

Challenges in Carbon/Carbon Composites Technologies

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Abstract

Carbon/Carbon Composites due to their far superior thermo-mechanical properties are used in a number of demanding applications. However, the material still is used only in specific high tech applications with few exceptions in general industrial applications. The material is extremely expensive and the major challenge is to reduce its cost. Various innovative processing routes are outlined to reduce the cost of processing.

Keywords : Carbon/carbon composites, Challenges, Carbon fibers, Processing, Low cost

1. Introduction

Materials, in general, play a pivotal role in achieving continuous development for the mankind in any sphere. In the recent past, we have seen the development of sophisticated steels and super alloys, which in turn have contributed enormously to the overall advancement of various technologies affecting every walk of life. The aircraft industry is one glaring example, where progress would not have been possible but for the availability of metal super alloys. It is but imperative therefore that further progress in engineering will obviously depend on the continued development of newer forms of materials.

With the advent of fibers, a new class of materials, known as composites, took birth somewhere around 1930's or so. Carbon in general, and carbon fibers in particular, holds a central position in this new class of materials (composites) especially when the composites have to be used at elevated temperatures and under extreme conditions.

Carbon is truly a unique solid that can be made to exhibit the broadest variety of structures and hence the properties. For example, carbon has the capability to be one of the best thermal conductors (κ of the order of 3000 W/mK) and at the same time can be one of the best thermal insulators (famous tiles used in space shuttle!). Second example is that it is one of the best braking materials and is being used in all modern military or civilian aircrafts. Contrary to this, carbon is known to be one of the best lubricating materials—offering no resistance at all. So on and so forth.

The most important point to be noted here is that the variation in the properties is brought, not by alloying as in the case of metals, but is achieved only through changes in the carbon structure itself.

Carbon/carbon composites, which consist of carbon fibers

as reinforcement and carbon generated from resin, pitch or organic gas on pyrolysis as matrix, due to their far superior thermo-mechanical properties like low density, high thermal conductivity and shock resistance, low thermal expansion, high specific strength and modulus find applications in aerospace and defense, mainly for aircraft brake discs, rocket re-entry nose tips, leading edges in high performance aerospace vehicles and parts of rocket nozzles [1-6]. About 70% of the production of carbon/carbon composites world wide is used at present for aircraft brake discs. Very few applications of carbon/carbon composites have been realized for general engineering industries. What can be reasons for this? Cost of carbon/carbon composites (compared to conventional materials used at present in different general engineering industries in spite of carbon/carbon composites having far superior specific performance compared to conventional materials) is thought to be the main reason. Carbon/carbon composites are still economically unviable compared to conventional materials used in these general applications. Different ways by which carbon/carbon composites can be made economically viable and competitive to conventional materials used in general engineering applications will be discussed.

2. Invention of Carbon/Carbon Composites

As a matter of fact, first carbon/carbon composite was prepared in 1958 at the “CHANCE VOUGH AIRCRAFT CO.” by accident [7]. A chemical analysis was being performed to determine the fiber content in an organic matrix composite. Part of the process was to expose the composite to air at higher temperature. However, by mistake, the lid of the crucible was left ‘on’ and as a result, instead of

oxidizing, the organic matrix composite got “pyrolized”. The resultant charred composite was found to exhibit structural characteristics and hence the birth “of carbon/carbon composites”.

3. Onset of Missile Age

First use of carbon in missiles was made during the World War II where fins of the missile were made using fine grain graphite. It was immediately realized that this form of carbon was not only brittle but also very low in strength. Soon another form of carbon called “pyrolytic carbon” was invented which is about 4~5 times stronger than fine grain graphite. Pyrolytic carbon though much stronger was found to show brittle fracture. Fig. 1 shows properties of various carbons versus testing temperature. Properties of some of the best alloys are also included.

As seen from Fig. 1 carbon/carbon composites, with mechanical properties exceeding those of pyrolytic graphite by three times, are a highly desirable material especially when the application is at elevated temperature. Without mentioning the quantitative numbers of mechanical and thermal properties, it may be concluded that in no other single material you can find combination of properties that are being offered by this wonder form of carbon i.e. carbon/carbon composites. All present day missiles make use of carbon/carbon composites.

4. Evolution of Carbon/Carbon Composites

Immediately after its accidental discovery in 1958, further development of this wonder material was nurtured under US

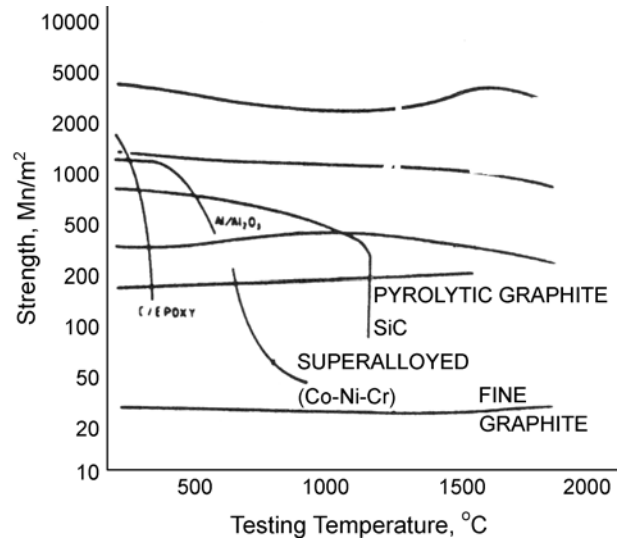


Fig. 1. Short term strength, S, of carbon materials including CFRC compared with C/epoxy and metals.

Airforce Space Programme as well as NASA’s Apollo Projects [5]. However, intensive research in its development was initiated only in late 1960’s [2-10] when the Space Shuttle programme got underway. Carbon/Carbon composites consisting of carbon fibers dispersed in a carbonaceous matrix sound extremely simple since both the constituents are of carbon. In reality however, carbon/carbon composites are extremely complex, both from the point of view of processing as well as performance. The prime reason for the complexity is attributed to the fact that the structure of each constituent can vary continuously from amorphous carbon (non-graphitic) to graphite.

Fig. 2 gives general information about various stages through which newly invented materials pass through. As is

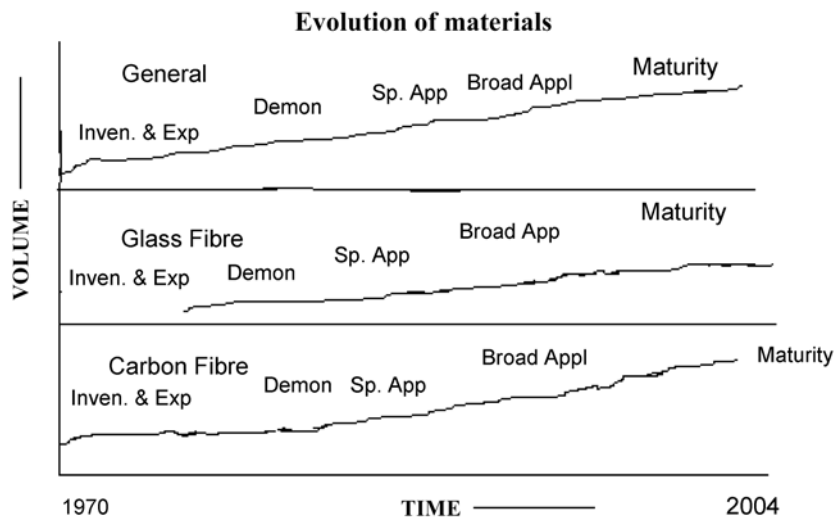


Fig. 2. Evolution of materials with time.

Table 1. Cost analysis of composites

	Carbon/Carbon, %	CFRP, %
Carbon Fibres	17	70
Weaving	17	5
Matrix	5	5
Processing	40	5
Protective Coating	12	Nil

clear from the top curve, a material achieves maturity only after broad or mass applications are established. In case of glass fibers, it has already reached a stage of maturity. In other words, the material has already been accepted as a standard material by the industry and is available at reasonable cost. One may conclude further that maximum possible properties have also been achieved and very limited amount of further R&D is required. As is evident, it has almost taken 60 years from the date of invention to achieve maturity in case of glass fibers.

Let us now look at the history of carbon fibers before going to carbon/carbon composites. Carbon fiber is on the threshold of achieving maturity in a gap of about 40 years. It is to be recorded here that the materials in general are seldom or very rarely invented from the point of mass application areas. Invariably, the materials are invented for specialized applications. However, to achieve the maturity of the material, it is not the specialized application but the mass application, which plays a crucial role. For a material to achieve maturity it must satisfy the following conditions at least:

- (i) Availability in standard and accepted forms
- (ii) Reasonable cost etc.

In the case of carbon/carbon composites, we are still struck at the stage of specialized applications. It is important to mention that large number of industrial applications has been attempted. *So what is holding carbon/carbon composites for going to broad application areas and hence achieving maturity?* Before we address this question, let us look at the cost analysis of carbon/carbon composites (Table 1).

4.1. Cost analysis

The cost of carbon/carbon composites depends not only on the cost of raw materials, processing etc. but also on the end product configuration. For the purpose of comparison; information about carbon fiber reinforced plastic is also included in the table.

In case of carbon/carbon composites, the major cost is contributed by the processing stage. It is 52% of the total cost as compared to only 5% in case of CFRP.

Thus unlike the well known standardized conventional metals industry, uniform price quotations for carbon/carbon are not possible. The products range in the price from a

single block of approx. \$500/Kg using an automated weaving process to \$3000/Kg using the labor-intensive weaving process. For complex developmental structure, the cost per Kg is between \$9000/-and \$11000/Kg. The cost of Brake Material, the major industrial application of carbon/carbon composites comprising about 70% of the total production of carbon/carbon composites worldwide, is about \$200/Kg.

5. Processing Step

The processing step is broadly made up of:

- (1) Understanding of interaction between carbon fibers and the matrix (established at the polymer stage).
- (2) Since the material has to be heat treated to the temperatures of the order of 3000°C, a couple of times during the processing of the product, it is very energy intensive.

Some of the other important parameters, which control the properties of carbon/carbon composites, are listed below:

- (i) Properties of reinforcing carbon fibers
- (ii) Properties of matrix material
- (iii) Fiber volume fraction and its distribution
- (iv) Interface between reinforcing fibers and the matrix

Out of these four, the first three have been very well documented in the literature [11-15] and fairly understood as well. The fourth parameter is still in the process of being understood. It controls not only the final mechanical and thermal properties of the composites but also the pseudo-plasticity of the composites as well [1, 11, 13-14, 17-23].

In order to obtain good load transfer between fibers and the matrix, an optimum adhesion at the interface is necessary. Although the surface roughness of the reinforcement contributes to the adhesion, surface bonding is one of the most important factors. It is influenced by the presence of chemically active groups on the surface of the fiber. A compromise between strong and weak bonding at the interface is necessary to optimize the efficiency with which fiber properties are utilized and the fracture toughness of the composites increased.

Fitzer *et al.* [21] have established a qualitative correlation between the amount of surface functional groups, fiber/matrix interface and mechanical properties of carbon-carbon composites. Dhakate and Bahl [20] have shown that it is the amount of reactive functional groups present on the surface of carbon fibers and not the total amount of surface groups present which influence the fiber/matrix bonding and mechanical properties of carbon/carbon composites. Present authors have studied the role of pyrolytic carbon coating on carbon fiber surface by developing carbon/carbon composites with and without pyrolytic carbon coating. A four-fold increase in the flexural strength was achieved using carbon fibers with a thin (≈ 0.1 mm) coating of pyrolytic carbon. It has been concluded that it is the porosity present at the

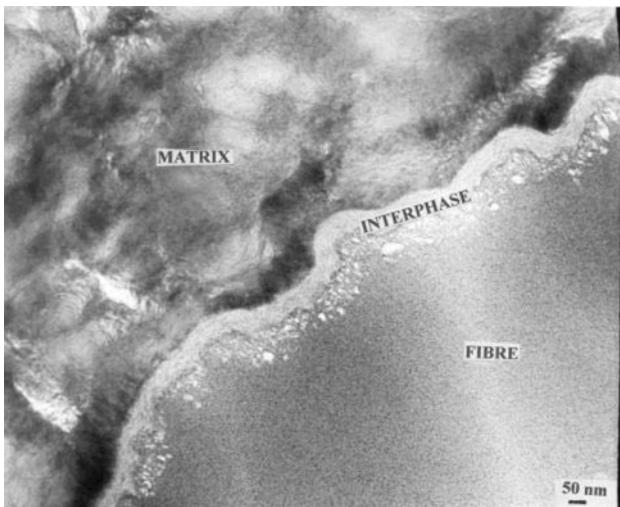


Fig. 3. Overview of interfacial zones between two fibers and the matrix in a 1000 °C carbonized C/C/C composite. The interphase texture is similar to that as α -deposited. The porous texture of the interphase is obvious, together with the elongation of the pores, inducing an anisotropic feature.

interface in case of C/C/C (carbon fiber/pyrolytic carbon coating/carbon matrix, Fig. 3) composites which account for the increase in mechanical properties of such composites compared to C/C (carbon fiber/carbon matrix) composites [22].

6. Global efforts in the area of reducing processing cost

As pointed out earlier, one of the major challenges to introduce carbon/carbon composites in general engineering/mass applications is to reduce the cost of these materials so as to bring it at par (performance/unit price) with other conventional materials. The major areas where reduction can take place is

- i) reduction in the cost of carbon fibers
- ii) reduction in the cost of processing

As far as reduction in the cost of carbon fibers is concerned, it has been happening all the time, but more vigorously between 1970 and 1990. The prices fell from approximately US\$ 1000/kg to about US\$ 20~40/kg for high strength and standard modulus carbon fibers [24, 25]. The prices have stagnated at this since then. The technologists seem to have exhausted all the available tools for any further improvement. Altogether new approaches are being tried using “higher throughputs”, “New Precursor chemistry”, Carbon fiber from Lignin” including Microwave Processing etc. etc. These new techniques are spearheaded by A. Ogale of Clemson University) USA [26], Zoltek Co. St Louis, USA and Oak Ridge National Laboratory in addition to

many others. The targeted cost is about US\$ 5~10/kg.

Regarding bringing down processing cost, there have been numerous efforts reported in the open literature. Ferguson *et al.* [27] have used single step processing of Pitch based carbon/carbon composites to reduce its cost. They have reported density of 1.2 to 1.7 g/cc and Flexural Strength and Flexural Modulus were 14 MPa and 9.4 GPa respectively. Kowbel *et al.* [28-30] have used novel one-step low cost manufacturing method to develop 2-D carbon/carbon composites, which eliminates the conventional densification step. This technology was applied to develop net-shape molded carbon/carbon pistons and valves. They could achieve density of the order of 1.45~1.6 g/cc on HTT 2500 °C. Fracture behavior, FS and strain to failure ratio were found to be greatly influenced by the heat treatment temperature and FS was of the order of 350 MPa. These composites appear to be more attractive than metals or organic composites for heat dissipation components. Luo Ruiying [31] has also tried to reduce the cost of processing of C/C composites by using the technology of rapid chemical vapor deposition. It was observed that C/C composites developed by this route are compact having FS of 320 MPa and showed pseudo-plastic behavior. Interface between carbon fibers and matrix carbon showed continuity and turbostratic structure as revealed by HRTEM. Wang, J. S [32] also observed that applications of carbon/carbon composites are restricted within narrow limits due to long processing cycle and high cost involved in their development. It is observed that research and development efforts are concentrated in manufacturing and processing technologies for low cost carbon/carbon composites around the world but these are still at exploration stage. Some of the more important cases are reported below:

6.1. Plasma enhanced vapor infiltration technique

Fig. 4 gives an overall diagram for processing of carbon/carbon composites using this technique. This technique provides a method for controlled densification of a porous article having either open or closed porosity. In this approach, densification is extremely rapid as compared with conventional techniques, and involves only a single processing cycle instead of multiple cycles of carbon deposition and surface machining. The densification time can be reduced to a few percent of that required in conventional processing methods.

A plasma is formed in a reactor by high frequency induction. The article to be densified is immersed into the plasma so that the plasma completely surrounds the article. A source of densifying species is introduced into the plasma. For example, methane gas can be taken. The source gas dissociates and the disassociated carbon thereafter gets ionized. Since the article is immersed into the plasma, it assumes a small negative potential relative to plasma. To

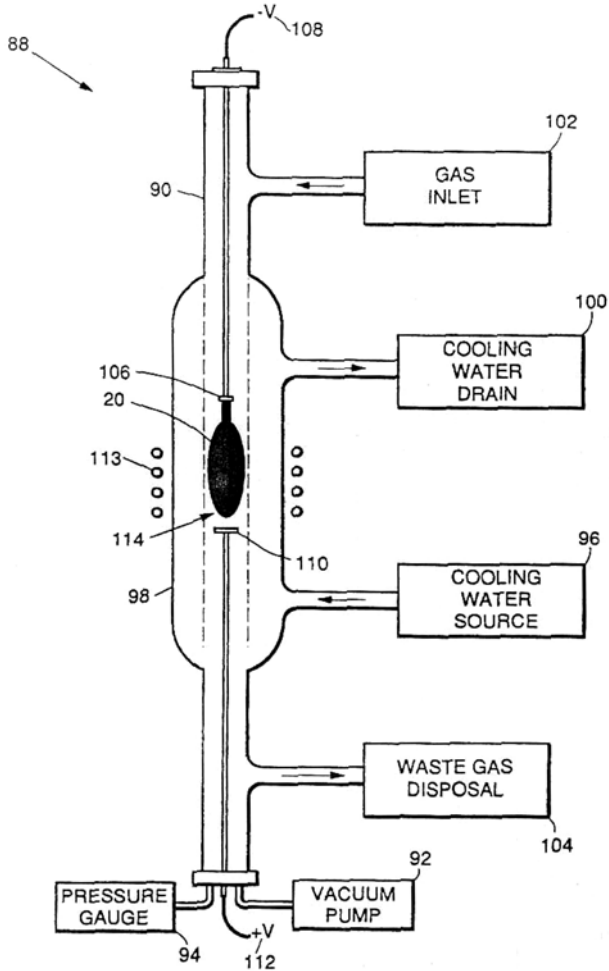


Fig. 4. Schematic of processing of C/C composites by plasma enhanced vapor infiltration technique.

increase the negative potential, a further negative bias to the article is given from outside. As a result of this, the total negative bias causes the ionized carbon species to be accelerated towards the article and these species finally deposit into the pores. Depending upon the thickness of the sample the negative bias has to be selected accordingly. As is obvious, the center (pore) of the article gets densified first. Thereafter one will have to reduce the negative bias to densify the article at a lesser depth. Extending this principle the applied bias voltage has to be gradually reduced to move the zone of densification towards external surface of the article.

This technique provides a very powerful way of densifying carbon/carbon composites [33] in a controlled way and at the same time in a very short time. For example, it has been reported [33] that the density of 1/2" thick and 6" dia preform was increased from 1.1 g/cc to 1.8 g/cc in a matter of four hours only. **Another important feature of this technique is that we can get near net shape products**

without involving any machining etc. It is a single step infiltration technique which cuts down both on processing time, required temperatures and avoids any machining as well.

6.2. Densification of porous structures using super critically fluid technique

In this process, the porous article is immersed in a liquid hydrocarbon [34] and is heated through induction so as to form, through decomposition of the hydrocarbon, carbon or pyrolytic graphite which is deposited in the pores or cavities of the porous article.

Fig. 5 explains the process of densification and Fig. 6 compares the density as a function of time in hours achieved through this process with that of a densification by deposition in the gaseous phase. The density of 1.75 g/cc can be achieved in a period of 2 hours compared to 80 hours by conventional method.

In this process the article (preform) placed on a rotary

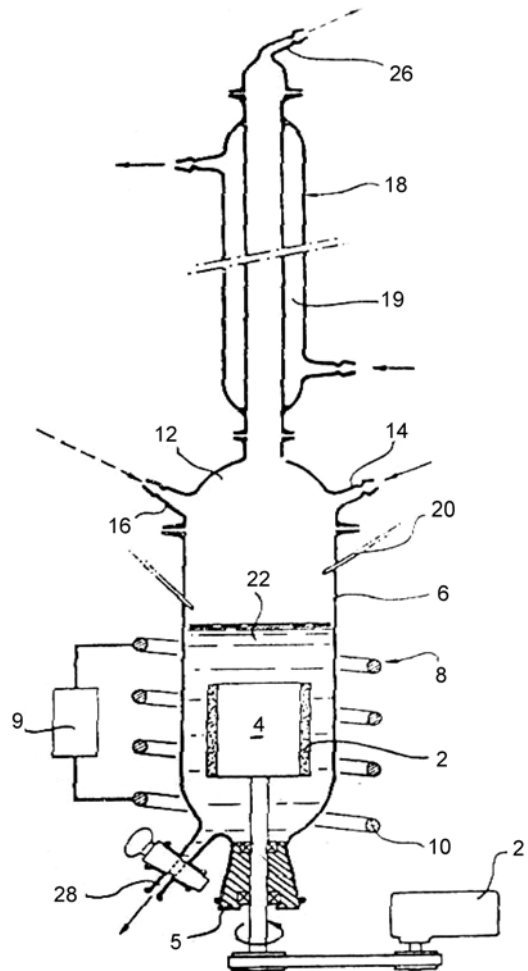


Fig. 5. Schematic of densification of C/C composites through super critically fluid technique.

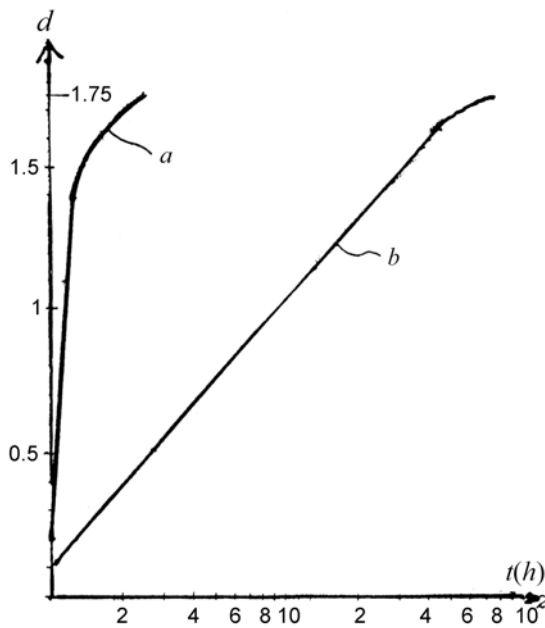


Fig. 6. Comparison of average impregnation speed of the porous structure by super critical fluid and CVI technique.

support is immersed in a liquid hydrocarbon, such as cyclohexane. The air present in the system is expelled and the system is heated through induction heater using inert gas. The heated liquid hydrocarbon vaporizes and then decomposes into carbon or pyrolytic graphite and hydrogen. Initially, the liquid hydrocarbon is heated by natural convection and evaporation therefore takes place only on the surface of the liquid. But with time gas bubbles form level with the porous structure and rise through the liquid mass producing natural circulation streams. At this time, the porous structure is separated from the liquid by a vapor film which diffuses through the porous structure and carbon or pyrolytic graphite is deposited in the pores.

As the temperature is a maximum initially close to the mandrel which is generally made of graphite, so carbon is deposited in structure in contact with mandrel and thus a densification gradient is obtained from the interior to the exterior of the structure. The rate of deposition of carbon is proportional to the concentration of the hydrocarbon.

Fig. 6 clearly shows that average impregnation speed of the porous structure by cyclohexane is approximately 100

times greater than that obtained by deposition in gaseous phase.

6.3. Single Step Fabrication of High Density Carbon/Carbon Composites

A special technique of processing high density composites has been developed [35] using a special type of a sample holder for heat treatment of polymer composite material up to 1000 °C. In this sample holder, green composites are placed and are covered from both the sides by two other plates. Finally, these three plates were sandwiched; keeping plate containing composite samples in the center. This sample holder was kept for carbonization up to 1000 °C in an inert atmosphere keeping the desired heating rates. These were heat treated to 2000 °C and 2600 °C in inert atmosphere. Bulk densities and shrinkage pattern of these composites are described in the Table 2.

It is really outstanding, achieving a density of +1.85 g/cc in a single shot i.e. without any impregnation whatsoever. In a normal processing, it usually takes 4-5 impregnation/carbonization/graphitization cycles to achieve the same value of density. As a result:

- The processing becomes extremely expensive.
- It is time consuming
- Repeated heating/cooling cycles deteriorate the mechanical properties of the final composite.

The processing technique invented during the present work overcomes all the above mentioned three drawbacks. If we look at the density values at 1000 °C HTT samples, composite with 50% fiber volume shows a slightly higher value as compared to composites of 35% and 20% fiber volumes, which may be due to higher fiber volume only as the density of VGCF (Pyrograph IIITM) is 1.95 g/cc. It is fascinating to note that reversal in density values takes upon heat treatment to 2000 °C. Sample with 20% fiber volume shows a density of 1.76 g/cc which is highest of all. This sample shows maximum volume shrinkage as well, 41%. It is well known in literature that soft carbons when heat treated above 1700 °C get graphitized and normally show 3-4% volume shrinkage in addition to exhibiting increase in bulk density.

In the present case, the volume shrinkage is very large giving an indication that some sort of compaction is taking place in addition to decrease in volume as a result of

Table 2. Physical Characteristics of C/C Composites

Fiber vol. VGCF (Pyrograph III TM) (%)	Volume changes (%)			Total	Bulk Density (g/cc)		
	600 to 1000 °C	1000 to 2000 °C	2000 to 2600 °C		1000 °C	2000 °C	2600 °C
20	-15	-22	-4.1	-41.1	1.35	1.76	1.85
35	-7.2	-15	-0.34	-22.54	1.38	1.60	1.56
50	-1.48	-8.7	-0.84	-11.02	1.54	1.66	1.52

graphitization. The samples were heat treated to 2600 °C in nitrogen atmosphere to see if any graphitization sets in. In case of 20% fiber volume composites, the increase in density from 1.76 to 1.85 g/cc is clearly due to further compaction largely .

7. Development of Oxidation Resistant Coatings

7.1. Coatings for temperatures above 1800 °C

Oxidation protection systems for carbon/carbon composites upto 1500 °C for long term duration based on carbides and nitrides of Si in combination with a variety of sealants and inhibitors are well established [36-38]. Long term oxidation protection at temperatures higher than 1800 °C requires materials and their combinations quite different than coatings based on silicon with silicon sealants as the reaction products disrupt the SiO₂ layer which is necessary for oxidation protection. The materials identified for coatings above 1800 °C are HfB₂ and mixtures of HfB₂ and SiC [39, 40]. Criteria for selection of oxide coatings are melting point, vapor pressure and CTE. ZrO₂, HfO₂, Y₂O₃ and ThO₂ have the required thermal stability for long term use at temperatures over 2000 °C while Al₂O₃ can be used at lower temperatures. Fig. 7 compares the thermal expansion coefficients of ceramic oxides, which are quite higher than that of carbon, making spallation of coatings during thermal cycles a significant issue [41, 42]. The potentially effective very high temperature coating system on carbon materials might contain a refractory oxide outer coating as an erosion barrier and an inner SiO₂ glass coating as an oxygen barrier and sealant for cracks in the outer coating.

The coating of refractory oxide over SiO₂ is not compatible with carbon or carbides. Therefore SiO₂ will have to be separated from carbon surface by a layer of refractory oxide. To have compatibility, the inner refractory oxide layer should be in contact with refractory carbide, which in turn would interphase with carbon substrate as carbon diffusion barrier. TaC, TiC, HfC and ZrC have low carbon diffusivities. Thus total four layer coating systems would be consisting of carbon substrate/refractory carbide/refractory oxide/SiO₂ glass/refractory oxide. Long term oxidation protection of carbon materials at temperatures above 1800 °C is a very complex problem as fundamental issues of thermodynamics and kinetics of materials limit the choice to a very small number. This needs long term and systematic basic research to identify the materials.

On advanced aerospace vehicles, applications for these composites are predominantly in hot structures, for which an expanded performance capability is needed relative to the Space Shuttle material. Simulation results for entry and cruise missions representative of an advanced aerospace vehicle showed a 40 hour cumulative exposure time to a 75

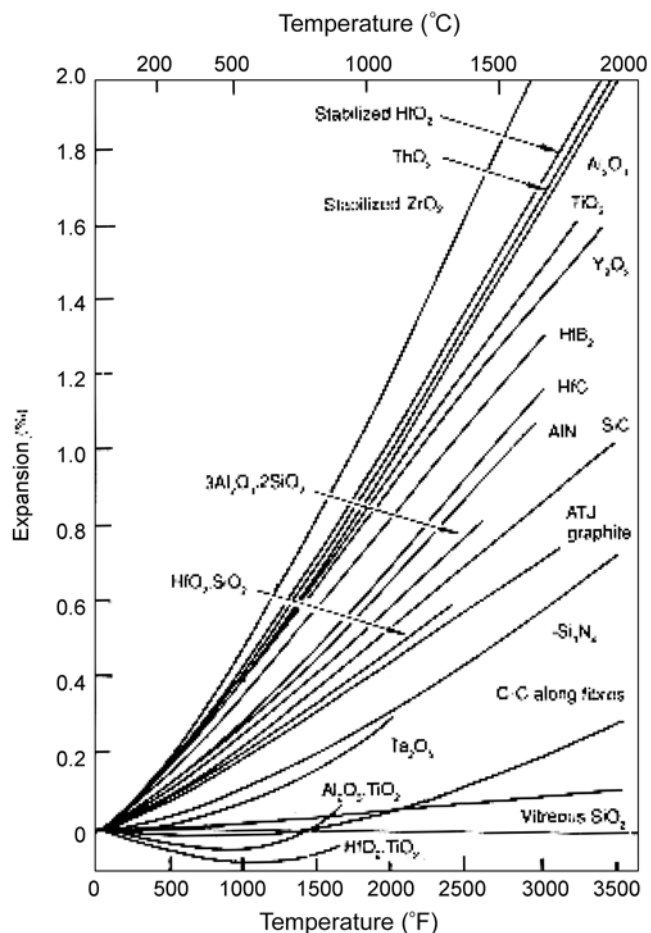


Fig. 7. Comparison of the thermal expansion characteristics of a number of refractory materials.

g/m² mass loss. (Table 3) [43].

Some of the resistance of the designers to use this material is due to:

- i) Lack of familiarity with the material
- ii) Limited availability of design data
- iii) Complex materials response modes to service environments
- iv) Lack of suitable design methodology

8. Carbon/Carbon Composites for Thermal Management

Another area where this material is facing considerable challenge is thermal conductivity. Theoretically, graphite is capable of offering thermal conductivity of ~3000 W/mK. Highly oriented CVD carbon is capable of offering a thermal conductivity as high as that of diamond. A value of ~2500 W/mK for carbon whiskers produced by vapor grown technique [44] and 1950 W/mK VGCF (Pyrograph I) has already been achieved [45]. However, to translate these

Table 3. Performance requirements of carbon/carbon composites for selected aerospace applications

	Space Shuttle nose cap and wing leading edges	Man-rated turbine engines	Aerospace vehicle hot structure
Number of missions	100	Many	≥ 100
Hot lifetime, hrs	100	4000(b)	≥ 200
Design temperature, °C	1500(a)	1450(b)	< 1600
Single-mission survivability temperature, °C	1600	–	> 1600
Operating environment	Air	Combustion products	Air
Operating pressure, atm	< 1	> 1	< 1
Mechanical property requirements	Low σ and E	High σ	High σ and E

(a) Actual flight experience 1450 °C

(b) Air Force Technical Objective Document FY88, Dec 1986

This table is taken from Ref. 43

lucrative properties in carbon/carbon composites is quite a challenge as of now.

In metals and most other single-phase materials, it is something straightforward. However, in two-phase materials like carbon/carbon composites, it becomes complex and complicated since the over all thermal conductivity of carbon/carbon composites would at least depend upon:

- (i) Thermal conductivity of carbon fibers used
- (ii) Thermal conductivity of the carbon matrix
- (iii) Interface between carbon fibers and the matrix
- (iv) Microstructure of the matrix
- (v) Processing conditions including final heat treatment temperature
- (vi) Porosity as well as pore size distribution.

Carbon fibers, due to preferred orientation of the graphene layers show not only high modulus, high strength but also high thermal conduction along the fiber axis. High modulus carbon fibers i.e. carbon fibers possessing high degree of preferred orientation of the graphene layers along the fiber axis are known to exhibit high thermal conductivity [46]. In short, if one has to make the choice of a suitable reinforcement, under the present context, one would use therefore high modulus pitch based carbon fibers.

As far as the choice of matrix is concerned, there are generally three types to choose from:

- (a) Carbon arising out of thermosetting resins
- (b) Carbon derived from thermoplastic resin
- (c) Vapor deposited carbon

Since the thermosetting resins are known to yield hard carbons [47], the choice either falls on thermoplastic resin (e.g. coal tar or petroleum pitch) or vapor deposited carbons.

There are a large number of publications [46, 48-53] wherein effect of various processing conditions as well as those of raw materials has been studied and reported from the point of view of thermal properties of carbon/carbon composites.

How precisely to predict the thermal conductivity of

carbon/carbon composites and also how to control achieving higher values is a big challenge before technologists today. Thermal conductivity of carbon/carbon composites does not follow rule of mixtures of its individual components i.e. carbon fibers and the carbon matrix. The interface and the porosity present dictate the over all thermal conductivity.

Contrary to what is reasoned above, it has been demonstrated [54] that it may not be sufficient and essential to use only high modulus carbon fibers and a soft carbon matrix for achieving high thermal conductivity in carbon/carbon composites. It has been possible to develop carbon/carbon composites with over all degree of graphitization exceeding 82% using PAN based T300 carbon fibers and phenolic resin as matrix precursor. Both the components are well known as hard carbons. Here carbon fibers have been used in the chopped form and not in usual continuous form, the implication being the availability of interface, which is manifold for the same carbon fiber volume. Complete transformation of matrix hard carbon to graphitic form took place, which eventually became responsible for many fold enhancement of thermal conductivity etc.

9. Conclusions

Carbon/carbon composites seem to have reached a stage, which is a threshold for its final journey to maturity. Challenges before the world community are reduction in the cost of processing of carbon/carbon composites using new and innovative techniques like plasma enhanced CVI, and densification through Super Critical Fluid Technique on commercial scale. Problem of oxidation resistance coatings on carbon/carbon composites has been solved to a large extent upto temperatures of 1700~1800 °C but beyond 1800 °C the problem still exists though lot of efforts are being made globally to solve this problem. Basic scientific understanding of the conduction of heat in carbon/carbon composites (two phase systems) needs to be understood in a

comprehensive way before its use as a standard material for thermal management applications.

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