

Carbon-Carbon Composites – An Overview

G. Rohini Devi and K. Rama Rao

Defence Research and Development Laboratory, Hyderabad-500 269

ABSTRACT

Carbon-carbon composites are a new class of engineering materials that are ceramic in nature but exhibit brittle to pseudoplastic behaviour. Carbon-carbon is a unique all-carbon composite with carbon fibre embedded in carbon matrix and is known as an inverse composite. Due to their excellent thermo-structural properties, carbon-carbon composites are used in specialised application like re-entry nose-tips, leading edges, rocket nozzles, and aircraft brake discs apart from several industrial and biomedical applications. The multidirectional carbon-carbon product technology is versatile and offers design flexibility. This paper describes the multidirectional preform and carbon-carbon process technology and research and development activities within the country. Carbon-carbon product experience at DRDL has also been discussed. Development of carbon-carbon brake discs process technology using the liquid impregnation process is described. Further the test results on material characterisation, thermal, mechanical and tribological properties are presented.

1. INTRODUCTION

Carbon is a unique element that can exhibit different properties in different forms. Some forms of carbon are extremely hard, like diamond, while some forms are extremely soft and ductile. Thus, in addition to its well defined allotropic forms (diamond and graphite), carbon can take any number of quasi-crystalline forms ranging from amorphous or glassy carbon to highly crystalline graphite¹. The latest form of carbon (C₆₀), discovered recently, is called Fullerene, named after Buck-Minister Fuller, the renowned American architect and philosopher. Fullerene is the roundest of all round molecules, more like a soccer ball, and has properties like high strength, ferromagnetic properties, superconductivity and is an excellent semiconductor². Full knowledge of its properties is still not acquired.

The changing Defence scenario and satellite launchings using re-usable launch vehicles led to vigorous search for light weight, ultrahigh temperature materials. For a long time, carbon has been known for its high temperature properties and is widely used in heating elements. In 1879, Thomas Alva Edison used

a crude form of carbon fibre for the first electric lamp³. Germans used graphite for the jet vanes in the V₂ rocket. But its application in structures was limited because of its failure even at low strains, thermal shock sensitivity, anisotropy and processing difficulties for large and complex shapes. The advent of carbon-carbon (CC) composites changed the scene drastically.

2. CC COMPOSITES

CC composites are a new class of engineering materials that are ceramic in nature but exhibit brittle to pseudoplastic behaviour. Carbon fibre reinforcements when embedded in carbonaceous matrix material results in CC composites. As in all composites, the aim is to combine the advantage of high specific strength and stiffness of carbon fibres with the refractory properties of carbon matrix. When the fibres are laid in near-net shapes with multidirectional reinforcements, the result is an ideal high temperature structure.

2.1 Unique Features

These composites are the best among all high temperature materials because they are thermally stable

and do not melt up to 3000°C, have high thermal conductivity and low thermal expansion (thus having high resistance to thermal shock) and retain their mechanical strength to the end. Also, these composites maintain good frictional properties over the entire temperature range with low wear. They have high fracture toughness and do not fracture in a brittle manner like conventional ceramics. A multimode mechanism of fracture occurs where the fibres break as well debond^{1,11}. The brake discs for high speed aircrafts like Mirage 2000, Concorde, Airbus-320 are some of the example where the favourable frictional properties were put to use. The first generation CC composites had the limitation of proneness to oxidation over long exposures. However, with the advent of second generation oxidation-resistant composites, this limitation was overcome. These unique features made it the most favourite material for re-entry nosetips, leading edge material for space shuttle wings, rocket nozzles, thrust vectoring nozzles using CC ball and socket joints and high performance turbojet engines.

2.2 International Status of CC Composites

CC composites have their origin in the jet vanes used in the German V₂ rockets. The jet vanes were made of graphite which eroded rapidly and had limited life. Subsequently, pyrolytic graphite, ATJ graphites were used, which when reinforced with carbon fibres gave birth to CC composites. Major work is going on in USA, France and former Russia. There is a great demand for CC brake discs for aircrafts, trains, trucks and even racing cars. The pioneers in this field are Bendix in USA and Dunlop in UK. Nigrafite, Moscow, is the leading organisation in former USSR. Little is known about its products, while Germany and Japan are in the race for industrial applications. Israel, Taiwan, and Egypt were reported to have initiated some R&D activities. A study conducted by Dr Robert A. Meyer on CC composite research in the Far East indicates that, apart from India, as many as 18 institutions in Japan, 4 in China, 3 in Taiwan, 3 in Korea, 2 in Australia are carrying out active research on different aspects of CC technology. His assessment indicates that the collective research efforts in Japan and other Asian countries will improve and, in time, surpass the research activities in the United States if the present financial support trend continues. Internationally, the stress is on industrial applications. CC composites are a

candidate material in glass industry, furnace and semiconductor industry as well as for preventing corrosion in chemical plants⁴. One interesting and innovative use is as tooling material for metallurgical superplastic stage forging process. Other high tech applications are as heat exchanger tubes for helium cooled high-temperature nuclear reactors, high temperature crucibles, fasteners, load bearing plates, rods and heating elements. Elemental carbon is known to have the best biocompatibility with blood and soft tissues. Thus it finds use in hip bone endoprosthesis, bone plates, osteosynthesis and artificial heart valves.

3 CC COMPOSITES: FABRICATION PROCESS AND STRUCTURAL DESIGN

3. Fabrication Process

Classically, carbon ceramics are fabricated by combining solid particles of pure carbon (known as primary carbon) with a preliminary binder which acts as a precursor for secondary carbon formed during the carbonisation process. In CC composites the carbon fibres (based on rayon/PAN/pitch) in the form of U-D, 2-D and multi-directional preforms are used as primary carbon rather than particulate fillers. There are two distinct techniques used to fill the interstices between the carbon fibres. These are (i) gas phase using a chemical vapour deposition process, and (ii) the liquid phase route using thermosetting resins or pitch (PIC). The fibres can be very stiff, highly oriented graphitic and dense (pitch precursor) or relatively flexible, stronger, less oriented graphitic and less dense (PAN precursor). Conversely, the matrix can be highly oriented and graphitic if produced from pitch, either isotropic or anisotropic if produced from CVD or usually isotropic if produced from thermosetting resin using phenolic resin⁵. A general rule of thumb employed by manufacturers is that the gas phase route is adequate for thin-walled parts and the liquid phase route is preferable for thick parts⁶. A combination of liquid and gas phase processes is also being followed. Fig. 1 gives the general process flow sheet.

3.2 Multidirectionally Reinforced Preforms

The main advantage of multidirectional CC composites is the freedom to orient selected fibres and amounts to accommodate the design loads of the final structural component and make them virtually

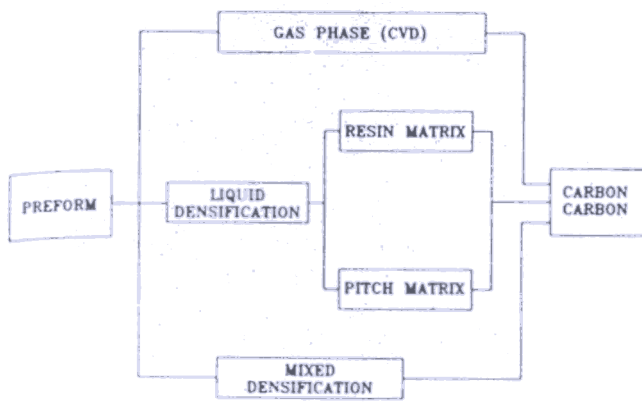


Figure 1. Densification by carbon matrix.

delamination-free. Multidirectional preform fabrication technology provides the means to produce tailored and near netshape composites, which meet the directional property requirement of an end item.

Thermal, mechanical and physical properties of the composites can be controlled by the appropriate design of substrate parameters such as fibre orientation, volume fraction of fibres in the required direction⁷. Preform weaving technology provides the ideal approach to tailor the structural composites. The simplest type of multidirectional structure is based on a three directional (3-D) orthogonal construction as shown in Fig. 2, consisting of multiple yarn bundles located within the structure described in cartesian co-ordinates. In any direction, fibre bundles are straight in order to obtain the maximum structural capability of fibre. The type of fibre, the number of fibre bundles per site, the fibre bundle spacings, volume fraction distributions, the woven bulk densities characterise the

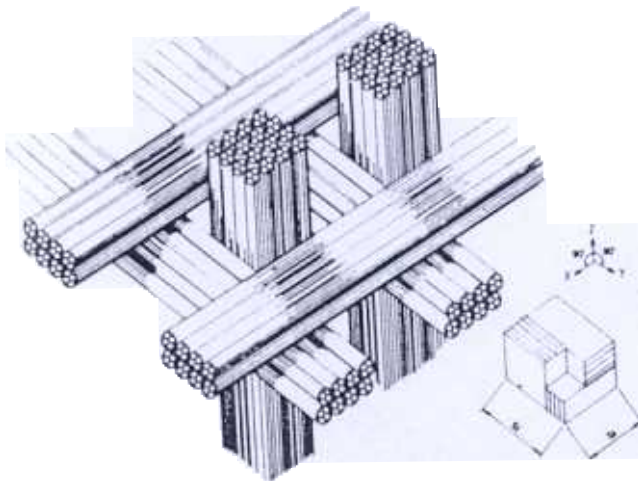


Figure 2 3-dimensional array.

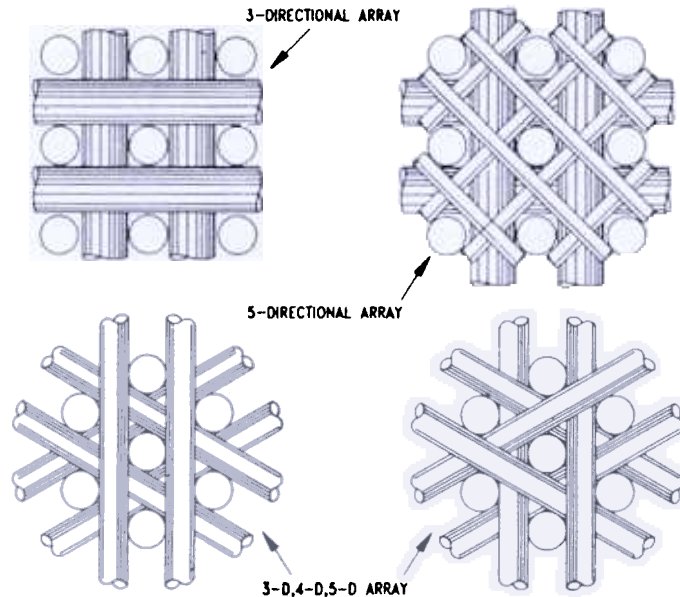
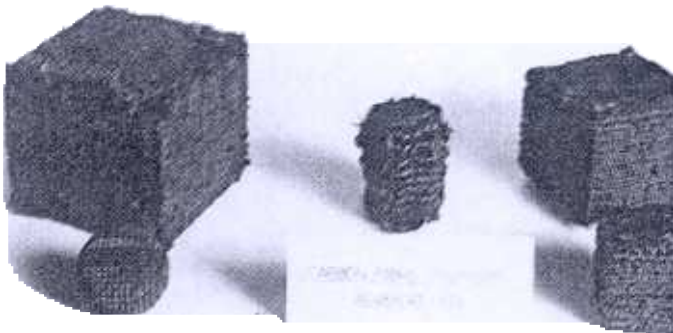
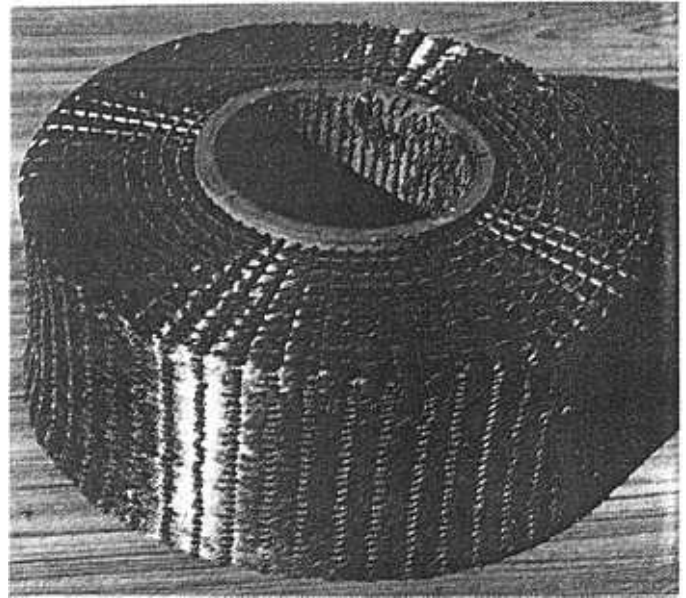
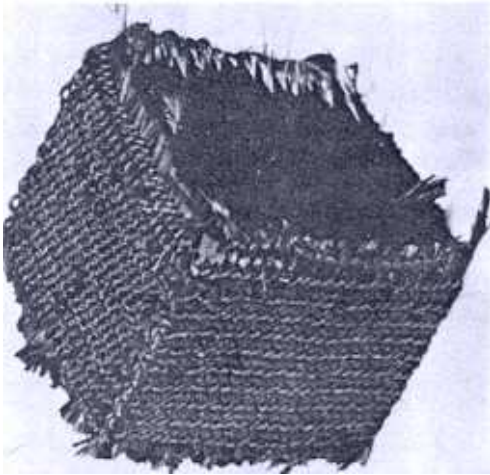
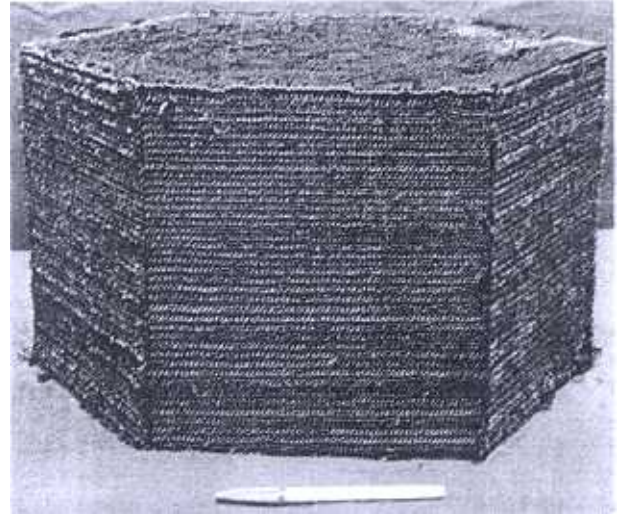
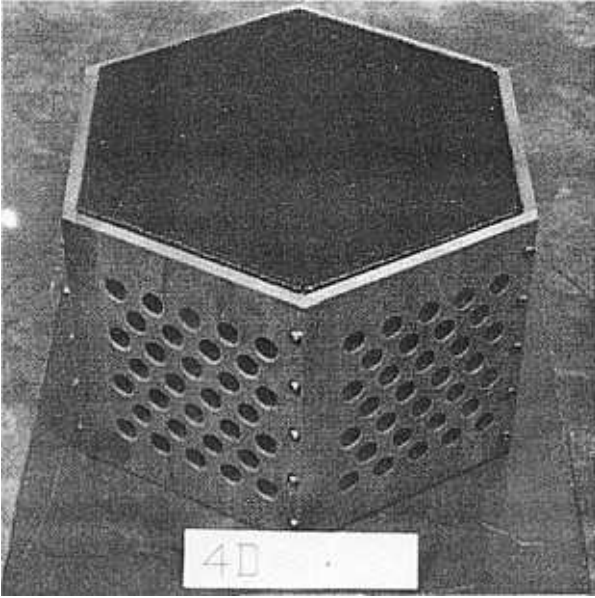


Figure 3. 3-D, 4-D, and 5-D arrays.

preform. These characteristics are calculated for a typical unit cell in the preform. Several weave modifications to the basic 3-D orthogonal designs are possible as shown in Fig. 3, to form a more isotropic structure in 4-D, 5-D, 7-D and 11-D. To enhance the composite properties between the planes, diagonal yarns are introduced. The multidirectional preform technology, also known as the fibre architecture, employs multidisciplinary approaches of structural engineering, mechanical engineering and textile technology to develop preforms in simple blocks, cylinders, cones, contours, surfaces of revolution and complex geometries and shapes. The techniques employed are conventional weaving with dry yarns, pierced fabrics, assembly of pre-cured rods, on manual, semiautomated and automatic loom set-ups and 3-D braiding and 3-D knitting. Countries like USA, France where this technology was pioneered, have kept this technology closely guarded due to its immediate adaptability to strategic products. With relentless efforts and innovation, DRDL has developed the multidirectionally reinforced preform technology for 3-D, 4-D, 5-D and 6-D preforms in blocks and cylinders with varying weave parameters. The technology and facilities are established to develop multidirectional preforms using manual and semi-automated looms. Figure 4 shows the possible material variants to preforming⁸. The weaving technology and defect characterisation techniques are developed to realise



preforms way

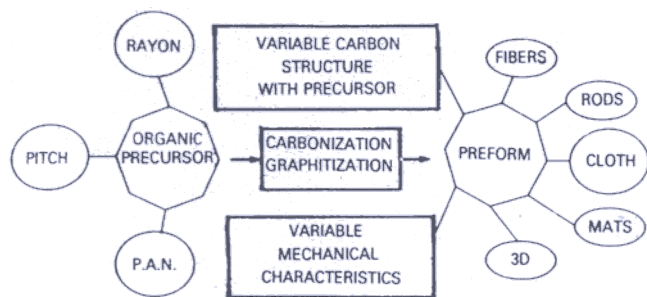


Figure 4. Material variants to preforming.

defect-free preforms. Efforts are being made to produce preforms in near-net shapes using automation techniques and 3-D braiding technology. Figure 5 shows six different multidirectional preforms woven at DRDL.

4. CC PROCESSING TECHNOLOGY

The CC densification process involves in-depth deposition of secondary carbon from different precursors using either gas phase impregnation or the liquid phase impregnation.

4.1 Gas Phase Impregnation (Chemical Vapour Deposition, CVD)

This technique uses volatile hydrocarbons such as methane, propane, benzene and other low molecular weight units as precursors. Thermal decomposition is achieved on the heated surface of the carbon fibre substrates resulting in a pyrolytic carbon deposit. This technique can be employed to deposit carbon on to dry fibre preforms or to densify porous CC structures produced by the liquid impregnation route, in which case it is referred to as chemical vapour infiltration. This process route was widely used by the Western countries for the production of thinner parts like aircraft brake discs and nozzles. CC process technology using CVD technique is yet to be established in our country.

4.2 Liquid Phase Impregnation Process

This process involves impregnation with liquid impregnants like coal tar/petroleum pitches and high char-yielding thermosetting resins.

The criterion for selection of impregnates is based on the characteristics like viscosity, carbon yield, matrix microstructure and matrix crystalline structure which are considerably influenced by the time-temperature pressure relationships during the process. The two general categories are aromatic, ring-structured,

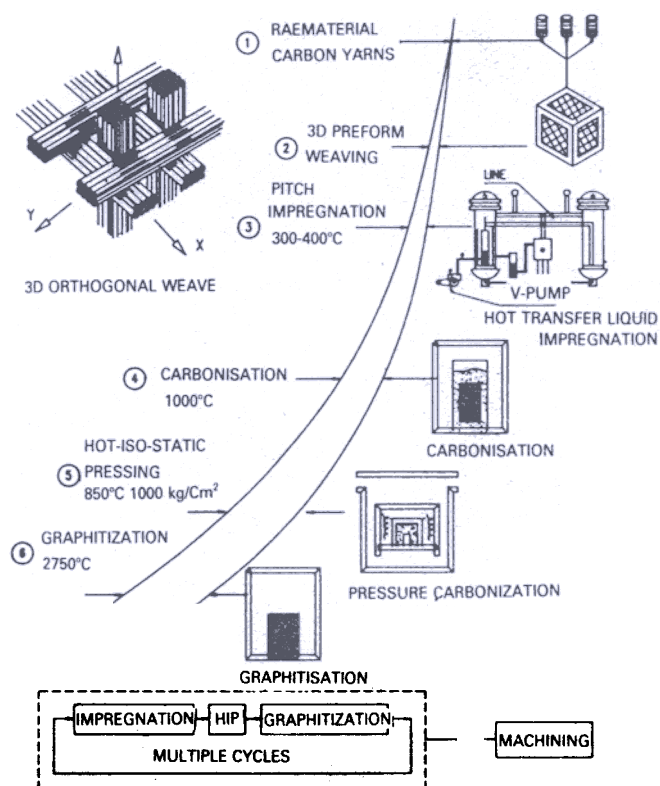


Figure 6. Carbon-carbon manufacturing process.

conventional thermosetting resins such as phenolics, furans and advanced resins like ethynyl pyrenes or pitches based on coal tar, petroleum and their blends. Figure 6 shows the CC manufacturing process using the multiple impregnation, carbonisation (1000°C), high pressure (1000 bars) carbonisation (HIP) and graphitisation (2750°C). In atmospheric pressure carbonisation, the carbon yields obtained from pitch are only around 50 per cent i.e. approximating those from high yield thermosetting resins. Yields as high as 90 per cent can be obtained by carbonising the pitch under high pressure of 1000 bars, thus making the process more efficient. Pressure applied during pyrolysis also affects the matrix microstructure. The higher the pressure the more coarse and isotropic will be the microstructure due to the suppression of gas formation and escape. High pressure also helps in lowering the temperature of mesophase formation in pitch, resulting in highly oriented crystalline structure. The HIP process is the only practical route to lower the production cost of CC composites⁹.

DRDL has established the state of the art facilities for the prototype production of CC products up to a maximum density of 2.0 g/cc. Apart from the basic process equipments, DRDL has designed and fabricated

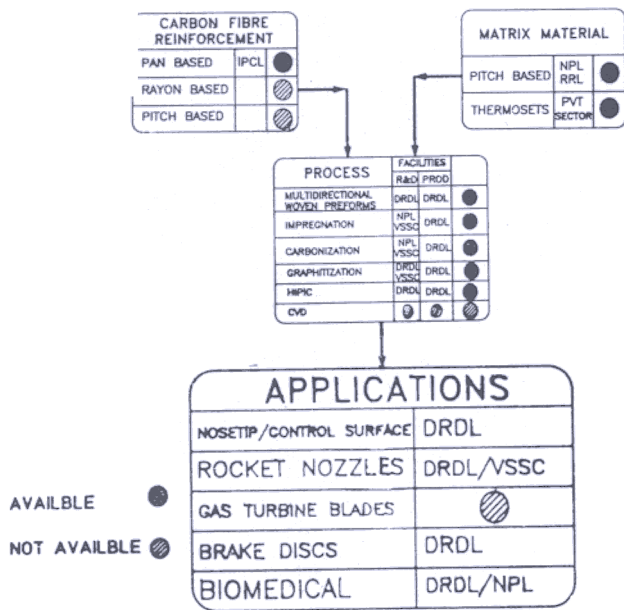


Figure 7. CC composites status in India.

several auxiliary support systems such as centralised nitrogen gas supply, closed loop process cooling station, and ventilation/pollution control devices. Also the CC technology group has established the machining facilities and standardised the machining parameters for CC composites.

CC process facilities like impregnator/carboniser exist with various institutions like VSSC and NPL. The present status of this technology is depicted in Fig. 7.

5. OXIDATION RESISTANCE

5.1 Oxidation Protection Mechanisms

Notwithstanding the attractive mechanical and thermal properties of CC at elevated temperatures, some of the potential applications like turbine structural components which require long term exposure to high temperature are restricted by the inherent reactivity of carbon towards oxygen beyond 500°C. A number of different oxidation protection mechanisms have been explored to improve the oxidation resistance of CC composites¹⁰. The techniques developed can be categorised as:

- (a) Surface coatings: single layer/multilayers, using chemical vapour deposition, pack cementation, physical vapour deposition (PVD) and plasma spray.
- (b) In depth protection includes solgel process, impregnation with inorganic salts (for limited

temperature range) and melt impregnation or in depth deposition of SiC matrix.

The materials and process known to give oxidation protection are given in Table 1.

With the external protection methods, the thermal expansion mismatch between carbon material and possible refractory coatings is the main problem to be overcome. Microcracks developed in refractory layers have to be sealed with glassy coatings. The best oxidation resistance was achieved in which CVD surface coatings were formed in addition to in depth protection.

Internal protection methods include (i) direct removal and or deactivation of catalytic impurities, and (ii) incorporation of oxidation inhibitors and total or partial substitution of matrix material.

A successful protection system comprises a coating, internal inhibitor and a compatible substrate since CC

Table 1. Various materials known to give oxidation protection

Short-term protection	Long-term protect up to 600°C
1. Pack cementation with SiC, SiO ₂ followed by impregnation with alkali silicates to seal the cracks.	Impregnation with inorganic salts, boron oxides, phosphates, and halogen compounds.
2. Sintering with SiC and B ₄ C.	Up to 1500°C
3. Impregnation with tetra ethyl-orthosilicate	(i) Chemical deposition of Rh,
Solgel process for addition of ceramic powders and glasses.	Tantalum, carbides and nitrides of titanium, silicon
5. Chemical vapour deposition of SiC	nitride, tungsten carbide.
6. Spraying of Ni and Si in nitrocellulose lacquer followed by sintering in vacuum to form Ni-Si metallic phase of SiO ₂	(ii) Cermets of refractory materials such as ZrB ₂ , MoSi ₂ or Si ₃ N ₄ and carbides, oxides silicides, nitrides of metals like tantalum.
7. Chemical reaction with molecular silicon to form SiC.	
8. Hafnium diboride, hafnium oxide, iridium for temperatures beyond 1700°C.	

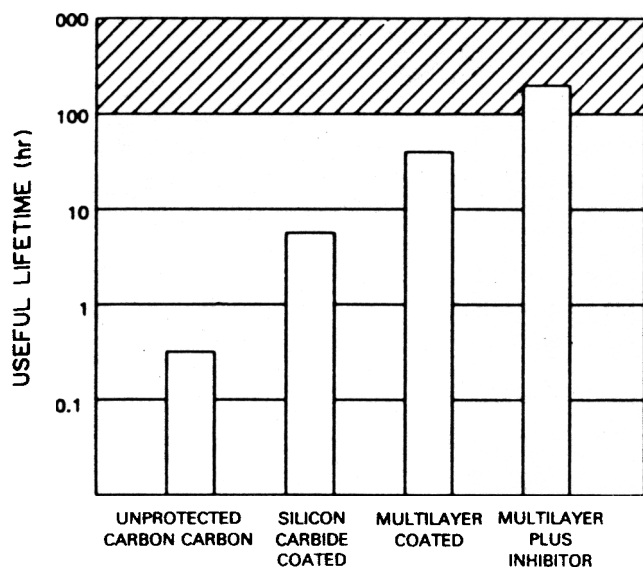


Figure 8. Process in oxidation protection research.

composites constitute a diverse class of materials with a wide range of mechanical, thermal and morphological properties. Selection of appropriate fibre, preform fabrication technique, matrix precursor and densification processing method is essential if good oxidation resistance as well as physico-chemical compatibility between substrate and coating is to be achieved. The progress on research on oxidation protection is illustrated by the bar chart shown in Fig. 8.

5.2 Higher Oxidation Resistance

Introduction of a ceramic matrix like SiC instead of carbon matrix in the carbon fibre preform gives higher oxidation resistance than that of oxidation-resistant CC. These composites known as C/SiC composites provide a good trade off between the high temperature capability of carbon fibres and the high oxidation resistance of ceramic matrices. Extensive work has been carried out by SEP FRANCE on C/SiC composites for liquid propellant rocket and air breathing engines, thrust vectoring nozzles, hot gas valves and tubes and space plane thermal structures. The third family of thermo-structural composites, viz. SiC/SiC , employ ceramic fibres (SiC) and ceramic matrix (SiC). These composites provide an excellent oxidation resistance for long durations and capable of withstanding thermal cycling for re-usable structures. SiC/SiC composites are used for liquid propellant rocket engine chambers, jet engines, gas turbine components and space thermal structures. However, SiC/SiC composites start losing the mechanical strength beyond $1200^{\circ}C$ unlike carbon

carbon composites. DRDL has documented the processing methods of C/SiC and SiC/SiC composites (see Fig. 9) and initiated research activities in this regard.

6. CC PRODUCT DEVELOPMENT EXPERIENCE

DRDL has initiated research and development activities in different aspects of CC technology for realising several hi-tech CC products. The main thrust of the effort is to establish the CC composite process technology and the study of the influence of: (i) different types of carbon fibres in various fibre architectures, (ii) impregnants, and (iii) process parameters of impregnation, carbonisation, high pressure carbonisation and graphitisation, on microstructure, physical, thermal, mechanical, thermo-structural and tribological performance of CC composites. DRDL has pioneered the challenging tasks of design, development and qualification of full scale products like 3-D and 4-D CC composites and CC aircraft brake discs. Research is under active progress for biomedical products like CC bone implants and heart valves. Collaborative research with NPL includes development of pitch impregnants and oxidation-resistant CC composites. Extensive process and material characterisation data have been generated during the development of the above mentioned products. While in multidirectional CC composites, preforming with multidirectional reinforcements is a complicated and challenging task, in bi-directional composites densification is a complex process involving optimum selection of process parameters like heating rates, temperatures, pressures and pressure gradients to avoid delamination due to evolution of pyrolysis gases, shrinkage and thermal stresses. DRDL has conducted systematic and planned experimentation to establish densification process parameters to get delamination-free 2-D composites. Development experience of CC composite brake discs is chosen to discuss some of the process and test data generated.

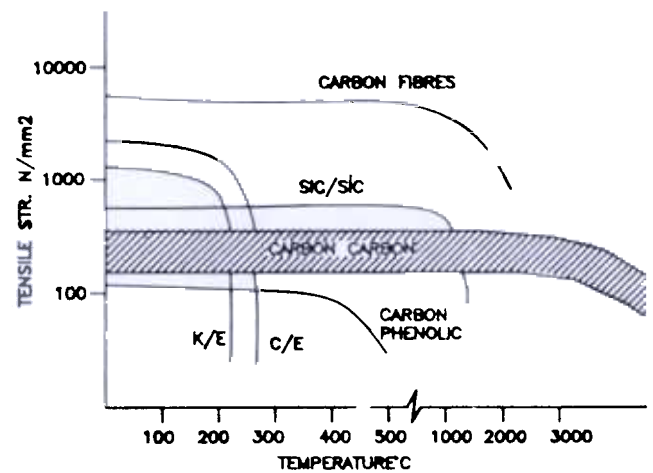
6.1 Development of CC Brake Discs

CC composites are used in aircraft brakes as thermostructural, frictional and as heat sink materials. Aircraft wheel brakes, in addition to producing the required drag by generating frictional torque, have to absorb large quantities of energy in the form of heat. CC composites have been developed to meet these

APPLICATIONS	
●	RE-USABLE THERMAL STRUCTURES FOR RE-ENTRY VEHICLES AND AIRCRAFTS
●	GAS TURBINE WHEEL AND BLADES
●	LIQUID ENGINE CHAMBERS AND
●	RAMJET NOZZLES, ROCKET NOZZLES AND THRUST VECTOR NOZZLES

TYPICAL PROPERTIES		
PROPERTY	2D C/SIC	2D SIC/SIC
DENSITY (gm/cc)	2.1	2.5
TENSILE STR (MPa)	300-350	300-400
TENSILE MOD (GPa)	90-100	200-230
MAX. TEMP OF USE IN AIR	1400°C	1600°C
DURATION OF USE AT ELAVATED TEMP HOURS	0.3-3.0	3 - 30

THERMO-STRUCTURAL COMPOSITES PERFORMANCE PROFILE



PROCESS TECHNOLOGIES	
CHEMICAL VAPOUR INFILTRATION	
POLYMER PYROLYSIS	
SOL-GEL PROCESSING	
PACK CEMENTAION	

Figure 9. Silicon carbide/silicon carbide composites.

property requirements. CC brake discs offer significant advantage in terms of high specific heat and high operating temperatures besides substantial reduction in the weight. Their use also offers long service life with also stable frictional properties through the entire range of operating temperatures. The design requirements call for light material with good heat sink properties, high temperature structural stability, thermal shock resistance, good frictional characteristics with low wear rates for long service. To achieve the above design requirements, a brake disc material should have the properties: (i) high specific heat, (ii) high thermal conductivity, (iii) low coefficient of expansion, (iv) good strength at elevated temperature, (v) high and stable coefficient of friction, and (vi) low wear rate.

CC composites possess all the above mentioned properties and are a promising candidate material for brake disc application. Tables 2 and 3 give the properties and design data of materials commonly used in brake discs, indicating the advantages of CC composites over conventional brake disc materials. Figure 10 shows the

Table 2. Properties of different disc materials

	CC	Steel	Copper
Density(g/cc)	1.7	7.8	8.9
Specific heat(J/g/K)	1.42	0.59	0.42
Thermal conductivity(J/m/s/k)	100-150	54	346
Coefficient of thermal expansion($k \times 10^{-6}$)	0.8	14	18
Tensile strength (Mpa)	66	410	240
Impact resistance (J)	0.7	110	55

Table 3. Comparison of typical brake design data

Design	Copper brake & organic pad	Steel brake & Ceramic Sintered pad	CC composite
Landing loading (KJ/kg)	240	450	390
RTO loading (KJ/kg)	540	750	960
Landing (coeff. of friction)	0.27	0.25	0.23
RTO (coeff. of friction)	0.17	0.18	0.15
Landing temperature(°c)	580	650	600
RTO temperature (°c)	1000	1000	1050
Relative weight	-	100	100
Relative volume	-	100	100

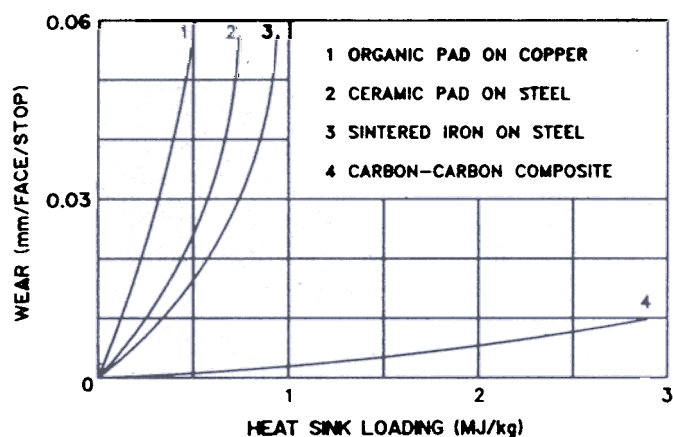


Figure 10. Comparative wear rates of brake disc materials.

advantages of CC composites over conventional brake disc materials in terms of wear.

CC brake materials usually comprise different types of carbons each of which exhibits different friction and wear characteristics. For example, carbon fibre reinforcement may be from PAN, rayon or pitch precursors in different fibre architectures to meet the thermostructural and tribological performance.

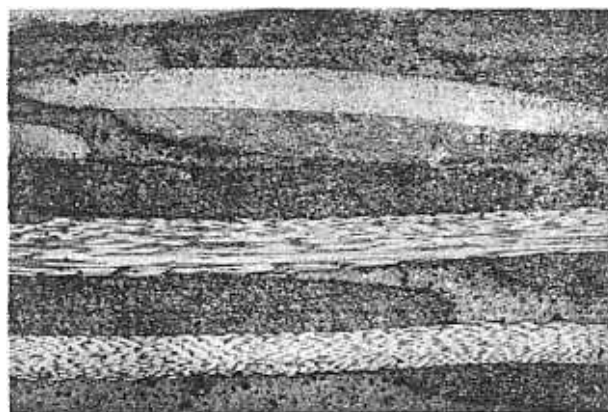
The carbon matrix can be directly formed using chemical vapour deposition or carbonising of resin or pitch precursors.

The type of microstructure formed depends upon the matrix precursors used. The commercial manufacturers in the west generally employ CVD matrix for the brake discs. Pitch-derived carbons are highly graphitisable and soft and exhibit excellent thermal properties but tend to undergo rapid wear. Nongraphitisable carbons from resins are vitreous and hard and exhibit large fluctuations in co-efficient of friction. The CVD carbon shows an intermediate behaviour. In view of the above, in DRDL, research is aimed at developing CC discs with an optimum combination of isotropic and an anisotropic matrices. Different types of fibre architectures with rayon/PAN carbon fibres, carbon fabric and carbon felts, and also PANOX composites, are processed with matrix combinations of resin/pitch, pitch and pitch/resin. Process parameter variations include atmospheric carbonisation, pressure carbonisation, pressure impregnation, and final heat treatment temperatures. All the composites processed are studied for microstructure, physical, thermal, mechanical and tribological behaviour. Some of the test data are discussed in what follows.

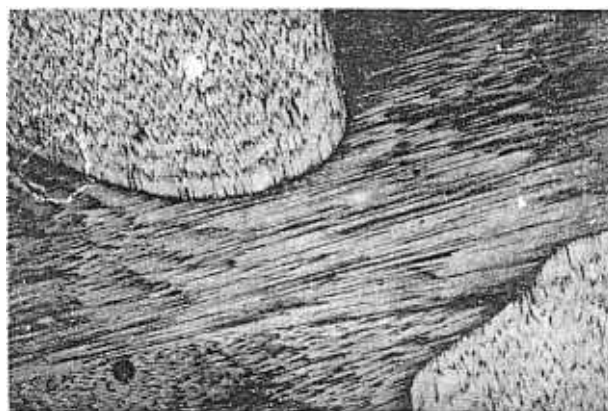
6.1.1 Microstructure

Microstructure plays an important role in the performance of CC composites. The study of microstructure gives vital information about distribution of fibres and matrix, pore distribution, isotropy and anisotropy of matrix formation and filling of cracks and extent of damage during either processing or testing. Microstructure of carbon-carbon composites has been studied using optical and scanning electron microscopes (SEM). Composite samples made with different types of fibres and matrices are studied at various stages of processing and also after subjecting to friction testing.

Figure 11 shows the optical micrograph of carbon/phenolic sample in transverse (TS) and longitudinal (LS) directions. Bulk phenolic resin matrix is seen between the layers and between carbon fibre tows (Fig. 11(a)). Bulk matrix has micropores which are produced during the curing of phenolic resin because of volatile evolution. Figure 11(b) shows the accumulation of resin near the tows crossover points, of fabric weave.



(a)

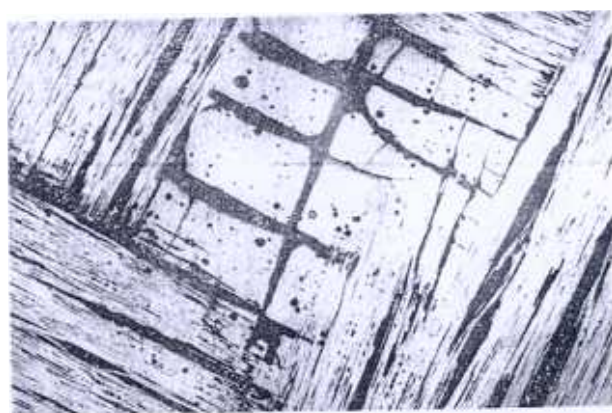


(b)

Figure 11. Optical micrograph of carbon/phenolic composite, (a) TS 50X and (b) LS 50X.



(a)



(b)

Figure 12. Optical micrograph of CC skeleton, (a) parallel 50X and (b) LS 50X.

Figure 12 shows the optical micrographs of carbon/carbon skeleton formed from carbon/phenolic composite after carbonisation. Gaps/cracks are seen in the transverse tows. No cracks are seen in the longitudinal tows. This indicates that the fibre is not getting damaged during skeleton formation but in transverse tows filaments are getting separated. Generally, cracks occur during pyrolysis as shrinkage of resin is restricted by the surrounding fibres. Strong chemical adhesion between fibre and resin has to be avoided to prevent the damage of carbon fibre during carbonisation.

A study of different matrix precursors namely resin (R) and pitch (P) for skeleton formation showed that the porosity is slightly higher in composites totally processed with pitch material possibly due to the higher density of carbon derived from pitch. The final density of the composite can be achieved faster with pitch matrix.



Figure 13. SEM micrograph of CC composite with total pitch-derived matrix (P+P), (L8 2000X).

During the graphitisation process, the carbon derived from pitch readily undergoes graphitisation and the extent of graphitisation depends upon the heat treatment temperature. Carbon derived from thermosetting resin is generally glassy in nature and becomes graphite only under stress-induced graphitisation. The pitch carbon is anisotropic as seen under polarised light. Figure 13 shows the SEM micrograph of the composite totally processed with pitch. Bulk matrix and matrix around the filament shows lamellar structure indicating good graphitisation. Figure 14 shows the SEM micrograph of the composite where skeleton was made using resin and subsequent densification was carried out by pitch. The bulk matrix (pitch) is in lamellar structure. But the matrix around the filament shows the lamellar structure to a limited extent only.

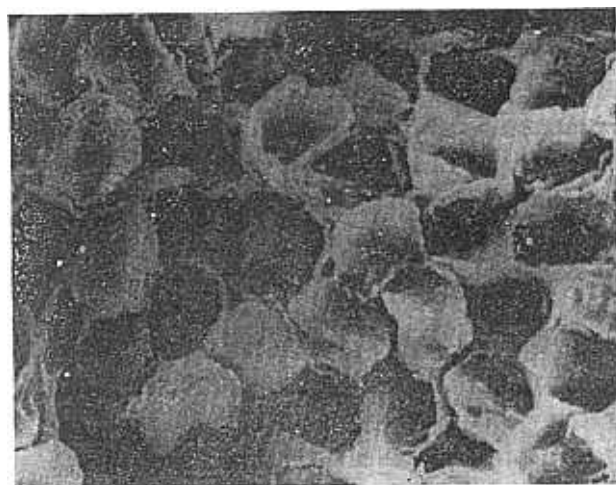
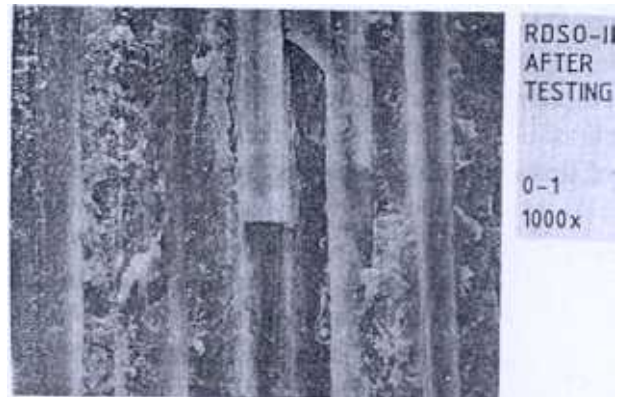


Figure 14. SEM micrograph of CC composite (R+P) (B-7 2000X).



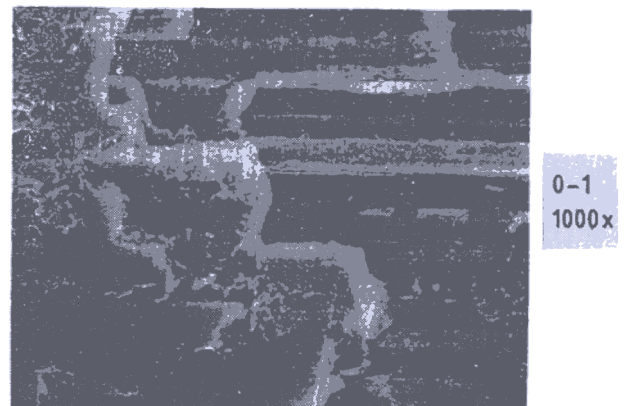
(a)



(a)



(b)



(b)

Figure 15 SEM micrograph of air-exposed surface of dynamometer tested CC disc samples.

Figure 16. SEM micrograph of friction surface of dynamometer tested CC brake disc sample.

Figure 15 (a) shows the effect of oxidation on CC composites. The figure shows the SEM micrograph of the sample from air-exposed and unprotected surface after carrying out dynamometer testing. Fibres are projected out leaving behind the matrix. This indicates that the matrix is more prone to oxidation than the fibres. Figure 15 (b) shows the extent of damage because of oxidation inside the composite after 60 dynamometer stops. Deterioration is to the extent of 2-3 mm deep only. This can be avoided by using a suitable oxidation resistant paint in the exposed portion. Figure 16 shows the SEM micrograph of rubbing surface of carbon-carbon brake disc sample. It shows the incomplete formation of friction film. Formation and maintenance of friction film is important to control the wear rate of the composite.

6.1.2 Densification Efficiency

For the fabrication of 2-D CC composites using liquid impregnation process, two types of preforming techniques are adopted: (i) preforming by compression moulding with thermosetting resin followed by carbonisation, and (ii) preforming directly by pitch impregnation followed by carbonisation. Subsequent densification can be either with pitch or resin.

Densification process efficiency in terms of density gain depends upon the skeleton density, porosity and pore size distribution, heating rate during the skeleton formation and other process parameters, like temperature of impregnation, pressure of carbonisation, temperature of graphitisation, sequence of densification of impregnation, carbonisation,

pressure carbonisation, intermediate graphitisation and machining. The stages involved in the processing of discs are: (i) skeleton formation, and (ii) densification using multiple pitch impregnation, carbonisation (atmospheric pressure/high pressure) and graphitisation. In the case of skeleton formation with resin, first carbonisation is very important. Optimised cycle is to be followed to avoid the delaminations. Heating rates as low as 4°C/hr are to be used in the pertinent zones. Selection of cycle parameters is highly dependent on product shape and size. A carbonisation cycle optimised for a small product cannot be directly used for larger discs. Delamination during skeleton formation was the main problem during the development of full scale products. This was solved by using appropriate heating rates during different stages of pyrolysis and loading methods. For the densification of skeletons, two options are available for carbonisation: (i) atmospheric pressure carbonisation, and (ii) high pressure carbonisation.

Atmospheric pressure carbonisation, it takes 15-17 densification cycles to get a density of 1.7 g/cc. In high pressure carbonisation process, it was possible to achieve this density just in there densification cycles after process optimisation. Figure 17 shows the differences in the densification by the various processes, viz. atmospheric carbonisation, high pressure carbonisation and an optimised densification sequence.

Graphitisation is another important step in the development of brake discs, which changes the microstructure of amorphous carbon to graphite like structure. The extent of graphitisation depends on the precursor used. Delaminations during graphitisation was a major problem during the initial development. This problem was solved by choosing appropriate heating cycle. Not only the heating rates but also the ultimate temperature has considerable bearing on the final thermal, mechanical and tribological properties of the composites. Graphitisation temperature affects the properties of resin carbon and pitch carbon to different extents.

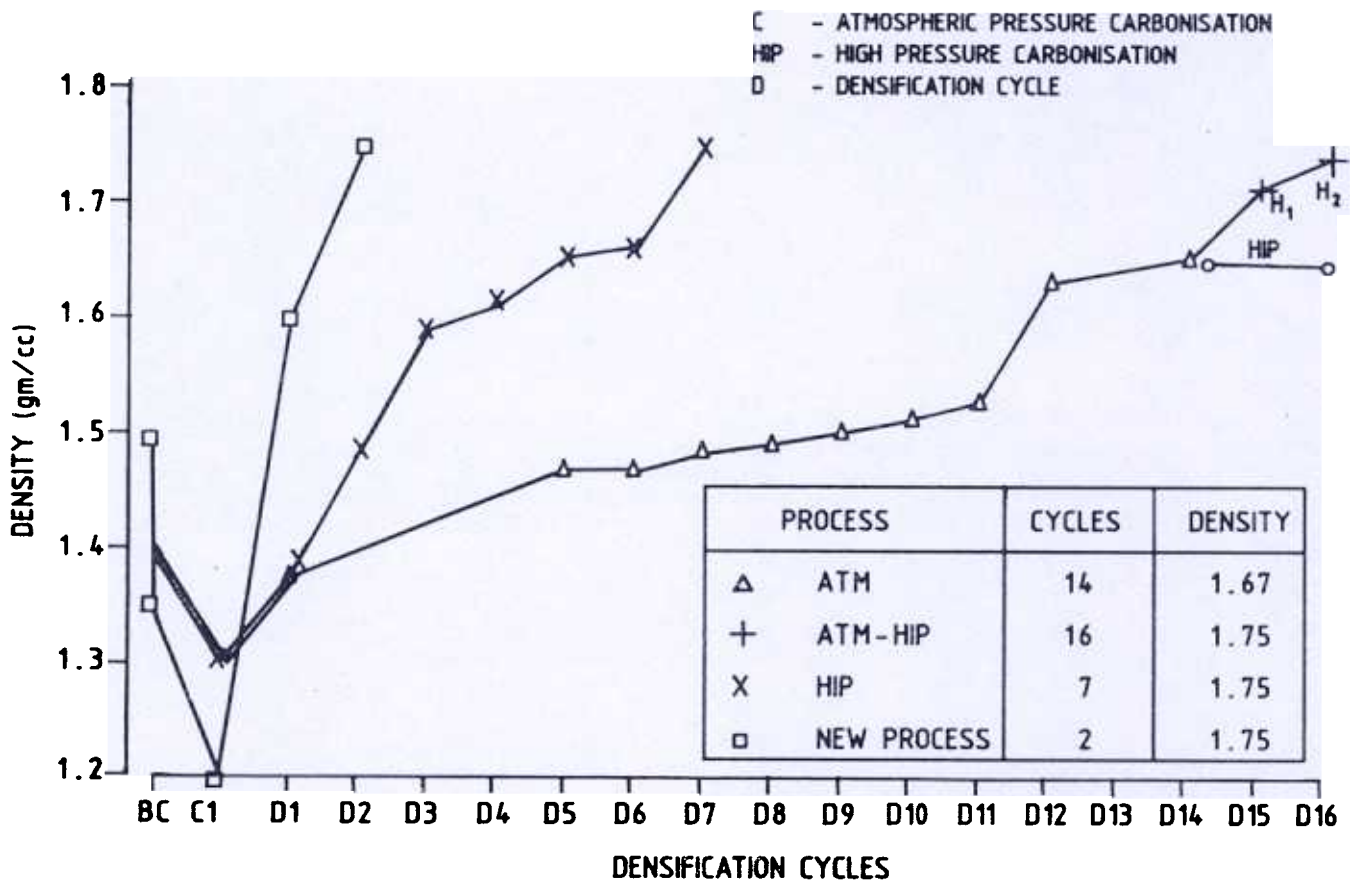


Figure 17. Densification progress by different routes and optimised process sequence.

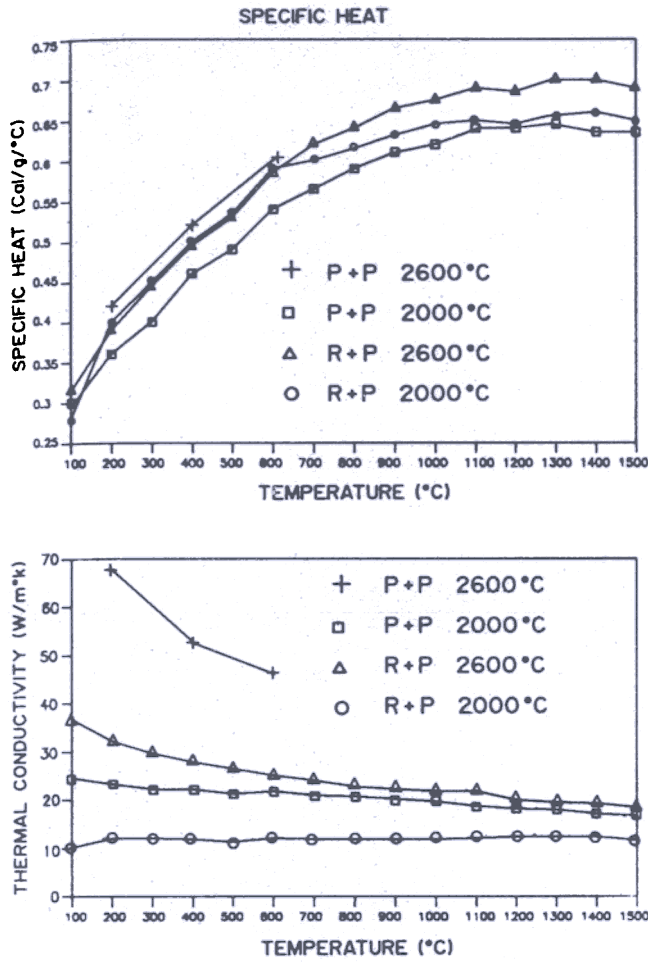


Table 4. Material characterisation of multidirectional CC composites

	3-D CC	4-D CC			
Fibre type	Rayon based	Pan based			
Fibre volume fraction (%)					
Total	44	38			
Z direction	27.7	13.8			
X-Y direction	8.14	Each			
U, V, W direction	—	8.0			
Physical properties					
(i) Density (g/cc)	1.80	1.816			
(ii) Porosity (%)	7.498	11.90			
Mechanical properties		Z	X-Y	Z	U
Tensile strength (MPa)	78.55	24.59	82.23	76.60	
Tensile modulus (GPa)	89.49	25.07	38.23	30.60	
Flexural strength (MPa)	95.97	38.21	101.46	—	
Flexural modulus (GPa)	19.29	5.72	35.59	—	
Compression strength (MPa)	96.00	73.19	83.11	40.54	
Thermal properties					
Specific heat (Cal/g °K)					
200 °C	0.32	0.32	0.42	0.40	
1000 °C	0.56	0.59	0.82	0.65	
1500 °C	0.58	0.596	0.88	0.72	
Thermal conductivity (W/M/K)					
200 °C	148	130	70	54	
1000 °C	83	80	49	37	
1500 °C	88	84	49	35	

Table 5. Generation of test data on indigenous CC materials

CC material	2	3	4	
Reinforcement type	PAN(HS)	PAN(HS)	PAN(HM)	PAN(HS)
Type of lay-up	Plain	Plain	Plain	Plain
Type of matrix	Resin/pitch	All pitch	Resin/pitch	Resin/pitch
Density (g/cc)	1.64	1.74	1.65	1.73
Open porosity (%)	7.80	7.23	6.69	7.42
Flexural strength (MPa)	214	206	149	191
ILSS (MPa)	11	14	—	11
Tensile strength (MPa)	105	120	132	—
Compression strength (MPa)	126	109	—	127

testing of CC brake discs for tribological behaviour. CC composite rotors (2 Nos) and stator discs (3 Nos), assembled alternately in a typical aircraft brake wheel assembly, are chosen for evaluation in the deceleration

Figure 18 shows the effect of graphitisation temperature on resin skeleton + pitch densification (R+P) and pitch skeleton + pitch densification (P+P). Increase in the graphitisation temperature increases the thermal conductivity as well as specific heat of the composite, the increase being more pronounced in case of P+P composites; it is 2-3 times higher when heat treatment temperature is raised from 2000 to 2600°C.

6.1.3 Physical Thermal and Mechanical Properties

Table 4 lists the physical, thermal & mechanical properties measured on the multidirectional composite samples. The test data on 2-D CC composites made with PAN-based high-strength and high-modulus carbon fibres, are presented in Table 5.

6.1.4 Tribological Behaviour

Study of co-efficient of friction and rate of wear for a given heat energy input under a specified brake pressure forms the main part of the dynamometer

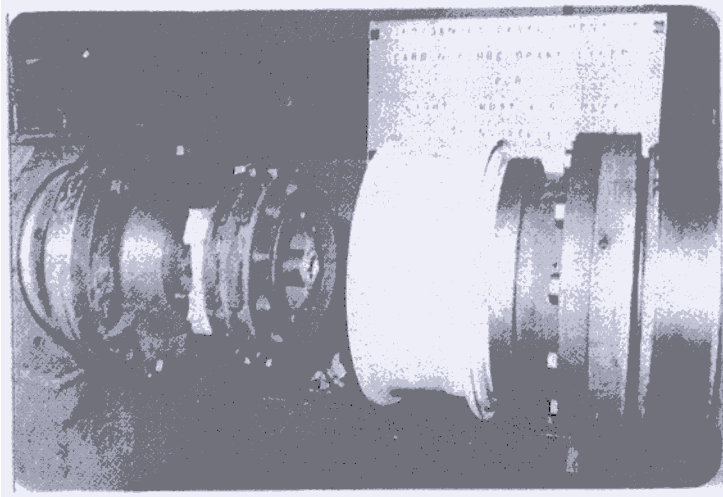
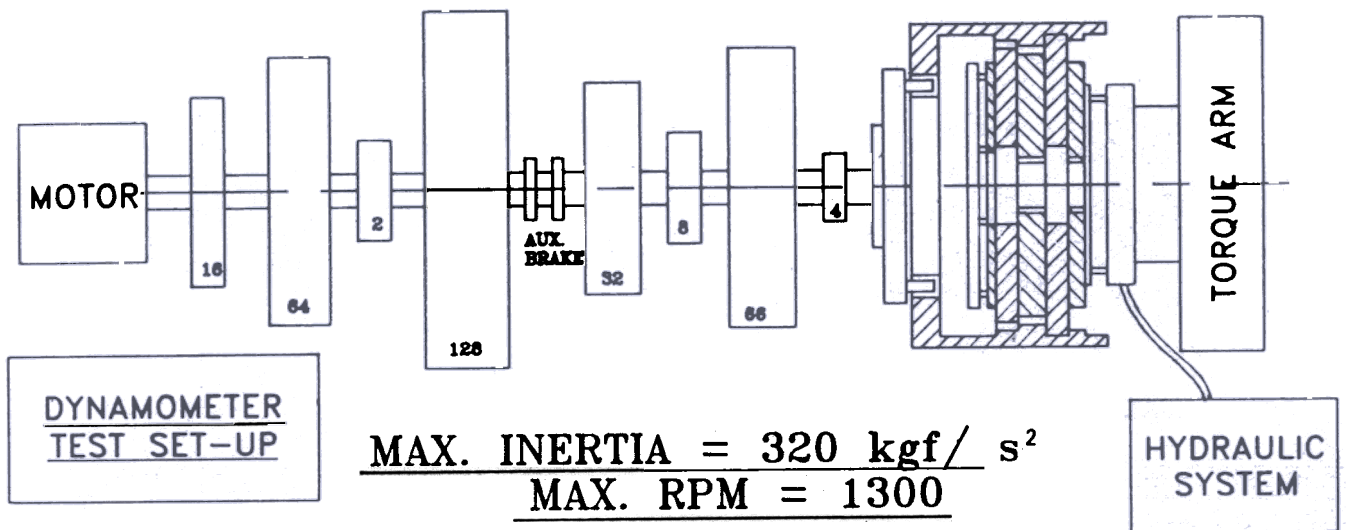


Figure 19. Test set-up for evaluation of CC brake disc in friction dynamometer.

dynamometer facility at RDSO Lucknow. The test set-up (Fig. 19) consists of 8 fly wheels of various gyrating masses on a shaft connected to a dc motor of

350 Kw. The rpm of the shaft can be varied up to 1300. The required gyrating mass inertia can be introduced by connecting a selected set of flywheels. The CC heat pack along with the aircraft wheel is mounted on the dynamometer. Brake pressure is applied through a hydraulic power pack. The system is connected to a control and recording system. Parameters like stop time, run down revolutions, brake pressure, torque, brake energy absorbed and bulk temperature of the heat pack are continuously monitored during a run. The performance of CC indigenous discs developed with different fibre orientations are evaluated.

Rosette and plain lay-up discs have been subjected to dynamometer testing simulating normal (21.2 MJ) and overload (29.8 MJ) landing kinetic energies of the aircraft. Figure 20 indicates the test set-up (schematic) with the test plan for 50 dynamometer runs with 45 normal and 5 overload stops.



TEST PLAN

1 BLOCK=9N.L+1 O.L
TOTAL BLOCKS 5 ON EACH ASSY.
TOTAL NO. OF RUNS = 50 (15 N,L+5 O,L)
1 R.T.O

TEST CONDITIONS

	NORMAL	OVER LOAD	R.T.O
WHEEL RPM	1196	1250	1250
MASS INERTIA kgf/s ²	136	174	276
BRAKE PRESSURE	90	1038	157

N.L = NORMAL LOAD, O.L = OVER LOAD
R.T.O = REJECTED TAKE OFF

Figure 20. Dynamometer test set-up.

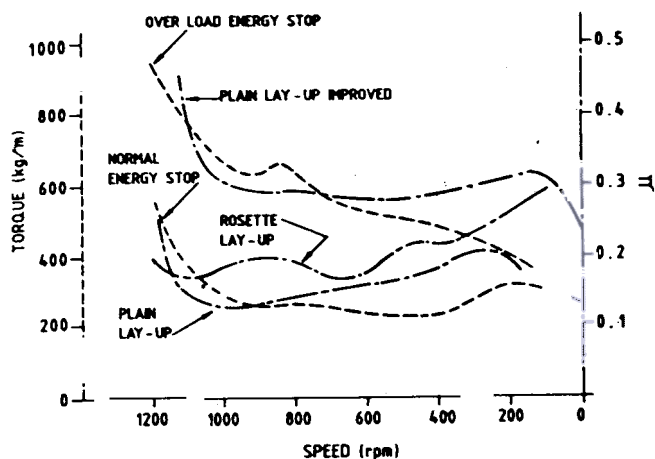


Figure 21 Variation of torque and coefficient of friction during typical brake runs.

In the case of rosette lay-up, coefficient of friction was in the range 0.20-0.26 and wear was 8 microns/face/stop. In the case of plain lay-up, coefficient of friction was in the range 0.13-0.18 and wear was on the higher side. Figure 21 shows the torque recorded as a function of speed during typical normal and overload stops, it also incorporates the variation of the coefficient of friction during a typical run carried out with rosette and plain lay-up discs. Analysis of the total test data in relation to the material characterisation pointed to the need for certain improvements with respect to degree of graphitisation, thermal conductivity and the fibre volume.

To improve the thermal conductivity, toughness and tribological properties, a new batch of discs are processed and successfully tested in the dynamometer simulating normal, overload and rejected take off conditions. Coefficient of friction was consistent in all the 30 normal energy stops and the value is 0.22-0.33 with a stop time of 22.5-32.5 seconds. Figure 21 shows the coefficient of friction recorded during a typical run of the improved material.

ACKNOWLEDGEMENTS

The authors wish to thank the Aeronautical Development Agency (ADA), Bangalore for sponsoring the project on 'Development of CC

Composite Brake Disc Technology' and also the Vikram Sarabhai Space Centre (VSSC), Trivandrum for extending the support for thermal characterisation.

REFERENCES

- 1 Fitzer, Erich. The future of carbon-carbon composites. *Carbon*, 1987, 25(2), 163-90.
- 2 Singh, Himmat. New generation carbon materials. Paper presented in National Workshop on Newer Carbon Products - Indian Scenario, held in Bangalore, 9-10 October 1992.
- 3 Delmonte, John. Technology of carbon and graphite fibre composites. Van Nostrand Reinhold Company, New York, 1981. pp. 388-419.
- 4 International symposium on carbon 1990 (Tsukuba).
- 5 Wright, Maurice A. Materially speaking. Materials Technology Center, Southern Illinois University, 1990.
- 6 Appell, Y.F. New composite materials for aerospace motor nozzles and their applications in the aircraft industry. French Aerospace Seminar held in Bangalore, 1981.
- 7 Mcallister, L.E. Multidirectional carbon-carbon composites. *In Hand book of composites*, 1983, Vol 4, pp. 109-75.
- 8 Choury, J.J. Thermostructural composite materials in aeronautics and space application French Aerospace 1989 (Aeronautical and Space Conference) 15-17 Febuary 1989, Delhi.
- 9 Klein, Allen J. Carbon-carbon composites: advanced materials and processes. *Metal Progress*. 1986, 130(5), 64-68.
- 10 Webb, Richard D. Oxidation resistant carbon-carbon material. Research Project abstract by AVCO Systems USA.
Huttner, W. Carbon-carbon--an inverse composite structure. *Z. Werkstofftech*, 1985, 16, 430-35.