



## Carbon-Carbon Composites – A Review

**Anilas K, Surendranathan A.O\***

Department of Metallurgical & Materials Engineering, National Institute of Technology Karnataka (NITK), Surathkal, P.O.Srinivasnagar-575025, Karnataka, India.

### Abstract

Carbon-carbon composites (CCC) stand unique in the class of composites as they possess some extra-brilliant properties which the other composites do not have. They combine the advantages of both carbon fibre and carbon matrix such as high specific strength, stiffness and in plane toughness as well as refractory properties like retention of mechanical properties at extremely high temperatures of the order of 3000°C. This has resulted in the exploitation of CCC as structural materials in space vehicles, heat shields, rocket nozzles and aircraft brakes. Properties like biocompatibility and chemical inertness have led to new applications in medicine industry. Carbon fibres may be combined in a wide variety of forms; woven, knitted, braided and filament wound, to provide 1, 2 and multidirectional reinforced composites. The matrix can be a vapor deposited carbon, glassy carbon or coke. Also, the degree and extend of graphitization may be varied considerably, imparting a wide range of thermo-chemical properties of composites.

Conventional fabrication techniques like Gas phase impregnation (Chemical vapour deposition (CVD)) and Liquid phase impregnations were employed in the early ages of CCC fabrication. Later on, modifications like liquid phase pre-impregnation (LPI), hot pressing, hot isostatic pressure impregnation carbonization (HIPIC) and isothermal CVI saw the improvement in properties of the fabricated CCC. Advanced techniques like Preformed Yarn (PY) method showed exceptional values for specific strength and fracture toughness. Successful modifications and modern techniques like application of temperature, densification, increasing the number of pitch/resin impregnation cycles, using carbon nano tubes/carbon nano fibres as reinforcements, growing multi walled carbon nano-tubes on woven carbon fibre, fibre architecture modifications, microwave curing, fibre sizing, ultra-high temperature ceramic coating are also reviewed to throw light on how these have improved the important mechanical properties, which make CCC as the “material of future”.

**Copyright:** © 2018 Unique Pub International (UPI). This is an open access article under the CC-BY-NC-ND License (<https://creativecommons.org/licenses/by-nc-nd/4.0/>).

**How to cite:** Anilas K, Surendranathan AO. Carbon-Carbon composites-A review. UPI Journal of Engineering and Technology 2018; 1(1): 8-19.

### Article history:

Received: 14-03-2018, Accepted: 22-03-2018,  
Published: 23-03-2018

**Correspondence to:** Surendranathan AO, Department of Metallurgical & Materials Engineering, National Institute of Technology Karnataka (NITK), Surathkal, P.O.Srinivasnagar - 575025, Karnataka, India.  
Email: [nathan@nitk.ac.in](mailto:nathan@nitk.ac.in)

**Key words:** CCC, Carbon-carbon composite/s, Design, Fabrication, Properties, Applications.

## 1. Introduction

The versatility of the element carbon has proved itself to be the “*material of future*”. It is a unique element that exhibits different properties in different forms - extremely hard like diamond and equally soft like graphite. It can take forms from crystalline graphite to amorphous or glassy carbon. Ultrahigh temperature properties and light weightness add advantage to its application while failures at lower strains, thermal shock sensitivity, anisotropy and processing difficulties limit the application in structures. This saw the birth of carbon-carbon composites (CCC).

Carbon-carbon composites have gained popularity (despite of their generally high cost) in high-performance products that need to be lightweight, yet strong enough to take harsh loading conditions such as aerospace components (tails, wings, propellers) and racing car bodies and brakes. Carbon-carbon composite is a key material in today's launch vehicles and heat shields for the re-entry phase of spacecraft. It is widely used in solar panel substrates, antenna reflectors and yokes of spacecraft. It is also used in payload adapters, inter-stage structures and heat shields of launch vehicles. Infact scientists have made up their minds not to repeat another Columbia space shuttle disaster. Furthermore, disk brake systems of airplanes and racing cars are using carbon - carbon composites, and the composite material with carbon fibres and silicon carbide matrix has been introduced in luxury vehicles and sports cars. The Boeing 787 and Airbus A350 structures including the wings and fuselage are composed largely of composites mainly, carbon-carbon composites. Carbon-carbon composites materials are also becoming more common in the realm of medicine like orthopedic surgery.

## 2. Fabrication Methods-Conventional

In conventional fabrication processes of CCC, carbon fibres based on rayon/PAN/pitch in the form of unidirectional, 2D and multi directional preforms are used as primary carbon. To fill the interstices between the fibres there are two routes.

- Gas phase route using chemical vapour deposition process.
- Liquid phase route using thermosetting pitch or resin.

Fibre orientation and volume fraction of fibre in the required direction controls the properties of the CCC fabricated. Fibre architecture (multidirectional preform technology) explains development of preforms in simple blocks, cylinders, cones, contours, surfaces of revolution and complex geometries and shapes [1].

### 2.1. Gas Phase Impregnation-Chemical Vapour Deposition (CVD)

In CVD, volatile hydrocarbons like methane, propane, benzene and other low molecular weight units are used as precursors. Thermal decomposition of the hydrocarbons takes place on the heated surface of carbon fibres and deposition of carbon takes place. This technique of depositing carbon on to dry fibre perform pyrolytically is referred to as chemical vapour infiltration/deposition.

### 2.2. Liquid Phase Impregnation Process

Here, impregnation is done with liquid impregnates like coal tar/petroleum pitches and high char yielding thermosetting resins. CCC fabrication may involve multiple impregnations for appreciable density, followed by hot isostatic pressing at temperatures  $\sim 750^{\circ}\text{C}$  and 100 MPa pressure. This is followed by carbonization at  $\sim 1000^{\circ}\text{C}$  and graphitization at  $\sim 2750^{\circ}\text{C}$ .

Reactivity of carbon towards oxygen beyond 500°C is of serious concern, and so a decent oxidation resistance is to be obtained. Techniques like surface coatings using CVD, pack cementation etc, SiC coatings and sol gel process, impregnation with inorganic salts, incorporation of oxidation inhibitors etc are some of them. [1]

Tongshik CHANG *et al.* [2] fabricated CCC by simple hot pressing (600°C and 49MPa) of pulverized coke powders (3.5µm) as matrix, carbonaceous bulk mesophase (BM) as binder and carbon fibres as reinforcements, in the form of sheets impregnated with epoxy resin, in unidirectional(UD) long fibres (UD1 – thinner, UD2 - thicker) and cloth(CL) forms (CL1 – thinner, CL2 - thicker).

From the results of water substitution method and 3-point bending tests, maximum density and strength was obtained at the optimum hot pressing pressure, i.e., 49MPa at 600°C. The maximum strength values of 119MPa and 130MPa were obtained for UD-1 ( $V_f = 30\%$ ) and UD-2 ( $V_f = 46.4\%$ ) at BM ratio 70% and 80% respectively in matrix. When CL-1 and CL-2 were used, strength was found to be lower than that of UD, at  $V_f = 20\%$ . However it can be concluded that both apparent density and bending strength increase with increasing  $V_f$  value.

### 2.3. Modifications in Conventional Fabrication Methods

A few modifications to the conventional liquid phase impregnation can be listed as follows:

- Before liquid impregnation, fibres are pre-impregnated with resin or pitch and carbonized at 350-800°C under a pressure of 100MPa. Liquid phase impregnation (LPI) in vacuum adds more pitch and resin, which will increase the density and inter-laminar shear strength. Graphitization at 2200-

3000°C opens up the closed pores and a further impregnation will lead to higher density.

- Hot isostatic pressure impregnation carbonization (HIPIC) is yet another development in this area, in which a high pressure of 100MPa is applied while carbonization and impregnation at 650-1000°C. This increases the carbon yield and maintains more volatile fractions of pitch in a condensed phase. It is then graphitized at temperatures more than 2000°C without pressure. This yields CCC with higher density.
- Hot pressing is yet another advancement, where carbonization at 650-1000°C and 76Mpa in an inert, reducing or vacuum atmosphere, is followed by graphitization at 2200-3000°C without pressure (Figure 1).

## 2.4. Advanced Fabrication Methods

### 2.4.1. Preformed Yarn (PY) Method

Unlike the conventional manufacturing methods like impregnation and CVD (chemical vapour deposition), a Preformed Yarn (PY) of 3 to 4mm diameter and 200 to 1000mm in length was prepared by N. Hirota *et al.* [4] in the PY method. PY has PAN-based carbon fibre as the reinforcement, coke powder and petroleum mesophase pitch (binder) as matrix precursor and polypropylene beads as the polymer that coats the carbon fibre and matrix precursor. Then, a PY block was made by unidirectionally aligning PY sheets, which were prepared by the piling up of chopped PYs. Hot pressing of the PY block in a metal mould was carried out at 600°C (10°C/min) which was then subjected to carbonization at 800°C and graphitization at 2000°C to obtain the final CCC.

- 3 point bending tests showed improved flexural strength and modulus than that obtained by conventional methods by a factor of  $\sim 1.3$ .

- Micro structural inspections reveal decent fibre / matrix bonding - the least number of pores were recognized.

P. Naik *et al.* [5] [6] [7] modified the method and used carbon fibre (PAN based) as reinforcement and pitch, coke and nylon-6 as matrix materials in PY method to fabricate CCC for aerospace brake pad application.

The PY [5] prepared here was a fibre bundle surrounded by coke and pitch which was enclosed in Nylon-6. Three types of samples having fibre weight percentage 30%, 40% and 50% were fabricated. This was followed by hot pressing at 600°C and heat treatment at 1800°C. To eliminate porosity and to attain the required density, one cycle of pitch impregnation was done on the samples by hot isostatic pressing.

From the hardness test, compression test, impact test and fracture toughness test, improved mechanical properties were obtained when carbon fibre volume % was increased.

Density of the obtained composite improved from 1.55g/cc to 1.60g/cc while the carbon fibre increased from 30 to 40%; which again showed an improvement to 1.65g/cc when carbon fibre was 50%. Similar trend was observed in the cases of hardness, compressive strength, flexural strength, impact strength and flexural modulus.

#### 2.4.2. CCC for Nuclear Reactor Applications

V. Ramani *et al.* [8] fabricated CCC by impregnation method in which performs made using PAN carbon fibres were stacked to 2D preform using phenol formaldehyde resin. Rectangular green performs were cut and carbonized at 1000°C to get highly porous sample, which were then densified using two cycles of impregnation of liquid formaldehyde resin under pressure in steps.

The following conclusions were drawn:

- CCC with density of 1470 kg/m<sup>3</sup> was obtained.
- XRD revealed that the composite was amorphous in nature.
- Higher the impregnation pressure, lower the porosity.
- X-ray tomography showed decrease in cracks and better matrix-resin bonding at higher pressures.
- SiC coating by CVD method was done for oxidation resistance, thermal shock properties and stability against hot corrosion, making it suitable for low temperature reactor applications.

### 3. Properties of CCC

CCCs are ceramic in nature but exhibit brittle to pseudo plastic behaviour. Carbon fibre reinforcements in carbonaceous matrix materials combine the advantages of both the reinforcement (high specific strength and stiffness), and the matrix (the refractory properties).

The total properties are:

- Thermal stability - thermally stable and do not melt up to ~ 3000°C.
- High thermal conductivity and low thermal expansion.
- High resistance to thermal shock.
- Good frictional properties at temperatures ~ 3000°C, low wear.
- High fracture toughness.
- Oxidation resistance.

All the above-mentioned properties make CCC the best applicable in high temperature applications like re-entry nose tips, leading edge material for space shuttle wings, rocket nozzles, and thrust vectoring nozzles, disc brakes of aircrafts and high performance vehicles and high performance turbo jet engines. CCC also find application in glass industry, furnace and semi conductor industry as well as for preventing

corrosion in chemical plants, high temperature crucibles, fasteners, load bearing plates, rods and heating elements. [1]

#### 4. Recent Developments to Improve the Mechanical Properties of Carbon Fibre Composites Fabricated by Different Techniques

##### 4.1. Application of Temperature

Various factors which affect the mechanical properties of CCC at elevated temperatures ( $\sim 2273\text{K}$ ) are:

- High temperature properties of fibre, matrix and the fibre-matrix interface.
- Effect of evolution of absorbed gases.
- Creep deformation.
- Thermal stress.

After suitable tensile tests and creep tests on the 2D laminate CCC specimens at elevated temperatures, G. Ken *et al.* [9] concluded that the tensile strength of CCC enhanced with application of temperature. This can be due to the above mentioned factors in which, predominantly, up to a temperature of  $1773\text{K}$ , the evolution of absorbed gases (degas phenomenon) have contributed to strength improvement of matrix. And for temperatures above  $1773\text{K}$ , it is creep deformation. The stress-strain curves were observed to be non-linear above  $1873\text{K}$  up to  $2273\text{K}$  (at low deformation rates)

##### 4.2. Densification

The densification of cross ply laminate CCC, fabricated by 3 processing routes namely, preformed yarn (PY), resin char (RC) and hot isotropic press (HIP), were carried out by H. Hiroshi *et al.* [10]. An increase in density of CCC from  $1.6\text{g/cm}^3$  to  $1.95\text{g/cm}^3$  was obtained. PY CCC were densified by repeating resin char cycles, pitch infiltration at  $60^\circ\text{C}$  in  $10\text{MPa}$  Argon, carbonization at  $600^\circ\text{C}$  in  $10\text{MPa}$  air and graphitization at  $2000^\circ\text{C}$  in vacuum. Resin charred CCC were densified by carbonization at  $600^\circ\text{C}$  and graphitization

at  $2000^\circ\text{C}$ . Resin charred CCC, reinforced by IM-600 and UM-46 fibres were denoted by RC-I and RC-U. Hot isotropically pressed CCCs were densified by repeated cycles and were denoted by HIP-1 to 5 in which the number stands for repeated cycles.

Tensile test, interlaminar shear test and in-plane shear tests were carried out on the above mentioned CCCs.

The following were the results:

- Tensile test – RC-I and RC-U exhibited increase in tensile strength while HIPs showed a decreasing trend.
- Stress-strain relation (Young's modulus) – Densified PYs showed improvement in Young's modulus; slight improvement in strength, while for HIPs, strength reduced when densified, due to degradation of fracture strain.
- Interlaminar shear strength-densified PYs and HIP-5 exhibit high values of shear strength exceeding  $10\text{MPa}$  while RC-I, RC-U and HIP-1, 2, and 3 showed low values of shear strength.

All the above results point out to the fact that a low interfacial strength between fibre and matrix lead to significant improvement in tensile strength of CCCs.

##### 4.3. Increasing the Number of Pitch/Resin Impregnation Cycles

Jan Chłopek *et al.* [11] fabricated CCC plates by prepeg method, where a preform was obtained from phenol formaldehyde resin and carbon fibres (in one or two directions) after pressing, curing and carbonization. This was followed by pitch impregnation of the preform (liquid impregnation technique or chemical vapour impregnation) at  $215^\circ\text{C}$ ,  $10\text{MPa}$ , carbonization at  $1000^\circ\text{C}$  and graphitization at  $2500^\circ\text{C}$ .

The mechanical properties of CCC improved with increase in the number of pitch/resin impregnation cycles as well as with increase in the extent of

graphitization. High temperature heat treatment ensures a more ordered graphite structure with improved density and strength. The elasticity of CCC is very important as far as biomedical applications are concerned. The type of fibres, orientation and porosity of fibres govern this.

#### 4.4. Carbon Nano Tubes and Carbon Nano Fibres as Reinforcements

Because of their superior mechanical properties, carbon nano tubes (CNT) and carbon nano fibres (CNF) were chosen as composite reinforcements by T. Shinn-Shyong *et al.* [12]. Ultrasonification, to mix CNTs and phenolic resin, was followed by vacuum bag hot pressing technique to fabricate phenolic resin composite. This was followed by heat treatments (carbonization at 1000°C in Ar atmosphere and then at 1400°C in He atmosphere) to convert into CCC.

3 point bending test was used to study the mechanical properties and fracture behaviour. Taking into consideration the improvement in flexural strength, CNF proved to be better reinforcements than CNTs due to better interfacial bonding with a better result for modulus measurement also. For CCC, CNT reinforcement (carbonized at 1000°C and 1400°C) proved to show better results than CNF reinforced. However CCC heat treated at 2400°C showed inferior flexural strength values.

#### 4.5. Carbon Fibre Felt and PAN Carbon Fibre Felt Reinforcements

With an aim to improve the mechanical strength, fracture toughness and thermal stress-resistant properties, S Sato *et al.* [13] developed two composites - CC-A (47 wt% carbon felt) and CC-B (34 wt% carbon felt). These were obtained by reinforcing and binding, pitch carbon felt and PAN carbon felt with coal tar pitch. Tensile strength, fracture toughness, Young's modulus (load elongation curve)

and thermal shock resistance were experimentally determined at 2400°C by suitable tests. These properties were compared with already prepared plate composites, CC-C, by piling 12 layers of rayon carbon fibre cloths and an isostatically molded fine grain petroleum coke graphite, IG-11. As reported by Tensile strength - CC-B has 20% higher than CC-A, and twice as compared to that of IG-11.

- Fracture toughness - CC-B has 20% higher than CC-A and four times as compared to that of IG-11.
- Thermal shock resistance-CC-A,B,C has three times larger than that of IG-11 (practically indestructible by thermal strain)

From the above results, it is evident that these materials can be used as high strength structural materials at high temperatures, for rocket throats, fusion reactor components, etc.

#### 4.6. The Effect of Fibre Architecture on the Mechanical Properties

CCC was fabricated at carbon/carbon advanced technologies by ACC-4 method. Carbon fibre (Union Carbide T-300 graphite yarns) and the matrix (ACC4 carbon) were used to produce CCC in the shape of panels and six different fibre layup architectures were tested. Tensile test, shear test and 3-point bending tests were conducted on the developed samples. J. Neumeister *et al.* [14] found out the following:

- Tensile test result showed that it is dependent on weave architecture as well, rather than fibre vol. % alone.
- Longitudinal strength and fibre efficiency was improved for woven composites (8HSW and Q-iso, PW) than aligned fibres (1D-al.1&2, 1D 0°/90°) although new fibre defects and waviness exist.
- Individual fibres failed in groups to contribute to the ultimate failure of the composite, which is tougher when the fibres are weaved.

- In woven architecture, shear cracks and defect growths are restricted and arrested. And hence woven CCCs are more consistent and geometrically uniform.

#### 4.7. Fracture Toughness as an Important Property

Toughness is the ability of a material to resist crack initiation and propagation from a pre existing flaw. T R Guess *et al.* [15] tested fracture toughness of two types of composites-CVD/FELT composite and CVD/FW composite and ATJ-S molded polycrystalline graphite (CVD – chemical vapour deposition).

- CVD/FELT composite was fabricated from a carbon felt substrate (9 vol %), obtained from rayon fibres, and a chemical vapour deposited carbon matrix. CVD cycles were repeated until the required density was obtained. Heat treatment at 2750°C in nitrogen atmosphere for 2hrs was done.
- CVD/FW composite was fabricated from continuous carbon filaments as substrate infiltrated with the matrix of CVD carbon. Densification was obtained by CVD of carbon from methane at 1100°C and 1atm pressure. Heat treatment at 3000°C in argon atmosphere for 2hrs was done.
- ATJ-S graphite was obtained readymade

Bending tests of single-edged notched specimen of each sample were conducted. 2 modes of failure of the specimen were observed:

- CVD/FWS (notch transverse to filament in specimen) specimen failed by de-lamination between layers.
- CVD/FWW (notch parallel to filament), CVD/FELT and ATJ-S specimen did not delaminate, but by crack propagation.

Comparing the LEFM (linear elastic fracture mechanics) parameters,  $K_c$  (stress intensity factor) and  $G_c$  (critical strain energy release rate), CVD/FWS

was ranked 1, followed by CVD/FELT, ATJ-S and CVD/FWW in terms of fracture toughness.

#### 4.8. Effect of Oxidation on Fracture Toughness of CCC

PAN derived carbon fibre (25% - volume fraction) reinforced pyro-carbon matrix composite was developed by CVI (chemical vapor infiltration technique) by ZHANG Chengyu *et al.* [16] which had a density of 1.83 kg/m<sup>3</sup> and residual porosity of 15%. Oxidation test of the composite was performed in static air at different temperatures of 673K, 773K, 873K and 973K and oxidation weight change was calculated. Also, the fracture toughness of the specimen was determined by single edge-notched specimen (SEN) at room temperature.

- Weight loss as a function of oxidation temperature showed that it increased rapidly with increasing temperature; more severe damage was there when exposed to higher temperatures;
- Fracture toughness decreased with increase in oxidation temperatures – 0.05% weight- loss resulted in 16.6% decrease in fracture toughness; which can reach up to 50% when weight loss is 12.5%.
- SEM images of fracture surfaces depict that oxidation occurs at fibre-matrix interfacial zones and progresses along the fibres into the body of the material. Lower the oxidation at the interface lower will be the decrease in fracture toughness.

### 5. Methods to Improve Fracture Toughness of Carbon Fibre Composites Fabricated by Different Techniques

#### 5.1. Growing Multi Walled Carbon Nanotubes on Woven Carbon Fibre

K.L. Kepple *et al.* [17] found that multi-walled carbon nanotubes (MWCNT) grown on woven carbon fibre (CF) sheets, as reinforcement, in carbon composite

showed excellent improvement in fracture toughness of the composite - 46% for warp direction samples and by 23% for fill direction samples. CNT was grown on CNF at 820°C in argon atmosphere followed by hydrogen, CH<sub>4</sub>, and acetylene atmospheres and then cooled to 666°C in Ar atmosphere. This was followed by ambient cooling to at least 200°C after which it is kept open. These temperatures did not affect flexural stiffness as seen in conventional techniques, in fact flexural modulus of the composite increased by a factor of 5% due to addition of CNT on CF. These composites can find their application in aerospace applications and performance automotive as they prove to be strong, lightweight, and can withstand extreme temperatures.

### 5.2. Graphene / Graphite-based Conductive Polyamide 12 Interlayer

E Barjasteh *et al.* [18] fabricated composite by VaRTM (Vacuum assisted resin transfer moulding)-a layer of polyamide, PA12 was placed in between every two layers of stitched unidirectional layers of carbon fabric. The resin was then infused into the preform at 110°C followed by curing for 2hrs at 180°C. The results indicated:

- Mode I fracture toughness (DCB (double cantilever beam) method) - 36% increase (for G<sub>IC</sub> initiation) and 70-75% increase (for G<sub>IC</sub> propagation).
- Mode II fracture toughness (three-point end notched flexure (3p-ENF) test) – 140% increase (for G<sub>IIC</sub>).
- SEM and XRD - uniform distribution of graphene/graphite on to the surface of the fabric.
- Surface resistivity and electrical conductivity - compared with different values of surface resistivity produced by different methods as per literature, interfacial trapping method produced minimum

surface resistivity and improved electrical conductivity.

### 5.3. Microwave Curing

The idea was put to use by Z. Jing *et al.* [19]. A high performance octahedron microwave oven was designed to serve the purpose. A unidirectional prepreg was combined with T700 carbon fibres and toughened BMI resin which was then subjected to curing in steps - 130°C for 60 min and 200°C for 240 min at a rate of 1°C/min, and cooling at a rate of 1.3°C/min in vacuum of 0.5Pa. The results can be summarized as:

- Mode-I fracture toughness measurement using DCB (Double cantilever beam) test showed that there was a huge, 133.5%, increase in fracture toughness and 61.2% increase in fracture resistance respectively.
- The processing time was cut to 44% of that of thermal forming and eventually the cost of manufacturing by a greater extend.
- DMTA (dynamic mechanical thermal analysis) brings out the fact that a better interfacial adhesion between carbon fibre and BMI resin was obtained in microwave curing.

### 5.4. Dissolvable Thermoplastic Fibres

Here, a phenoxy fibre in solid form was incorporated into a carbon fibre perform as a chopped fibre interleave. Composite laminates were fabricated by vacuum infusion technique in which the phenoxy fibre remained in solid state during infusion but dissolved and phase separated when resin (Epon 828) was added while curing (180°C at 3°C/min in the presence of 4,4'-diaminodiphenylsulfone as curing agent). To study the properties of epoxy/phenoxy blends without the presence of reinforcing fibre, neat resin specimens were prepared with 5 wt% and 10 wt % chopped phenoxy added to epoxy. Doris W.Y. Wong *et al.* [20] performed the following to conclude :



- SEM fractography points out the presence of phase separation in epoxy/phenoxy blends.
- Mode I fracture toughness tests (DCB) showed drastic results as  $G_{IC}$  value doubled with 5wt% of phenoxy fibre and increased tenfold when 10wt% phenoxy fibre was added.
- 10 wt% phenoxy sample showed improved damage resistance and tolerance than 5 wt% one.

### 5.5. Silk Fibroin Nano Fibres

V. M. Cuong *et al.* [21] used silk fibroin nanofibres (nSF) produced by electrospinning as nano material reinforcement in epoxy (diglycidyl ether of Epon 828) matrix in different contents (0, 0.03, 0.05, 0.1 wt%) at 90°C for 60mins. Thereafter, sonication using ultrasonic homogenizer and curing (at 130°C for 1hr and 150°C for 3hr) was done to obtain the composite.

- 3 point bending test showed 28.3% improvement in fracture toughness, with the addition of 0.1 wt% of nSF.
- Micro droplet test showed a 20% increase in IFSS with the addition of 0.1 wt% nSF.
- Tensile tests show an improvement of 4.8% of tensile strength with the addition of 0.05 wt% nSF.
- At 0.1 wt% nSF, Mode II interlaminar fracture toughness increased by 30%.

### 5.6. Fibre Sizing

Out of different methods to improve the fracture toughness of composites, fibre coating technique is considered to be one of the simplest and feasible methods. Fibre coating (sizing) modifies the mode of failure, i.e. the potential energy absorbing capacity which in short determines the fracture toughness. B.

L. Wen *et al.* [22] sized T700 CF (12K carbon fibre) by a thermoplastic polymer (PPEK) solution mixed with a compactible amine monomer (DHPZDA). The results obtained were:

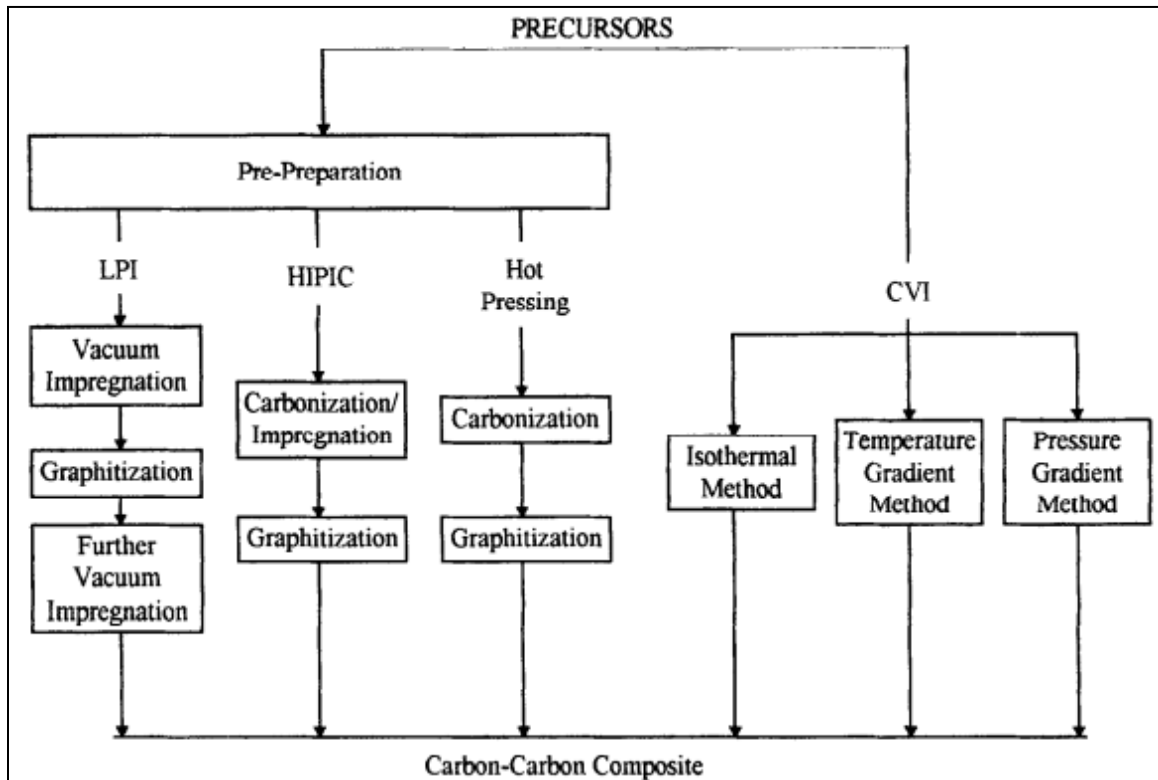
- The strength testing of single fibre showed no reduction in tensile strength of carbon fibre even after sizing.
- The load – displacement curve showed that the interfacial shear strength (IFSS) of resized CF composite improved by 15.48%.
- The force – displacement curve in the micro-bond test showed that the interfacial fracture toughness of resized CF composite improved by 56.1%.

### 5.7. Ultra-High Temperature Ceramic Coating

To overcome the limitations of CCC, oxidation and ablation at high temperatures (~ 2000K), ultra high temperature ceramic material (ZrB<sub>2</sub> and ZrC) coatings are applied on CCC. Z. Yulei *et al.* [23] utilised supersonic atmosphere plasma spraying (SAPS) combined with reaction melt infiltration (RMI) as the technique to coat. ZrB<sub>2</sub>-Si-ZrC coating was applied by SAPS, followed by RMI to fill holes after SAPS coating and densification of the coating.

The summary of the results are:

- Ablation resistance tested by oxyacetylene torch system with heat flux of 2400kW/m<sup>2</sup> for 120s recorded appreciable mass and linear ablation rates as -0.016X10<sup>-3</sup> g/s and 1.30 μm/s.
- SEM and BSE images prove the integrity maintained without any crack or void even after expose to temperature of 2300K during coating.



**Figure 1.** Modifications in conventional fabrication methods [3].

## 6. Summary and Conclusion

In this paper a comprehensive review on carbon-carbon composites is presented addressing the fabrication techniques, advancements in fabrication techniques, properties and applications. High costs in the fabrication processes have restricted the abundant use of carbon – carbon composites. And of this reason, there is always a greater opportunity for researchers to develop new fabrication techniques which will effectively reduce the cost of fabrication without compromising the extra brilliant properties of this class of composites. The PY method is one such method which has brought down the cost of fabrication to a greater extend when compared to the conventional methods.

Specific strength and fracture toughness are the key properties that almost every structural applications like aerospace demand. CCC has never failed to meet these and an improvement in the existing values of

these properties will surely make them ideal and would be the best choice of engineers.

## 7. Conflicts of Interests

The author(s) report(s) no conflict(s) of interest(s). The author along are responsible for content and writing of the paper.

## 8. Acknowledgments

NA

## 9. References

1. Rohini Devi G, Rama Rao K. Carbon-carbon composites. Defence Science Journal 1993; 43(4): 369-383.
2. Tongshik C, Akimitsu O. Fabrication of Carbon-Carbon Composites by Using Carbon Fibers Impregnated with Resin and Their Mechanical Properties. Transactions of the Iron and Steel Institute of Japan 1987; 27(3): 229-237.
3. Windhorst T, Blount G. Carbon-carbon composites: a summary of recent developments and applications.

- Materials & Design 1997; 18(1): 11-15.
4. Hirotaka N, Takao N, Hidefumi H. Manufacture carbon of unidirectional composites carbon fiber reinforced method by preformed-yarn method. TANCO 1999; 1999(186): 7-12.
  5. Naik P, Londe NV, Surendranathan AO, Jayaraju T. Carbon-carbon composites by preformed yarn method. International Journal of Mechanical and Materials Engineering 2011; 6(1): 133-139.
  6. Naik P, Ibrahim M, Surendranathan AO, Mujeebu M. Development and characterization of carbon-carbon composite for aircraft brake pad using preformed yarn method. World Journal of Engineering 2011; 8(3): 259-266.
  7. Naik P, Surendranathan AO, Ravishankar KS. Preparation of yarn based carbon-carbon composites and their properties. International Journal of Science Technology and Management 2015; 4(1): 156-163.
  8. Venugopalan R, Sathiyamoorthy D. Development of Carbon / Carbon Composites for Nuclear Reactor Applications. BARC News Letter 2012; 325: 16-20.
  9. Goto K, Ohkita H, Hatta H, Iseki H, Kogo Y. Tensile strength and creep behavior of carbon-carbon composites at elevated temperatures. 16<sup>th</sup> International Conference On Composite Materials, Japan, 2007.
  10. Hatta H, Suzuki K, Shigei T, Somiya S, Sawada Y. Strength improvement by densification of C/C composites. Carbon 2001; 39(1): 83-90.
  11. Jan C, Stainisław B, Augustyn P. Mechanical Properties of Carbon-Carbon Composites. Carbon 1993; 19(4): 251-257.
  12. Tzeng S, Lin Y. Mechanical Properties of Carbon-Carbon Composites Reinforced With Carbon Nanotubes or Carbon Nanofibers. 16<sup>th</sup> International Conference on Composite Materials, Japan, 2007.
  13. Kurumada A, Iwaki H, Komatsu Y. Tensile Properties and Fracture Toughness of Carbon-Fiber Reinforced Carbon Composites. Carbon 1989; 27(6): 791-801.
  14. Neumeister J, Jansson S, Leckie F. The effect of fiber architecture on the mechanical properties of carbon/carbon fiber composites. Acta Materialia 1996; 44(2): 573-585.
  15. Guess TR, Hoover WR. Fracture Toughness of Carbon-Carbon Composites. Journal of Composite Materials 1973; 7(1): 2-20.
  16. Zhang C, Yan K, Qiao S, Li M, Han D, Guo Y. Effect of oxidation on fracture toughness of a carbon/carbon composite. Journal of Wuhan University of Technology-Mater. Sci. Ed. 2012; 27(5): 944-947.
  17. Kepple KL, Sanborn GP, Lacasse PA, Gruenberg KM, Ready WJ. Improved fracture toughness of carbon fiber composite functionalized with multi walled carbon nanotubes. Carbon 2008; 46(15): 2026-2033.
  18. Barjasteh E, Sutanto C, Reddy T, Vinh J. A graphene/graphite-based conductive polyamide 12 interlayer for increasing the fracture toughness and conductivity of carbon-fiber composites. Journal of Composite Materials 2017; 51(20): 2879-2887.
  19. Zhou J, Li Y, Li N, Hao X. Enhanced interlaminar fracture toughness of carbon fiber/bismaleimide composites via microwave curing. Journal of Composite Materials 2017; 51(18): 2585-2595.
  20. Wong DWY, Lin L, McGrail PT, Peijs T, Hogg PJ. Improved fracture toughness of carbon fibre/epoxy composite laminates using dissolvable thermoplastic fibres. Composites Part A: Applied Science and Manufacturing 2010; 41(6): 759-767.
  21. Manh CV, Choi HJ. Enhancement of Interlaminar Fracture Toughness of Carbon Fiber/Epoxy Composites Using Silk Fibroin Electrospun Nanofibers. Polymer-Plastics Technology and Engineering 2016;

55(10): 1048-1056.

22. Barkoula NM, Alcock B, Cabrera NO, Peijs T. Fatigue properties of highly oriented polypropylene tapes and all-polypropylene composites. *Polymers and Polymer Composites* 2008; 16(2): 101-113.

23. Zhang Y, Wang H, Li T, Fu Y, Ren J. Ultra-high temperature ceramic coating for carbon/carbon composites against ablation above 2000K. *Ceramics International* 2018; 44(3): 3056-3063.