

# Chapter 15

## C/C and C/SiC Composites for Aerospace Applications

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**Abstract** This chapter deals with different aspects of the carbon fibre-reinforced carbon composites (C/C) and carbon fibre-reinforced silicon carbide composites (C/SiC), especially for aerospace applications. The reinforcement and matrix materials and the process technologies developed for these composites are discussed. Typical mechanical and thermal properties at room and high temperatures are also presented, together with some actual and potential aerospace applications. Some products developed in India are also included.

**Keywords** Carbon matrix composites · Processing · Properties · Applications

### 15.1 Introduction

Carbon fibre reinforced carbon matrix (C/C) composites and carbon fibre reinforced ceramic matrix composites (C<sub>r</sub>CMC) offer some significant advantages over conventional high temperature materials for aerospace applications [1–7]. The development of C/C composites started in 1958, with US Air Force sponsored work, which later received significant impetus from the Space Shuttle programme. The C/C composite technology remained as guarded secret technology for the USA and European countries until other industrial applications were explored. Currently,

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many countries, including India, have their own technologies with manufacturing units in civilian and military sectors.

The C/C composites possess high thermal stability, high specific strength, high stiffness, high thermal conductivity, low coefficient of thermal expansion, high fracture toughness and good fatigue and creep resistance. Also, these composites maintain good mechanical properties, including frictional properties, over the entire operational temperature range [1, 3, 4, 7]. The combination of these properties makes them the preferred materials for different aerospace structural components like ailerons, flaps, landing-gear doors and other structural parts. However, they have a major drawback, the lack of stability above 500 °C in oxidizing environments. This limits their application in such environments to short durations [4, 8, 9]. On the other hand, these composites can withstand very high heat fluxes for short durations, making them suitable for re-entry nose tips and leading edges of ballistic hypersonic missiles [2, 9].

In order to make C/C composites suitable for extended durations and repeated use, the oxidation resistance is improved by (i) oxidation-resistant coatings, or (ii) modifying the composite matrix by adding third phase oxidation-resistant ceramic fillers or (iii) converting the carbon matrix to carbides like SiC. The latter type of composite, carbon fibre reinforced SiC matrix composites, are called C/SiC composites.

The C/SiC composites possess most of the good properties of the C/C composites and in addition have better oxidation and erosion resistance. Moreover, the density of the carbon is lower than the density of many metallic and non-metallic materials, and this makes them ideal for lightweight applications.

## 15.2 Carbon Reinforcements

The reinforcements (usually fibres) are the mainstay of any composite product. The constituents in C/C (and C/SiC) composites have distinctly different structures. In C/C composites each constituent can range from non-crystalline carbon to semi-crystalline, or highly crystalline graphite. Thus by varying the starting materials and processing, composites can be made with a wide range of structures and properties.

### 15.2.1 Carbon Fibre Reinforcements

Since the processing temperature of C/C and C/SiC composites is very high, the fibres must not degrade during processing. This means that the fibres should be initially produced at a higher temperature than the subsequent composite processing temperature.

Carbon fibres can be produced from different precursors and are in this respect classified into the following categories [3, 4]:

- Rayon-based carbon fibres
- PAN-based carbon fibres
- Pitch-based carbon fibres
  - Mesophase pitch-based carbon fibres
  - Isotropic pitch-based carbon fibres
- Gas-phase-grown carbon fibres.

However, on the basis of the final heat treatment temperature, the fibres can be broadly categorized in the following three categories:

1. **Type-I.** These fibres are called high-heat-treatment carbon fibres (HTT), where the final heat treatment temperature should be above 2000 °C. These fibres have a high elastic modulus.
2. **Type-II.** These fibres are called intermediate-heat-treatment carbon fibres (IHT), where the final heat treatment temperature should be around 1500 °C. These fibres have high strengths.
3. **Type-III.** These fibres are called low-heat-treatment carbon fibres, where the final heat treatment temperature is around 1000 °C. These fibres have low modulus and strength.

The properties of the different fibre grades depend upon the precursor, the processing technology and the final heat treatment temperature.

Table 15.1 lists representative properties of the commercially available carbon fibre grades taken from the website of M/s Troyaca and M/s Toray. These data are shown only to illustrate the wide property ranges of the fibres, and consequently may vary slightly from the actual values. **N.B:** It is always necessary to obtain certified properties from the manufacturers for design use.

**Table 15.1** Properties of carbon fibres (extracted from the website of M/s Troyaca and M/s Toray)

Trade name	Density (g/cm <sup>3</sup> )	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation (%)	CTE (m/m°C)	Thermal conductivity (Cal/cm s°C)
T300	1.76	3530	230	1.5	$-0.41 \times 10^{-6}$	0.0250
T700S	1.80	4900	230	2.1	$-0.30 \times 10^{-6}$	0.0224
T800S	1.80	5880	294	2.0	$-0.56 \times 10^{-6}$	0.0839
T1000G	1.80	6370	294	2.2	$-0.55 \times 10^{-6}$	0.0765
M40 J	1.77	4410	377	1.2	$-0.83 \times 10^{-6}$	0.1640
M50 J	1.88	4120	475	0.8	$-1.10 \times 10^{-6}$	0.3720
M60 J	1.93	3920	588	0.7	–	–

CTE coefficient of thermal expansion

### 15.2.2 Other Carbon Reinforcing Materials

Although most of the high performance composites use carbon fibres, other forms of carbon are also used as secondary reinforcements:

**Graphite whiskers:** These are pure graphite single crystals having a high degree of crystal perfection and possessing very high strength [10]. The whiskers can be used as a secondary reinforcement in combination with the primary reinforcement of carbon fibres. However, handling and uniform dispersion of the graphite whiskers may require additional precautions and skill.

**Carbon Black:** Carbon black is available in the form of spheres with fused aggregates below 1  $\mu\text{m}$  size. It is produced by the combustion of hydrocarbons under oxygen-deficient conditions. Carbon black can be used as filler in the C/C composite matrix [4] to reduce the number of coal tar infiltration cycles (discussed in subsection 15.4.2). However, its use is mainly dictated by the desired properties of the final composite.

**Carbon nanotubes:** Owing to their unique properties, carbon nanotubes (CNTs) are potentially an ideal reinforcement candidate. Theoretical and experimental results have shown some unusual mechanical properties of CNTs, with Young's modulus as high as 1.2 TPa and tensile strength 50–200 GPa [11]. These features make CNTs an attractive option for secondary reinforcing materials in C/C or C/SiC composites. However, CNTs have been used mostly as fillers in experimental nanocomposite materials to enhance their properties [12, 13].

**Carbon nanofibres:** Carbon nanofibres are non-continuous filaments that differ from conventional carbon fibres with respect to both their dimensions and mechanical properties. Carbon nanofibres are produced using catalytic chemical vapour deposition (CVD) as well as by a combination of electrospinning of an organic polymer and thermal treatment [14, 15]. The diameter of these fibres is reported to be in the range of 100 nm and their aspect ratio is reported to exceed 100. They could become of great practical—as well as scientific—importance, owing to their combination of high specific area, flexibility and mechanical strength.

### 15.3 Carbon Fibre Preforms

Development of fibre scaffolding with carbon fibres orientated in specific amounts and directions is called the preforming process. The preform enables the subsequent composite product to have the desired strength in different directions. Since carbon fibres are also thermally anisotropic, the preforms should be designed such that the composite will absorb all the thermal stresses during processing and result in a delamination-free product. Also, multidirectional preform fabrication technology provides the means to produce a tailored and near-net shape composite that meets

the directional thermomechanical property requirements of the final composite product.

The preforming technology employs multidisciplinary approaches of structural engineering, mechanical engineering and textile technology to develop preforms. The preforms can be simple blocks, cylinders, conical contours, surfaces of revolution and complex geometries and shapes. They can be made using layers of conventional fabrics with dry yarns pierced through them in the third direction, or assembling semi-cured/pre-cured yarns (pre-pregs), using manual, semi-automated or automatic machines. An illustration of some complex preform schemes [1, 4, 16, 17] is given in Fig. 15.1. Some of these, and others, are briefly described in the following paragraphs.

**UD preforms:** These are the simplest preforms and are obtained by arranging either dry or resin-impregnated fibre tows (bundles of fibres) parallel to each other and employing pultrusion or filament winding in combination with a polymeric binder to obtain the required shape and volume fraction of fibres. The polymeric binder can be converted into the carbon matrix by heating the preform under inert atmosphere above 1000 °C.

These initial products are porous and can be densified using the standard C/C composite processing techniques described in Sect. 15.4.

**2-D preforms:** These are fabricated by stacking carbon fibre cloth in a suitable fixture, mostly in the dry condition. The stack is subjected to further processing along with the fixture till the preform acquires a stable shape and rigidity suitable for handling. The 2-D preforms can have different stacking arrangements where

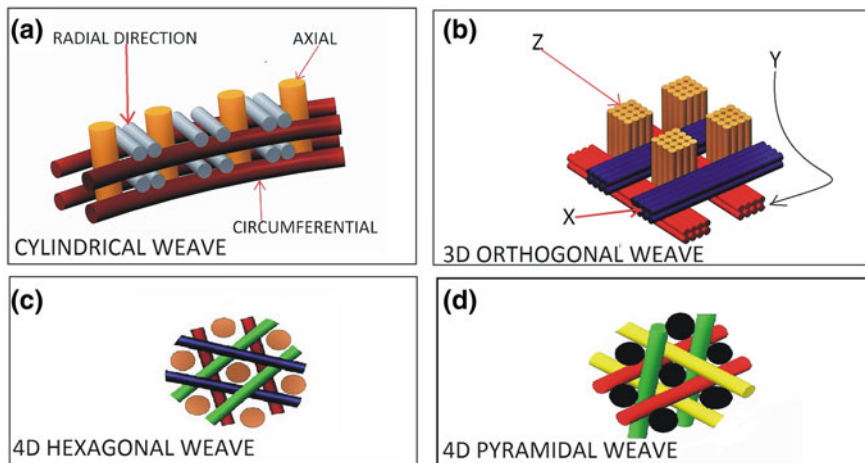


Fig. 15.1 Some typical schemes for developing multi-directional carbon fibre preforms

fibre tows are arranged in a regular fashion with different combinations of fibre orientations.

This type of preform provides good mechanical and thermal properties in two directions parallel to the fibre axes, but the composite properties are poor in the through-thickness direction. Also, 2-D preforms are somewhat susceptible to delamination during pyrolysis. However, the delamination problem can be overcome by using an optimum resin system and optimum heating cycle for the given fibre volume fraction,  $V_f$  or by making 2.5-D preforms.

**2.5-D preforms:** The delamination problem in 2-D preforms can be eliminated by stitching the carbon fabric stack with carbon fibre tows. Since the  $V_f$  of the stitching fibres is relatively low compared to the other main direction fibres, such preforms are called 2.5-D. Stitched preforms are suitable for many C/C and C/SiC composite products. These preforms are used for general applications owing to their straightforward fabrication process [18].

**3-D preforms:** 3-D orthogonal preforms are fabricated using multiple fibre tow bundles that are appropriately located within the structure in three mutually perpendicular directions. In each direction the fibre bundles are straight in order to obtain their maximum structural capability. This type of preform obviously can provide greater isotropy compared to UD, 2-D and 2.5-D preforms. However, the strengths of 3-D preforms are relatively low. 3-D preforms of different shapes and sizes can be made using automated machines.

3-D preforms are used for applications like rocket nozzles and hypersonic vehicle leading edges. 3-D cylindrical weave pattern based preforms are used for some near net conical or cylindrical shapes, but they have the disadvantage of non-uniform  $V_f$  across the thickness, and this affects the mechanical and thermal properties.

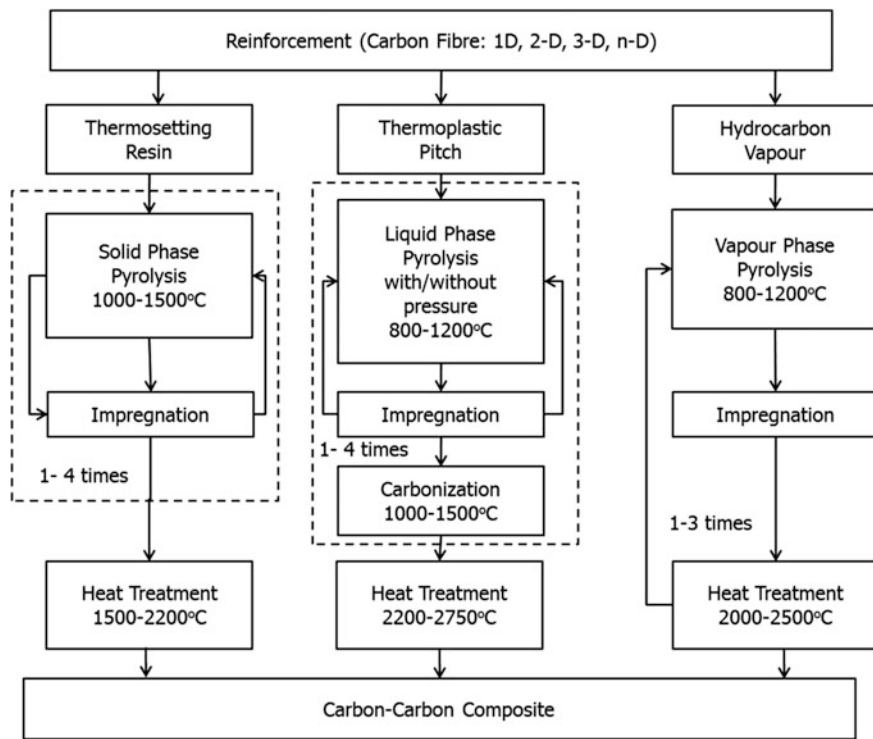
**4-D preforms:** These are modified versions of 3-D preforms that achieve even better isotropy, but at the cost of lower  $V_f$  and properties. 4-D preforms are made by introducing diagonal yarns between the main fibre planes.

In addition to 4-D preforms, 5-D and 7-D can also be developed. 4-D and 5-D preforms are mostly used when greater isotropy is required for re-entry missile nose tips and for throat inserts in rocket nozzles.

## 15.4 C/C Composites Processing

Carbon has a high melting temperature (above 3000 °C), and so the fabrication techniques used for polymer matrix or metal matrix composites cannot be used for C/C composites. Many research articles have been published on the processing of C/C composites, but the actual knowledge of fabrication—for both C/C and C/SiC composites products—remains proprietary and exclusive to a few research institutions.

A general flow diagram of C/C composite processing is shown in Fig. 15.2 [3, 19]. The processing is based either on vapour phase pyrolysis of some selected



**Fig. 15.2** Schematic layout of typical methods used to fabricate C/C composites from different reinforcement materials

hydrocarbons, or pyrolysis of solid/liquid carbonaceous precursors. The selection of the processing route and suitable precursor for the matrix development are the most important parameters [1, 4, 19]. The carbon matrix precursor materials can be divided into two categories:

1. Gaseous precursors, usually hydrocarbons such as methane and propane.
2. Liquid precursors (carbonaceous resins). The liquid route can be further categorized as
  - Aromatic thermosetting resins like phenolics and furfural.
  - Thermoplastic resins, including pitch material obtained from coal or petroleum.

The process route selection depends mostly on the availability of the processing equipment and types of preforms. For example, UD and 2-D based preforms are generally processed using thermosetting resin systems. 2.5-D and 3-D preforms of limited thickness are processed using chemical vapour deposition or infiltration processes (CVD/CVI), while thicker products of 3-D, 4-D and 5-D composites are processed using pitch impregnation.

The following factors should be taken into account while selecting the matrix precursor:

- The solid carbon yield of the precursor must be high. This minimizes the number of densification (infiltration and pyrolysis) cycles. It also helps in controlling delamination particularly in 2-D composites.
- The liquid or molten matrix precursor must readily wet the carbon fibres.
- The molten precursor should have a low viscosity to aid penetration into the preforms.
- On pyrolysis, the resulting matrix should (obviously) acquire a favourable microstructure for optimum mechanical and thermal properties.

### 15.4.1 Chemical Vapour Impregnation (CVD/CVI)

This processing route has been widely used by Western countries for the production of C/C composite thinner parts like aircraft brake discs and extendable rocket nozzle cones. The chemical vapour impregnation (CVI) technique evolved from chemical vapour deposition (CVD), which was generally used to obtain uniform coatings or films with tailored composition by decomposition of gaseous compounds. In this chapter the CVD/CVI term has been used for the process where entire matrices are developed by infiltrating fibre preforms with volatile hydrocarbon matrix precursors such as methane, propane, benzene and other similar low molecular weight hydrocarbons. Thermal decomposition of the hydrocarbons takes place on the heated surfaces of the carbon fibre preforms, resulting in deposition of pyrolytic carbon. This technique can be used to deposit carbon onto dry fibre preforms or on partially densified C/C composite structures produced by the liquid impregnation route [3, 4].

There are several variants of the CVD/CVI processes. These are summarized in the following paragraphs.

**Isothermal CVD/CVI:** This process is the most widely used for fabricating C/C composite products. The carbon fibre preform is positioned in a vacuum furnace having a gas management system. The temperature of the preform is raised to within the range of 700–1000 °C, and the precursor gases are passed through the preform. For CVD the pressure in the furnace is maintained in the range 10–100 mbar, while for CVI the pressure should be lower.

The gases decompose and solid carbon is deposited over the fibre surfaces and between the fibre bundles. In general, the deposition is higher at the surface of the preform than in the core, since surface deposition hinders diffusion of the gaseous precursors into the core. The result is non-uniform through-thickness deposition and an overall low density composite with closed porosity.

To achieve uniformly dense composites, the pores at the surface need to be opened up by intermittent surface machining, i.e. the CVD/CVI process is discontinuous. The final density is usually in the range 1.5–1.7 g/cm<sup>3</sup>. Even so, a



density gradient usually remains when using *isothermal* CVD/CVI. For thin sections this type of the process is acceptable, but for somewhat thicker sections thermal gradient CVD/CVI is recommended.

**Thermal gradient CVD/CVI:** For this process a known temperature gradient is maintained from one side of the preform to the other. The side where the precursor gases enter the preform is maintained at a lower temperature, while at the other side the temperature is maintained high enough to allow deposition, see Fig. 15.3.

As deposition continues the temperature front shifts towards the entry-point of the precursor gases, such that the whole preform is uniformly densified. The densification rate is improved by applying (i) a pressure gradient to the preform, i.e. by replacing slow diffusion mass transfer by the much faster convection mass transfer within the pore network, or (ii) an inverse temperature gradient [20, 21]. Another efficient way to increase the densification rate is to immerse the heated fibre preform in a boiling liquid precursor under reflux (calegation process) [21, 22]. However, design of the equipment and precise temperature control is a challenging task which needs to be carefully considered while selecting this process.

For still thicker C/C composites, the liquid impregnation process is recommended.

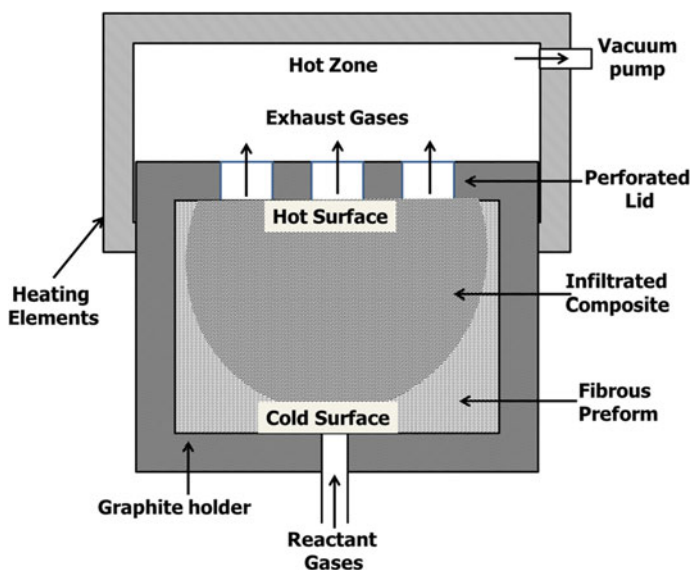


Fig. 15.3 Schematic of the thermal gradient CVI process

### 15.4.2 *Liquid-Phase Impregnation Process*

This process is based on impregnation with coal tar/petroleum pitches or high-char-yielding thermosetting resins into the fibre preforms, followed by pyrolysis. Figure 15.2 shows the schematic of the C/C composite manufacturing process using multiple impregnation; carbonization (1000 °C); high pressure (about 1000 bars) carbonization (HIP); and graphitization (up to 2750 °C).

Although it is possible to densify the C/C composite even by atmospheric pressure carbonization, the process requires several cycles of impregnation and carbonization because the carbon yield of the pitch is only around 50 % if pyrolysed at atmospheric pressure.

To reduce the total processing time, high pressure carbonization is recommended since the yield of the coal tar pitches is reported to be 80–90 % if pyrolysed at 750–1000 bars. Not only is the process time reduced, but high pressure helps in lowering the temperature of the mesophase formation in the pitch, resulting in a highly oriented crystalline structure of the matrix. The higher pressure carbonization also results in a coarser and isotropic microstructure, since it suppresses gas formation and prevents its escape, and therefore results in a higher char yield. In fact, the HIP process is the only practical route to lower the production costs of C/C composites.

## 15.5 Properties of C/C Composites

C/C composites for aerospace applications need to be characterized for general properties like strength and stiffness, fracture toughness, thermal conductivity and CTE, and for specific functional properties depending on the service requirements. For example, the composite needs to be characterized for frictional properties if it is intended to be used for brake pads, and ablation and erosion resistance if it is used for propulsion applications.

A large data base is available in the literature covering mechanical, thermal, electrical, frictional and fatigue properties of C/C composites. The effects of fibre type,  $V_f$ , preform type and the process route have also been reported. The mechanical properties of C/C composites are controlled by the properties of the constituents and their volume fraction bonding, and crack propagation mechanisms, whereas the thermal properties are governed by thermal transport phenomena. Moreover, both the fibres and matrix are likely to undergo a change in properties during processing, depending on the final heat treatment temperature, preform type and the resulting thermal stresses. It is therefore very difficult to compare the properties reported in different studies. However, characteristic and more or less representative properties are discussed in Sects. 15.5.1. and 15.5.2.

### 15.5.1 Mechanical Properties of C/C Composites

Extensive work has been done to enhance the fibre properties in these composites. The strength and stiffness of C/C composites are dominated by the type of fibre and preform. These composites are complex owing to many changes (physical and chemical interactions) that occur during processing. Broadly speaking, the properties change with the combined effect of the following factors:

- Fibre types: precursor, heat treatment temperature limit.
- Matrix precursors: resin, pitch, CVD/CVI or hybrid.
- Preforms: type of weave, and number of fibre directions, etc.
- Fibre volume fraction in a particular direction.
- Final density of the composite.

In general, C/C composites having strong fibre/matrix bonding fail catastrophically without fibre pull-out, while those having a desired interface fail in a mixed tensile and shear mode, resulting in high strength [1, 4, 19, 23].

Differences in the interfacial bonding, owing to residual stresses, are considered to be the prime factor for the different tensile strengths of 2-D and 3-D C/C composites. The residual stresses are induced mainly due to the following reasons:

1. Carbon fibres have different CTEs along the axis and in transverse directions. Along the axis the CTE is close to  $0.1 \times 10^{-6}/^{\circ}\text{C}$ , while in the transverse directions it is about eight times higher. The difference between these two CTEs varies slightly, depending on the fibre type and its thermal history.
2. Shrinkage of the matrix during the carbonization and graphitization stages. The as-infiltrated polymeric resin or pitch precursor has a density of the order of  $1.0 \text{ g/cm}^3$  which converts to a higher density (about  $2.0 \text{ g/cm}^3$ ) carbon matrix during pyrolysis. If the fibre preform does not allow uniform shrinkage during pyrolysis, then microcracks and residual thermal stresses occur. Also, the amount of microcracks and residual stresses will depend on the fibre and preform types.

Owing to the above factors, even composites having the same raw materials and processing route yield different properties depending on the preform types.

Thermal residual stresses in 3-D composites are reported to be much higher than those in 2-D composites [24]. Thus for 3-D composites, the cracks would be expected to be much larger and more numerous, and the interfacial strength much weaker, than in 2-D composites. Hence 3-D composites have lower strength than 2-D composites. Also, the properties of UD C/C composites are reported to be higher than those of 2-D and 3-D composites. A typical set of properties for UD C/C composites made with high strength and high modulus fibres is shown in Table 15.2 [25].

**Table 15.2** Typical properties of UD C/C composites [25]

C/C properties	Parallel to the fibre axis		Perpendicular to the fibre axis	
	High strength	High modulus	High strength	High modulus
Flexural strength (MPa)	1250–1600	825–1000	20	30
Tensile strength (MPa)	600	572	4	5
Tensile modulus (GPa)	125	220	–	–
Compressive strength (MPa)	285	380	25	50
Shear strength (MPa)	20	28	–	–
Work of fracture (kJ/m <sup>2</sup> )	70	20	0.4	0.8

### 15.5.2 Thermal Properties of C/C Composites

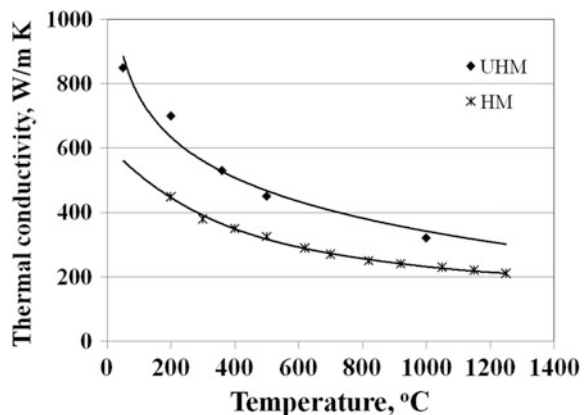
The complexity of C/C composites makes estimation of their thermal properties difficult. The thermal conductivity depends more on the crystalline nature of the fibres than the type of fibres. The thermal conductivity dependence of C/C composites on temperature is shown in Fig. 15.4. This shows that the thermal conductivity of C/C composites made with UHM (highly crystalline high modulus fibres) is higher than that of those made with HM (high modulus) fibres.

## 15.6 Example Applications of Aerospace C/C Composites

### 15.6.1 C/C Composite Brake Pads

Carbon in the form of graphite is a well-known solid lubricant. It is perhaps surprising that such a material can also be a promising friction material for a

**Fig. 15.4** Thermal conductivity of UD carbon-carbon composites made with different pitch based carbon fibres



**Fig. 15.5** Typical assembly of C/C brake discs



braking system. A typical C/C composite brake disc assembly is shown in Fig. 15.5.

The fact is that many of the characteristics that make carbon an attractive anti-friction material are equally required to make it an attractive friction material. Carbon, when rubbed against itself, exhibits a wide range of coefficient of friction ( $\sim 0.1$  to over  $0.5$ ) depending on the material type, load, speed and temperature [19, 26]. In comparison to steel, a carbon brake pad has low density ( $1/4$  of steel density), high heat capacity ( $2.5$  times that of steel), and a thermal conductivity almost equivalent to those of metals.

C/C composite brake pads rapidly conduct heat away from the rubbing surface and help in forming a smooth and stable wear controlling friction film: this is desirable to facilitate smooth braking and reduce the wear rate, which in turn increases the service life. In addition, carbon has a high thermal shock resistance owing to its high thermal conductivity and low coefficient of thermal expansion, and also does not soften at high temperatures. These properties make C/C composites ideal friction materials for brake design. Over  $70\%$  by volume of C/C composites are used as aircraft brake discs. M/s Dunlop, UK, provided the first major breakthrough for C/C composite brakes: these were introduced in the 1970s for the Concorde undercarriage. The Dunlop design with C/C composite brake discs saved more than  $600$  kg ( $\sim$ equivalent to seven passengers) as compared to steel brakes [27]. Many advanced tactical and commercial transport aircraft also use C/C composite brake materials [4, 26], as does the Indian Light Combat Aircraft (LCA).

### 15.6.2 C/C Nozzle and Throat

M/s SNECMA Propulsion Solide has developed C/C composite throat and lightweight exit cones for rocket nozzles [28]. The C/C composite exit cone concept significantly simplified the whole nozzle design. Lightweight C/C composite nozzles have significantly reduced the overall weight compared to phenolic nozzle

systems [28], which must be much thicker because they ablate and erode much more. Typical C/C composite nozzle and exit cone are shown in Figs. 15.6 and 15.7 respectively.

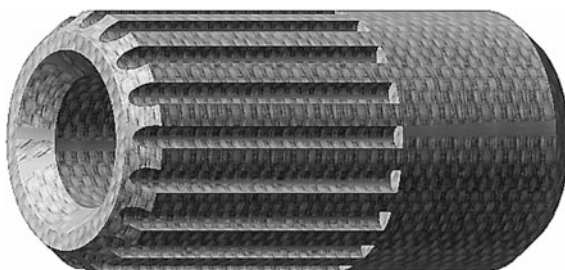
**Fig. 15.6** Typical C/C composite exit cone



**Fig. 15.7** A typical C/C composite nozzle test article



**Fig. 15.8** A typical model showing transpiration cooled article



### 15.6.3 C/C Combustion Chamber

Transpiration-cooled C/C composite combustion chamber test article has been demonstrated [6, 29]. This cooling method is used for high heat fluxes and long times. A typical model showing transpiration cooling is shown in Fig. 15.8.

In a transpiration cooling system the coolant is injected into the hot gas flow through a porous structure like a semi-densified C/C composite, as opposed to a discrete structure with film cooling. Transpiration cooling also decreases the heat flux to the structure, and this could benefit its useful life.

## 15.7 C/SiC Composites

C/SiC composites exhibit excellent thermo-erosive properties up to 2000 °C [17, 18, 30]. Their high strength-to-weight ratio and oxidation resistance make them ideal candidates for highly demanding engineering applications such as high performance heat shields, structural re-entry components, ultrahigh temperature heat exchanger tubes, rocket nozzles and brake discs.

The choice of silicon carbide as a matrix is based on its high melting point ( $\sim 2500$  °C), excellent mechanical properties at high temperatures related to its covalent character, relatively good oxidation resistance up to about 1500 °C, and stability in fast neutron environments [22, 31]. Also, silicon carbide can be easily inserted into a fibre preform by a variety of techniques, discussed in Sect. 15.8.

However, when used in C/SiC composites, the SiC matrix undergoes multiple microcracking when loaded in tension beyond stress levels of only 100–200 MPa [22]. The density and widths of the microcracks depend on the fibre architecture, the fibre/matrix bonding and the applied load. The role of the fibre/matrix interface becomes very important for C/SiC and other ceramic matrix composites, since the matrix tends to react with the carbon fibres. The interface and its usefulness in C/SiC composites are described next.



### 15.7.1 C/SiC Fibre/Matrix Interface/Interphase

Fibre/matrix interfaces have a strong influence on the mechanical properties and lifetime of the ceramic composites. The interfacial domain remains a critical area because load is transferred from the matrix to fibres (and vice versa) through the interface. The fibre/matrix interface is the key to tolerating stress-induced damage and enhancing the toughness in brittle matrix composites.

The essential property of a composite interface where the matrix has a lower failure strain than the fibres, is the ability to disbond in the presence of matrix microcracks. The concept of weak interfaces has been widely used to increase the fracture toughness of the composite. However, the weak interface may be detrimental to the composite strength: a high strength requires efficient load transfer from fibre to matrix, which requires a strong interface. Hence the requirements of tough composites and strong composites are opposite in nature, and this must be considered when choosing a composite for the desired properties.

The interface is a thin film (about 500 nm) of a compatible material having low shear strength, and which is coated onto the fibre surfaces before composite processing, whereby the matrix is either deposited by a gaseous route or infiltrated by a liquid route. The main function of the interface is to arrest and/or deflect matrix microcracks, hence protecting the fibres from premature failure. The interface should also help in transferring the loads, when necessary.

All intended requirements of the fibre/matrix interface may not be met by a thin single layer. In such cases multiple thin layers of the same or different materials have been used. Thus the interface in actual composites is better described as an interface zone of finite dimensions called an interphase. Within the interphase the mechanical properties differ from the fibre and matrix.

Different kinds of interphase concepts have been suggested and are shown in Fig. 15.9 [32–34]:

- (a) Single layer pyrocarbon (PyC) or hexagonal boron nitride (BN) interphases.
- (b) Porous SiC single layer interphase.
- (c) Multi-layered  $(X/Y)_n$  interphase, with  $X = \text{PyC}$  or  $\text{BN}$ , and  $Y = \text{SiC}$  (schematic).
- (d) Crack deflection in a multi-layered (PyC–SiC) interphase.

Layered crystal structures like PyC–BN or a layered microstructure, e.g.  $(\text{PyC–SiC})_n$  or  $(\text{BN–SiC})_n$ , have been proposed as the best interphase materials, with the layers deposited parallel to the fibre surface via CVD. On the one hand, these layers are weakly bonded to one another, but on the other hand the interphase bonds strongly to the fibre surface [32–34]. Crack deflection occurs within the interphase, preferably parallel to the fibre surface. However, as Fig. 15.9 suggests, this ideal crack deflection is not always achieved. Even so, the load transfer capacity is improved compared to simple fibre/matrix bonding when this bonding is weak.



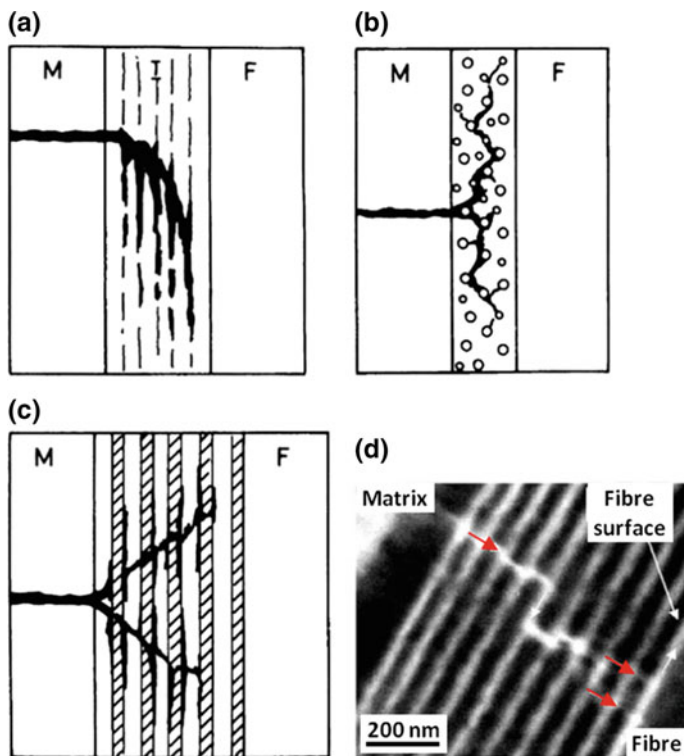


Fig. 15.9 Typical interphase schemes as described in [33, 34]

### 15.7.2 Oxidation

When C/SiC composites are exposed to an oxidizing atmosphere at medium or high temperatures, the matrix microcracks facilitate the depth-diffusion of oxygen towards the oxidation-prone interphases and fibres. Therefore the SiC matrix *and* the fibre/matrix interphase should be designed to hinder oxygen diffusion. The general guidelines are:

1. Introduction of boron-like elements in the SiC matrix. These form molten oxide phases over a wide temperature range, filling the cracks and acting as self-healing materials.
2. Proper design of the interphase to reduce the crack openings.

With respect to (1), boron oxide phases are efficient at relatively low temperatures (500–1000 °C) whereas silica-rich phases are more appropriate at high temperatures (1000–1500 °C). However, these solutions work only when the

environmental oxygen partial pressure is high enough to support passive oxidation. Then the molten oxide phases cover the surface of the composite and prevent further oxygen diffusion into the structure.

## 15.8 C/SiC Composite Processing

### 15.8.1 Chemical Vapour Impregnation (CVD/CVI)

As stated earlier, the solid matrix deposits on the preform fibre surfaces owing to a vapour phase reaction of the precursor gases under appropriate conditions. For CVI of C/SiC composites, methyltrichlorosilane (MTS) is generally used as precursor for SiC. The deposition of SiC takes place between 850–1200 °C in vacuum. The process requires several weeks of continuous deposition in order to obtain a dense C/SiC composite. There are several advantages of the CVD/CVI process:

1. The process temperature is lower than that of other processes, e.g. CVI can be accomplished at 800–1000 °C, whereas the process temperatures of liquid silicon infiltration (LSI) and polymer infiltration and pyrolysis (PIP) are in the range 1400–1600 °C.
2. Multi-directional preforms can be infiltrated, since gas can diffuse into any shape and size preform.
3. CVI results in better thermal and mechanical properties of the composites.

The CVD/CVI process yields mostly  $\beta$ -SiC deposits with controlled composition and microstructure. There are also some disadvantages: long process cycles; through-thickness density gradients; and requirements for accurate control of the precursor gas flow, temperature, pressure and handling of hazardous byproduct gases. It typically takes 40–60 h to infiltrate a 4–5 mm thick carbon preform to obtain residual porosity of the order of 15 %.

### 15.8.2 Polymer Infiltration and Pyrolysis (PIP)

The PIP process uses polycarbosilane (PCS), which is a ceramic precursor that converts into SiC if heated under an inert atmosphere or in vacuum above 1200 °C [35–39]. Polycarbosilane has also been used to fabricate continuous SiC fibres by melt spinning, followed by cross-linking and pyrolysis [40].

The PIP process has several advantages, including good matrix composition control, low densification temperature, and the possibility to join different parts of a complex-shaped component. There are a few disadvantages: multiple infiltration/pyrolysis/densification cycles (typically 10 cycles to achieve a density of 2.0 g/cm<sup>3</sup>) are required; local non-availability of the ceramic precursor in large

quantities; and large shrinkage of the matrix during pyrolysis, leading to matrix cracking. Also, interior cracks are difficult to fill during subsequent infiltration cycles.

The composites obtained by PIP have superior interfacial bonding, and this process has been proposed for making large C/SiC composite rocket nozzles. Such large products can be made by employing a variety of techniques, including filament winding, vacuum assisted resin transfer moulding, autoclave moulding, hydraulic press moulding or matched die moulding. Symmetrical as well as non-symmetrical composite components can be made.

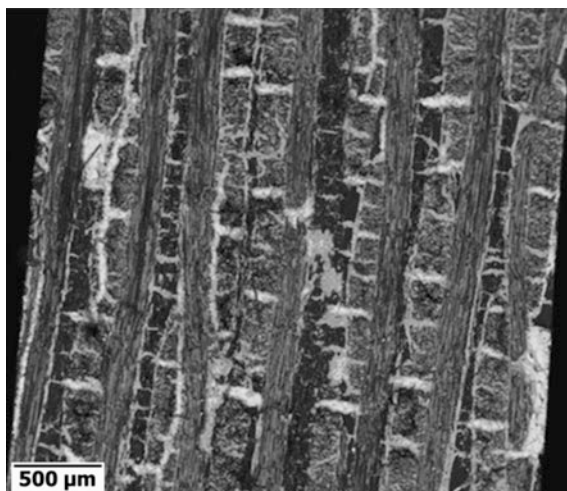
### 15.8.3 Liquid Silicon Infiltration (LSI)

Liquid silicon infiltration has been used to fabricate C/SiC composites for different applications. LSI is the most common and economical method to fabricate C/SiC composites of reasonable quality, especially for short-life components and thicker and complex shapes. Investigations of the LSI process have been carried out by many researchers [17, 18, 30, 41].

The processing by LSI consists of the infiltration of a porous C/C composite preform with molten silicon using a vacuum or inert gas furnace. Carbon and silicon react to form SiC at temperatures in the range of 1450–1650 °C. This process leads to the development of C/SiC composites with lower component fabrication time and therefore reduced component costs.

LSI-based, 3-D stitched, 3-D woven and 4-D C/SiC composite technology and product development work has been carried out extensively in India by Suresh Kumar et al. [17, 18, 42]. Several products have been realized using the process: of these, Jet-vanes for thrust vector control of rocket motors and a throat insert for a

**Fig. 15.10** Typical microstructure of an LSI-based C/SiC composite



liquid propulsion based rocket motor have been included in the C/SiC composite application Section, see Sects. 15.10.2 and 15.10.4.

A typical microstructure of an LSI-based C/SiC composite is shown in Fig. 15.10.

## 15.9 Properties of C/SiC Composites

Much work has been done in achieving the highest possible use of the fibre properties in C/SiC composites. Nevertheless the properties vary widely, depending on the manufacturing process and the test results from different R&D laboratories. This should not be surprising, since C/SiC composites are obviously heterogeneous, consisting of fibres, matrix and pores. Also, the fibres and matrix can have a variety of microstructures.

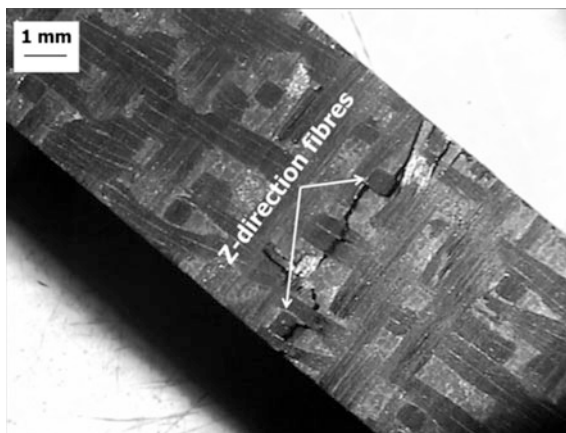
Typical properties for 2-D C/SiC composites are given in Table 15.3. Note that the thermal properties are experimental values, since calculations of these properties are difficult owing to the complex microstructures. It is seen from Table 15.3 that the CVD/CVI process gives superior mechanical properties compared to the LSI process: this is generally the case. Even so, different researchers have reported different ranges of properties even for the same processing technique. A typical image of a 3-D stitched C/SiC composite sample tested under three-point bending is shown in Fig. 15.11. It can be seen that the crack initiated from the stitching points.

**Table 15.3** Properties of bi-directional (2-D) carbon fibre reinforced C/SiC composites [17, 18, 42, 43]

Composite configurations		With carbon fibres (2-D composites)			3-D stitched C/SiC
Property	Units	PIP -based C/SiC	CVD-based C/SiC	LSI-based C/SiC	LSI -based C/SiC
Fibre content ( $V_f$ )	vol.%	42–44	42–47		40–45
Density	$\text{g/cm}^3$	1.7–1.8	2.1–2.2	2.4	2.1–2.2
Flexural strength	MPa	250–330	450–500	180–200	140–180
Tensile strength	MPa	200–250	300–380	80–190	70–90
Young's modulus	GPa	60–80	90–100	60	–
Strain to failure	%	0.3–1.1	0.6–0.9	0.15–0.35	0.10–0.15
Compressive strength	MPa	590	–	–	–
Thermal conductivity	W/m K	11.53 (100 °C)	–	30–35	50–15 <sup>a</sup>
Specific heat	J/kg K	900 (100 °C)	–	800 (RT)	750–900
CTE					
Parallel ( $\times 10^{-6}$ )	$\text{m/m}^\circ\text{C}$	2–3	3	1.0–1.5	–0.5–1.5
Perpendicular ( $\times 10^{-6}$ )	$\text{m/m}^\circ\text{C}$	4–7	5	5.5–6.0	3.0–4.5

<sup>a</sup>Derived from the thermal diffusivity value (25–1200 °C) in the through-thickness direction

**Fig. 15.11** A typical image of 3-D-stitched C/SiC composite failure under 3-point bend testing



Thus on the one hand, stitching the fabric stack improves the integrity of the resulting composite and also the through-thickness thermal and mechanical properties; but on the other hand, stitching limits the in-plane strength. This means that it is important to optimize the preform to achieve the required thermal and mechanical properties.

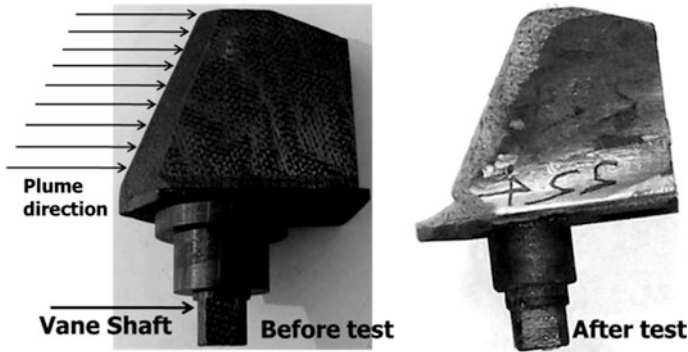
## 15.10 Applications of Aerospace C/SiC Composites

C/SiC composites are used primarily for hot structures in aerospace systems. The applications correspond to very severe service conditions, namely high temperatures and corrosive environments, in which they must often demonstrate long service lives. However, for some space and missile systems the required life may be short, and the erosion resistance is paramount. C/SiC composites have also been proposed for high performance braking systems.

Some examples of C/SiC composite aerospace applications are given in the following subsections.

### 15.10.1 Thermal Protection Systems (TPS) and Hot Structures for Space Vehicles

The temperature during orbiter vehicle re-entry reaches up to 1800 °C. C/SiC composite thermal protection systems (TPS) have performed over more than two decades in spacecraft structures and numerous technology-driven projects in Europe, the USA and Japan. The hot structure of the cancelled X-38 (NASA's experimental space vehicle) was to be made from C/SiC composites. A nose cap, nose skirt, two leading edges segments and two body flaps for steering the vehicle



**Fig. 15.12** C/SiC Jet-vanes for a thrust vectoring system [18]

were manufactured and qualified by a German consortium [44–46]. Similarly, the thermal structure of the cancelled Hermes (European Space Plane), which would have experienced high mechanical loading and surface temperatures as high as 1300 °C, was intended to be made from a C/SiC composite [47].

### 15.10.2 Jet-Vanes for Rocket Motors

Jet-vane materials must have good erosion resistance to particulate flow in addition to high thermal shock resistance (i.e. high thermal conductivity, low CTE and good strength). Erosion of the Jet-vanes depends on the fibre architecture, the ratio ( $V_f$ ) of fibres to matrix, and testing/operation conditions. The formulation of the composite microstructure requires optimizing the conflicting demands for high fracture toughness (high carbon content) and high resistance to abrasion/erosion (high SiC content) [18].

A leading Indian laboratory has developed Jet-vanes made from LSI-based 3-D stitched C/SiC composites with excellent thermal and mechanical properties [17, 18, 42] and good resistance to erosion by solid rocket motor (SRM) plumes. A typical Jet-vane before and after testing is shown in Fig. 15.12. A CVD-applied SiC coating could further protect the Jet-vanes from oxidation and the blast of alumina particles deriving from the burnt solid fuel.

### 15.10.3 C/SiC Nozzles and Components for Rocket and Jet Engines

C/SiC composites have been developed as materials for combustion chambers and nozzles. The main advantages are an increased maximum combustion temperature

**Fig. 15.13** Typical C/SiC thruster test articles



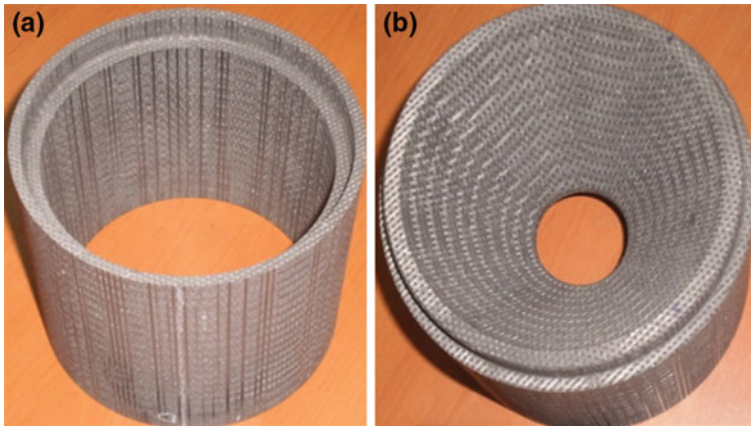
(up to 1500 °C) without requiring anti-oxidation coatings, and improved resistance to thermal cycles. C/SiC composite rocket engine for a bipropellant propulsion system has been used elsewhere [48]. Typical C/SiC thruster test articles are shown in Fig. 15.13. Such nozzles can be easily fabricated using the PIP process. A nozzle design demonstrator for the upper stage engine of the Ariane 5 has also been manufactured (by filament winding) using the PIP process.

Flame-holders, exhaust cones and engine flaps for military jet engines made of C/SiC composites have been reported [30, 43]. Outer flaps of the SNECMA M 88-2 engine have been made from C/SiC composites using the CVI process. These have provided 50 % weight savings over corresponding nickel-base superalloy flaps (Inconel 718).

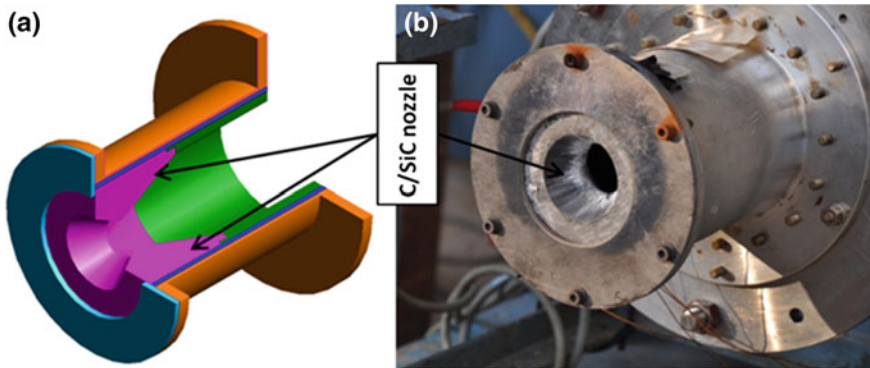
#### ***15.10.4 C/SiC Composite Nozzle Throats***

Especially for liquid propulsion systems, the exhaust gases contain huge amounts of H<sub>2</sub>O molecules that would attack and oxidize C/C composites unless they were coated. The alternative for short-life components is to use LSI C/SiC composites. A variety of thrusters made from C/SiC composites for a liquid rocket engine and a solid rocket motor were qualified as an effort to develop the AESTUS C/SiC expansion nozzle [49]. LSI-based 4-D C/SiC nozzle throats and inserts with uniform thermomechanical properties have been developed in a leading Indian laboratory and tested satisfactorily for 30 s with a mixed hydrazine/nitrogen tetroxide fuel propulsion system [49]. The throat insert and nozzle configurations are shown in Figs. 15.14 and 15.15. The 4-D C/SiC composite nozzle throat performed much better than conventionally used high density graphite or C/C composites under similar testing conditions. It is envisaged that in future the largest sales volume of the fibre ceramic production, besides brake discs and in ballistic protection systems will be based on C/SiC and C/C-SiC composites.





**Fig. 15.14** A typical 4-D C/SiC composite thruster chamber (a) and throat insert (b) [17]



**Fig. 15.15** 4-D C/SiC composite nozzle tested under liquid propulsion [17]

### 15.11 Indian Scenario for C/C and C/SiC Development

C/C and C/SiC composites have been under investigation for a long time. Several leading agencies around the world have developed state-of-the-art facilities for the reinforcements, preforming, processing and characterization of these composites. Several products have been realized for defence and aerospace applications under the aegis of NASA, Russia and the European Union. With reference to India, a significant amount of work has been done in the last two decades. C/C composite brake discs and reentry ballistic missile nose tips have been fabricated and qualified for different defence programmes. C/SiC composite Jet-vanes and throat inserts have been made using the LSI process. The PIP and CVD/CVI processes have also been employed to obtain C/SiC composite specimens and test articles. Research



continues to develop a reliable design methodology, automation of the preforming process, non-destructive testing (NDT), life estimations, and the joining of C/C and C/SiC composites with other materials.

## 15.12 Summary

Carbon fibre-reinforced composites occupy an important position in aerospace. This chapter provides a brief description of the processing, properties and applications of C/C and C/SiC composites. It also provides some basic information on the raw materials used for the matrix and fibre reinforcements. Some aerospace applications of these composites are given as well.

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