STRENGTH AND FRACTURE MECHANISMS IN CARBON-CARBON COMPOSITES

A. Chennakesava Reddy

Dept. of Mech. Engg., JNTU College of Engineering, Anantapur

ABSTRACT

This paper highlights on strength and fracture mechanisms of carbon-carbon composites consisting of a carbon matrix reinforced with carbon fibers. Carbon matrices derived from thermosetting resin precursors (TSRP) are amorphous and isotropic in their properties and microstructure. In carbonaceous liquids, polymerization results in the production of large sheet-like molecules, which upon reaching a critical size, phase separate into an ordered liquid crystal structure called mesophase. Crack propagation within a mesophase carbon will tend to the layered structure. The weak bonds between fiber and matrix are frequent in carbon – carbon composites. Higher values of tensile and compressive strengths are reported along the fiber direction with the increase in the degree of chemical bonding resulted due to interfacial coupling.

Introduction

An early application of carbon-carbon composites was to replace polycrystalline graphite as the nose tip in rockets, which become incandescent and erode during reentry. The addition of the reinforcing fiber to the bulk carbon significantly improved the durability of the nose tip when it was subjected to intense thermomechanical stresses. Successful use of these of composites led to the development and use of carboncarbon composites in rocket nozzles, exit cones, the nose cap, leading edge of the wings and brakes in aircraft and racing cars. Because of the retained specific strength at high temperature, carbon-carbon composites treated with oxidation resistant coatings are under active investigation as casings, containers and blades in gas turbine engines. Carbon is very compatible with tissues within the human body, thus carbon-carbon composites are being considered for prosthesis applications because the strength, modulus and shape can be easily tailored to the specific requirements. The heat storage capacity by weight for carbon is 3.7 times that of steel, hence it can be used as a braking material [1]. Some important carbon composites used in various aerospace applications are given Table-1.

The type reinforcement in carbon-carbon composites include: random fibers, unidirectional fibers, braided yarns, stacked 2-D fabrics, pierced fabrics to provide increased interlaminar shear properties, orthogonal 3-D geometries in either Cartesian or cylindrical coordinates, or in multi-directional weaves designed to improve the off-axis properties and to minimize the empty spaces that occur at filament cross-over locations. Commercial carbon fibers are manufactured from rayon fabrics, isotropic pitch, mesophase pitch, and acrylic fiber precursors [2]. The strength and modulus of these reinforcing materials vary considerably depending on the raw material and the processing conditions [3, 4].

Sl. No.	Type of composite	Application
1	Carbon-carbon composites	•Nose tip in rockets
		• Rocket nozzles
		• Exhaust cones
		•Nose cap
		•Leading edges of the wings
		• Brake discs
2	Carbon fiber reinforced plastic composites	• Control surfaces
		• Cowlings
		• Under carriage doors
		• Fairings
		• Fin box
		• Dual launch structures
		• Solar array wings
		Parabolic reflector
3	Graphite reinforced aluminum composites	• High gain antenna boom
4	Carbon/Boron reinforced epoxy composites	• Rudder

Table-1: Some aerospace applications of carbon composites

The matrix can vary from an isotropic carbon obtained from thermosetting resins to a highly oriented anisotropic carbon that arises from the mesophase developed during low temperature $(300 - 450^{\circ}C)$ pyrolysis of pitch precursors. Matrix material can also be formed by chemical vapor deposition of carbon obtained by cracking hydrocarbon vapors in the range of 1000 to 2000^oC.



Fig.1 Schematic representation of alignment of mesophase layers with graphitic orientation in needle coke particles (10µm).

Interfaces

Bulk carbons or graphites are used in the manufacturing of nuclear reactors and furnace electrodes. They are made by mixing and forming a mass of carbon particles (filler) with a carbon rich liquid (binder pitch) and baking to volatilize the non—carbon species. Binder pitch transforms into a mesophase in which large planar molecules, precursor to graphite layers in the final carbon, align themselves in layers. Covalent bonds can form only when at least one phase has graphite orientation and the graphite lamellae are not parallel to the interface. Fig.1 shows the alignment of mesophase layers with graphite orientation. Weaker bonds viz., Van der Waals, dipolar occurs between layers, which are parallel to the graphite layers [5].

Not only bonding occurs across interfaces but also coupling viz., mechanical interlocking and friction. The interfaces in carbon-carbon composites exist in many different levels: between filaments and matrix, between yarns, between the microstructures that exist within the matrix, between different layers of matrix, between matrix and voids and by the deposition of additional matrix during the impregnation cycles [6]. The interfaces are the result of matrix selection and the processing conditions.



Fig.2 Scanning electron micrograph showing an isotropic TSRP carbon matrix (10µm).

Carbon Matrices

Carbon matrices derived from thermosetting resin precursors (TSRP) are amorphous and isotropic in their properties and microstructure. Fig.2 illustrates the microstructure of an isotropic TSRP carbon matrix. TSRP matrices can be made graphitic and anisotropic by the heat treatment or the application of stress or both. Stress is applied in a composite by the adjacent filaments during the pyrolysis stages of processing. At this stage, the organic precursor is undergoing heteroatom elimination and is shrinking. Any covalent bonds between phases that involve heteroatoms will be rearranged or destroyed. If the organic matrix is in contact with filaments, it cannot shrink freely. This results in tensile stress at the filament-matrix interface, which in turn may align the matrix molecules near the filaments. Upon heating to graphitization temperatures, the matrix will tend to become graphitic. The final heat treatment temperature influences the degree of graphitic structure. For a heat treatment of 2400⁰C, a graphitic structure (lamellar) as shown in Fig.3 will result. The inter fiber matrix becomes more graphitic with increasing heat treatment temperature. These effects are attributed to stress-graphitization. It is not clear, whether the major contribution to stress-graphitization is by stresses caused by matrix shrinkage during pyrolysis or creep-induced flow and stress relaxation at high temperatures. This leads to conflict conclusions and much research is required to understand the conditions under which TSRP matrices develop graphitic orientation.



Fig.3 Scanning electron micrograph of a TRSP matrix heat-treated to 2400°C to from graphitic carbon (20µm).



Fig.4 Schematic representation of mesophase alignment around carbon fibers after low-pressure impregnation and heat setting (10µm).

Graphite Matrices

All graphitic matrices are usually derived from carbon containing substances, which pass through a fusion or liquid stage between 300 and 450° C. In carbonaceous liquids, polymerization results in the production of large sheet-like molecules, which upon reaching a critical size, phase separate into an ordered liquid crystal structure called mesophase. On heat treatment above 450° C, the mesophase undergoes further polymerization, and hardening. The mesophase converts into graphitic carbon in the temperature range 2000 2400° C. The interfaces within carbons derived from pitches which form mesophase are complex due to imperfect ordering of the discotic, nematic structure. Fig.4 shows the schematic structure of graphitic matrix.



Fig.5 Schematic representation of orientation and disclinations of some basal planes in a bulk mesophase. Cracks assumed to be propagating from right to left ($10\mu m$).



Fig.6 Schematic representation of shear crack at region between perpendicular fiber bundles (10µm).

Defects in Carbon-Carbon Composites

A number of defects are disclinations in carbon-carbon composites. Crack propagation within a mesophase carbon will tend to the layered structure. Blunting and bifurcation of cracks can occur by interaction of the crack tip with various disclinations [7]. The influence of the disclinations structure on the crack path is shown in Fig.5. It can be observed that an interlaminar crack is propagated between layers of carbon cloth. Fig.6 reveals that the matrix forms a sheath around fibers despite the presumed availability of edges to which the mesophase might have formed covalent bonds. In some carbon-carbon composites, it was observed that active edge atoms are more at fiber surfaces. If the edge atoms are active as shown in Fig.7, the interface region will not survive heat treatment. But, a thin layer of matrix is bonded to the fiber and the cracks are seen in the matrix rather than at the interface.



Fig.7 Microstructure of pitch matrix after heat treatment to 2700° C. Specimens were etched with oxygen plasma (20μ m).



Fig.8 Scanning electron micrograph of transversely aligned matrix (10µm)

The graphite matrix is highly anisotropic. Fig.8 shows the variation of microstructures of pitch precursor matrix between carbon filaments. The lamellar structure is resulted on account of low-pressure impregnation whereas transversely aligned matrix forms between the filaments due to high-pressure infiltration. The possible orientations of matrix at the fiber-matrix interface are illustrated in Fig.9.



Fig.9 Schematic representation of different matrix orientation around a fiber



Fig.10 Microstrain of a TSRO matrix carbon-carbon composite cycled between room temperature and 250° C.

Strength in Carbon-Carbon Composites

The weak bonds between fiber and matrix are frequent in carbon – carbon composites. The fiber-reinforced composites differ from the particle reinforced composites. Particle reinforced composites have some strong bonds. If the interlayer bonds throughout the lamellar matrix are weak, cohesive failure in the matrix is likely to occur, with effects similar to those of adhesive failure at the interface [8]. Some weakness of fiber matrix interfaces may be a requirement of successful carbon-carbon: too strong a bond, produced sometimes between TSRP matrices and carbon fibers, may promote excessive damage to fibers during heat treatment; also, too good a bond may promote brittle behavior [9]. Because of weak matrix and interfaces, unidirectional carbon-carbon composites have low transverse tensile and shear strengths. Carbon-carbon composites are multi directionally reinforced; cloth-reinforced 2-D laminates or spatially reinforced 3-D and 4-D composites to reduce the risk of delamination in service.

Interfacial coupling of fiber to matrix and filament that are caused by shrinkage of the matrix during processing and by differences in thermal expansion. Therefore, it is important to select the fiber type, the

matrix and the processing conditions to maximize the effectiveness of this coupling. Higher values of tensile and compressive strengths are reported along the fiber direction with the increase in the degree of chemical bonding that results due to interfacial coupling. The coefficient of thermal expansion between room temperature and 500° C is negative for filaments having a high degree of graphitic planes aligned parallel to their axes. In contrast, the coefficient of thermal expansion of TSRP matrix is both isotropic and positive and is four times the absolute value of filaments and the matrix. This must be resisted by the interfacial coupling [10]. The variation of microstrain with temperature is shown in Fig.10. A composite specimen of yarns embedded in a TSRP matrix was heated and cooled between room temperature and 250° C for a number of cycles. On the first cycle, the curve has a negative slope as would be expected for a composite that is dominated by filament expansion.

Conclusions

- 1. Carbon matrices derived from thermosetting resin precursors (TSRP) are amorphous and isotropic in their properties and microstructure. TSRP matrices can be made graphitic and anisotropic by the heat treatment or the application of stress or both.
- 2. All graphitic matrices are usually derived from carbon containing substances, which pass through a fusion or liquid stage between 300 and 450° C. In carbonaceous liquids, polymerization results in the production of large sheet-like molecules, which upon reaching a critical size, phase separate into an ordered liquid crystal structure called mesophase.
- 3. A number of defects are disclinations in carbon-carbon composites. Crack propagation within a mesophase carbon will tend to the layered structure. Blunting and bifurcation of cracks can occur by interaction of the crack tip with various disclinations.
- 4. The graphite matrix is highly anisotropic.
- 5. The weak bonds between fiber and matrix are frequent in carbon carbon composites. The fiber-reinforced composites differ from the particle reinforced composites.
- 6. Some weakness of fiber matrix interfaces may be a requirement of successful carbon-carbon: too strong a bond, produced sometimes between TSRP matrices and carbon fibers, may promote excessive damage to fibers during heat treatment; also, too good a bond may promote brittle behavior.
- 7. Higher values of tensile and compressive strengths are reported along the fiber direction with the increase in the degree of chemical bonding resulted due to interfacial coupling.

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