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Effect of Fiber Weight Fraction on Mechanical Properties of Carbon–Carbon Composites

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This article presents the synthesis of carbon–carbon (C/C) composites by preformed yarn (PY) method, by varying the percentage of carbon fiber weight fraction. The PY used was carbon fiber bundle surrounded by coke and pitch which was enclosed in nylon-6. Three types of samples with fiber weight fractions of 30, 40, and 50%, respectively, are fabricated and mechanical properties were studied. In each case, the PY was chopped and filled into a die of required shape and hot pressed at 500°C to get the preform composite. To obtain the carbonized and graphitic structure, the specimen was heat treated at 2500°C followed by soaking for 10 to 12 hrs. Further, two cycles pitch impregnation was done by hot isostatic pressing, to eliminate the voids and to increase the density hence to obtain good mechanical properties. The characteristics such as hardness, flexural strength, and impact strengths were studied. It is observed that, as the carbon fiber percentage increases, the properties also get improved, provided sintering is done at fairly higher temperatures such as 2700°C. The superiority of the new class of C/C composites made by the proposed PY technique over those obtained by the conventional methods is also demonstrated. POLYM. COMPOS., 33:1329–1334, 2012. © 2012 Society of Plastics Engineers

INTRODUCTION

Carbon–carbon (C/C) composites are composed of carbon fibers in a carbon matrix. They are lightweight materials and have superior thermal shock resistance, toughness, ablation and high-speed friction, and load bearing properties. C/C composite which is one of the advanced materials is widely used for high performance, brake systems of aircraft and racing cars, bolt, nut in the furnace, etc. [1]. C/C composites have been emerged in response to sustained aerospace and defense needs. The conventional C/C composites manufacturing takes more time because of more number of pitch impregnation cycles involved to achieve higher density. The extremely high price of C/C composite due to such production characteristics made it costlier to the industrial application [2, 3]. Institute of Production Technology of Tokyo University in Japan developed innovative Preformed Yarn (PY) technology, which improved manufacturing process drastically from existing infiltration and other methods and resulted in reduction of manufacturing cost [4–6]. Substantial investigations have been carried out and are still underway, on the development and characterization of C/C composites. A comprehensive review on all those works is beyond the scope of this article. Despite their high strength and toughness at elevated temperatures, carbon fiber-reinforced carbon matrix composites (C/C) have never been applied to primary load-bearing structures. Instead, they have been used in structures which require only heat resistance, for example nose cones of re-entry space vehicles and heat resistant components of rocket nozzles [7–10]. One of the reasons for the limited use of C/C is the lack of reliability studies for their long-term use. Mechanical behavior is one of the most important design properties for primary load bearing structures intended for long-term use [11–19]. However, only a few studies on the impact behavior of C/C have been reported and the governing mechanisms of the impact behavior have not been successfully described.

In this study, an attempt is made to introduce the PY method, to synthesize C/C composites for structural and other applications. Moreover, as far as the authors are aware, manufacturing of C/C composite by varying the

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percentage of fiber weight fraction and impact test has not been reported so far. The PY developed in the current study is carbon fiber bundle surrounded by coke and pitch which is enclosed in nylon; three types of samples with fiber weight percentages of 30, 40, and 50%, respectively, are fabricated. The PY is subjected to hot pressing at 500°C followed by heat treatment at 2500°C. Subsequently, to eliminate the voids, two cycle pitch impregnation is performed on the samples, by hot pressing. Characterization of the specimens is then carried out in terms of hardness, flexural strength and impact properties.

EXPERIMENTAL PROCEDURE AND PROCESSING

Experimental Details

Preformed yarns (PY), which contain the carbon fiber bundle as well as the matrix of coke and pitch binder which is surrounded by Nylon-6. After completion of prototype PY machine it was planned to prepare at least three types of preformed yarns with three different compositions. Composition I (Sample 1) consists of 50 wt% carbon fibers (CF), 35 wt% matrix (50 wt% coke powders, 50 wt% binder) and 15 wt% thermoplastic sleeve. In compositions II (Sample 2) and III (Sample 3), the ratio of the binder in matrix is increased to 60 wt% (fiber 40%) and 75 wt% (fiber 50%), respectively, whereas the fiber content is decreased accordingly. The materials used in the manufacture of carbon–carbon composites are: Carbon fiber as reinforcement which is having tensile strength of 3.5 GPa, it is an organic fiber precursor (Polyacrylonitrile), known as PAN, is the commonly used precursor. Polyacrylonitrile (PAN) C-fibers with a tow size of 6K were used in the processing of the investigated C/C. The other materials used are pitch and coke as matrix materials, and nylon-6 as a sleeve material. Three different samples of C/C composite with different carbon fiber content such as 30, 40, and 50% weight fractions were prepared using above said PY machine. Unidirectional composition samples of C/C composite with different weight percent carbon fiber content were prepared.

By cutting the endless preformed yarns, chopped yarns were obtained, which were used to produce carbon fiber reinforced C/C composites. These PY enable ease to fabricate primary work pieces such as unidirectional (UD) sheets. Further, the C/C composite were produced by hot press molding method using these yarns and performs.

Processing Methods

The materials selected for this study is UD-reinforced C/C composites. The C-fibers in the investigated C/C composites are unidirectional configuration. In the UD-C/C, the C-fibers (in the form of PY) are kept in one direction which is perpendicular to hot press direction. The investigated C/C was fabricated through hot press mould method at 500°C subsequently impregnation of the C-fiber preforms by pitch at 240°C under a pressure of 1 bar for 5 hr. Then the pitch impregnated C-fiber performs was subjected to 700°C hot isostatic pressing at 800–1000 bar pressure for 1 day. This was followed by carbonization at 1000°C for 2 day and graphitization at 2500°C for 1 day, each under 1 atm. pressure. Pitch impregnation is normally repeated till maximum density is attained. The process cycle from pitch impregnation to graphitization was repeated for three times, until a density of 1.78 g/cc was obtained in the investigated C/C composites.

Characterization

Hardness. Initially, the three different weight fractions of C/C composites materials are taken and the test specimens were cut as per the ASTM-E-10 and are placed in the standard Brinell hardness tester. Brinell hardness test consists of indenting the composite surface with a 2.5 mm diameter steel ball at a load of 62.5 kg, and the load is applied for a standard time usually 15 secs and the diameter of the indentation is measured with a low power microscope after removal of the load. The hardness readings were taken for each specimen at different locations to circumvent the possible effects of fibers segregation. The average of four readings of the diameter of the impression was taken for calculation. The surface on which the indentation is made should be relatively smooth and free from dirt or scale. The Brinell hardness number (BHN) is expressed as the load P divided by the surface area of the indentation. And the experiment was repeated for all the three composition of samples. Rockwell hardness of the samples are also measured using Rockwell hardness tester. The average of three readings was used.

Flexural Strength. Three-point bending tests of C/C composites were carried out using computerized universal testing machine in accordance with ASTM D 790 standard at a deformation rate of 1 mm/min. The specimen for flexural test is shown in Fig. 1a.

Impact Strength. Un-notched Charpy impact tests were conducted on each specimen using impact tester. The

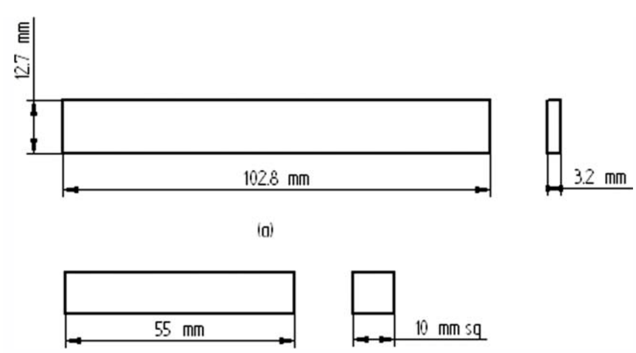

FIG. 1. (a) Flexural test specimen, (b) Impact test specimen.

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average of three readings is reported. The schematic diagram of impact test specimen is shown in Fig. 1b.

The flexural strength and flexural modulus values were determined for each composite. The test consists essentially of a hammer with a given amount of energy striking a notched test piece of fixed dimensions and recording the energy required to fracture the test piece at a specific temperature. The experimenter should also record whether the fracture mode was ductile or brittle.

RESULTS AND DISCUSSIONS

SEM/XRD Analysis

For all the samples SEM photographs were taken to study how the fibers are oriented in matrix and are shown in micrographs. From Fig. 2 (50% fiber) all the carbon fibers are oriented in unidirectional and measured the diameter of carbon fibers with an average value of about 6.95–7.1 μm but all the carbon fibers are not completely surrounded by the carbonaceous matrix and some fibers are find oriented other direction; this is due to breaking of fibers while hot press.

From Fig. 2, it can see that the carbon fibers are oriented in parallel direction to the carbon matrix and binders are completely surrounded the carbon fibers. It can be seen that the diameter of the carbon fibers are measured and is about 7.76 μm, The SEM analysis reveals that the interfacial bond between the carbon fibers and carbon matrix is not so good in sample and hence the properties of the materials are less than the expected values. This is because of many factors such as insufficient matrix or binder, hot press conditions, number of pitch impregnation cycle, and final heat treatment are those that plays an important role in manufacturing C/C composites. Optimization of all these factors needs to be done for better interfacial bond strength.

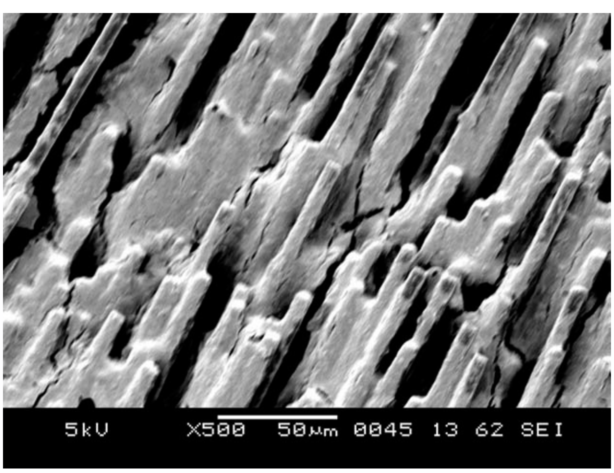

FIG. 2. SEM micrographs showing unidirectional orientation-50 wt% fiber.

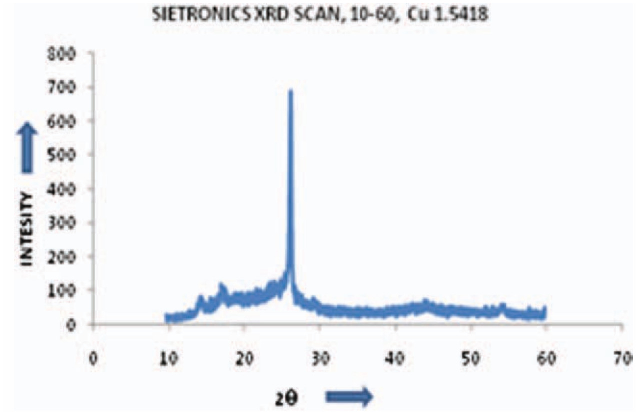

FIG. 3. XRD pattern of the sample with 30 wt% carbon fiber. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The X-ray analysis of the carbon–carbon composites have been taken to determine the presence of elements in C/C composites and extent of graphitization. All the samples show same X-ray pattern as in Fig. 3.

Hardness. The hardness test was carried out on all the three samples. Both BHN and Rockwell hardness tests were conducted. Hardness values of C/C composites as a function of fiber content are shown in Fig. 4. From the test results as shown in Table 1, it is seen that the increased in percentage from 30 to 50% of carbon fiber reinforcement increases the hardness of the material about 16%.

This is because of the higher percentage of strong fiber uniformly distributed throughout the matrix. Hardness is a resistance to penetration, wear, a measure of flow stress and resistance to cutting and scratching. The increasing the percentage of carbon fibers, is a slight increase in the hardness of the composites. It is generally known that, when fibers or other types of reinforcement are incorporated into a binder, the presence of the reinforcement can

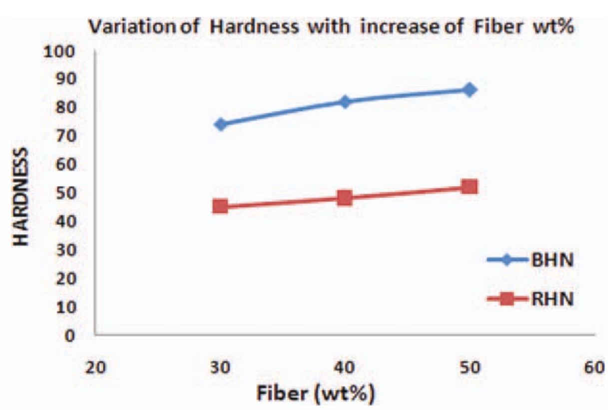

FIG. 4. Hardness values of C/C composites as a function of fiber content. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TABLE 1. Hardness values of three types of C/C composites.

Sl. No.	1	2	3
wt % of fiber	30	40	50
BHN	74	82	86
Rockwell hardness	45	48	52

affect the curing process; this can affect the properties of the cured binder. Increasing the carbon fiber content has also improved the hardness value as shown in Fig. 4. The increment of the hardness value for 30, 40, and 50 wt% carbon fiber reinforcement are BHN 74, BHN 82, and BHN 86, respectively, same trends were observed in Rockwell also. This shows that the mixture of carbon fiber and the binder are highly homogeneous and fiber per unit area is more as the weight percentage of fiber increases; this could be observed from SEM micrographs. Increasing the carbon fiber content restricted the percentage of binder available for the cross linking and resulted in a rigid interface, thus improves the Hardness. By these results, we can also conclude that for all the three samples carbonization process have been completed and the graphitization process have been initiated and almost completed it can be revealed from X-RD data. This shows that increase the fiber content resulted in increased load carrying capacity of C/C composites. Because fiber is stronger than matrix hence, this behavior is expected. The fiber distribution strengthening effect expected to be retained even at elevated temperature and for expected time period, because the fibers are not reactive with the matrix phase.

Flexural Strength. The variation of flexural strength and modulus values of C/C composites with increasing fiber fraction is shown in Table 2. Figure 5 shows the flexural strength and modulus of C/C composites for 30, 40, and 50 wt% fiber contents.

The effect of fiber on the strength of composite was investigated by flexural tests as a measure of the bonding of fiber to the matrix as well as mechanical strengthening. The flexural strength increased from 184 MPa in the case 30% fiber to 203 MPa for 50% fiber. The flexural modulus increased from 35 GPa in the case of 30% fiber to 46 GPa for 50% fiber. For fiber contents between 30 and 50 wt%, the modulus increased steadily. It is observed that the Flexural strength and Flexural modulus of the composites is increased by about 10 and 30%, respectively, as

TABLE 2. Flexural strength and modulus values of three types of C/C composites.

Sl. No.	wt % of fiber	Flexural strength (MPa)	Flexural Modulus (GPa)
1	30	184.87	35.39
2	40	197.14	40.10
3	50	203.30	46

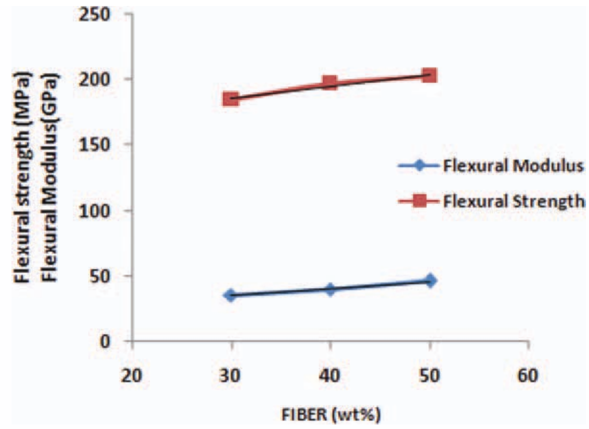

FIG. 5. Flexural strength and modulus of C/C composites as a function of fiber content. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the reinforcement content increases from 30 to 50 wt%. This shows that a strong fiber-matrix bond exists for all fiber contents.

The flexural strength C/C composites increased sharply to 203 MPa and the modulus increased to 46 GPa for 50% fiber. The values decreased gradually for lower fiber contents. It is clear that the flexural properties of C/C were enhanced due to the addition carbon fibers. The PY C/C composites exhibited higher impact and flexural strengths compared to conventional C/C composites.

Some important characteristics of composites have to be considered to correlate the results obtained. The quality of the interface in composites i.e. the fiber-matrix bonding and the interface stiffness play a very important role in the material's capability to transfer stresses and elastic deformation from the matrix to the fibers [10, 19]. This is especially true for composites because, they impart a high portion of interface. If the fiber-matrix interaction is poor, the fibers are unable to carry any part of the external load. The yield strength of fiber composite can be higher than that of the matrix alone; when there is a good fiber-matrix bonding [20]. A high interfacial stiffness corresponds to a high composite modulus. Hence, the increase in flexural strength and modulus as observed for the C/C composites suggests that stresses are efficiently transferred via the interface.

Fracture surface of the C/C composites are shown in Fig. 6a and b, reveals a brittle behavior characterized by smooth areas. In contrast to the smooth surfaces typically show a microrough structure characterized by flow patterns aligned in the direction of the main crack propagation, along with hackle like features. Such matrix deformation may occur by an energy-consuming mechanism in C/C composites and the mechanism has been reported by several authors.

Other energy consuming mechanisms are fiber debonding, rarely find fiber pulled out and the initiation of secondary cracks at local inhomogeneities indicated by curved region.

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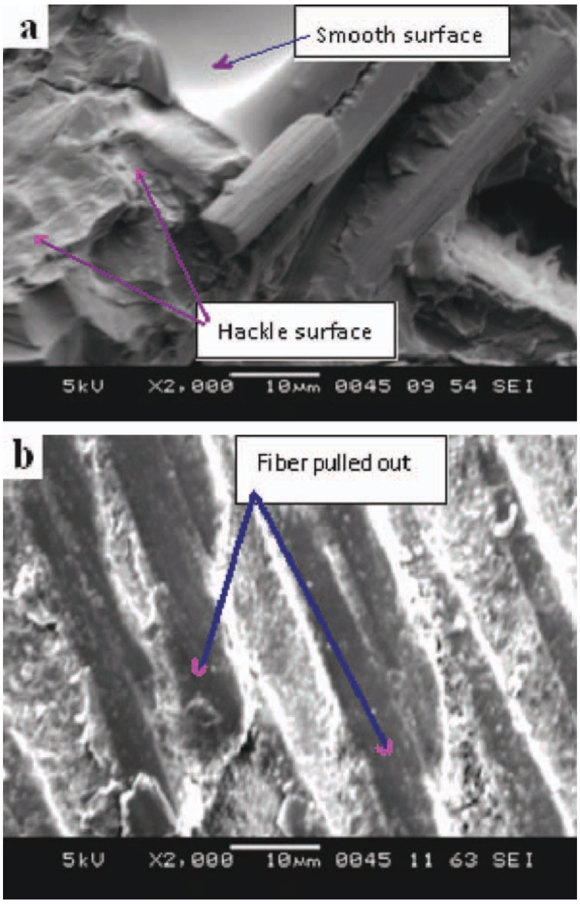

FIG. 6. (a) Fracture under three point bending (b) Fiber pulled out region. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

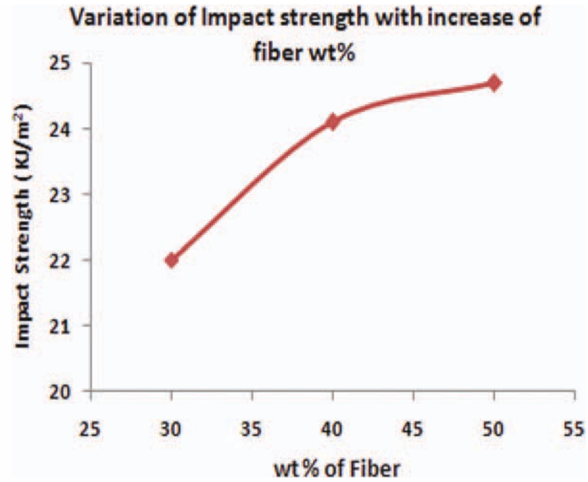

FIG. 7. Charpy Impact strength of C/C composites as a function of fiber content. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Impact Strength. Charpy impact tests are high speed fracture tests measuring the energy to break a specimen under bending conditions. The specimens are deformed within a short time and therefore exposed to high strain rates. In this work, the un-notched specimens were selected for impact testing, keeping in mind that the notches may induce stress concentration at their vicinity and the impact strength of the C/C may be further reduced. Table 3 and Fig. 7 shows the impact behavior C/C composites. The 30 wt% C/C composites the impact strength reported is 22; in the case of 40 wt% of fiber content strength increased gradually to 24.1. For 50% fiber composites, the impact value increased to 24.69. It is clear that; the amount of fiber increases resulted in the increased impact strength of C/C composites. It is observed that the compressive strength of the composites is increased by about 9% as the reinforcement content increases from 30 to 40 wt%. As the reinforcement content increases further (50 wt%), the compression strength of composite is increases from 9 to 12%. In general, the fiber enhance the impact properties of composites [5] while, fibers with high percentages induced more strength and hardness as we seen. We also confirmed that C/C composites do not become so brittle under high strain-rate conditions. The main mechanisms we find here is fiber debonding and fiber pulled out.

TABLE 3. Impact strength of three different fiber wt % of C/C composites.

Sl. No	1	2	3
wt % of fiber	30	40	50
Charpy impact strength (KJ/m^2)	22	24.1	24.69

CONCLUSION

The properties of C/C composites are dependent on fiber weight or volume percentages and binder ratios. Unlike polymer matrices, carbon matrices contribute significantly to the ultimate properties of the composites, especially in case of pitch and coke derived carbon matrices. The effect of increasing in the percentage of carbon fiber in preformed yarn C/C composites has shown the remarkable increase in various physical and mechanical properties of composites over conventional C/C composites.

By observing the flexural and impact strength data we can conclude that as the percentage of fibers increases these properties of the composites also increases for all the three weight percentage of composites. This is because of the ability of the C/C to operate at high temperature and loading conditions. Increasing the weight percentages of carbon fiber contributed significantly increases load bearing capacities and exhibited better structural properties. Composite with 50 wt% carbon fiber shows pseudo plastic nature of failure and higher load bearing strength.

NOMENCLATURE

BHN	Brinnel hardness number
C/C	carbon–carbon
PY	preformed yarn
SEM	scanning electron micrographs
UD	unidirectional
XRD	x-ray defraction

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