

Microstructural and compositional characterisation of the pyrocarbon interlayer in SiC coated low density carbon/carbon composites

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Abstract

SiC coated carbon bonded carbon fibre (CBCF) composites, a special class of carbon/carbon composites for thermal insulation, were investigated. Successful deposition of SiC requires the CBCF material to be first given a pyrocarbon coating. SiC coating on pyrocarbon coated CBCF was assessed using several analytical techniques. X-ray diffraction identified the coating as β SiC. The fibre orientation in two perpendicular planes was determined using X-ray microtomography, and it was found to be random in one plane whereas there was a preferred orientation in the other plane. A comparison was made between the uncoated and pyrocarbon coated substrates in terms of surface roughness, purity and crystallinity, using white light interferometry, neutron activation analysis/secondary ion mass spectrometry and transmission electron microscopy, respectively. The higher roughness, greater purity and increased levels of crystallinity of pyrocarbon coated CBCF are considered to be responsible for the successful deposition of a SiC coating on this material.

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1. Introduction

Carbon bonded carbon fibre (CBCF) composites are a special class of low density, highly porous carbon/carbon composites used for thermal insulation.¹ They are produced from rayon based carbon fibres which have the lowest thermal conductivity of all forms of carbon fibres and exhibit excellent refractory properties. In CBCF composites the short fibres are bonded together at the intersections of adjacent fibres with carbon char produced from carbonised phenolic powder by a high temperature heat treatment.

CBCF is a highly porous material, the typical density is in the range 0.1–0.5 g/cm³, and its layered structure (a consequence of an early stage in the manufacturing process) means that it

is anisotropic. The 2-D planar random structure of CBCF is shown in Fig. 1, and this material has been described in detail elsewhere.^{2,3}

The mechanical properties of CBCF material are relatively poor but are adequate for the applications for which CBCF is used, namely thermal insulation. Properties which make these composites suitable for thermal insulation include high porosity/low density, which results in low thermal conductivity, and anisotropic thermal conductivity (higher in the direction perpendicular to the heat flow). Experimental thermal conductivity data for CBCF composites will be presented in a later study. Often, the fibres within CBCF materials are given a coating of smooth laminar pyrocarbon using a chemical vapour deposition (CVD) process. This coating provides improved mechanical properties to the CBCF,^{2,3} and the material is then called CVD CBCF. CBCF composites can also be produced with a high level of purity (<0.2%), which is important in applications such as high purity single crystal growing furnace insulation, as any impurities in the insulation can contaminate the crystals being grown.

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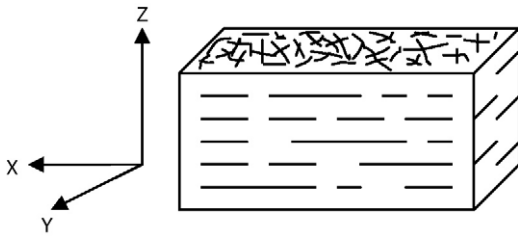


Fig. 1. 2D planar random structure of CBCF material.

The usage of CBCF based materials has been limited within the thermal insulation sector due to problems with oxidation (carbon/carbon composites oxidise readily above around $450\text{ }^{\circ}\text{C}^4$) and degradation within other hostile environments such as a hydrogen atmosphere which can lead to the formation of methane. This reactivity means that CBCF materials are currently only used in applications which involve an inert atmosphere or vacuum.

In an attempt to improve the performance of CBCF materials within these reactive environments, prototype materials have been produced where CBCF and CVD CBCF substrates have been infiltrated with a protective coating of silicon carbide using chemical vapour deposition. The silicon carbide coating does not encapsulate the substrate, rather the entire porous framework is infiltrated and coated. The CVD of silicon carbide is only successful when employing CVD CBCF as the substrate. When silicon carbide deposition has been attempted with CBCF substrates (i.e. with no pyrocarbon layer), the samples have disintegrated into green powder. It should be noted that within this paper, the silicon carbide coated CVD CBCF substrate material is, for simplicity, referred to as silicon carbide coated CBCF. To date no information is available on either the microstructure or the properties of the silicon carbide coated CBCF material.

The present work is the first characterisation study of the silicon carbide coated CBCF material focusing on material microstructure and on the effect of the pyrocarbon layer on the SiC coating. A variety of techniques, including scanning and transmission electron microscopy, X-ray diffraction and X-ray microtomography, were used. Explanations have also been sought as to why silicon carbide deposition is only successful for the CVD CBCF and not for the uncoated base CBCF material. Avenues of investigation included a comparison of the relative purity levels, surface roughness and crystallinity of the two substrates (CBCF and CVD CBCF).

2. Experimental procedure

2.1. Manufacture of materials

The production of CBCF materials has been described in detail elsewhere.¹ A flow diagram of the manufacturing process is shown in Fig. 2. The CVD process used to deposit pyrocarbon onto CBCF (forming CVD CBCF) is conducted at $1000\text{--}1100\text{ }^{\circ}\text{C}$, and employs nitrogen as the carrier gas and methane as the precursor gas. Typically, a $0.25\text{ }\mu\text{m}$ thick pyrocarbon coating has been deposited for the materials investigated

in this study. The CBCF materials were manufactured and supplied by Mersen Scotland Ltd, UK. The infiltration of SiC was carried out by means of an industrial CVD process using methyltrichlorosilane (MTS) as the precursor gas. All results for silicon carbide coated materials within this paper are for the CVD CBCF substrate, as when attempts at deposition were made using the CBCF material, the samples lost their structural integrity and were not suitable for testing. The composite is denoted as silicon carbide coated CBCF.

2.2. Characterisation of microstructure

Samples for scanning electron microscopy (SEM) were prepared by cutting cubes using a scalpel and mounted on metal stubs using carbon tape. The electrical conductivities of all samples (CBCF, CVD CBCF and silicon carbide coated CBCF) were sufficient for examination without prior coating. Typically, SEM samples would have dimensions of $5\text{ mm} \times 5\text{ mm} \times 5\text{ mm}$. The samples were examined in a JSM5610LV SEM at acceleration voltages ranging from 5 to 30 kV.

The transmission electron microscope (TEM) used was a JEOL 2000FX TEM. TEM samples were prepared using a focused ion beam generated by a FEI FIB200-SIMS, ion microscope. The first step was to attach fragments of the silicon carbide coated CBCF to copper grids. The grids were placed within the apparatus and suitable regions within the fragments were located and then thinned using the beam.

X-ray diffraction (XRD) analysis was conducted on the silicon carbide coated CBCF material. The diffractometer used was a Philips PW2273 diffractometer, and it was operated at 40 kV and 40 mA with a copper tube to give copper K_{α} radiation. The samples were cut using a scalpel and were tested in block form with dimensions of $3\text{ mm} \times 5\text{ mm} \times 5\text{ mm}$. The scans were conducted for 2θ values between 0° and 90° , at increments of 0.04° , with each increment lasting for 1 s.

The fibre orientation within the silicon carbide coated CBCF material was determined using X-ray microtomography (XMT).

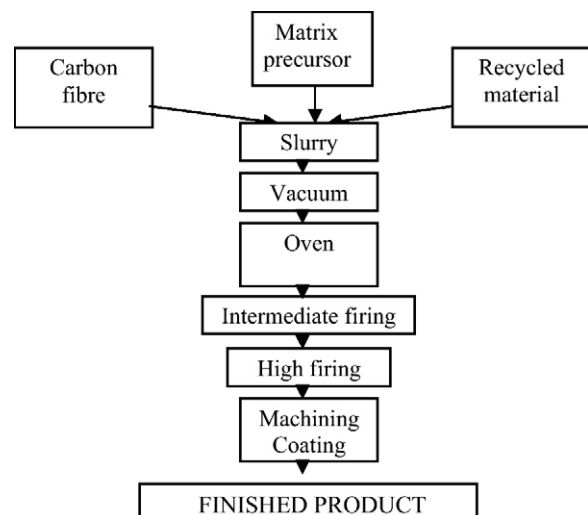


Fig. 2. Flow diagram summarising the manufacturing process for CBCF materials.

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