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Development and Evaluation of Carbon-Carbon Threaded Fasteners for High Temperature Applications

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ABSTRACT

Carbon-Carbon (C-C) threaded fasteners have been developed for joining C-C composites experiencing temperature above 1500 °C. The fasteners were fabricated from spun yarn graphitized carbon fabric through resin route and from needle punched carbon felt through pitch route techniques. The preforms were processed with multiple cycles of impregnation, carbonisation, and graphitisation up to densities of 1.78 gcm^{-3} and 1.90 gcm^{-3}. Densification process cycles were repeated to obtain composites with reduced porosity and improved thermo-mechanical properties. Material evaluation of C-C fasteners is discussed and compared with existing graphite fasteners used in industrial and aerospace sectors. Scanning electron microscopy was also carried out to study microstructure of fractured specimens.

Keywords: Carbon-carbon components, carbon-carbon fasteners, material evaluation, microstructure

1. INTRODUCTION

Joining of carbon-carbon (C-C) components for ablative applications poses a very special challenge in the field of C-C technology. C-C fasteners are the new brigade in joining of C-C composites for high temperature (>2500 °C) applications[1]. The joint integrity of C-C components that can be attained at this temperature depends principally on the material of reinforcement, matrix and the matching of thermo-mechanical properties at the joint with the base material. The degree of success that can be attained depends principally on the material of reinforcement since it largely affects the temperature serviceability of composite. Carbon has the highest sublimation point of all elements. It sublimates at about 3627 °C[2]. This ensures the serviceability of C-C materials upto 3000 °C. By using C-C fasteners continuity of reinforcement at the joint will be achieved as the joint strength and material properties of the fastener will be close to that of the parent composite. In the present work, C-C fasteners were developed from spun yarn graphitized (SYG) carbon fabric and needle punched (NP) carbon felt. SYG fabric is a special type of fabric woven with yarns made using stretch breaking technology. Although it's a 2-D continuously woven fabric, due to protruding fibers on the fabric surface it provides some kind of a 3-D reinforcement improving shear strength of material. NP felt is made using the novel technique of needle punching wherein, each part of the felt, through the thickness, has equal amount of transferred fibers of length 60-65 mm. This provides the felt its good through the thickness homogeneity. Due to 3-D fiber arrangement, they are resistant to delamination and have greater integrity to resist tensile, shear and compressive forces of deformation in high temperature applications[3,4]. The C-C fasteners developed with current approach gives better tensile and shear properties. The fiber architecture eases machinability and construction of uniform threads. Results on temperature serviceability of C-C fasteners upto 1500 °C, mechanical properties at room temperature (RT) and thermal properties from RT to 1200 °C are presented and compared with conventional graphite fasteners.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

The SYG fabric of 1.5 K tow size, fiber diameter of 8 µ and density of 1.70 gcm^{-3} was used as reinforcement in 2-D composite. The precursor resin for carbon matrix composite was resole phenolic resin of viscosity 150 cps. Initially a 2-D carbon phenolic (C-Ph) composite was moulded in a hydraulic hot press at 150 °C for 3 h. 2-D C-C was obtained by densifying 2-D C-Ph. Needle punched felt of 1000 gm^{-2} is made from Pan-ox (oxidized polyacrylonitrile) fiber of diameter 13 µ. The felt was first heat treated (HT) to 800 °C to shrink its fiber diameter to 8 µ, converting pan-ox fiber to carbon fiber (Fig. 7.). Layers were then directly cut from the HT felt and integrated to make two preforms. These preforms were then stacked in a fixture and processed. Thus, 2-D C-C fasteners were contrived from resin route technique whereas, NP C-C (A) and (B) fasteners were contrived from direct pitch route technique. The fiber volume fraction in all the preforms was between 30 per cent to 33 per cent approximately.

2.2 Processing

The investigated materials were densified through the liquid impregnation technique which involves multiple cycles

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of impregnation at high pressure with coal tar pitch followed by carbonisation and graphitisation. The preforms were carbonised at 1000 °C where the primary matrix material begins to breakdown only leaving its carbon backbone. The consequence is a large bulk shrinkage and opening up of voids. Re-impregnation cycles are carried out to fill porosity in the material and to obtain a strongly bonded fiber matrix (F/M) interface. Because debonding at F/M interface can provide a mechanism of energy absorption during mechanical deformation [5]. Graphitisation at 2500 ºC is then accomplished to restructure and graphitise the matrix. At this temperature the molecules realign, forming a graphite-like structure with improved thermal conductivity, stiffness, tensile and shear strength in a composite. The 2-D C-C and NP C-C (A) preforms were densified upto 1.78 gcm^{-3}. To study the effect of additional densification on properties, NP C-C (A) was densified to 1.90 gcm^{-3} and identified as NP C-C (B).

3. TESTING

The investigated materials were evaluated for mechanical properties at RT by a universal testing machine. Thermal properties were measured from RT to 1200 ºC. The test methods and specimen dimensions followed are given in Table 1. Density was measured using the relation, density = mass/volume. Porosity was measured using Pore master 60 GT, Quanta chrome Instruments, USA. A densified graphite billet of density 1.78 gcm^{-3} was taken for testing and comparison of material properties with C-C.

Table 1. Specimen dimensions and test methods

Property	Specimen dimensions (mm)	Test method
Density	10 x 10 x 10	As given in Section 3
Porosity	25 x 5 x 5	As given in Section 3
Tensile strength	150 x 10 x 8	ASTM D 3039
Compression strength	13 x 13 x 13	ASTM D 695
Thread shear strength	400 x 400	ASTM D 3039
Bolt tensile strength	M6	ASTM F 606
Co-efficient of thermal expansion	25 x 5 x 5	ASTM E-228
Thermal diffusivity	Ø12.7 x 2.5	ASTM E-1461

4. RESULTS AND DISCUSSION

4.1 Density and Porosity

The density and porosity results given in Table 2 indicate that 2-D and NP C-C (A) of density 1.78 gcm^{-3} had per cent porosity of 12 and 13. NP C-C (B) of density 1.90 gcm^{-3} had porosity of 10 per cent.

Table 2. Measured values of density and porosity

Materials	Density (g cm^{-3})	Porosity (per cent)
2-D C-C	1.78	12
NP C-C (A)	1.78	13
NP C-C (B)	1.90	10
Graphite	1.78	13

4.2 Tensile and Compression Strength

Tensile strength results are shown in Table 3. It is inferred that 2-D C-C exhibited highest tensile strength (TS) whereas NP C-C (A)/(B) and graphite were found to be less by 35 per cent, 22 per cent and 88 per cent (Fig. 1)[4,6]. TS of C-C composite depend upon fiber reinforcement, F/M adhesion and orientation of fiber in tensile direction. The inherent strength and modulus of SYG yarn is superior to Pan-ox felt. As a

Table 3. Mechanical properties of investigated materials

Materials	Tensile strength (MPa)	Compres-sion strength (MPa)	Thread shear strength (MPa)
2-D C-C	80-90	102	23.6
NP C-C (A)	58	92	23.7
NP C-C (B)	70	102.5	31.8
Graphite	11	43	14.2

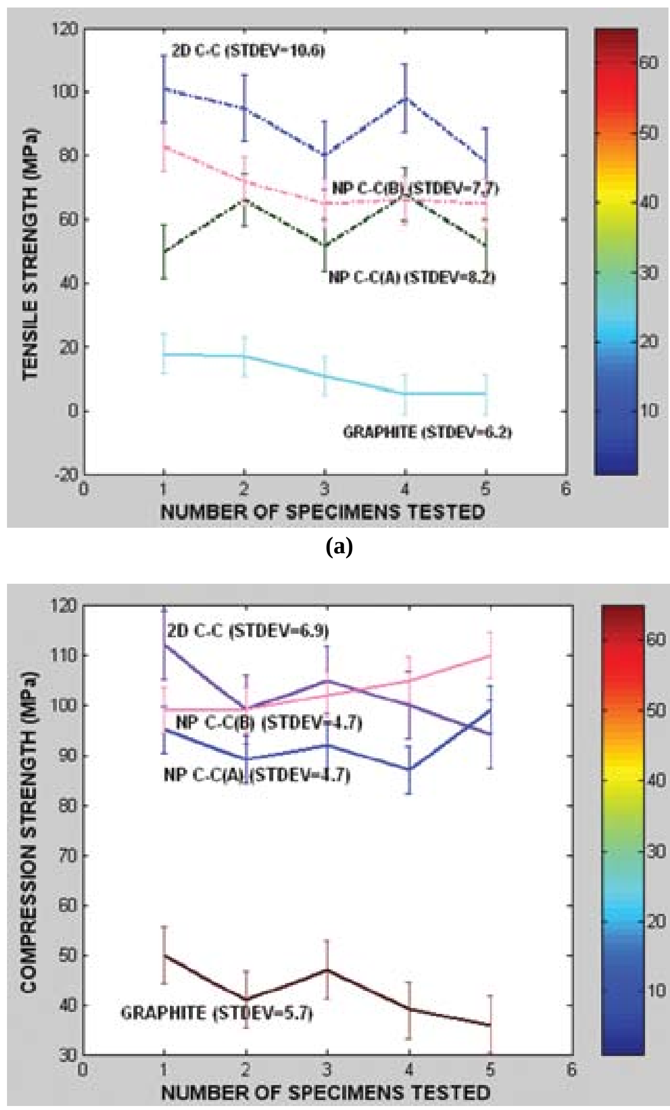


Figure 1. Strength of investigated specimens, (a) tensile and (b) compression.

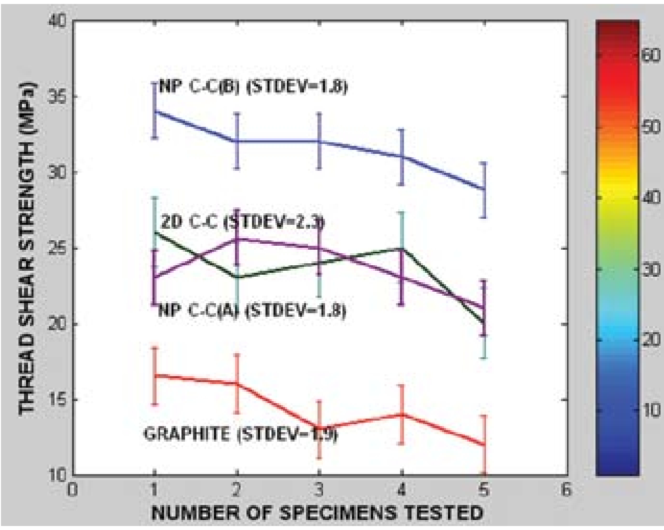


Figure 2. Thread shear strength of investigated specimens.

result, 2-D C-C weave architecture provides better network continuity and anisotropy to the structure to withstand larger stresses. NP C-C has an isotropic structure constituting of randomly distributed staple fibers making it structurally not as strong as woven preform[3,7]. Therefore, when an increasing tensile load is acting in the direction of fiber the fracture strength of weaker plane that is matrix is approaching. Weaker plane constitute matrix as they have low modulus and are brittle in nature. The stronger plane constitutes the reinforcing fibers with greater strength (3.5 GPa) and stiffness (220-240 GPa) than matrix. Therefore, the much stiffer fibers will be the principal load bearers. The matrix in addition to having the task of binding together the composite will deform under load and distribute the majority of stress to the fibers. Strong fiber matrix bonding also supports increase of fracture strength. 2-D C-C strong fiber architecture and good F/M bonding is responsible for its highest TS[5]. It was estimated that tensile strength of 2-D and NP C-C were 88 per cent and 82 per cent higher than graphite. Compressive failure in composites is matrix dominated. 2-D and NP C-C fractured

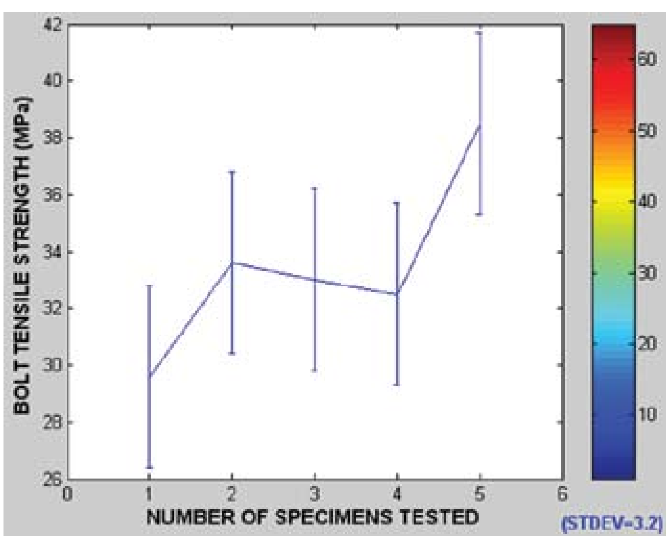


Figure 3. NP C-C (B) bolt tensile strength.

due to matrix crumbling and interlaminar shear failure. 2-D fractured specimens were observed to show pseudo-plastic failure[8]. Whereas, NP fractured specimens were observed to show catastrophic failure. Compression strength (CS) of all C-C materials is almost comparable due to same fiber volume fraction. CS of 2-D, NP C-C (A) and (B) showed an increase of 61 per cent, 53 per cent and 58 per cent over graphite. The tensile and compression modulus were measured to a value of 20-30 GPa and 1-2 GPa.

4.3 Thread Shear Strength

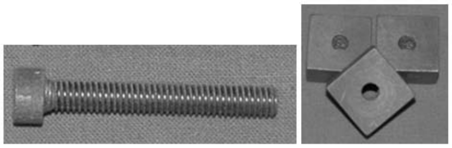
The cutting of metric threads on cylindrical rods cut from the C-C billets was done with die cut and tap tools for external and internal threading (Fig. 4(a)). The thread shear strength (TSS) of a fully engaged thread was evaluated on screws, bolts and nuts individually. The shear area, A, was calculated from relation given (1)[1,3].

$$A = F / (0.5 * \pi * d * h) \quad (1)$$

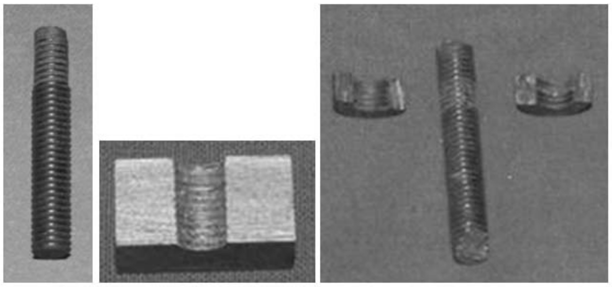
where, F = maximum shear load, d = major diameter of bolt or nut, h = height of thread engagement.

The height of thread engagement is the distance of the threads overlapping in radial direction. It is one of the key strength aspects. If bolt and nut are of the same material the minimum thread engagement length should be 65 per cent of the nominal diameter. The factors that have an important effect on the TSS are formation of threads and variation in the dimension of the threads. Due to continuous reinforcement in 2-D C-C the intermittent occurrence of fibers and matrix makes it difficult to machine continuous threads as the large matrix pockets get chipped off during machining. In NP C-C the fiber architecture consist of each individual fiber of size 8 μ, uniformly interacting with the matrix. The surface area of short fibers interacting with the matrix is much greater in NP C-C than in continuously woven 2-D C-C. This builds a strong bonding at the fiber-matrix interface reducing the size and number of matrix pockets. This makes machining of continuous and finer threads easier. From the test results (Table 3) it was inferred that TSS of NP C-C (B) was superior by 35 per cent to 2-D C-C and NP C-C (A) (Fig. 2). As density is same, TSS of 2-D C-C and NP C-C (A) is comparable but 67 per cent higher than graphite. The thread shear load bearing capacity of NP C-C (B) was 125 per cent higher than graphite.

The mode of failure in screws, nuts and at the threaded joint (Fig. 4(a)) was investigated as shearing of threads[1]. It was also observed that C-C bolts possess the mechanism of interlocking of threads. When the bolt failed in the joint assembly at RT, due to application of ultimate load the powder sheared, got self-interlocked and arrested the movement of threads. This enabled the joint to further take a compressive load of 400 N before failure. To observe this phenomenon, NP C-C (B) studs were used for fastening elements in high vacuum furnace for 300 h under 400 N load and 1500 ˚C temperature. Post test analysis of threads indicated no shearing or cracking on its surface (Fig. 4(d)). Also, TSS remains unaffected. NP C-C (B) has higher capacity of sustaining load and temperature than existing can be projected as a reason for negligible distortion.



(a)



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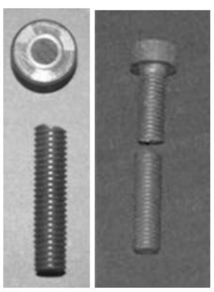
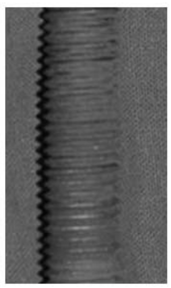


(c)



(d)

Figure 4. (a) C-C bolt and nut (size M6), (b) Thread shear failure in screw, nut and bolt, (c) Bolt failure, and (d) Stud post test (under 400N load at 1500 °C for 300 h).

4.4 Bolt Tensile Strength

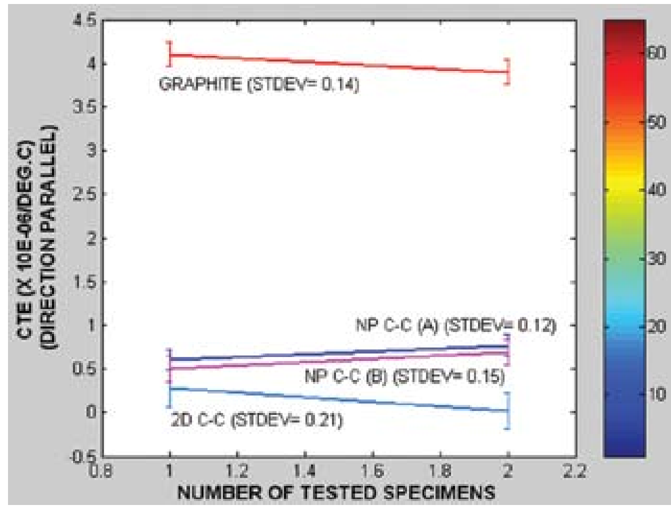
From the results obtained, NP C-C (B) was found to be more promising thus bolts of size M6 were tested for bolt tensile strength. The tensile strength (TS) was determined from formula given in Eqn (2),

$$TS = P/A \quad (2)$$

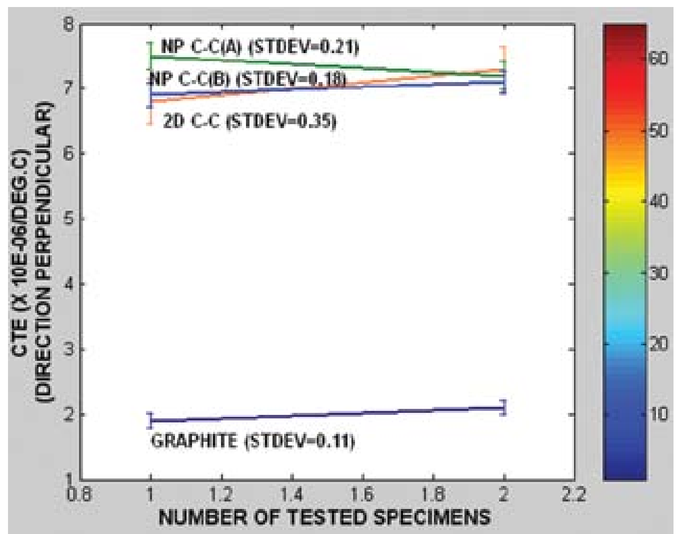
where P = tensile load, A = tensile stress area of bolt was calculated from the relation given in Eqn (3)[3,4].

$$A = P/(\pi/4*(d-0.649p)^2) \quad (3)$$

where P = maximum shear load, d = major diameter of bolt, p = thread pitch. The tensile strength of bolt was evaluated as 33.4 MPa [1,10]. Post test investigations showed that the bolt failed through the shank and pitch diameter. Some of the samples failed at the bolt head area which indicated the presence of bending stresses acting on that area (Fig. 4(c).)[3].



(a)



(b)

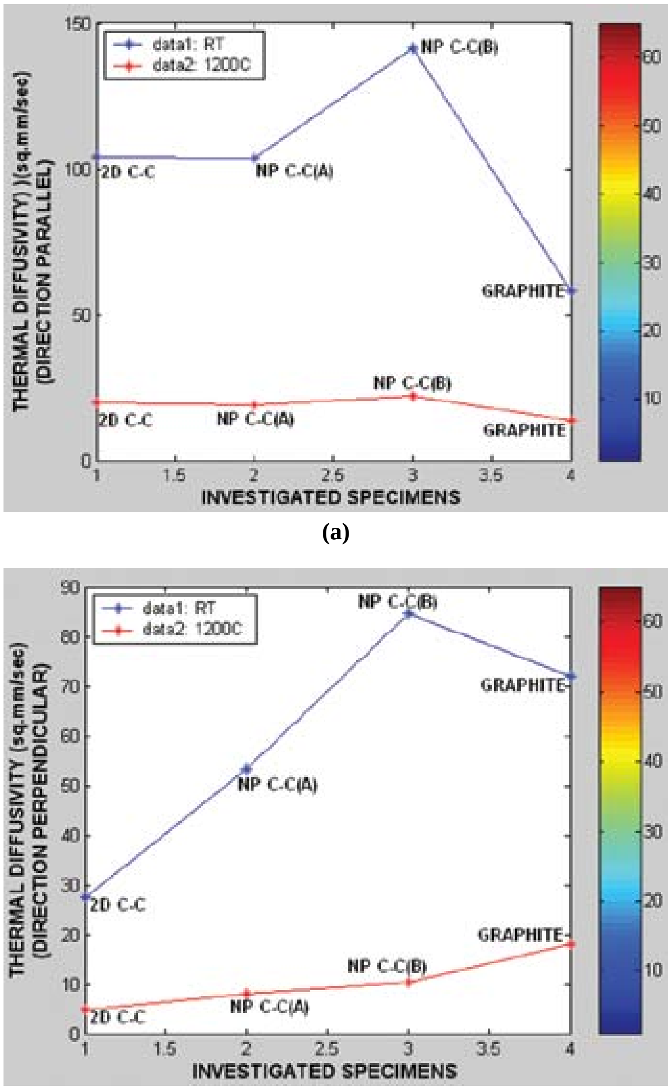
Figure 5. CTE of investigated specimens in direction (a) parallel (b) perpendicular.

4.5 Co-efficient of Thermal Expansion

Results given in Table 4 indicate that graphite has co-efficient of thermal expansion (CTE) lowest in perpendicular and highest in parallel directions than C-C materials. The

Table 4. Thermal properties of investigated materials

Materials	Co-efficient of thermal expansion (x (10*E-06/°C)		Thermal diffusivity (mm^2/s) at RT		Thermal diffusivity (mm^2/s) at 1200 °C	
	Parallel	Perpendicular	Parallel	Perpendicular	Parallel	Perpendicular
2-D C-C	0.15	6.9	104	27.5	20	4.8
NP C-C (A)	0.67	7.3	103.8	53.3	19	8
NP C-C (B)	0.58	7	141.5	84.6	22	10.3
Graphite	4	2	58	72	14	18



(a)

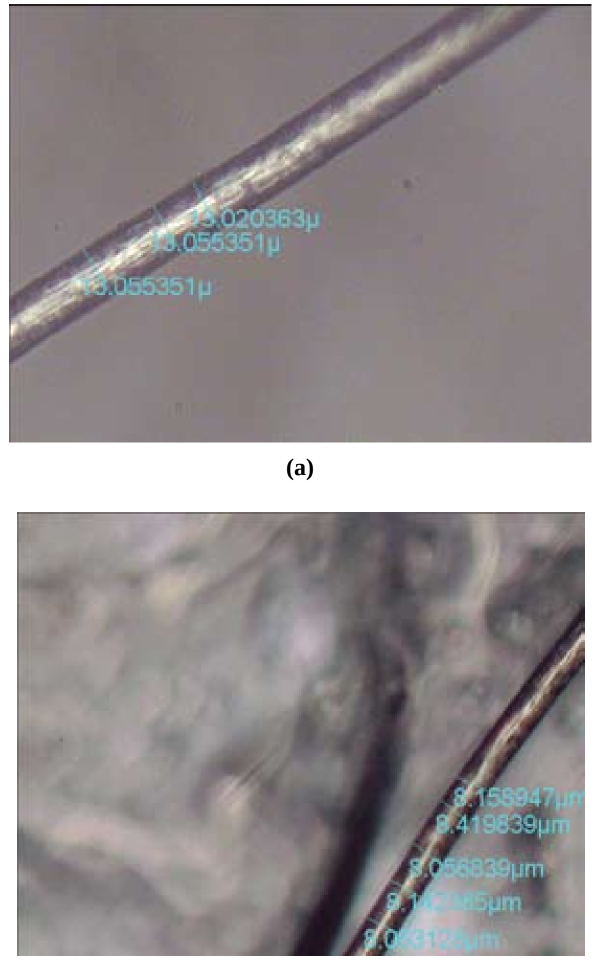
(b)

Figure 6. Thermal diffusivity of investigated specimens in (a) parallel and (b) perpendicular directions.

thermal stresses generated at the time of application will be minimal as CTE of C-C composites are always in the range of - 0.04 x 10^{-6}/°C to 8 x 10^{-6}/°C [9].

4.6 Thermal Diffusivity

Thermal diffusivity (α) was measured at RT and 1200°C (Table 4). It was observed that thermal diffusivity values at 1200 °C have decreased by 80-87 per cent from RT values for 2-D C-C, NP C-C (A) and (B) in parallel and perpendicular directions. Graphite α value has decreased by 75 per cent from RT to1200 °C in parallel and perpendicular directions. In C-C composites the carbon fiber is the channel of heat transmission so its direction, dimension and distribution in the preform have an important effect on the thermal diffusivity and conductivity of the materials. NP C-C (B) has a higher level of structural integrity and the fibers are uniformly distributed in the preform in XY (parallel) and Z (perpendicular) directions making it compact and homogeneous when compared to other materials. Also due to increased density it has very less micropores. Due to which NP C-C (B) has highest diffusivity in XY and



(a)

(b)

Figure 7. Optical micrograph of Pan-ox fiber (a) before HTT (b) after HTT.

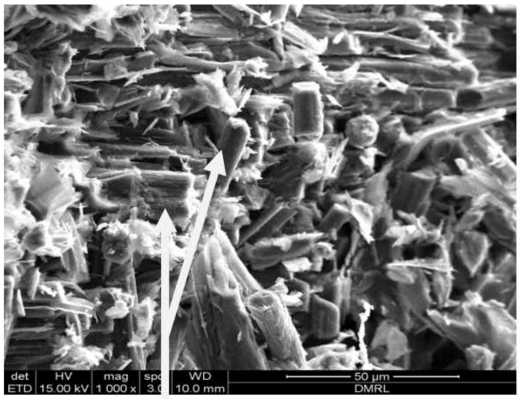
Z directions. For C-C composite the vibration of the crystal lattice is the source of heat conduction. According to quantum theory, the crystal lattice vibration can be described by the phonon interaction[9]. Phonon interaction in the C-C composites is divided into 3 types:

a. Phonon-phonon interaction
b. Phonon defect interaction
c. Phonon interface interaction

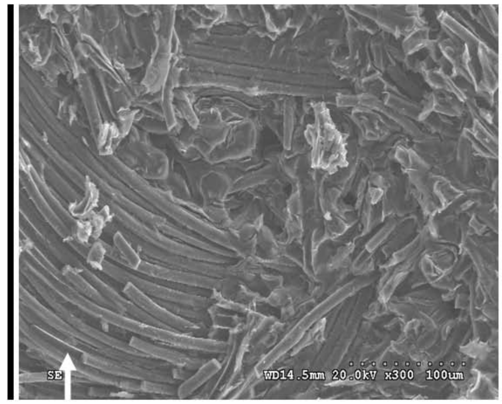
And in phonon-phonon interaction the mean free path is the most sensitive to temperature. As the temperature goes up the phonon vibration frequency will be quickened to make the collision possibility increase so, the mean free path decreases rapidly, which leads to rapid decrease of its thermal diffusivity.

4.7 Microstructure

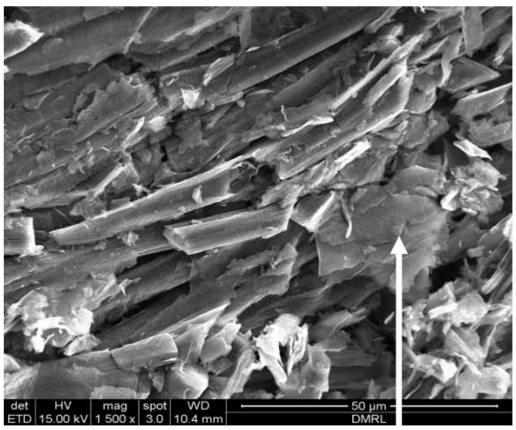
C-C fractured specimens were examined using a Carl Zeiss scanning electron microscope (SEM). Figure 8 shows SEM of all fractured specimens. Fiber architecture in 2-D that is continuously woven yarns in warp and weft directions are seen Fig. 8 (a). Random fiber distribution in NP C-C can be seen in Fig. 8 (b). Good F/M bonding was observed in all the composites. In 2-D C-C during pyrolysis, though the fiber and matrix both undergo chemical reactions with evolution



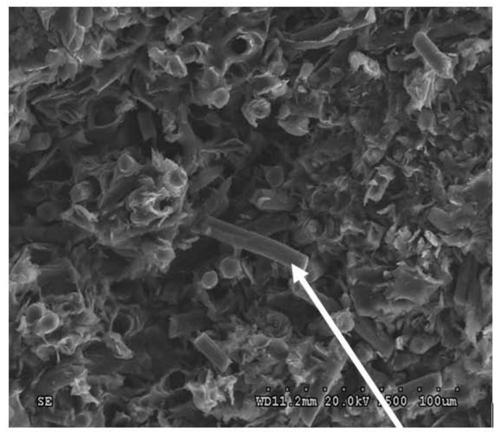
(a) Fibers in 2-D C-C



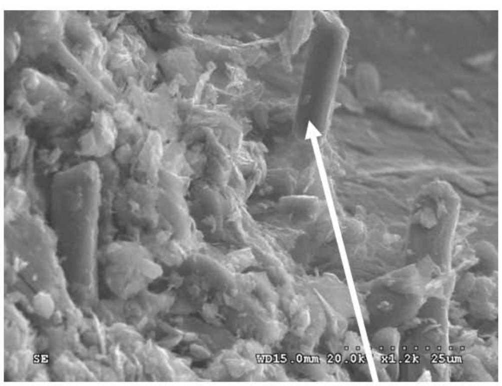
(b) Staple fibres in NP C-C (B)



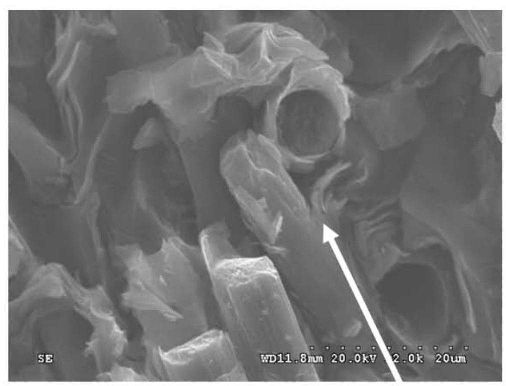
(c) Matrix



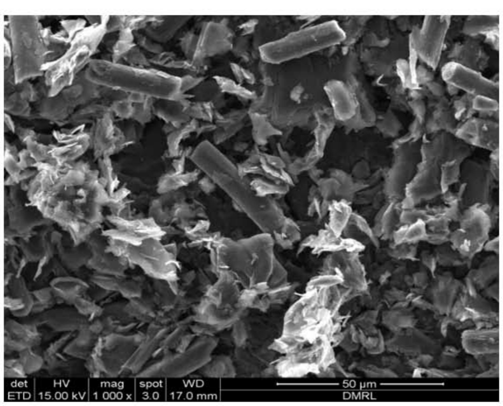
(d) Fiber pull out



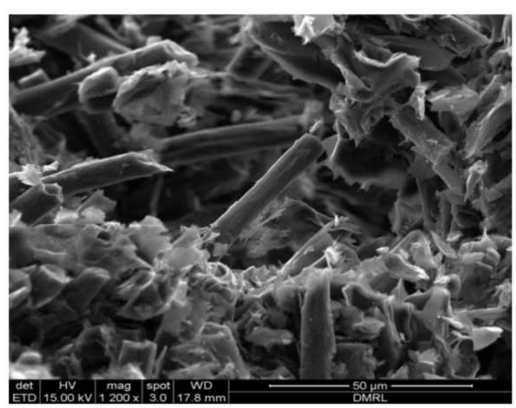
(e) Protrusion of sheared fibers



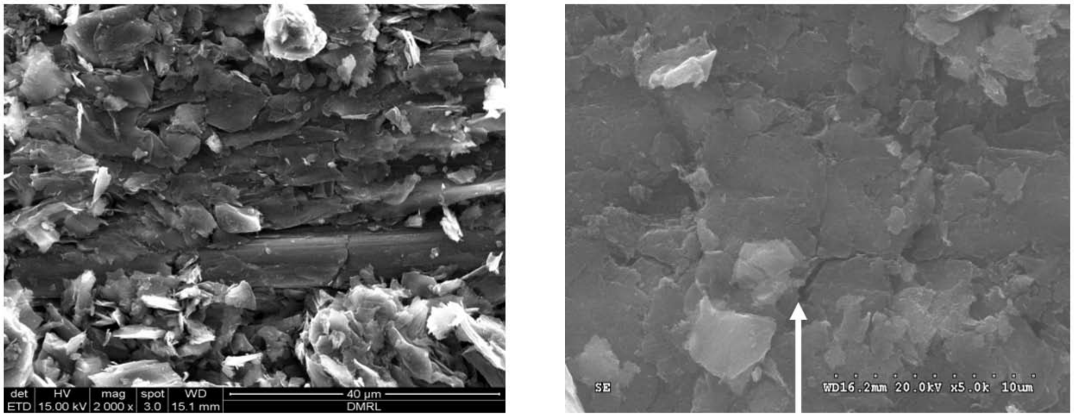
(f) Tearing of fiber



(g) Broken fibers



(h) Fiber network in NP C-C



(i) Matrix crack in 2D C-C **(j) Matrix crack in NP C-C**

Figure 8. SEM images depicting mode of failure in fractured C-C specimens.

of byproducts and get carbonized, the fiber matrix bonding is maintained and both fiber and matrix are easily distinguishable (Fig. 8 (c)). In no case was the coalescence of the two phases observed. Previous studies on carbonization of carbon fiber reinforced thermosetting resin composites has shown that strong F/M bonding leads to excessive shrinkage cracks in the matrix. However, in the present studies no matrix shrinkage cracks were observed. The fibers in 2-D C-C are almost surrounded by the matrix, because of the strong bonding in the interface. Mode of failure in 2-D C-C was found to be in the form of broken fiber and matrix cracking (Fig. 8(i)). Due to the presence on staple fibers the F/M interface bonding is stronger in NP C-C (B) than 2-D C-C. As a result the mode of failure is catastrophic with a little fiber pull-out. From the study of the micrographs it was understood that the mode of failure in NP C-C (B) was mainly due to fiber tearing, fiber pull out and matrix cracking (Figs. 8(d), 8(f), and 8(j)). Protrusion of fibers due to excessive shearing was also observed (Fig. 8 (e)). Matrix cracking at regions of fracture may be attributed to cohesive bond failure of the matrix. NP C-C (A) F/M interface bonding was not as strong as in 2-D or NP C-C (B). The fractured specimen was observed for breaking and tearing of fibers. The regions with broken fibers seem to have experienced fiber to matrix bond failure.

5. CONCLUSIONS

The C-C threaded fasteners can be developed and used for the joint integrity of C-C components only. They possess the mechanism of self- interlocking of threads in the base material thus configuring the joint assembly as one. From the experimental results it is concluded that material properties of NP C-C (B) fasteners are marginally superior to conventional graphite fasteners. Temperature sustainability was found to be greater than 1500 °C in service. These fasteners are suitable where high temperature fastening with good load bearing capability is required.

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