasonably well
 230 because of the regularizing effect of the discretization.

231 The extension to D dimensions is straightforward. The diffusion process is
 232 described by the equation

$$233 \quad \frac{\partial I(\mathbf{x}, t)}{\partial t} = \operatorname{div}(G \cdot \nabla I(\mathbf{x}, t)), \quad (7)$$

234 where $\mathbf{x} \in \mathbb{R}^D$ and G is a square $D \times D$ diffusivity matrix. Equation (3) is (7)
 235 with G a diagonal 2×2 matrix with equal diagonal elements $g(|\nabla I|)$. Diffusion
 236 filtering is usually implemented using simple finite differences to approximate
 237 the image derivatives within an explicit or Euler-forward scheme.

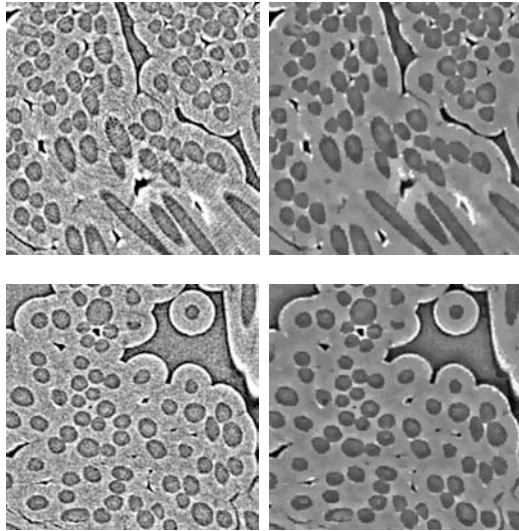


Fig. 5. Two orthogonal slices of the tomograph in Figure 1 before (left) and after (right) filtering with 3D anisotropic diffusion.

238 Figure 5 shows the results of applying (7) with (4) and $D = 3$ to the tomograph
 239 in Figure 1, and they are quite satisfactory. The relevant edges have been
 240 preserved while most of the noise has been wiped away. Note that the filter is
 241 equally efficient on the two types of noise present in the image, the random
 242 uniform noise and the circular patterns around the tomography axis.

244 However good the results in Figure 5 regarding noise reduction and mesoscale
 245 feature preservation, fibres cannot yet be separated on the basis of individual
 246 voxel grey levels. Many fibres show brighter nuclei inside darker boundaries,
 247 whose grey levels can also be encountered in the matrix. Our approach is
 248 masking all pixels within edges. For that purpose we use a differential profiler
 249 [57] along voxel rows. A differential profile is obtained by constructing an
 250 array of integers where every element represents the lowpass filtered gradient
 251 of the grey levels along a scan line. This is easily implemented using exclusively
 252 integer arithmetic. Let $I_{x,y,z}$ be the greyscale image. Let P_l be an integer vector
 253 to hold the profile of a scan line, and assume without loss of generality that
 254 the scan direction is along $+x$. Combining forward differencing

$$255 \quad P_l = I_{l,y,z} - I_{l+1,y,z} \quad (8)$$

256 with lowpass filtering

$$257 \quad P_l = \frac{1}{3}(P_{l-1} + P_l + P_{l+1}), \quad (9)$$

258 and forgetting about the dividing constant, we get

$$\begin{aligned} P_l &= I_{l-1,y,z} - I_{l,y,z} + I_{l,y,z} \\ &\quad - I_{l+1,y,z} + I_{l+1,y,z} - I_{l+2,y,z} \\ &= I_{l-1,y,z} - I_{l+2,y,z} \end{aligned} \quad (10)$$

259 and thus the whole procedure to obtain the profile is reduced to a sequence
 260 of integer subtractions of grey levels. Near-zero profile segments reflect parts
 261 of the image where no significant variations of grey level occur. Positive peaks
 262 reflect decreasing grey levels, such as when entering a fibre, and negative peaks

263 increasing bright, such as when leaving a fibre. The narrower the peak, the
 264 faster the variation. The higher the peak, the greater the variation. Thus,
 265 peak shape and magnitude tell us everything we need to know about intensity
 266 transitions along a given direction.

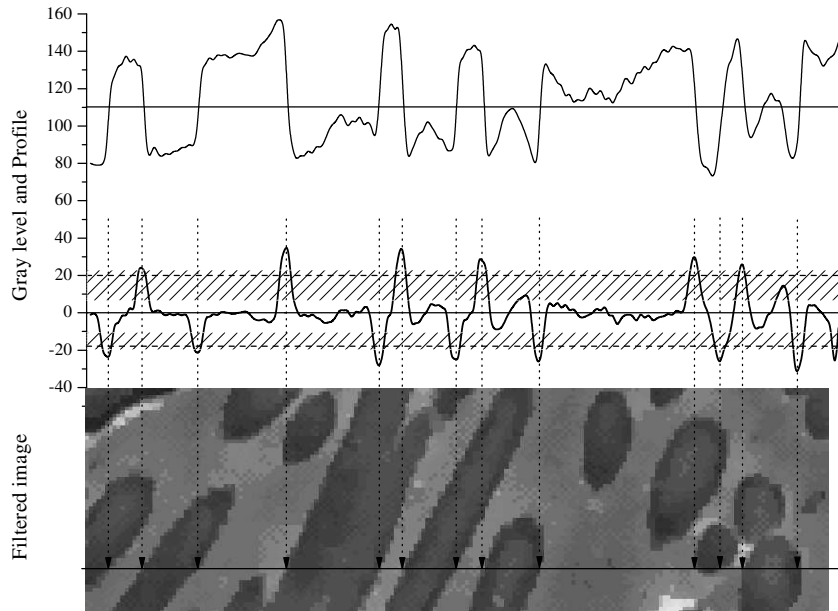


Fig. 6. Edge detection by profiling scan lines in slices of the tomograph in Figure 1 after filtering with 3D anisotropic diffusion. The patterned areas in the profile graph show the range of possible thresholds for successful detection of all fibres in the given line.

267 Figure 6 shows a grey level profile (above) and the corresponding differential
 268 profile (center) along a line of the tomograph in an arbitrary direction (below).
 269 A tentative grey level threshold is plotted on the grey level profile (upper
 270 graph), to show the lack of robustness of a per-pixel approach due to the
 271 brighter nuclei of some fibres. The vertical arrows show the edges in the image
 272 corresponding to the peaks above or below tuneable thresholds (dashed lines)
 273 in the differential profile (lower graph). The range of possible thresholds that
 274 would achieve successful detection of all fibre edges in the line is highlighted

275 in the graph. The upper (in absolute value sense) bounds for the thresholds
276 are dictated by two criteria: 1) The positive threshold should be low enough
277 as to not miss any positive peak at the entrance of a fibre, and 2) the negative
278 threshold should not miss any negative peak at the exit of a fibre. The lower
279 bound for the positive threshold depends only on the peaks outside fibres
280 (positive peaks inside fibres are irrelevant): the threshold should not allow
281 any peak not lying at the entrance of a fibre. Conversely, the lower bound for
282 the negative threshold depends only on the peaks inside fibres (negative peaks
283 outside fibres are irrelevant): the negative threshold should be high enough as
284 to exclude any negative peak not at the exit of a fibre.

285 To obtain a mask of fibre voxels, therefore, the voxel rows along the three
286 possible orientations in the 3D image are profiled, such that all voxels along
287 a differential profile from a positive peak to a negative peak are marked as
288 foreground voxels, and the rest as background voxels. In the resulting mask,
289 some fibres are “broken”, i.e. the dark boundary does not surround the entire
290 fibre, and therefore some profiles may result in spurious rectilinear spikes pro-
291 truding from the fibre mask, due to the lack of a negative peak, or well similar
292 rectilinear structures may be missing from the interior of a fibre due to the
293 lack of a positive peak, or a spurious negative peak with sufficient magnitude
294 inside a fibre.

295 Here is where spatial correlation among the different orientations in the 3D
296 image enters, as interior fibre voxels not marked in a profile in a given direction
297 are likely to be marked in the profiles along any of the transversal directions,
298 and thus the *a priori* knowledge of the morphology of the fibres is enough, in a
299 simple postprocessing stage, to get rid of the majority of the thin, rectilinear,
300 spike-like 2D artifacts protruding from or entering “broken” fibres.

301 Porosity in the matrix is segmented by the same procedure, only the thresholds
302 for the peak detection in the differential profile are tuned to the characteristic
303 edges of the porosity regions, where transitions are more pronounced than in
304 fibres, see the darker lacunar areas with a brighter rim in Figure 5. This is
305 due to the contrast saturation effect in the matrix/air interface induced by
306 the increased sensitivity of the tomograph in order to resolve carbon/carbon
307 interfaces.

308 Both the fibre and the porosity masks can be “cleaned” with a labelling al-
309 gorithm if their inspection reveals it to be necessary. Cleaning the masks con-
310 sists in getting rid of remnants of matrix material that may be adhered to the
311 outside face of fibre or porosity boundaries due to imprecisions in the exact
312 localization of edges. This is achieved by 1) labelling all connected foreground
313 voxels in the mask (fibres or porosity) with a unique label subject to the
314 condition of their original grey levels being below a given threshold, and 2) la-
315 belling as background voxels (i.e. delete from the mask) all connected groups
316 of remaining unlabelled voxels in the mask if any voxel in the group touches
317 a background voxel (matrix). This ensures that the “bright voxel hunt” takes
318 effect only in groups of voxels attached to the exterior boundary of fibres or
319 porosity, without affecting the bright cores of fibres. The result is a purified
320 mask of fibre or porosity voxels where bright voxels are only permitted in the
321 interior of fibres.

322 The masks thus obtained, see Figure 7 and Figure 8 for some partial render-
323 ings of fibre bundles and porosity, can be combined into a unique material
324 description mask, detailing the microstructure of the material. All voxels in
325 the tomograph are classified as belonging to one of the three phases in the
326 material: matrix, fibre, or porosity. This mask can then be directly fed into

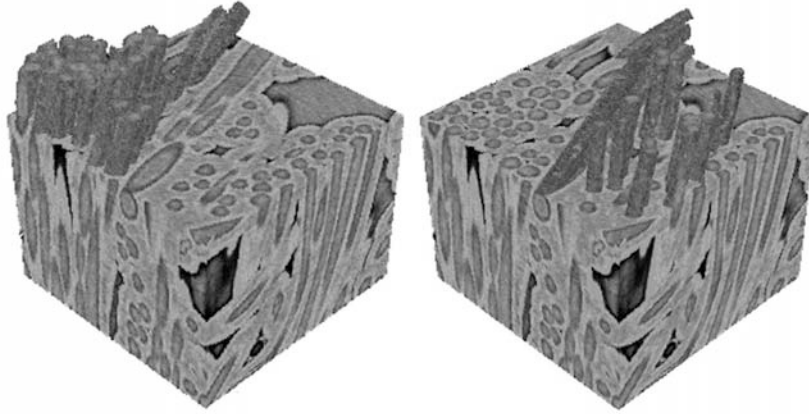


Fig. 7. Partial renderings of some fibre bundles, using the original grey levels in the tomograph.

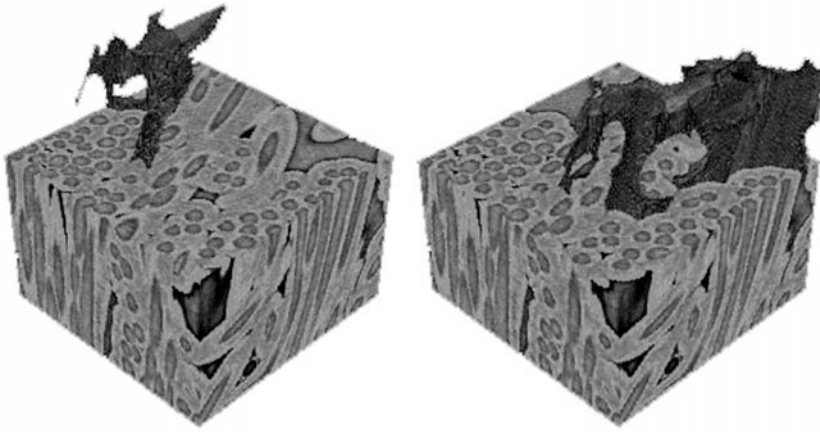


Fig. 8. Partial renderings of the porosity formed during the CVI process, using the original grey levels in the tomograph.

327 models requiring a precise description of the microstructure of the material.

328 Thus, for instance, a Finite Element Model can be constructed just by assim-

329 ilating the finite elements to the classified voxels.

330 3.3 Fibre individuation: The heavy ball

331 The microstructure of the sample is contained in the mask, indeed, but still

332 some further processing is necessary in order to be able to perform a com-

333 plete characterization of the microstructure. Some usual characteristics, such
334 as void content or volume fractions, can be directly computed from the phase
335 masks. However, others not less important such as the distribution of fibre
336 lengths, orientations, curvature, or waviness, cannot. Fibres have to be identi-
337 fied individually in order to be able to measure individual characteristics such
338 as those just mentioned.

339 A connected component labelling algorithm could do it, if we could warrant
340 that individual fibres do not touch each other in the mask. However, this is not
341 the case. Fibres touch each other both because of the limited resolution of the
342 image and because some of them actually touch each other in the material.
343 Therefore a connected component algorithm is able only to separate fibre
344 bunches, groups of fibres either touching each other or close enough as to not
345 be resolved by the spatial resolution of the imaging system, see Figure 7.

346 We use a novel method, the *heavy ball*, to successfully separate individual
347 fibres. It treats the fibre mask as a solid block drilled by wormholes, the
348 fibres. The hole mouths are located in each side of the block, and a zero drag,
349 zero gravity, high inertial ball is pushed into each hole in turn. The ball size is
350 adapted to the diameter of each hole. The ball is forced to move continuously
351 until it either finds the end of the fibre or leaves the block. The ball run along
352 each fibre can be used to label the fibre with a unique identifier for subsequent
353 characterization, or directly to compute the required parameters.

354 For locating the ball entrance points and estimating the required ball size,
355 the ellipse fitting procedure depicted in [20, 58], see [59], is applied to each
356 outer boundary (six 2D binary images) of the binary fibre mask. The method
357 requires the labelling of the connected components in each boundary. During

358 the labelling, the centroid and the covariance matrix of the coordinates of the
 359 pixels in each connected component is computed. The centroid (average loca-
 360 tion) gives the center of the fitted ellipse, and the root of the lesser eigenvalue
 361 of the covariance matrix Σ gives its minor axis, which coincides with the fibre
 362 radius, r :

$$363 \quad r^2 = 2(\Sigma_{11} + \Sigma_{22} - \sqrt{\Delta}), \quad (11)$$

364 where

$$365 \quad \Delta = (\Sigma_{11} + \Sigma_{22})^2 - 4(\Sigma_{11}\Sigma_{22} - \Sigma_{12}\Sigma_{21}). \quad (12)$$

366 Touching fibres in the 2D image have to be split before the ellipse fitting
 367 procedure. Otherwise a single enclosing ellipse will be fitted to an entire group
 368 of touching fibres in the 2D image. The splitting of touching fibres can be
 369 performed by means of mathematical morphology operators, using successive
 370 erosions and dilations, such that fibres are shrunk until disconnection and
 371 then expanded again constrained to the original image. Disconnecting touching
 372 groups of pixels is a well known application of mathematical morphology [22].

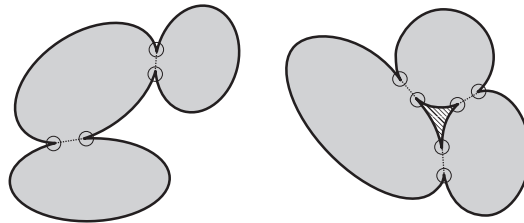


Fig. 9. Ellipse splitting procedure: left) Simple case; right) Complex case not consid-
 ered in [58]. Small circles mark convex perimeter points. Dotted lines mark splitting
 lines. The greyed area within the group on the right is enclosed by an internal
 perimeter.

373 An alternative approach is described in [58]. The perimeters —foreground

374 pixels with at least one background neighbour— of the connected components
375 in the 2D images are sequentially traversed and their curvature computed at
376 each perimeter pixel. Pixels where the curvature becomes convex are recorded.
377 If two fibres touch each other, there will be two convex points. A straight line
378 joining the points can be drawn to split the fibres, so they can be labelled
379 as two different components. If three or more fibres touch, it could be just
380 a trivial extension of the two-fibre case, and then a line is drawn for each
381 consecutive pair of convex points, see Figure 9 (left). However, it could also
382 be a special case, not considered in [58], where three or more fibres touch
383 each other, thus totally enclosing a background region inside the group, see
384 Figure 9 (right). In this case, an odd number of convex points is found when
385 traversing the external perimeter of the group. If this is the case, the internal
386 perimeter (the perimeter of the enclosed background) has to be traced also,
387 and the splitting lines should be drawn between pairs formed by a convex
388 point from the external perimeter and the closest convex point in the internal
389 perimeter, see Figure 9 (right). As many convex points have to be found in
390 the internal perimeter as in the external perimeter. If they are not, another
391 background area is enclosed within the group, and so on.

392 Therefore, if the aggregation pattern of the fibres in a given sample is not
393 too complex, the convex point search is probably faster than mathematical
394 morphology. Otherwise, mathematical morphology is probably a safer choice.
395 No matter what method is used, splitting the touching fibres is a fast opera-
396 tion. Note that it is only performed on the external sides of the 3D domain
397 of the fibre mask, \mathcal{M}_f , six 2D binary images. Moreover, the purpose of ellipse
398 fitting is locating entrance points for the heavy ball, and this is performed
399 sequentially. Fibres traversed from the first side are likely to emerge on any

400 of the sides. Fibres are marked as they are traversed, to avoid repeated runs.
 401 Thus, as the process progresses through the sides, each side has less and less
 402 ellipses left to be fit.

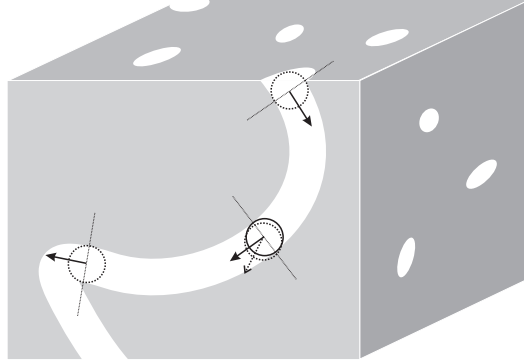


Fig. 10. Identification of individual fibres with the heavy ball method. Solid arrows show the direction of the previous move. The dotted arrow and circle represent a candidate movement. The thinner dotted line marks the limit for candidate moves depending on the previous move. Ellipses in the mask surface are best fits to the fibre sections and determine the radius and initial location of the ball.

403 Once the center and radius of the “wormhole” have been estimated, a heavy
 404 ball of the appropriate size —same radius as the fibre, i.e. minor axis of the
 405 fitted ellipse, r , equation 11— is placed centred in the hole mouth. Figure 10
 406 illustrates the method. The ball has inertia, i.e. at step k it remembers the
 407 previous move, vector \mathbf{m}^k . This memory is initialized opposite to the normal of
 408 the current mask boundary, $\mathbf{m}^0 = -\hat{\mathbf{n}}$. The ball moves looking for the widest
 409 passage, one voxel at a time. The set of candidate locations for the centre of
 410 the ball, \mathcal{C}^k , is made of all the voxels C_i adjacent to the current centre C^k
 411 such that the direction of advance is at most at right angles with the previous
 412 movement. This is verified by means of the scalar product of the vector of the

413 previous move and the vector linking the current centre and the candidates:

$$414 \quad \mathcal{C}^k = \left\{ C_i \in \mathcal{M}_f \mid \|\overline{C^k C_i}\|_0 = 1 \wedge \overline{C^k C_i} \cdot \mathbf{m}^k \geq 0 \right\} \quad (13)$$

415 For each step, the number $s(C_i)$ of fibre voxels overlapped by the ball in each
416 candidate location C_i is computed:

$$417 \quad \mathcal{O}_i^k = \left\{ P \in \mathcal{M}_f \mid f(P) = 1 \wedge \|\overline{C_i P}\|_2^2 \leq r^2 \right\} \quad (14)$$

$$418 \quad s(C_i) = I(\mathcal{O}_i^k) \quad (15)$$

419 where $f : \mathcal{M}_f \subset \mathbb{Z}^3 \rightarrow \{0, 1\}$ is the binary fibre mask.

420 If the maximum overlap is verified for more than one candidate location, the
421 one closest to the previous direction is chosen, i.e. the one maximizing the
422 scalar product of the intended and the previous movement:

$$423 \quad C^{k+1} = \arg \max_{C_i \in \mathcal{C}^k} \left\{ s(C_i) \wedge \overline{C^k C_i} \cdot \mathbf{m}^k \right\} \quad (16)$$

424 This step by step advance is repeated until the centre of the ball leaves the
425 fibre, either because the fibre ended inside the mask, or because the ball is
426 leaving the mask, $C^k \notin \mathcal{M}_f$.

427 During the advance of the ball the centre locations $\{C^k\}$ can be recorded
428 —for instance for a curvature analysis—, the length of the moves accumu-
429 lated, $\sum_k \|\mathbf{m}^k\|_2$, —to estimate the length of the fibre—, the fibre voxels $\cup \mathcal{O}^k$
430 labelled with a unique identifier —for further characterization in a later occa-
431 sion or involving several fibres, such as nearest neighbour and average distance
432 computations—, and appropriate statistics can be computed, such as extreme
433 locations of the fibre centres $\{C^k\}$, or variations in the fiber width or size

434 by inspection of the cross sections at right angles with the current movement
435 vector, \mathbf{m}^k .

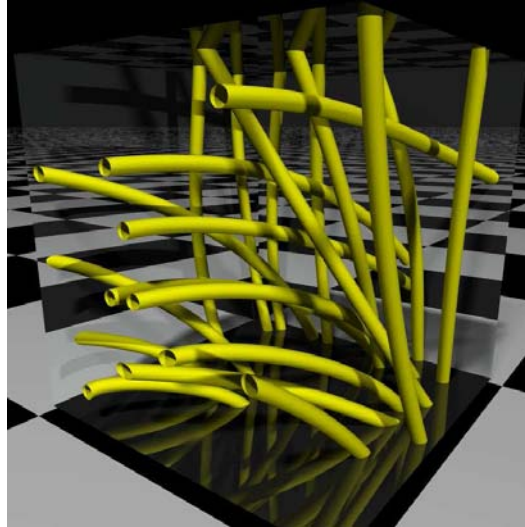


Fig. 11. Rendering of some of the fibres in the sample in Figure 1 after separation with the heavy ball. Only some fibres are shown to avoid visual clutter.

436 Figure 11 shows some isolated fibres in the sample in Figure 1, where the
437 output of the heavy ball algorithm has been used to produce a ray tracing
438 rendering.

439 3.4 Labelling algorithms

440 Several steps in the methodology depicted above use connected components
441 labelling algorithms, both in 2D and 3D.

442 2D labelling is required for the ellipse fitting procedure. The components tar-
443 geted by the algorithm are, if the previous processing was successful, small to
444 medium compact rounded shapes. Here the simple classical recursive labelling
445 algorithm [60, 61], also known as region growing or region burning, is likely
446 to work fast and well. Moreover, the labelling aims at computing moments of

447 each connected component, and this can be easily done during the labelling
448 with the recursive method. The limited size of the components is likely to pre-
449 vent any stack overflow problem arising from its recursive nature, the cause
450 why it is usually discarded in benefit of the two-pass iterative algorithm.

451 In the classical two-pass iterative method [62,63], first the image is traversed
452 in raster scan order assigning temporary labels to the components, and then
453 a second pass resolves conflicts in components with multiple labels, thus re-
454 quiring an equivalence table to record label conflicts, usually handled with
455 a Union-Find data structure [64]. Therefore, in exchange of the robustness
456 implicit in its iterative nature, it is far more complex than the recursive algo-
457 rithm, and characterizing components during the labelling is not straightfor-
458 ward, as components are not labelled sequentially and labels are not resolved
459 until the second pass.

460 Recently a hybrid algorithm has been proposed [65,66], that has proven to
461 be faster and more robust for compact objects than the classical recursive
462 algorithm, whereas maintaining its simplicity and versatility. The 2D labelling
463 in the ellipse fitting procedure is therefore a good instance for adoption of the
464 hybrid technique.

465 3D labelling is used to purify the phase mask. Labelling in 3D, however, can
466 be trickier. The components are not moderately sized anymore. We are dealing
467 now with three dimensions, and the recursion depth and the required stack
468 grow accordingly. Thus serious doubts arise about the robustness of the algo-
469 rithms relying on recursion. The classical recursive algorithm can be rewritten
470 in iterative form, by using a custom stack. However, this comes at the cost of
471 increased complexity.

	Rec. depth		Time (ms)	
Recursive	281 607		146.9	+62%
Iterative (Stack)	252 341	89.6%	167.3	+84%
Hybrid along x	27 617	9.8%	108.9	+20%
Hybrid along y	26 246	9.3%	99.7	+10%
Hybrid along z	15 481	5.5%	90.9	
Two-pass	N/A	N/A	497.8	$\times 5.5$

Table 1

Performance data in the labelling of the fibre mask with different labelling algorithms.

472 We analyzed the performance and robustness in 3D of the different alternatives
473 in the labelling of the phase mask. Table 1 summarizes the results, including
474 different scan directions for the hybrid algorithm, which is anisotropic, in the
475 sense that it favours a direction of choice. Note the effect of the inherent
476 anisotropy of the fibrous components, even when many fibres in the sample
477 deviate significantly from the main orientation, z , specially towards y , see
478 Figure 11 and axes in Figure 1. All data were obtained on a iPentium M 2.13
479 GHz, 1 GByte RAM, OS MS WindowsXP. Both time and stack figures were
480 clearly favourable to hybrid labelling. A detailed analysis can be found in [66].

481 4 Discussion

482 The study case accompanying the description of the method just described
483 shows its effectivity. It deals with a sample showing all the problems typical of

484 a C/C composite synchrotron tomograph, and demonstrates how the method
485 is able to tackle them and results in a detailed description of the microstructure
486 of the sample, either in the form of a phase mask suitable for numerical models
487 working on explicit spatial domains, or up to the point of a detailed individual
488 characterization of each fibre in the sample. Specifically, the heavy ball is not
489 only able to separate the fibres, but it also produces a set of data permitting a
490 whole range of measurements, from simple statistics, such as fibre length and
491 orientation, to the parameterization of fibres as 3D curves, surfaces, or solids,
492 see Figure 11.

493 With respect to the computational overhead, clearly the heaviest stage is the
494 anisotropic diffusion filtering. Completing the 30 to 40 iterations needed to
495 get results such as those in Figure 5 on the 200^3 sample takes several minutes.
496 However, techniques do exist capable of accelerating significantly anisotropic
497 diffusion [67].

498 The differential profiler is very fast, the whole procedure in all sections along
499 the three directions, including the correlation stage, takes about 2 seconds.

500 With respect to the stages involving labelling algorithms, time figures are pro-
501 vided in the previous section, and a detailed analysis of these techniques can
502 be found in [65,66]. If the right choice is made in the selection of the labelling
503 algorithm, labelling times for moderately sized samples are very small. How-
504 ever, it should be noted that, in spite of the high reduction in stack overhead
505 of the hybrid technique, it still uses recursivity, and this could be a source
506 of trouble as sample sizes grow. For the 3D labelling of big samples, it may
507 be necessary to resort to block techniques, where the sample is labelled by
508 blocks [68], or to use an iterative technique.

509 Regarding the heavy ball, computation time depends on the length and radius
510 of the fibre, but the average fibre, 200 voxel long and 14 voxel in diameter,
511 takes about 60 ms, including dumping the center locations to a text buffer
512 for export. Future implementations of the algorithm can yet be optimized
513 for speed, for instance by avoiding repeated computations in the overlap for
514 candidate moves at each step.

515 With respect to parametric complexity, tuneable parameters are only found
516 in two of the stages, the preprocessing filtering and the differential profiler.
517 Anisotropic filtering uses three parameters, the noise scale σ in (6), the feature
518 scale κ in (4), and the number of iterations. These usually require careful fine
519 tuning when dealing with applications related to enhancement of images with
520 multiple levels of detail and where the visual appearance of the final result
521 is a major factor. However, 1) in our case the noise scale and the feature
522 scale are well defined and well apart, and 2) we use anisotropic diffusion to
523 provide suitable input for the differential profiler, which has a high degree of
524 robustness and is itself tunable.

525 The result is that, as long as the noise is sufficiently eliminated and the
526 mesoscale features of the microstructure are well preserved, the sensitivity to
527 the parameters of the diffusion filter is very low. Differences among parameter
528 configurations can be handled by the tuneable thresholds in the differential
529 profiler. We recommend plotting a couple of profiles anywhere through the
530 sample every, say, ten iterations of the anisotropic diffusion filter, so that the
531 progress in the edge enhancement and noise reduction can be tracked, a suit-
532 able stopping point for the diffusion can be chosen, and adequate thresholds
533 for the profiler can be set.

534 Regarding precision, anisotropic diffusion has the interesting property of pre-
535 serving not only the existence of edges at the feature scale, but also their
536 location. With the differential profiler, on the other hand, it is possible to de-
537 termine with high accuracy the position of edges, and also to choose the outer
538 side of the features (first threshold crossing point for ‘entry’ peaks and second
539 for ‘exit’ peaks), the inner side (vice versa), or exactly the edge (maximum
540 between threshold crossing points), see Figure 6.

541 The only potential source of trouble are brighter cores within the fibres which
542 sometimes may percolate to the outside of the fibre through a broken edge,
543 creating a connection with the matrix material and leaving a gap for the
544 differential profiler. However, correlations among profiles in the three spatial
545 directions decrease their probability, and, moreover, the path of the heavy ball
546 is not likely to be affected by these spots. Nevertheless, in significant numbers
547 they could affect some of the statistics. An inspection of the phase mask will
548 reveal the extent of the problem, if any, and advice about the opportunity
549 of a finer tuning of the profiler thresholds or the anisotropic diffusion before
550 characterization.

551 Note also that the procedure described for the application of the heavy ball
552 would miss any fibre entirely embedded *inside* the sample. If a low but signif-
553 icant probability of short fibres entirely embedded in the sample is expected,
554 the interior of the fibre mask may be scanned after completing the procedure,
555 looking for fibre voxels that were not tracked by the heavy ball, in order to
556 apply it there. Alternatively, if the material is expected to have a large pro-
557 portion of short (with respect to sample size) fibres, the heavy ball can be
558 applied as if peeling an onion: first the heavy ball is applied to all six outer
559 boundaries of the sample, then the procedure is repeated a little deeper into

560 the sample for any fibre section not yet tracked, and so on, until the volume
561 being inspected is too small as to contain any fibres.

562 Finally, note that the diffusion process in the preprocessing stage involves
563 mixing contributions from neighbouring voxels. This implies the existence of
564 border effects near the image boundaries. Edge detection in the profiler, in
565 turn, uses finite differences involving separated voxels, and it is not able to
566 detect edges if they are just at the boundary of the image. Moreover, in the
567 correlation among profiles, the extreme ends of profiles count with much less
568 information than the rest. All this added up results in the outer boundaries of
569 the resulting mask having a high degree of unaccuracy in the determination
570 of phase type. Therefore, it is a good practice to discard a few layers of voxels
571 around the mask before any further processing.

572 **5 Conclusion**

573 We have detailed and illustrated a multistage methodology able to extract and
574 characterize the true microstructure of a fibrous C/C composite using phase
575 contrast X-ray microtomography. Since C/C composites are probably a worst-
576 case benchmark, the method can also be applied to other cylindrical fibre
577 composites. The processing chain is made of 1) a preprocessing stage where
578 3D anisotropic diffusion filtering is used to reduce the noise while preserving
579 the features in the sample; 2) a differential profiling method to detect areas
580 between intensity edges on every 2D cross section parallel to the reference
581 system, which are then refined by correlating the outputs in 3D; 3) an optional
582 “cleaning phase” using a 3D labelling algorithm to purify the resulting mask;
583 and 4) a novel fibre separation process which requires an ellipse fitting procedure

584 applied to the outer boundaries of the 3D mask followed by the use of what
585 we call a heavy ball to individually identify each fibre and characterize it.

586 We hope to have contributed to demonstrate the ability of phase contrast
587 X-ray tomography to produce sufficient information as to extract the true
588 microstructure of composites with constituents with close densities, such as
589 C/C composites, when appropriate image processing techniques are used.

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