MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MESOPHASE PITCH-BASED C/C COMPOSITES

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Abstract

Microstructures and mechanical properties of 2D PAN fiber-reinforced mesophase pitch-based C/C composites were studied with the aid of polarized light microscopy (PLM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and mechanical testing experiments. Results show that the matrix carbon exhibits optical anisotropy under PLM. The mesophase pitch carbon exhibits lamellar structure and its orientation is roughly parallel to the carbon fiber axis. Besides, the micro-cracks inside the matrix and those at the fiber-matrix interface are also parallel to the carbon fiber axis. The non-brittle fracture behavior of the composites is related to multiple crack deflections caused by the interface and micro-cracks within the matrix carbon. The flexural strength of composites is up to 257MPa.
Keywords: Carbon composites; Mesophase pitch; Graphitic carbon

1 Introduction

Carbon/carbon (C/C) composites are composed of carbon fibers, carbon matrix and different interfaces [1, 2]. The microstructure of each constituent has a great effect on the mechanical properties of these composites, Especially the structure and performance of interfaces affect the physical, chemical, and mechanical properties of C/C composites largely [3, 4]. Therefore, the interface engineering can be applied to develop high-performance materials [5, 6].

The main problems for the applications of C/C composites are their large brittleness and poor toughness, which might result in the occurring of the calamity and outburst as they are used as structural materials. Mesophase pitch has the characteristics of nematic liquid crystals and can be used to construct an interface with suitable bonding strength, which is advantageous to improve the mechanical properties of C/C composites and weaken their brittleness. In this study, using mesophase pitch as matrix precursor, C/C composites were prepared by the liquid impregnation and carbonization technique. The microstructures and mechanical properties of the as-obtained composites were studied.

2 Experimental

2.1 Preparation of C/C composites

The preforms were prepared by lamination technique with 3K PAN carbon woven cloth. The fiber volume fraction of performs was controlled to about 40%. The precursor of the matrix was mesophase pitch with the softening point of 283.7℃ and the mesophase content of 100 %, produced by Mitsubishi

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Gas Chemical Company, INC. The liquid impregnation-carbonization technique was used to prepare mesophase pitch-based C/C composites. The carbonization pressure was 40MPa. To meet the desired density and properties, a multi-cycled impregnation/carbonization/graphitization is required. Finally, the C/C composites were formed.

2.2 Measurement of mechanical properties of C/C composites

Samples for mechanical properties test were cut from the as-prepared composites, with the rectangular cross-section of 10 mm in width, 4 mm in thickness, and 50 mm in length. Three-point bend test was performed using the 1195-typed Instron Electric Mechanical Tester with the load direction perpendicular to the surface of carbon cloth, and the span was 40 mm.

2.3 Observation of microstructure

Metallographic examinations were carried out by PLM using an OLYMPUS PM-T3 microscope. C/C composite samples were embedded and fixed with epoxy resin and curing agent before polishing. The fracture surfaces of C/C composites were observed using a JSM-6460 scanning electron microscopy. The original morphology was examined by SEM in a JSM-6700F microscope with a field-emission gun. Thin foils for TEM involved were prepared using mechanical dimpling followed by argon ion milling. Firstly, specimens were sliced to a thickness of 200μm from blocks of the composite using a diamond saw. Three millimeter diameter discs were cut from such slices using a drill press. These discs were mechanically dimpled (Gatan 656 mechanical dimpler) to a thickness of 60 μm. The dimpled discs were then atom milled using a GATAN 600 ion miller until perforation occurred. The TEM was carried out in a JEOL-2010 high resolution transmission electron microscope operating at 200 kV.

3 Results and discussion

3.1 Microstructure of mesophase pitch-based C/C composites

Fig.1 shows the polarized texture structure of mesophase pitch-based C/C composites. It is clear that the matrix carbon exhibits optical anisotropy under PLM. The mosaics-type pitch matrix was formed inside the fiber bundles and the domain-type pitch matrix was formed among fiber bundles.

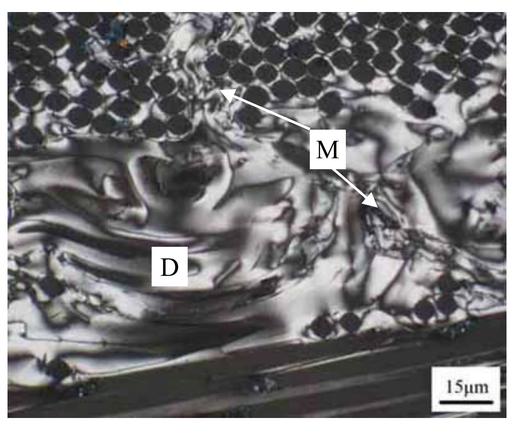

Fig.1 Polarized light micrographs of mesophase pitch-based C/C composites ----M: mosaic-type pitch matrix; D: domain-type pitch matrix

Fig.2 shows the original SEM image of mesophase pitch-based C/C composites. The fiber-matrix interface was discontinuous. Some pitch carbon lamellae were well bonded to fiber as shown in Fig.2 at the single arrow, while other pitch carbon lamellae were poorly bonded or even entirely separated from the fiber as shown at the double arrow. This interface morphology is similar to “fissured-type” interface, as described by Ragan and Marsh [7]. The mesophase pitch carbon in the C/C composites present lamellar structure and overlap together in three dimensions. The orientation of carbon lamellae is parallel to the carbon fiber axis, forming POG (Parallelly Oriented Graphite) structure [8].

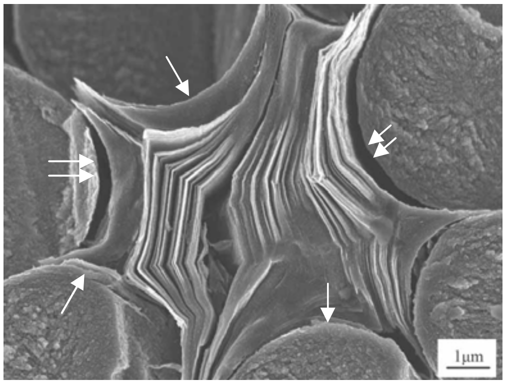

Fig.2 Original SEM micrographs of mesophase pitch-based C/C composites

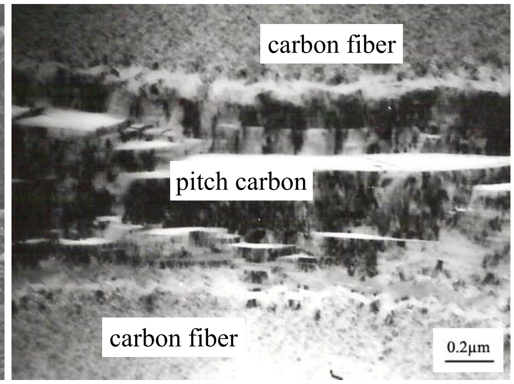

Fig.3 Low magnification TEM micrographs of mesophase pitch-based

Fig.3 is the lower magnification TEM morphology of mesophase pitch-based C/C composites. The interface between fibers and mesophase pitch carbon in the C/C composites is discontinuous due to the existence of some microcracks at this interface. The mesophase pitch carbon in the C/C composites present lamellar structure and its orientation is roughly parallel to the carbon fiber axis. Moreover, numerous micro-cracks parallel to the fiber axis were formed at the fiber-matrix interface and within the mesophase pitch carbon.

3.2 Mechanical properties and fracture features of mesophase pitch-based C/C composites

Table 1 shows the mechanical properties of mesophase pitch-based C/C composites. Eight samples were used for testing of each property of composites and the average value of each property was listed in Tab.1.

Table 1 Mechanical properties of mesophase pitch based C/C composites

Property	Density /g·cm^{-3}	Flexural strength /MPa	Flexural modulus /GPa
Average value	1.78	257.6	45.2

Fig.4 shows the typical curves of load-displacement of mesophase pitch-based C/C composites. It can be seen that the composites exhibits the tough fracture characteristic. Prior to fracture, the load-displacement curve presents non-linear feature. During the fracture process, the load is declined not abruptly but gradually in steps.

Fig.5 is the SEM photographs of fracture surface of mesophase pitch-based C/C composites. It can be seen that the fracture surface morphology is the like-step structure and the pull-out of fibers is obvious. In the C/C composites, the fiber-matrix interface morphology is similar to the “fissured-type” interface. So the mixed fracture occurs during the fracture of the C/C composites, that is, the interface fracture and cohesive fracture of matrix take place simultaneously [9]. The alternative expansion of the cracks along the radial direction or the axial direction of the fibers happens, which induces the like-step fracture surface as shown in Fig.5. Under this situation, the matrix transforms the load moderately, so the flexural strength of the composites is higher and the composites exhibit the tough fracture characteristic.

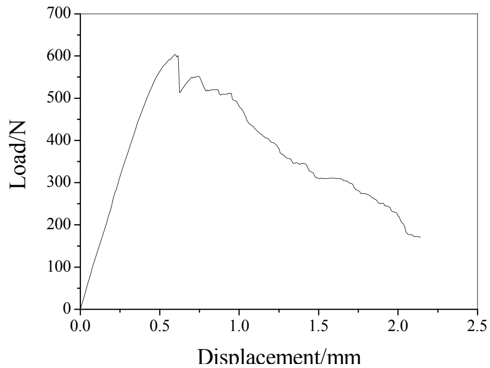

Fig.4 the curve of load-displacement of mesophase pitch-based C/C composites

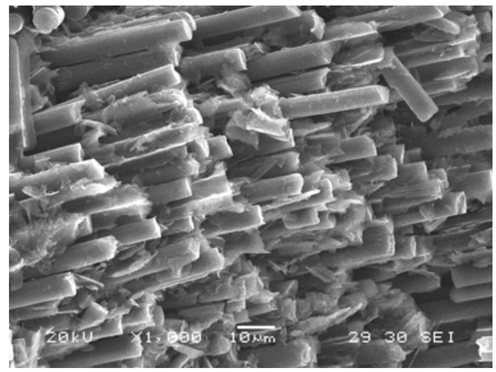

Fig.5 SEM photographs of fracture surface of mesophase pitch-based C/C composites

4 Conclusions

2D PAN fiber-reinforced mesophase pitch-based C/C composites were prepared by the liquid impregnation-carbonization technique. The mesophase pitch carbon exhibits lamellar structure and its orientation is roughly parallel to the carbon fiber axis. The non-brittle fracture behavior of the composites is attributed to multiple crack deflections caused by the interface and micro-cracks within the carbon layers. The flexural strength of C/C composites can be increased by improving the bonding of the interfaces in the composites.

Acknowledgements

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