Investigations on anisotropic fracture mechanics of graphitic foams

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A B S T R A C T

This work investigates destructive (crush) compressive and shear behaviour of Poco-HTCTM, which is a porous graphitic carbon foam. This material is anisotropic, and compressive measurements were made in both out-of-plane and in-plane directions. A camera filmed the tests to visually study crack formation and growth at macro-scale. Scanning electron microscopy images of fracture surfaces were recorded to examine post-failure material formation at micro- and meso-scales. In another series of tests, cyclic uniaxial compression measurements were performed in the elastic regime to characterise this behavior. Some of the samples were crushed after the cyclic test to measure strength.

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1. Introduction

Porous graphitic carbon foam is an emerging material with a very high thermal conductivity to density ratio, that is approximately seven times higher than that of copper [1]. This raises the possibility to make ultra-light and efficient thermal management systems. Highly-aligned graphitised-carbon base material (with 800–1900 W/m.K thermal conductivity) brings high bulk thermal-conductivity to graphitic foams (135–245 W/m.K), while the porous structure reduces density [2,1,3]. Furthermore the bulk material exhibits very low thermal expansion [3], has low atomic number which makes it relatively transparent to radiation, and has high modulus to density ratio [4] compared to other foams.

Carbon foam is currently being considered in the development of a thermo-mechanical support structure for the upgrade of the inner-tracker of the ATLAS detector at the Large Hadron Collider at CERN, Geneva [5]. The above properties make carbon foam an ideal choice for part of this structure. The carbon foam is used to conduct heat from electronics into a 2 mm diameter titanium tube with evaporative CO2 cooling. The tube is sandwiched between two pieces of foam, and the part is placed between two thin ultra-high-modulus carbon-fiber facings. The detector will sit on the facings in a high radiation area, and must survive more than 10 years without maintenance.

The CO2 coolant will cool the tube to −30 °C. The thermal contraction of the tube will exert forces on the carbon foam. There is a risk that these forces will lead to fractures at the interface, which would result in deterioration in thermal performance. Since preventing mechanical damage is crucial for maintaining thermal properties, there is a particular interest in the fracture mechanics of the foam.

Most research has focussed on thermal performance, rather than mechanical performance of carbon foams [6–8]. The majority of existing studies on mechanical performance are limited to measurements on elastic bulk parameters such as Young’s and shear modulus [9,10]. Chen et al. [11] have measured crush strength resulting from different precursors and manufacturing techniques, but with very little work on understanding graphitic foams under force in detail. One of the most detailed studies on this topic was made by Gowthaman et al. [12,13]. They performed crushing tests on graphitic foams, and presented camera records showing fracture lines and scanning electron microscopy (SEM) records from the fracture surfaces. Since graphitic foams are highly anisotropic, the fracture response is dependent on material direction. Although the work is very useful to describe fracture behavior, the tests were performed in a single material direction and limited to characterisation of fracture in other directions. Consequently, this highlights the need to further investigate fracture mechanics in the anisotropic case. This study extends the understanding of the failure mechanism by presenting measurements made in both material directions, and made with different loading modes.

Destructive compression (crush) and shear tests were performed. The crushing test was conducted in both out-of-plane...
and in-plane direction to understand anisotropic behavior. The tests were recorded with a video camera to visually analyse fracture mechanics at macro-scale. SEM images were captured from the fracture surfaces to examine post-failure material formation at micro- and meso-scales. In another group of measurements, cyclic compressive loads were applied in the elastic regime to characterise the elastic behavior, such as Young’s modulus and elastic limits. Also, the strengths were measured by subjecting some samples to crush after cyclic tests.

### 1.1. Material and micro-structure

Poco–HTC is a graphitised-carbon foam produced from a mesophase–pitch precursor. It is licensed, and manufactured by Poco Graphite Inc [14]. Poco–HTC is the improved version of Pocofoam with higher thermal conductivity and density. Table 1 gives some properties of both foams.

<table>
<thead>
<tr>
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<th>Poco–HTC</th>
<th>PocoFoam</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density [g/cm³]</td>
<td>0.85 ± 0.05</td>
<td>0.55</td>
</tr>
<tr>
<td>Ligament density</td>
<td>2.23</td>
<td>2.23</td>
</tr>
<tr>
<td>Porosity [%]</td>
<td>61.8</td>
<td>75.3</td>
</tr>
<tr>
<td>Open porosity [%]</td>
<td>95</td>
<td>96</td>
</tr>
<tr>
<td>Therm. cond. [W/(m.K)]</td>
<td>245</td>
<td>135</td>
</tr>
<tr>
<td>Out-of-plane (x)</td>
<td>245</td>
<td>135</td>
</tr>
<tr>
<td>In-plane (y)</td>
<td>70</td>
<td>45</td>
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The precursor and processing details affect the internal structure of the end product, which in turn determines the bulk properties. The structure of the Poco–HTC sample is illustrated in Fig. 1, which was recorded with SEM. This figure highlights what the terms ligaments, junctions, cell-openings (pores), micro-cracks on walls and around cell-openings, and layer-spacings around folded layers refer to. Due to the foaming process, the resultant material has bubbles elongated in the vertical (out-of-plane) direction, which is taken as z-axis, while the x and y-axes are used for the horizontal (in-plane) directions.

Poco–HTC consists of highly graphitised material. The cell walls at mid-height of the bubble have graphite planes parallel to the bubble walls and are perfectly compacted. Where these planes meet at the top and bottom of the bubble (junctions), the graphite structure folds and has many micro-defects. Fig. 2 shows that the molecular layers are much less well aligned at junctions and contain cleavages between planes of graphite. This feature was illustrated in [8] with higher magnification for a graphitic foam similar to the Poco–HTC. It was reported in [8] that higher graphitization rate causes micro-cracks as separation of the graphitic layers. These layer-spacings run parallel to the planes of graphite, affect neither crystal size nor thermal conductivity. However, these defects are expected to mechanically weaken the foam [6].

There are also cracks and defects in the cell-walls probably caused by thermal stresses arising during the heat treatment process. These cracks occur at the boundaries of the planes. However these are much less frequent than the micro-defects at the junctions.

If the gaseous volume is large enough, the bubbles join at holes in the cell walls, making an open-cell foam. These holes in the walls, referred to as cell openings, are initially smooth and circular. However, later heat treatments can lead to fracture and micro-cracks at cell openings.

Both the elongation of bubbles and the alignment of graphite planes along the bubble walls lead to anisotropic behaviour of the bulk material. The micro-cracks, folds, and other defects have a major impact on bulk material properties compared to what would result from perfect graphite.

### 2. Measurements

Tests were performed to capture material destructive compression and shear behavior. The intention was also to capture elastic behavior for calculating material constants.

The crush tests used monotonically increasing uniaxial compression up to complete failure of material. These tests failed to characterise elastic behavior due to using samples cut from the top (low density) surface of the foam block. A new group of samples were cut from the bottom of the foam block to minimise density variation, and were subjected to a different compressive loading scheme: A cyclic loading scheme, in which the load is increased in stages, and released after each stage, before moving on to the next, higher-load stage (Fig. 3). The Young’s modulus was extracted from the cyclic compression test. Both crush and cyclic compression tests were applied in the out-of-plane and in-plane directions to characterise anisotropy.

The Iosipescu shear tests were used to study destructive shear. The material was placed in a fixture to apply shear load in the in-plane direction on the out-of-plane face (Fig. 4). The foam has a porous surface, so it was not possible to install strain gauges, therefore elastic properties could not be derived from shear tests.

A camera filmed the destructive compression and shear tests.

#### 2.1. Samples for compression Tests

Samples were machine-cut from a large foam block (about 300 × 30 × 300 mm³). The out-of-plane cyclic test samples had 20 × 10 × 20 mm³ dimensions. The in-plane cyclic test samples were 10 × 20 × 20 mm³. The out-of-plane (z) size is always the middle of the three dimensions given here; the surface being compressed is always 20 × 20 mm², while the height in the machine is 10 mm. The crush test samples were measured 20 × 20 × 20 mm³, and cut from the top of the block.

These samples are large enough to minimise edge effects due to open bubbles, debris from machining etc. No cover plates were attached to avoid effects of glue leaking into the surface cells.

#### 2.2. Samples for Iosipescu shear tests

The Iosipescu specimen is a rectangular beam with a symmetric V-notch at its center. A fixture with proper configuration is used to transform applied machine load into the pure shear load acting on a central section [15]. Furthermore, V-notches intensify stress at the center and localise failure at this section.

The Iosipescu sample is 80 × 20 × 8 mm³ (Fig. 4). V-notches have a 90° angle and are 12 mm apart. Thus, the shear surface covers a 12 × 8 mm² area. Since our Poco–HTC block is only 30 mm thick, we constructed our sample from two aluminium blocks 27.5 mm long, glued either end of the Poco–HTC sample with length 25 mm.

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2 By micro-scale we mean structures smaller than cell-walls (but much bigger than atomic scale); by meso-scale we mean structures at the size of a cell; and macro-scale treats the block sample as a whole.

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*The foam samples used in these tests were cut from the top of the main foam block, where the material has slightly lower density. Local cell crushing occurred at this surface. This in turn introduced sudden changes in the stress–strain response before the actual crushing stress. Consequently, crushing tests showed some irreproducibility when characterising elastic behavior.*
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