# Chapter 8
# Carbon Fiber/Carbon Matrix Composites

We devote this chapter to a very special kind of composite that consists of carbon fibers embedded in a carbonaceous matrix. The carbon fiber in these composites can be continuous or short. We described the processing of carbon fibers in Chap. 2. Carbon is a very versatile material. It can exist in a variety of forms; amorphous, graphite, diamond, fullerenes, graphene, nanotubes, etc. Below we summarize the salient features of some important forms of carbon.

*Diamond*. Carbon in the diamond form has an FCC structure; it is also called diamond cubic. Each C atom is covalently bonded to four carbon atoms at the vertices of a tetrahedron and the interatomic distance = 0.154 nm. Carbon in the diamond form has isotropic properties. It is one of the hardest materials.

*Graphite*. Graphitic form of carbon has a hexagonal structure. Carbon atoms are arranged in layers. The bonding between atoms in the plane of the layer is covalent but the bonding between layers is weak, van der Waals type. This difference in the bonding in $a$ and $c$ directions makes graphite is highly anisotropic. Young's modulus in the basal plane ($a$ direction) is ~1,000 GPa, while in a direction perpendicular to the basal plane ($c$ direction) it is ~35 GPa. Tensile strength, thermal expansion, and thermal conduction are also anisotropic in a similar manner.

*Pyrolytic graphite*. Pyrolytic graphite is produced from a gaseous phase via pyrolysis of hydrocarbons. The pyrolysis of hydrocarbons results in carbon, which is deposited on a hot substrate. Pyrolytic graphite has a highly oriented structure and therefore is highly anisotropic.

*Carbon black*. A generic term for carbon powder of extremely fine size (nm range) and very high surface area. It is essentially elemental carbon (>97 %), electrically conductive, and finds extensive use as a reinforcement in tires and other rubber products as well as in printing inks and paints.

*Carbon nanotubes*. These are hollow, cylindrical tubes of carbon atoms with diameter in the range of 5–20 nm. They are frequently referred to as CNTs.

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CNTs can have very large aspect ratios. They show some very unusual properties, such as extremely high stiffness and strength.

Carbon is an excellent high temperature material when used in an inert or nonoxidizing atmosphere. Potential high temperature applications call for 10 h to a few 1,000 h at greater than 1,000 °C and at times approaching 2,200 °C. The major drawback of carbon is that it reacts with oxygen, forming gaseous oxides of carbon. Unlike other nonoxide ceramics, carbon powder cannot be sintered. Thus, the carbon matrix in a carbon fiber/carbon matrix composite is generally obtained by chemical vapor deposition (CVD) of carbon or thermal decomposition of a source of carbon such as pitch or phenolic resin. Generally, many pores form during this conversion of a hydrocarbon to carbon. Thus, a dense and strong pore-free carbon/carbon composite is not easy to fabricate. A very wide range of characteristics, however, can be obtained in these composites, the principal characteristic being the high temperature capability of these composites in *inert* environments. Being very light, carbon/carbon composites show superior specific strength at high temperatures in *inert* atmospheres.

Although, carbon/carbon composites were originally developed for the space program, they have now become high performance engineering materials, with special high temperature applications. More specifically, these high temperature applications involve brakes for aircraft and automobiles, dies for use in hot-pressing, high temperature fasteners, heating elements in furnaces, liners and protection tubes, etc. Carbon/carbon composites are expensive mainly because of the long processing times involved. Essentially, we take a carbon fiber reinforced polymer matrix composite and convert the polymer matrix by pyrolysis to carbon or we deposit carbon from gaseous reactants into interstices of a woven carbon fiber preform. The process can take days, making the final product very expensive. In what follows we describe the processing techniques, properties, and major applications of carbon/carbon (C/C) composites.

## 8.1 Processing of Carbon/Carbon Composites

Three methods are commonly used to fabricate carbon/carbon composites:

1. A woven preform of carbon fiber is impregnated, under heat and pressure, with thermoplastic pitch, followed by pyrolysis of pitch into carbon. Generally the cycle is repeated to obtain the desired density. A special version of this technique is called high pressure impregnation carbonization or HIPIC (see below). Pitches are mixtures of hydrocarbons of different molecular weights. Yield of carbon from the pitch precursor increases with increasing average molecular weight of the pitch but high molecular weight means high viscosity which makes penetration of the fibrous preform difficult.
2. A carbon fiber/polymer matrix composite is made by one of the conventional PMC fabrication techniques (see Chap. 5), followed by conversion of the resin, generally a thermoset, to carbon by pyrolysis. Again, reimpregnation and repyrolysis are

used to attain the desired density of the composite. Phenolics are one of the common resins used because of the comparatively high carbon yield.

3. CVD of carbon is made from a gaseous phase onto and in between the carbon fibers in a preform. Hydrocarbon gases become unstable with respect to carbon at high temperatures (>550 °C).

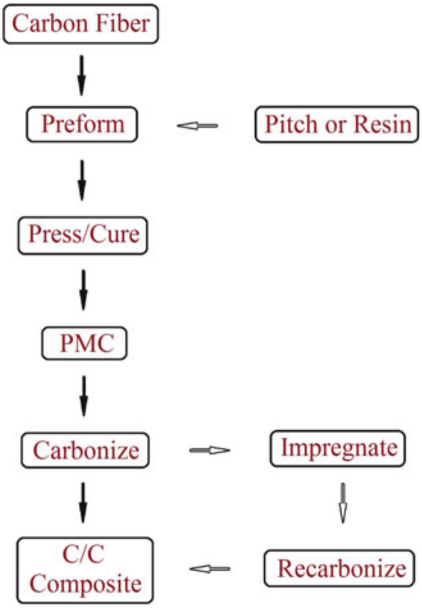

**Fig. 8.1** Processing of carbon/carbon composites by pyrolysis of carbon fiber/polymer composites

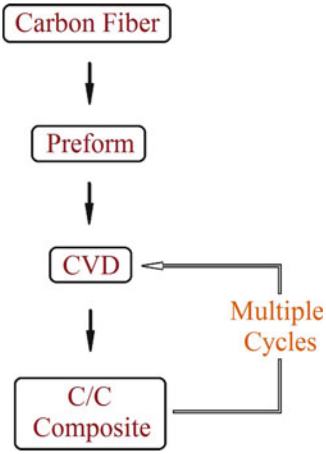

**Fig. 8.2** Processing of carbon/carbon composites by chemical vapor deposition (CVD) process

Methane, a common precursor for CVD, decomposes as per the following reaction:

$$CH_4(g) \rightarrow C(s) + 2H_2(g).$$

Low pressure (or addition of inert diluent gases such as $H_2$, $N_2$, or Ar to the gaseous stream) is used to improve diffusion because it increases the mean free path of the gaseous molecules.

Figure 8.1 shows the process corresponding to the pyrolysis of a PMC, while Fig. 8.2 shows the CVD process. In both processes, multiple impregnation cycles are required to increase the density of the composites, i.e., reduce the amount of porosity in the composite to an acceptable level. Invariably, in all the processes, there occurs a large weight loss (between 10 and 60 %) during the pyrolysis step, which results in a large amount of shrinkage porosity. In addition, there is the complexity of the anisotropic nature of the carbon fiber (properties are different in the axial and radial directions). The carbon matrix, obtained by pyrolysis, is essentially isotropic. This leads to generation of thermal stresses in each cycle, because of thermal mismatch between the fiber and the matrix, which leads to the formation of microcracks in the C/C composites after multiple cycles of impregnation and pyrolysis.

### *8.1.1 High Pressure Processing*

Application of isostatic pressure in an autoclave during carbonization of pitch can increase the yield of carbon from 50 % at atmospheric pressure to over 90 % at 100 MPa. This high pressure processing is also called high pressure impregnation carbonization or HIPIC. Commonly, partially impregnated preform and excess pitch are placed in a sealed metal can, which in turn is placed in the pressure vessel. The temperature is increased to 550–650 °C and pressure in the range of 5–100 MPa is applied. The thin metal can acts like a rubber bag in the processing of PMCs in an autoclave. The process can take as much as 24 h. After HIPIC, the metal container is stripped off and the impregnated composite is generally subjected to a graphitization treatment at temperatures above 2,300 °C.

Properties of C/C composites depend a great deal on the volume fraction of porosity present. Essentially, the pores present are of open-cell type. An easy parameter to follow the evolution of porosity in C/C composites is the density of the composite. One generally starts with a carbon fiber preform of density of 1 $g/cm^3$. After about four cycles of impregnation, we attain a density of 1.4 $g/cm^3$. However, under conditions of HIPIC, it is possible to reach a density of 1.8 $g/cm^3$ after four or five cycles, see Fig. 8.3. It is possible to reach a density of 1.8 $g/cm^3$ after four or five cycles, see Fig 8.3. It is worth pointing out here that the theoretical density of carbon is 2.25 $g/cm^3$. Carbon fiber itself can have density between 1.6 and 2.2 $g/cm^3$. A value of 2.2 $g/cm^3$ for carbon fiber is very uncommon; only mesophase pitch-based carbon fiber can show such a value after high temperature graphitization

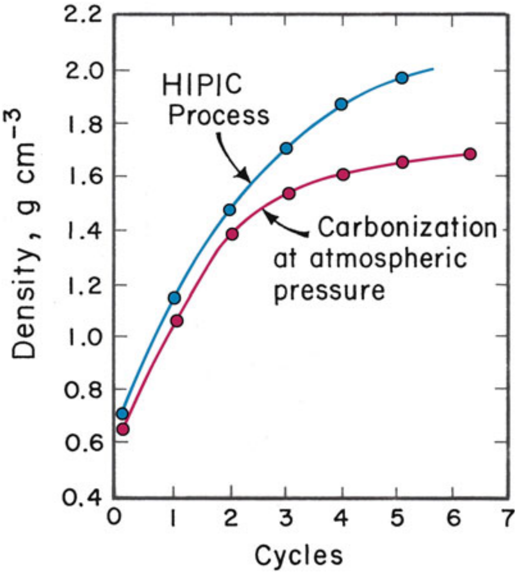

**Fig. 8.3** Change in density of carbon/carbon composites as function of number of impregnation cycles. Under conditions of high pressure impregnation (HIPIC), higher density is achieved than under atmospheric impregnation (after Taverna and McAllister 1971)

treatment. For the fabrication of C/C composites, up to a density of 1.4 g/cm$^3$, atmospheric processing and HIPIC have about the same efficiency. For density values greater than 1.4 g/cm$^3$, HIPIC shows much better efficiency than processing at atmospheric pressure.

## 8.2 Oxidation Protection of Carbon/Carbon Composites

By far, the greatest drawback of carbon/carbon composites is the formation of gaseous oxides of carbon upon reaction with oxygen in air at temperatures as low as 450 °C. Potential applications of carbon/carbon composites may call for use in service at temperatures exceeding 1,000 °C and even approaching 2,200 °C for times ranging from 10 h to a few thousand hours. There are two main approaches to protect carbon/carbon composites against oxidation (Buckley 1988; Luthra 1988; McKee 1986, 1987; Strife and Sheehan 1988):

1. Use inhibitors to slow down the rate of reaction between carbon and oxygen. This involves modification of matrix through addition of some oxidation inhibitors (e.g. B, Si, Zr, or their compounds).
2. Use diffusion barriers to prevent oxygen from reaching carbon and reacting with it. This involves deposition of ceramic coatings on the surface. These coatings

are generally multilayer coatings of functionally gradied materials of carbides, nitrides and oxides of Si, Zr, Ta, Al, etc.

The diffusion barrier coating should have the following characteristics:

- Oxidation resistance
- Low oxygen permeability and volatility
- Good adhesion to carbon
- Chemical compatibility with carbon
- Thermal expansion match with carbon

Figure 8.4a shows the critical factors that should be considered in an oxidation protection system for carbon/carbon composites (Strife and Sheehan 1988), while Fig. 8.4b shows, schematically, a typical multilayer oxidation protection system on a C/C substrate.

The two approaches involving the use of inhibitors and diffusion barriers are frequently combined: use diffusion barriers and internal glass forming inhibitors and sealants. Silicon-based ceramics, such as SiC and $Si_3N_4$, are commonly used as the primary oxygen barrier coatings. Sealants have the added advantage that they also seal thermal stress cracks originating because of the thermal mismatch between C/C composite and silicon carbide coating. Tetraethylorthosilicate (TEOS) is commonly used to impregnate such cracks (McKee 1986; 1987). TEOS leaves oxidation resistant silica in the cracks. Addition of boria $B_2O_3$ can inhibit the oxidation (Ehrburger et al. 1986; McKee 1986).

Extended oxidation protection of carbon/carbon composites can be obtained by using boria containing dense $Si_3N_4$ or SiC coatings and boron-rich inner coating. Borate glasses act as sealants and fill up the cracks that form in the outer coatings because of thermal expansion mismatch. An oxidation protection system used for extended protection under thermal cycling conditions, with peak temperatures reaching 1,400 °C, is shown in Fig. 8.4a (Strife and Sheehan 1988). Moisture sensitivity of borate glasses, corrosion of outer coatings by glasses, glass volatilization, and unacceptably high oxygen permeability are some of the negative factors. Alvey and George (1991) used multilayer Zr/Pt coating that formed $ZrPt_3$ on annealing on carbon/carbon composites. Their results showed that this coating protected carbon and a carbon fiber/phenolic resin composite from a 2,800 °C flame. They attributed this performance to the oxidation resistance of $ZrPt_3$ and to the reflectivity of the coating, which reduced the thermal flux on the substrate to a short time.

## 8.3 Properties of Carbon/Carbon Composites

Properties of carbon/carbon composites depend on the type of carbon fiber used (high-modulus or high-strength type), fiber volume fraction, the fiber distribution, and the amount of porosity. One-, two-, and three-dimensionally woven carbon fibers may be used. Carbon fiber can be woven in a variety of weaves for

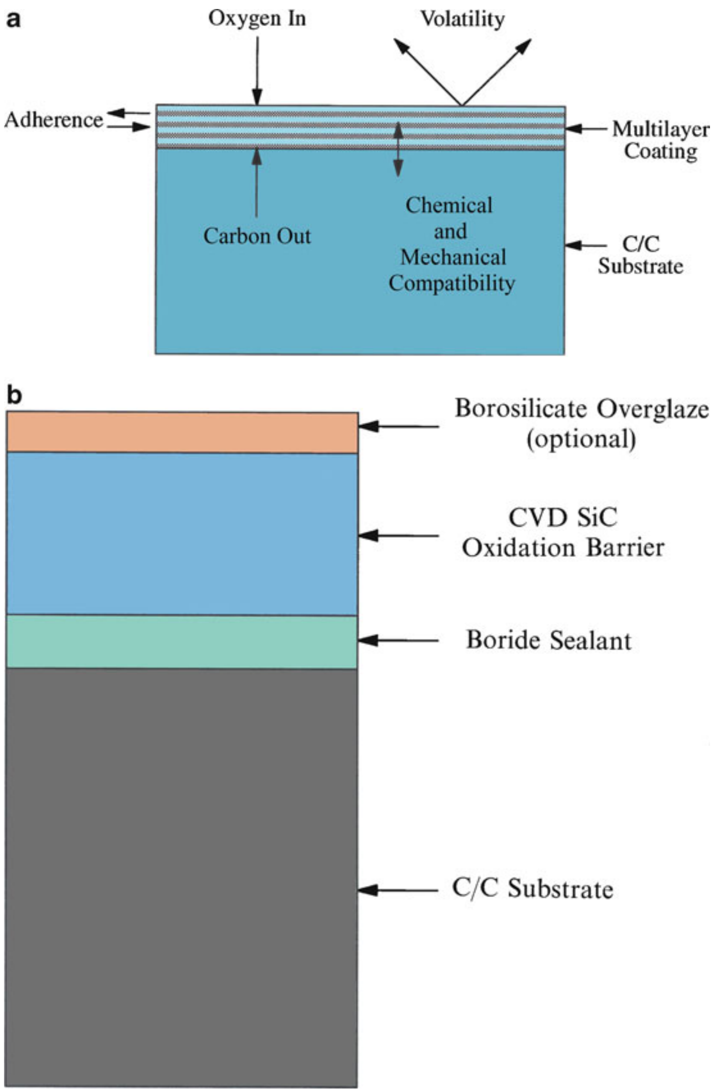

**Fig. 8.4** (**a**) Critical factors that should be taken into account for an oxidation protection system for carbon/carbon composites (after Strife and Sheehan 1988). (**b**) Schematic of a typical multilayer oxidation protection system on a C/C substrate

reinforcement in two or more dimensions. Two main types of weave are plain and satin. A *plain weave* has one warp yarn running over and under one fill yarn and is the simplest weave. *Satin-type weaves* are more flexible; that is, they can conform to complicated shapes easily. Figure 8.5 shows a two-dimensional (2D) plain weave and a five-harness satin weave (McAllister and Lachman 1983). Modifications of the basic three-dimensional (3D) orthogonal weave involving different fiber

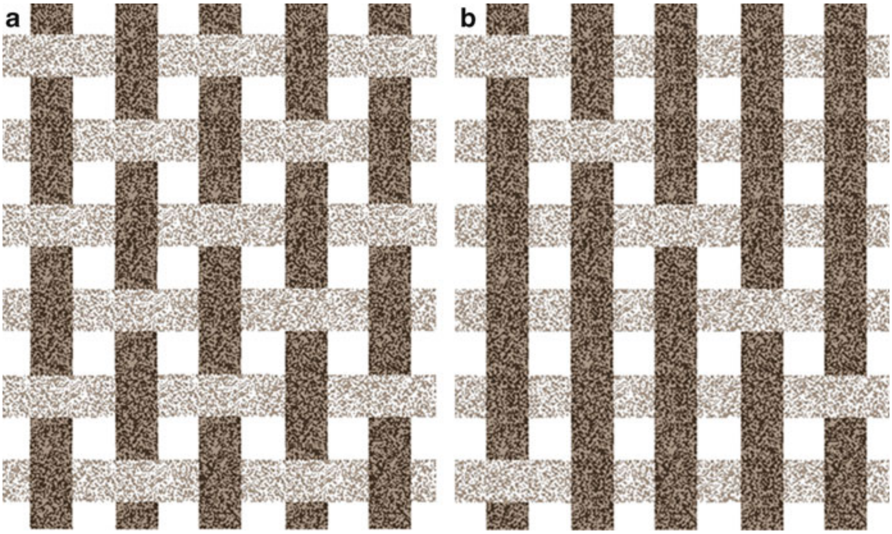

**Fig. 8.5** Two-dimensional (2D) fabric: (**a**) plain weave; (**b**) five-harness satin

directions, as many as 5, 7, or 11 are possible, which give a highly isotropic final composite (McAllister and Lachman 1983). A five-harness satin weave, shown in Fig. 8.5b, has one warp yarn running over four fill yarns and under one fill yarn.

The precursor, processing, and high temperature involved all influence the carbonaceous matrix significantly. Table 8.1 summarizes the room-temperature properties of some carbon/carbon composites. The 1D values refer to a high-modulus carbon fiber, a final heat treatment at 1,000 °C, four to six densification cycles, and a fiber volume fraction of 55. The properties of a woven carbon fabric depend on the weave pattern and the amount of fibers in the *x*, *y*, and *z* directions. Figure 8.6 shows schematically the stress-strain curves, at room temperature, of 1D, 2D, and 3D carbon/carbon composites (Fritz et al. 1979). The fracture mode changes from semibrittle (1D) to nonbrittle (3D). The latter is due to the existence of the continuous crack pattern in the composites. Because carbon/carbon composites are meant for high-temperature applications, their thermal expansion behavior is of great importance. As expected, fibers control the thermal expansion behavior parallel to the fibers, while perpendicular to the fiber axis the carbonaceous matrix controls the expansion behavior. The amount of porosity in the matrix will also influence the thermal expansion behavior.

### 8.3.1 Thermal Properties

Carbon/carbon composites can have high thermal conductivity, especially if mesophase pitch-based carbon fibers which have highly oriented carbon fibers

**Fig. 8.6** Stress–strain curves (schematic), at room temperature, of 1D, 2D, and 3D carbon/carbon composites (from Fritz et al. 1979, used with permission). The fracture mode changes from brittle (1D) to nonbrittle (3D)

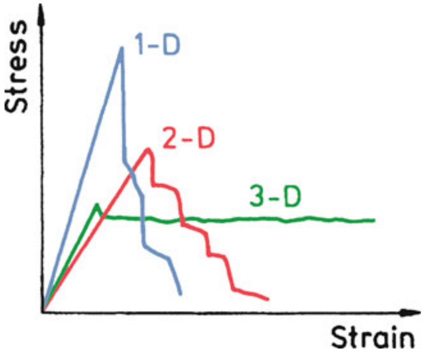

**Table 8.1** Mechanical properties of carbon/carbon composites at room temperature

| Weave | Flexural strength (MPa) | Young's modulus (GPa) | Interlaminar shear strength (MPa) |
|---|---|---|---|
| 1D, 55 % $V_f$ | 1,200–1,400 | 150–200 | 20–40 |
| 2D, 8 H/S weave, 35 % $V_f$ | 300 | 60 | 20–40 |
| 3D, felt, 35 % $V_f$ | 170 | 15–20 | 20–30 |

*Source:* Adapted with permission from Fritz et al. 1979

with a graphitic structure are used. The reader should recall that these composites show highly anisotropic properties, high thermal conductivity along the fiber and low conductivity in transverse direction. This means that one must have proper fiber configuration to obtain the desired thermal conductivity.

### 8.3.2 Frictional Properties of the Composites

Carbon/carbon composites have good tribological properties, in addition to adequate strength, good toughness, and high thermal conductivity. These characteristics make it possible to use them in applications such as bearing seals, electrical brushes, and brakes for aircraft and automobiles.

### 8.3.3 Ablative Properties

Space vehicles, on reentry into the atmosphere, are subjected to intense aerothermal loads (external surface pressure, skin friction, and aerodynamic heating). Intense heating occurs which leads to extremely high temperatures. We use thermal protection systems (TPS) made of suitable materials in order to keep space vehicle and occupants and equipments safe. Ablative TPS is commonly used because it is quite effective in expending thermal energy. In an ablative TPS, we sacrifice the material and thus absorb the thermal energy. There is a variety of ablative materials available. The one of interest to us here is the high temperature ablative material of which C/C composites are a prime example. They are used in manned space vehicles, hypersonic vehicles, and missiles.

Please see problem 8.6 at the end of this chapter about the Columbia shuttle disaster which involved C/C composites.

**Fig. 8.7** A three-dimensional carbon/carbon frustum (courtesy of Fiber Materials Inc.)

## 8.4 Applications of Carbon/Carbon Composites

Major applications of carbon/carbon composites involve uses at high temperatures, for example, as heat shields for reentry vehicles, aircraft brakes, hot-pressing dies, and high-temperature parts such as nozzles. Figure 8.7 shows a fully processed 3D carbon/carbon frustum. Hot-pressing dies made of carbon/carbon composites are commercially available, while brake disks are used in conventional aircraft, although their first use in civilian aircraft occurred in the Concorde supersonic aircraft. Brakes for racing cars and some high end luxury cars are the latest entry. Heat shields and nozzles are made of multidirectionally reinforced carbon/carbon composites. As pointed out, carbon/carbon composites can withstand high temperatures in an inert atmosphere. Lack of oxidation resistance is a major problem, and a great deal of effort has been put into the development of oxidation-resistant coatings for carbon fibers, with SiC coating being the primary coating material.

Other applications of carbon/carbon composites include their use as implants and internal repair of bone fractures because of their excellent biocompatibility. They are also used to make molds for hot pressing. Carbon/carbon molds can withstand higher pressures and offer a longer use life than polycrystalline graphite.

### *8.4.1 Carbon/Carbon Composite Brakes*

Brakes of any kind use friction between contact surfaces to stop a moving component. In the process, the kinetic energy of a moving object is converted into heat, which needs to be absorbed by the brake materials. Most commonly, the essential components of a braking system are: a rotating part (such as a wheel, axle, disk, or brake drum) and a stationary part that is pressed against the rotating part to slow or stop the moving object. The transmission of kinetic energy from the moving vehicle to heat in the brake pads generates high temperatures in the friction pad material and brake components. Efficient brake systems must be able to withstand the thermal shock, dissipate heat quickly (i.e., high thermal conductivity or more appropriately high thermal diffusivity) and maintain consistent braking performance. Any brake design must consider the following items (Awasthi and Wood 1988):

- Friction members must generate stopping torque (over a range of environmental conditions: rain, snow, dust storms, etc.).
- Heat sinks must absorb the kinetic energy of the aircraft.
- Structural elements should be able to transfer torque to the tires.

Brakes are one of the major applications of carbon/carbon composites. An example of a carbon/carbon brake assembly used on a Boeing 767 plane is shown in Fig. 8.8. Brakes, aircraft or automobile, have the following general requisites (Awasthi and Wood 1988):

(a) Oxidation resistance
(b) High thermal capacity
(c) Good strength, impact resistance, strain to failure
(d) Adequate and consistent friction characteristics
(e) High thermal conductivity

When the brakes are applied, multiple-disk brakes with alternating rotors and stators are forced against adjacent members by hydraulic pressure. Friction between rotating and stationary disks causes them to heat up to 1,500 °C (surface temperature can be as high as 3,000 °C) over a short time period, i.e., one needs excellent thermal shock resistance. In commercial processing of carbon/carbon brakes, a fabric made of woven carbon fibers with fiber tows inserted in the thickness (or $z$-direction) direction is used.

**Fig. 8.8** A carbon/carbon brake assembly that is used on a Boeing 767 airplane (courtesy of Alied-Signal Corp.)

In view of the requisites listed above, any braking material must be a good structural material, an efficient heat sink, and must have excellent abrasion resistance. Let us consider an example from the aircraft field which will help illustrate the formidable demands made on a braking material. Many civilian aircraft use carbon/carbon composite brakes. Let us take Boeing 767 as an example to help illustrate the formidable demands made on a braking material. In the event of an aborted takeoff, an extremely large amount of kinetic energy must be dissipated in about 30 s by the eight brakes on the aircraft (see the example below for an estimate of the temperature rise in an RTO). A rejected takeoff (RTO) is, indeed, the worst case scenario but then the braking material must be able to meet such requirements. It is estimated that about 3,000 RTOs occur every year in the USA.

The weight savings that result from replacement of conventional brakes by carbon/carbon brakes helps one understand why the aircraft industry has taken up the C/C brakes (Awasthi and Wood 1988). In a large aircraft, a conventional multiple stator and rotor arrangement (a sintered high friction material sliding against a high temperature steel) weighs about 1,100 kg. Carbon/carbon brakes (both the stator and the rotor being made of carbon/carbon composite) weigh about 700 kg, resulting in a weight savings of 400 kg. Also, as a braking material, carbon/carbon composites are superior to high strength bulk graphite. Carbon/carbon composites have much higher strength at the same density.

The Formula 1 racing cars usually use a new set of brake linings for each race. Aircraft brakes, on the other hand, are required to survive 2,000–4,000 landings.

An Airbus A380 has 16 sets of brakes; each brake costs something between US$ 35,000 and 45,000. However, use of carbon/carbon composite brakes results in weight savings relative to the conventional steel brakes; 250 kg on a Boeing 737. This allows more payload and/or or fuel savings.

*Example* One of the functions of the brake system is to absorb the heat energy developed during braking. Consider the Boeing 777 plane. Find out its mass and takeoff speed from the Web. Compute the kinetic energy of the plane when it is about to takeoff. Assume that the plane has carbon/carbon brakes. The pilot is asked to abort the takeoff for some reason. Under these conditions, assuming that all of the kinetic energy is converted to thermal energy, compute the temperature rise in this aborted takeoff.

**Answer** From the website of Boeing, we find that the maximum takeoff weight of a Boeing 777 plane is 247,000 kg. The takeoff speed can range from 270 to 345 km/h. Let us take a speed of 78.6 m/s. Putting these values in to the equation for kinetic energy

$$K.E. = \frac{1}{2}mv^2,$$

where $m$ is the mass of the plane and $v$ is its velocity. Substituting the values of mass and velocity in the above expression, we get the kinetic energy to be 763 MJ.

This is the kinetic energy that is converted into heat and must be absorbed in the plane during a rejected take off. To find the change in temperature, we use the following relationship

$$Q = \Delta T C_p m,$$

where $Q$ is the kinetic energy converted to heat, $\Delta T$ is the change in temperature, m is the mass of the brakes, and $C_p$ is the specific heat of the carbon brakes. The mass of the brakes is 700 kg. The specific heat of the carbon brakes is $1.6(\mathrm{kJ/kg} \times \mathrm{K})$. Putting all these values in the above expression, we get a change in temperature of 954 °C.

### 8.4.2 Other Applications of Carbon/Carbon Composites

Among other aerospace applications of carbon/carbon composites, one may cite their use in rocket propulsion components and reentry thermal protection of a spacecraft. Figure 8.7 shows the frustum of a cone of a rocket made of a 3D carbon/carbon composite. A 3D composite has fibers in $x$, $y$, and $z$ directions. The top layer in contact with the high temperature environment gets eventually sacrificed. Among nonaerospace applications, there are examples of use of carbon/carbon composites as implants as well as internal fixation of bone fractures because of their excellent biocompatibility. They are also used for making molds for hot pressing. Carbon/carbon molds can withstand higher pressures and offer a longer life than polycrystalline graphite. Figure 8.9 shows an example of a mold made of carbon/carbon composite that replaced a conventional heat resistant tool steel material. In general, the high cost of carbon/carbon composites limits applications to aerospace and other specialty applications. The limitation of low oxidation resistance is a serious one, and most applications do need some sort of protection against oxidation.

**Fig. 8.9** A mold made of carbon/carbon composite (courtesy of SIGRI)

### *8.4.3 Carbon/SiC Brake Disks*

This is new type of brake, developed by SGL Co. The matrix consists of silicon carbide (SiC) and elemental silicon (Si). The reinforcement fiber is carbon (C). Silicon carbide provides the hardness required for wear resistance while the carbon fibers provide the fracture toughness. The strain at fracture of such C/SiC composites ranges from 0.1 to 0.3 %, which is quite high for ceramics. The low weight, high hardness, resistance to thermal shock, and nonbrittle fracture behavior make for an excellent braking material.

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## Problems

8.1. The terms voids and cracks are frequently used interchangeably but in reality they are not synonymous. Specifically, in regard to C/C composites, distinguish between voids and microcracks in terms of their form and origin.
8.2. TEOS is often used as a glass forming sealant to heal the microcracks in C/C composites. However, there are some limitations on efficiency. Explain what the limitations are and why.
8.3. Invariably C/C composites will need to be joined to other conventional materials. Describe the different joining approaches that can be used to accomplish this; give the pros and cons of each technique.
8.4. C/C composites are frequently made of carbon fibers woven in more than three directions. What effect does this have on crack propagation, fracture surface appearance, and toughness of C/C composite?
8.5. Glass-forming sealants help close the crack-like defects in C/C composites. Are there any deleterious effects of such sealants?
8.6. Describe the NASA's Columbia shuttle disaster in which all seven astronauts lost their lives in 2003. Highlight the role of carbon/carbon composites in this disaster. How and why did the disaster occur? Describe the modifications instituted by NASA to prevent such a disaster in the future.