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Influence of Temperature and Time Shifts on the Densification of Randomly Oriented Carbon/Carbon Composite

Thakur Sudesh Kumar Raunija

Materials and Mechanical Entity, Vikram Sarabhai Space Centre, Indian Space Research Organisation,
Thiruvananthapuram - 695 022, India
E-mail: thakurskr@gmail.com

ABSTRACT

The efforts were made to improve the hot-pressing method of carbon/carbon (C/C) composite fabrication. The C/C composite was fabricated by hot-pressing the slurry moulded compact followed by carbonisation. The temperature and time shifts during hot-pressing were made in order to see their effect on the densification. Their effect was checked through visual inspection, density measurement, scanning electron microscope (SEM) analysis, and mechanical properties evaluation. The results showed that the temperature shift yielded in significant increase in the density after hot-pressing. Further, the density pick up instead of fall after carbonisation was noticed. The results further showed that the time shift too yielded in significant enhancement of density after hot-pressing and showed the pattern after carbonisation similar to the temperature shift. And also showed that the shifts of both temperature and time yielded in significant increase in density and moderate increase in mechanical properties.

Keywords: Temperature shift, time shift, densification, carbon/carbon composite, mechanical properties, hot-pressing method

1. INTRODUCTION

Carbon has its presence in almost every strata of the human habituated planet of the universe. Almost each and everything of daily usage this or that way is associated with carbon. It is very soft on one side and hardest among all materials on the other side. Its usage is also very vast. In advanced and strategic systems, it finds a significant place. The most advanced and strategic areas of space and defence are almost incomplete without carbon. It is used to fabricate several components of these sectors.

Various forms and types of carbon are used for such applications. However, composites of carbon are the materials which are widely used for these applications. There are several composites of carbon. The first layer of carbon composite binary tree includes carbon fiber reinforced metal matrix composites, carbon fiber reinforced polymer matrix composites, carbon fiber reinforced ceramic matrix composites, and carbon fiber reinforced carbon matrix composites. Further, these composites of carbon are classified based upon processing conditions, aspect ratio of carbon fibers, etc. Among all these types of carbon composites, the carbon fiber reinforced carbon matrix composites or more popularly known as C/C composites have their own importance. Mainly, because carbon with potential variations in structures can result in development of reinforcement as well as matrix systems with desired properties[1,2]. This is evident from very high modulus and conductivity pitch based carbon fibers and very high strength PAN based carbon fibers. Further, these are the composites which are most suitable for high temperature applications due to increase in their specific strength with temperature. They were first synthesised for such areas of applications in aerospace and defence[3].

Over a period of time, their usage has been extended from these high end applications to various other societal applications[3]. These include bipolar plates of proton exchange membrane (PEM) fuel cell[4], components of an engine[5], components of a distillation column[5], components of a nuclear reactor[6], various fixtures and dies[7], heating elements[8], crucibles, fasteners[4], vanes of a hot gas motor[9], etc. Though almost all of these products and components have been successfully fabricated out of C/C composite and their functioning has been demonstrated, the usage of them on commercial scale is still not viable. The major hurdle is their marathon processing. As a consequence, the processing is energy, finance and labor intensive. The efforts have been made[10-16] and still being made to bring newer methodologies, which can produce rapid and cost effective C/C composites. In our previous work[17], we have demonstrated a highly rapid methodology to produce rapid and cost effective C/C composite. The heart of this technology as described earlier is hot-pressing. In our another work[18], we have studied various parameters of hot-pressing in detail. Further, in another work[19], the influence of various hot-pressing parameters on the densification behaviour of this rapid and cost effective C/C composite was reported. However, some of the parameters of this crucial step were left untouched which are addressed in this work. These are temperature and time shifts during hot-pressing. How these two particular parameters are crucial and important in the densification of C/C composite were explored in this work.

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2. EXPERIMENTAL

2.1 Materials used

High strength polyacrylonitrile (PAN), and high modulus and high conductivity pitch based carbon fibers were used as reinforcement for making randomly oriented discrete carbon fiber reinforced carbon matrix composite. The diameter and length of PAN based T-800 and pitch based P-75 carbon fibers were 8 µm and 10 µm, 9 mm and 3 mm, respectively. Petroleum pitch derived mesophase pitch (named as Isroaniso matrix precursor) was used for deriving carbon matrix. The characteristics of reinforcement and matrix precursor were given elsewhere[18]. Distilled water was used as slurry media.

2.2 Fabrication of C/C Composite

The schematic process flow chart of the fabrication of C/C composite compacts is given in Fig. 1. The brief description of the process is as follows: Carbon fibers were chopped into discrete lengths using a chopping machine. The methodology of chopping continuous carbon fibers into sharp edge discrete length carbon fibers is reported elsewhere[20]. The chopped carbon fibers were split by mechanical means. The methodology for the splitting of chopped carbon fibers is reported elsewhere[17]. Mesophase pitch was powdered. Uniform slurry was then prepared by agitating the powdered mesophase pitch and chopped carbon fibers in distilled water. Slurry thus prepared was vacuum moulded in a metallic mould, and the preform along with mould was dried in an air oven. The preform thus made was hot-pressed to bind the reinforcement by deriving carbon matrix from mesophase pitch matrix precursor as a result of pyrolysis during hot-pressing and subsequent elevated carbonisation thereafter. Both the operation of hot-pressing and carbonisation were carried out under inert atmosphere maintained by purging argon gas. The hot-press and carbonisation furnace used for this study were custom built and Thermosystem make, respectively. Custom built hot-press used in the processing of composite samples is given in Fig. 2.

The details of the hot-pressing operation are given in Fig. 3 whereas details of elevated carbonisation are given in Fig. 4. Total two compacts were fabricated. First compact was fabricated by shifting the hot-pressing temperature from 650 °C to 700 °C whereas the second compact was fabricated by shifting hot-pressing soak time @ 700 °C from 1 h to 3 h. All the other parameters except the aforementioned were kept similar to the optimum parameters of the compact (OE-8) fabricated in our previous work[18]. The compacts with temperature and time

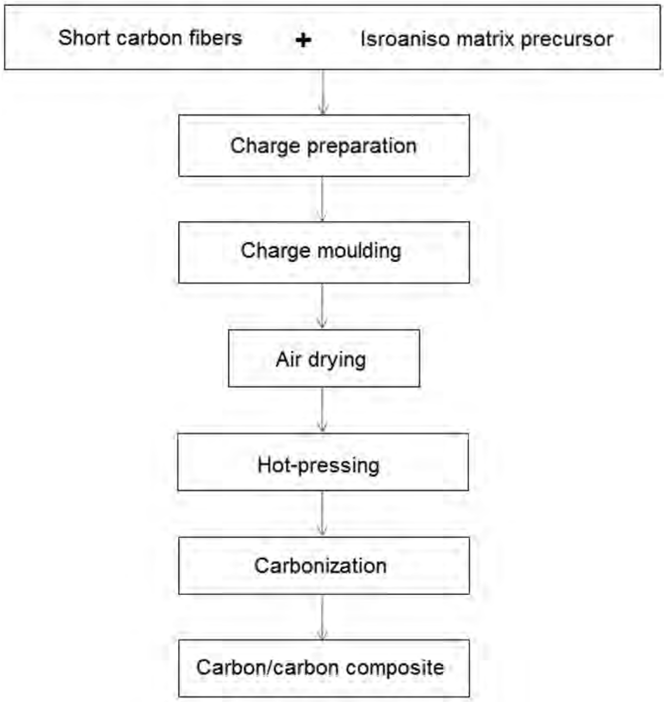


Figure 1. Schematic representation of process flow chart.

Figure 2. Custom built hot press used in the processing of composite samples.

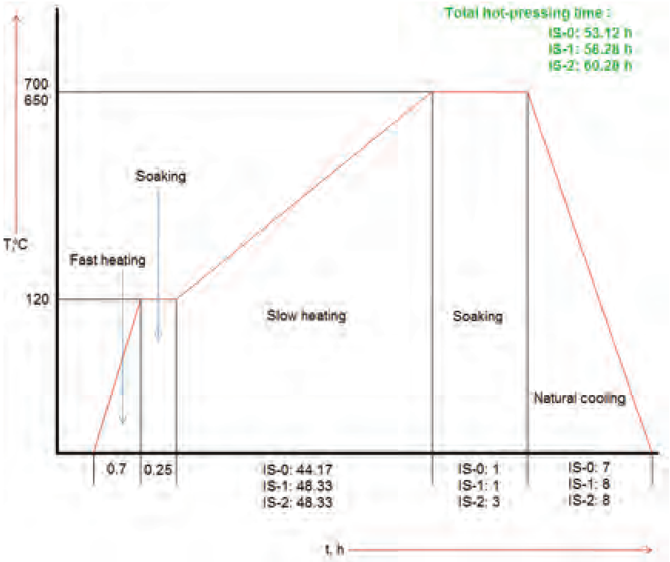


Figure 3. Hot-pressing plan for all the samples.

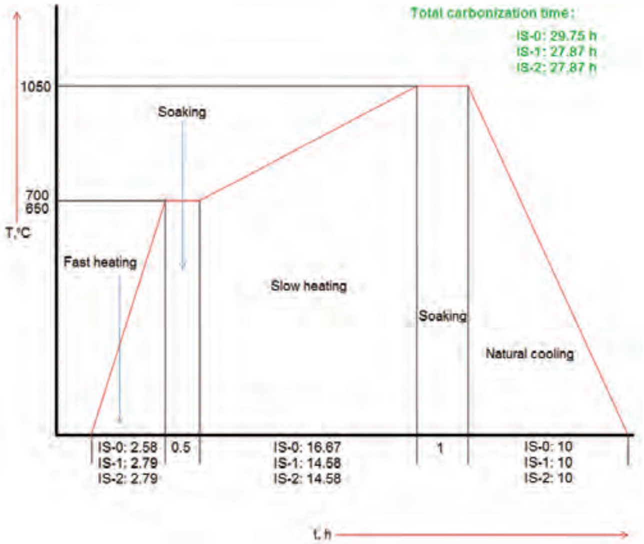


Figure 4. Carbonisation plan for all the samples.

shifts were named as IS-1 and IS-2, respectively. The results of these two compacts were compared with the one reported in previous work[18]. The compact of previous study was named here as IS-0 for clarity and better understanding.

2.3 Density

The bulk density of all the compacts after hot-pressing and carbonisation was calculated through mass volume formula described as fallows:

$$\rho_b = \frac{m}{v} \tag{1}$$

where

ρ_b = bulk density of the compact,

m = mass of the compact,

v = volume of the compact.

2.4 Visual Inspection

All of the C/C compacts were inspected visually for any kind of defects in the form of matrix to reinforcement distribution, cracks, pores, delamination, etc. Inspection of preforms, hot-pressed and carbonised compacts was done.

2.5 Microstructural Analysis

Carl Zeiss make, SMT EVO 50 model SEM was used for examining the surface morphology of the compacts. The microstructure analysis of the compacts after hot-pressing and elevated carbonisation was done using SEM. The SEM images were taken under variable pressure using 80 P air pressure. The filament of LaB6 was employed in the SEM for taking the images.

2.6 Mechanical Properties

Flexural and compressive strengths were measured using universal testing machine (Instron 5500R standard). Flexural and compressive strengths were tested according to ASTM C 1161-02C and ASTM C 695-91(Reapproved 2005), respectively. The details of the optimum dimensions of test specimens used were given elsewhere[18]. The fabrication of test specimens was done using Charmills Forbo 390 wire electron discharge machine (wire EDM).

3. RESULTS AND DISCUSSIONS

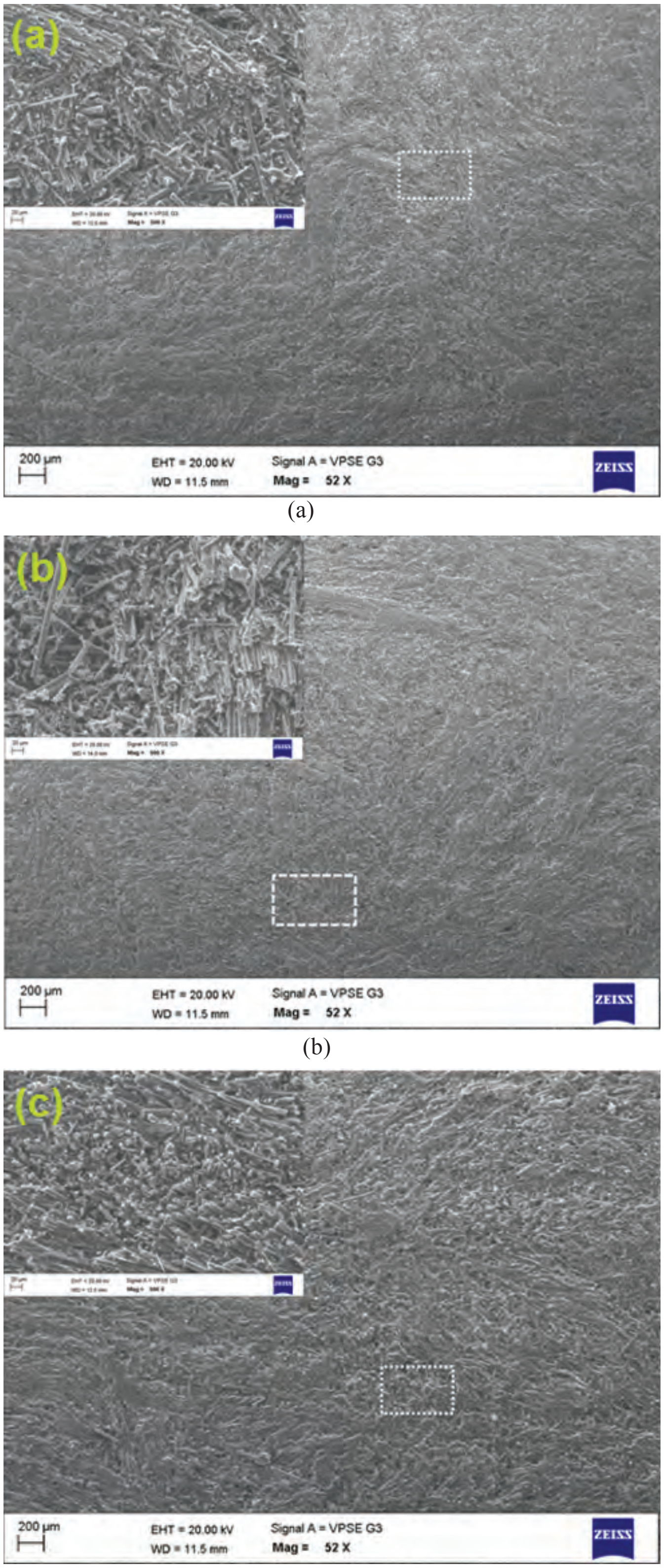
Author achieved higher density C/C composite along with good mechanical properties just after hot-pressing and elevated temperature carbonisation[18]. Besides, the processing time (90 h) of C/C composite was remarkably very short. The density and mechanical properties achieved were high compared to internationally reported methodologies for short fiber reinforced C/C composites[18]. Further enhancement, however, in density and mechanical properties was done by the application of low pressure densification cycles[19]. Though significant improvement in the final density and mechanical properties of C/C composite was recorded, the enhancement in the density and mechanical properties was not so lucrative compared to increase in processing time. Hence, to increase the density and mechanical properties without adding much densification cycles, the further work on hot-pressing can be done. On this line, the author took up this work as a supplementary work of his previous works[18,19]. As various parameters play an important role in the densification of hot-pressed artefacts, the author thought of working on some of the important parameters in order to further enhance the degree of densification after hot-pressing and elevated carbonisation itself. Some of the parameters of hot-pressing like pressure, heating rate, and reinforcement wt per cent were explored in detailed by the author in his earlier work[18]. The left out parameters were temperature of hot-pressing, soaking time at maximum hot-pressing temperature, etc. These parameters were studied in this particular study. The influence of these parameters on the microstructure, density and mechanical properties is discussed in subsequent sections.

3.1 Influence of Temperature Shift

First the author started with temperature shift. In this particular experiment, all the parameters except temperature shift from 650 °C to 700 °C, during hot-pressing, were kept similar to the compact IS-0 (OE-8) fabricated in his previous work[18]. As a consequence of this shift, the net increase in the processing time, as depicted in Figs. 3 and 4, was 3.28 h. The usefulness of this increase in processing time w.r.t. property gain is discussed in subsequent sections.

After hot-pressing and elevated temperature carbonisation, the visual inspection of compact IS-1 as compared to compact IS-0 doesn't signify any appreciable change in the texture. However, for further investigating the influence of temperature shift on micro level, SEM images were taken. The SEM micrographs are depicted in Fig. 5. From Figs. 5(a) and 5(b), it can be seen that the microstructure of compact IS-1 is not much changed compared to that of compact IS-0. However, some sort of smoothness in the microstructure of compact IS-1 compared to that of IS-0 is seen. The main causes of smoother microstructure are discussed in subsequent sections.

After microstructure verification, the influence of temperature shift on the density was checked. The density after hot-pressing was found to increase with temperature shift. Further, meager increase in the density of compact IS-1 after elevated temperature carbonisation was recorded. The density comparison of compacts is given in Fig. 6. From Fig. 6, it can be clearly seen that the compact IS-1 experienced



(a)

(b)

(c)

Figure 5. Scanning electron microscope micrographs of (a) IS-0, (b) IS-1, and (c) IS-2.

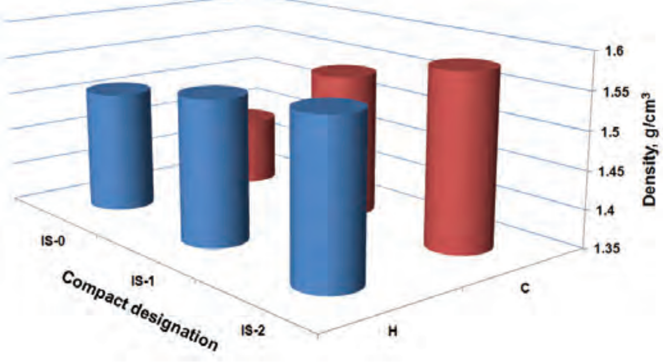


Figure 6. Density variation of compacts.

an extra densification (6.94 per cent more) as compared to compact IS-0. In comparison to increase in the processing time (3.64 per cent) from IS-0 to IS-1 (Figs. 3 and 4), the increase (6.94 per cent) in the density is quite high. This extra densification of compact IS-1 as compared to compact IS-0 might be the reason for the smoother microstructure of compact IS-1 as compared to compact IS-0. The causes responsible for the extra densification of compact IS-1 as result of temperature shift are discussed in subsequent sections.

The phenomena of extra densification in compact IS-1 with temperature shift can be understood with the graphs of real time process parameters such as temperature, pressure and height vs time. These graphs are shown in Figs. 7, 8, and 9. The primary densification for both the compacts (IS-0 and IS-1), as shown in Figs. 7 and 8, began as soon as pressurisation was started at around 250 °C, a temperature around the softening point of current matrix precursor. For compact IS-0 as shown in Figs. 8 and 9, the primary densification only could happen whereas for compact IS-1 secondary densification can also be seen.

Further, the maximum densification for compact IS-0, as depicted in Fig. 9, happened at around 370 °C. The pressurisation, thereafter, was stopped as per the plan. Since the compact IS-0 experienced a maximum temperature of 650 °C, even that also for a short while, the significant pressure build up at 650 °C (Fig. 7) was not noticed. As a consequence, the secondary densification (Fig. 8) could not happen. However, symptoms of minute pressure increase during hot-pressing of compact IS-0 at and around 650 °C, as depicted in Figs. 7 and 8, were seen. These symptoms increased the curiosity of the author to try the temperature shift in order to see its influence on pressure build up and secondary densification as a result of it.

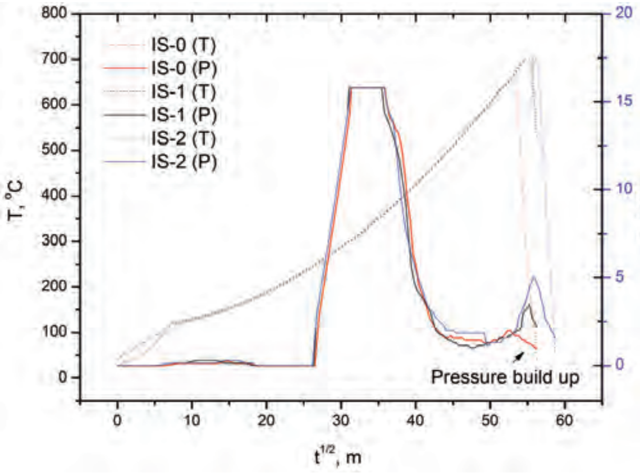


Figure 7. Pressure and temperature vs time profile of compacts.

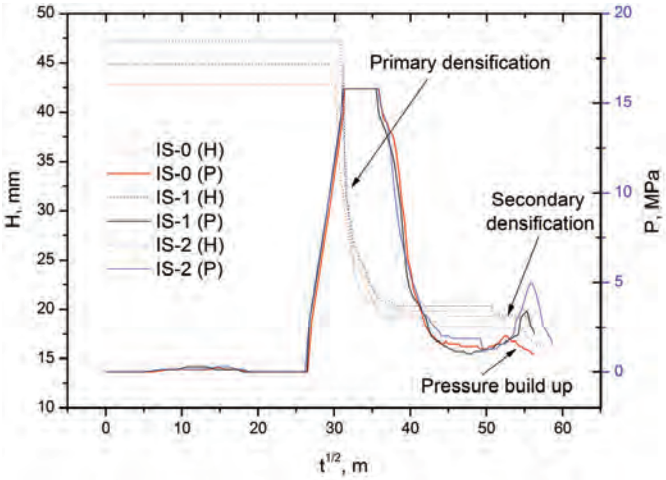


Figure 8. Height and pressure vs time profile of compacts.

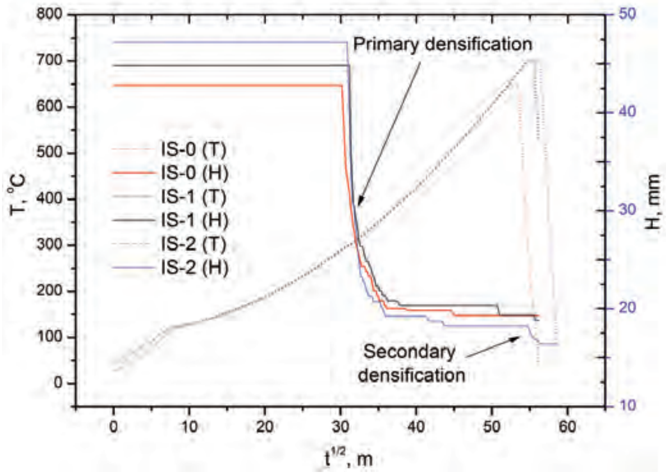


Figure 9. Height and temperature vs time profile of compacts.

The temperature shift, therefore, as mentioned above was made for compact IS-1. As a result of this, the compact IS-1 along with primary densification, at a temperature similar to compact IS-0, experienced secondary densification during temperature shift. The secondary densification (Fig. 9) was observed above 650 °C. This secondary densification was responsible for the extra densification of compact IS-1 after hot-pressing as compared to that of IS-0. The main cause of this secondary densification might be the pressure build as depicted in Fig. 7 as a result of temperature shift. This enhanced pressure resulted in extra densification of compact IS-1 in the form of secondary densification as depicted in Fig. 8.

Further, the primary densification would have happened in liquid phase because the matrix precursor used melts at around 220 °C and it remains in liquid phase while passing through discotic-nematic liquid crystal system until a temperature of around 400 °C is reached. Above 400 °C, for prolonged heating, these liquid crystals collide and coalesce to form larger regions of extended order until whole liquid is transformed to anisotropic phase which subsequently solidifies to form carbon at around 500 °C - 600 °C[8]. Since at around 600 °C almost the entire matrix becomes solid and it undergoes carbonisation[21] by liberating hetro-atoms like H, N, O and S in the form of gases like H_2O, CO_2, CO, CH_4, N_2, SO_2, etc.[5], the densification at this moment can happen only due to shrinkage. Hence, the secondary densification would have happened as a result of shrinkage caused by the liberation of hetro-atoms and the pressure exerted by the ram as a consequence of the liberation of hetro-atoms.

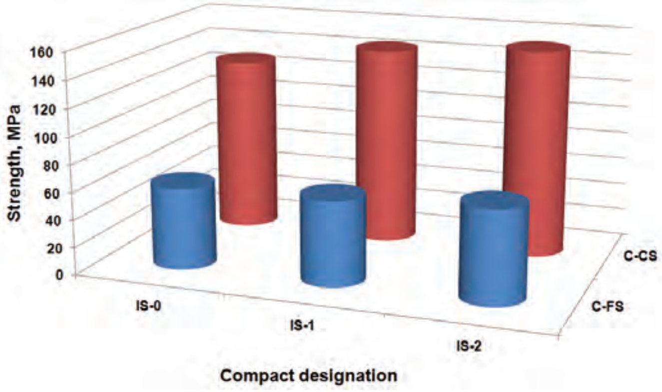


Figure 10. Flexural and compressive strengths of all the compacts.

Further, as mentioned earlier, the density of compact IS-1 compared to compact IS-0 was found to increase after elevated temperature carbonisation at 1050 °C also. The root cause behind this phenomena can be understood as follows: Generally, carbonisation involves the reactions like dehydrogenation, condensation, isomerisation, etc.[5]. These reactions start at a temperature of around 400 °C and continue till 1050 °C. However, the soaking at 700 °C for longer duration helps in finishing of these reactions at lower carbonisation temperature. Since the heating rate during hot-pressing was 0.2 °C/min, the compact IS-1 would have got enough time (from 650 °C to 700 °C) to finish the reactions like dehydrogenation, condensation. As a consequence of it, most of the hetro-atoms coming out from these reactions might have been liberated during hot-pressing itself. Due to this, the compact IS-1 would have experienced only isomerisation reactions in the form of re-arrangement with minimal liberation of hetro-atoms during elevated temperature carbonisation. As a result of this re-arrangement, the further shrinkage in the compact IS-1 would have been taken place which resulted in the enhancement of the density during elevated temperature carbonisation.

The influence of temperature shift on the mechanical properties of the compacts is shown in Fig. 10. From Fig. 10, it can be seen that the increase in the flexural strength of compact IS-1 is reasonably good (3.33 per cent) compared to that of IS-0. However, the increase in the compressive strength of compact IS-1, as depicted in Fig. 10, is remarkably high (12.4 per cent) compared to that of IS-0. In comparison to increase in processing time (3.64 per cent) from compact IS-0 to IS-1 as a result of temperature shift (Figs. 3 and 4), the increase in flexural strength is remarkably very high (12.4 per cent). The main causes behind enhancement of compressive and flexural strengths are the improved density of compact IS-1 compared to compact IS-0. It can be noticed that the increase in compressive strength is higher than flexural strength. The main cause behind this is the usage of short carbon fibers as reinforcement. As it is known, the short fibers act as filler and contribute more in compressive strength. Further, the density enhancement in short fiber composites contributes more in their compressive properties than flexural properties.

The rise in pressure with temperature shift and the densification as a consequence of this pressure rise indicate the possibilities to further enhance the degree of densification as a result of temperature shift. But, practically, it may not be possible. The main cause behind this is the die material. At higher temperature, die steel may not be useable as a die material. Graphite may be used. But, the usage of graphite may restrict the application of pressure. Additionally, the wear of graphite may be hindrance in commercial viability. Hence, 700 °C temperature may be suitable by considering all of the aforementioned causes. However, time shift on this particular temperature to extend the liberation of hetro atoms was tried which is discussed in the subsequent sections.

3.2 Influence of Time Shift

As mentioned in previous section, temperature shift was found to be very promising in the enhancement of the density and smoother microstructure of C/C compact due to occurring of secondary densification. It was concluded that it can further improve the density and microstructure. But due to practical constraints, temperature shift could not be further changed. The left option was time shift. As a consequence, time shift at 700 °C from 60 min to 180 min was made in compact IS-2. All the parameters except time shift were kept constant as per compact IS-1. Over visual inspection of compact IS-2, the significant change in the appearance compared to those of IS-0 and IS-1 was not seen. Similar to compact IS-1, the further influence of time shift on micro level was checked through SEM micrographs. As it can be seen, from Figs. 5(a), 5(b) and 5(c), the microstructure of compact IS-2 is very similar to those of compacts IS-0 and IS-1. However, some degree of smoothness in the microstructure of compact IS-2 can be seen compared to those of compacts IS-0 and IS-1.

The influence of time shift on the densification of compact IS-2 is shown in Fig. 6. From Fig. 6, it can be observed that the increase in density of compact IS-2 is quite higher than that of compact IS-1 as compared to compact IS-0. In comparison to increase in the processing time (5.81 per cent) from compact IS-0 to IS-2 as a result of time shift (Figs. 3 and 4), the increase in the density is remarkably very high (9.72 per cent). The cause behind this, as discussed in previous section, is the occurrence of secondary densification due to pressure build up. However, in the case of compact IS-2, as shown in Fig. 7, the pressure increases gradually with time shift, and the pressure reaches its maximum after 165 min and decreases gradually thereafter even the time shift remains for 15 min @ 700 °C. It shows that further increase in time shift may not aid in enhancement of secondary densification.

Aforementioned results may increase the curiosity of applying extra pressure during this particular band of temperature and time shifts in order to further increase the densification. But that may not help in densification because the matrix precursor used here possesses higher molecular weight and it is well reported by Hosomura and co-workers[22] that the pressure after certain limit has very little effect on the densification of high molecular weight pitch matrix precursors. Additionally, extra unidirectional pressure may result in the cracks. Hence, the pressure other than the pressure build up during hot-pressing as a result of temperature and time shifts was not applied. Further, this moderate rise in pressure was allowed as a result of temperature and time shifts in place of directly applying at low temperature in order to densify the compact.

The influence of time shift on mechanical properties is shown in Fig. 10. From Fig. 10, it can be noticed that the time shift enhanced the flexural and compressive strengths of compact IS-2 compared to that of compacts IS-1 and IS-0. In comparison to increase in the processing time (5.81 per cent) from compact IS-0 to IS-2 as a result of time shift (Figs. 3 and 4), the increase in compressive strength is very high (17.83 per cent). It is again caused by the extended degree of densification due to pressure build. The values of density and mechanical properties achieved after temperature and time shifts were found to be very close to those of reported by Baily and co-workers[12]. But, the processing time is very less in our methodology than that reported by them.

Further, it can be observed that the pressure rises, as depicted in Figs. 7 and 8, for all of the compacts as soon as the temperature reaches around 100 °C. This trend shows the evaporation of moisture present in the preform. At around 100 °C, it starts evaporating and continues till the temperature reaches 170 °C. The water vapours as a result of this happening would have exerted pressure on the ram. It signifies one important thing. Instead of going @ 0.2 °C/min beyond 120 °C for removing moisture, the higher heating rate can be preferred till the softening point of matrix precursor and for removing moisture the soaking at elevated temperature for sufficient duration can be given. It may help in reducing the processing time. Negative side of this approach, sometimes, may affect the microstructure. This particular possibility may be explored up later.

4. CONCLUSIONS

From the campaign of experimental proceedings following conclusions can be drawn:

i. The influence of temperature and time shifts was checked on the short carbon fiber reinforced mesophase pitch derived carbon matrix composite.
ii. Both the shifts were found very promising in enhancing the densification of the compact.
iii. Further, these shifts made the microstructure smoother.
iv. These shifts helped in improving the mechanical properties as a result of better densification.
v. It was demonstrated that the short carbon fiber composites with a density as high as 1.62 g/cm^3, and with flexural and compressive strengths as high as 68 MPa and 152 MPa, respectively can be achieved through hot-pressing followed by elevated temperature carbonisation in less than 100 h.

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CONTRIBUTOR

Mr Thakur Sudesh Kumar Raunija received his BE (Chemical Engineering), in 2008, from Chhotu Ram State College of Engineering (Now Deenbandhu Chhotu Ram University of Science and Technology), Murthal, Sonepat, Haryana, India. Currently pursuing his PhD from Indian Institute of Technology Delhi. Presently working as a Head, Carbon and Ceramics Laboratory, Materials and Mechanical Entity, Vikram Sarabhai Space Centre, Indian Space Research Organisation. He is principally working on the development of short carbon fiber reinforced carbon and ceramic composites, ceramic and metal-ceramic coatings, ablative systems, carbon fibers, carbon matrix precursors, propellants, etc.