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Tensile properties and fracture behaviour of carbon fibre filament materials

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Abstract Monotonic tensile properties and fracture behaviour of carbon fibre filament materials, namely single/mono- and multi-filaments (two and four filaments) as well as virgin carbon tows have been evaluated and discussed. Micro composite or single fibre approach is used in this study, which facilitated the evaluation of tensile properties and nature of fracture of carbon filament materials in a relatively short time with a large number of inexpensive trials. Tensile tests have been conducted on these filament materials at ambient temperature and laboratory air atmosphere. Load–elongation and the corresponding stress–strain plots thus obtained have been analysed to understand the tensile behaviour. The peak tensile strength of single carbon filament is found to be 3.8 GPa, and the value of the resilience obtained is 19 MJ/m^3. The peak tensile strength was found to increase moderately with further increase in number of filaments. However, the value of resilience was found to increase with increase in the number of fibres, which is attributed to the controlled failure of multi-filaments. On the other hand, the tensile strength of virgin carbon tow without matrix was found to be 1.13 GPa, and the value of the fracture energy was determined to be 9.9 MJ/m^3, which is nearly one fourth or even less than the corresponding values of the mono- and multi-filaments. The data obtained in the case of the virgin carbon tows were further analysed to evaluate the Weibull statistical parameters.

Introduction

Technological demand for materials that exhibit improved strength and stiffness has led to considerable research and development in the field of fibre-reinforced composites. In this direction, carbon fibres find use in aerospace systems due to their high strength, high stiffness and good toughness (when made into structural components) in addition to retention of high mechanical strength at ultra high temperatures (>1200 °C) for a number of advanced engineering applications [1–4]. Carbon heated in the range of 1500–2000 °C (carbonization) exhibits the highest tensile strength (5.8 GPa), while carbon fibre heated from 2500 to 3000 °C (graphitizing) exhibits a higher modulus of elasticity (530 GPa). There are several categories of commercially and semi-commercially available carbon fibres: standard modulus (250 GPa), intermediate modulus (300 GPa), and high modulus (>300 GPa). The tensile strength of different yarn types varies between 2000 and 7000 MPa. Typical density of carbon fibre is 1750 kg/m^3. Apart from these attractive properties, carbon also retains mechanical properties upto very high temperatures (upto 2000 °C). Unfortunately, carbon readily combines with oxygen at temperatures in excess of 400 °C, thus limiting its use unless oxidation resistant coatings or other appropriate

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form of oxidation protection are provided [5]. In case of the composites reinforced with carbon fibres, continuous fibres are used and fibre volume fraction usually exceeds 40%. Therefore, characteristics of the fibres are the most influential factor on the strength of the composites. The determination of carbon fibre strength also becomes important as the strength values are gauge length dependent. Finally, tensile strength of carbon fibres shows a large scatter and remarkable size dependence according to weakest link analogy.

Virgin carbon tows are the primary constituents for the carbon-fibre reinforced carbon (C–C_f) and silicon carbide (SiC–C_f) composites, which provide excellent thermo-mechanical properties at temperatures up to 2000 °C. Their high strength-to-weight ratio, which sustains up to temperatures as high as 2000 °C, makes them potential candidate materials for numerous high temperature applications, including the heat shields and structural components for re-entry space vehicles, high performance brake discs, and ultra-high temperature heat exchanger tubes [4, 6–9]. Mechanical properties of these composites are strongly dependent on the properties of the reinforcing fibres. Uniquely, fibre reinforcements offer great potential for improving strength as well as toughness of ceramic materials [7, 10, 11]. Primary reason for this interest lies in the assumption that the strong ceramic fibre can prevent catastrophic brittle failure in composites by providing various energy dissipation processes through progressive or gradual crack advance [12].

The aforementioned clearly points to a significant dependence of strength in carbon fibre filaments and the virgin tows to its processing parameters. Hence, the fibre strength determination is of primary importance because it dictates the ultimate strength of the composite. Data on the tensile strength and resilience of single filaments as well as of multi-filaments and tows are the basic necessary property inputs for composite design. Though time consuming and cumbersome, it is worth to evaluate the strength and resilience subjected to uniaxial tensile loading, as it can be later compared to the strength of the fibre in tows. This article presents the results of the investigations conducted on the room temperature mechanical behaviour of carbon fibres under tensile loading conditions. Ultralow capacity load cells (0–10 N) have been used to record the instantaneous load on the mono- and multi-filament specimens; while, load cells of slightly higher capacity (up to 1 kN) have been used for the determination of tensile properties of virgin carbon tows. Load–displacement data were recorded and analysed in terms of peak tensile strength, fracture energy and fracture behaviour. It is important to note that all the materials used in this study are from single source of carbon fibres, as one of the aims of the study is to bring out the changes in tensile strength and fracture energy for the development of carbon fibre-based composites.

Experimental details

Carbon tows of length 8 cm, more than the length of the window for fixing the specimen, were cut from the bundle. Each carbon tow was then pasted in a dish containing large quantity of acetone. Acetone is used to remove the sizing agents that are used during manufacturing of the carbon fibres. The mono-filaments or a single fibre is then extracted carefully from other fibres and pulled out using special forceps. Much care has to be taken during separation of a single fibre as it would break and fly in the atmosphere which causes health hazards. The paper window for mounting the specimen is prepared by cutting a chart paper of 100 mm length and 30 mm width. In order to mount a specimen having a gauge length of 50 mm, a rectangle window of same length is used. The specimen (single fibre) is mounted on the paper window by pasting it at the centre of the window on both ends using special type of adhesive (loctite-406). Finally, one more window of same dimensions is taken and pasted on top of the previous window in a fashion such that the specimen is sandwiched between the two paper windows. The adhesive is then allowed to harden before testing the specimen to avoid slipping. The specimens are mounted on paper windows as they can not be tested as such owing to their small diameter (6 μm).

The tow samples used in this study were made up of carbon multi-filaments consisting of 12,000 mono-filaments, which were supplied by Tae Kwang Ind. Co. Ltd., Korea. Average diameter of the fibres in the tow was 6 μm. Specimens for tensile testing are prepared by following the procedure outlined in Ref. [13]; however, with a higher gauge length of 50 mm and using paper tabs for gripping with a suitable adhesive. The tow samples were prepared by cutting the required length of the tow from the bundle of the fibres, which were then mounted onto the paper window tabs using suitable adhesive material. The samples are fixed at the centre of the paper window at both the ends. Special care was taken during sample preparation to achieve near-perfect alignment.

Support and mounting of specimens to avoid biaxial stress on the specimen is one of the major difficulties arising during tensile testing of single fibres due to the small diameters of the fibres. Carbon mono- and multi-filaments were tested according to the single fibre tensile test procedure (outlined in Refs. 14 and 15). The fibre specimens of average diameter of 6 μm along with the rectangular paper windows were mounted on INSTRON 1185 universal test system. Freely hanging pneumatic grips have been used for gripping the specimens, which exert tension along the fibre axis. This is of great importance to obtain reliable results as non axial loading causes bending stress which results in decreased strength values. The fibre sample along with the paper window was attached to the tensile grips and finally, the paper window was cut immediately before applying the load. All samples were tested at room temperature and at a cross head speed of 0.1 mm/min, corresponding to a strain rate of 6.67 × 10^{-4} s^{-1}. Tests were conducted in laboratory air atmosphere at an ambient temperature of 25 °C. An ultralow capacity Instron load cell of 10 N maximum load capacity was used to measure the load applied to the specimen as the load encountered during testing was in the range of 0.5–1 N. Special care was taken to accurately align the fibre on the paper mount so that off-axis loading was eliminated. Load–displacement plots were recorded and analysed to obtain the peak tensile strength and fracture energy of the materials. Similarly, the multi-filament samples were tested by pasting two or more filaments to the paper window and gripping the paper windows to the pneumatic grips. The tests have been carried out at the same cross head speed of 0.1 mm/min. In all cases, a minimum of three tests were conducted and the average values are reported.

Virgin carbon tows too were tested in a similar manner using a servo hydraulic universal testing machine model 8801, Instron, UK. The test set up consisted of special features such as alignment fixture and anti rotation device to avoid any biaxial stress on the specimens. Wedge action grips, having special type of coating to provide cushion to the composite specimens in the grip area, were used. The gauge length of the specimens was fixed at 50 mm. Tests were conducted at a cross head speed of 0.5 mm/min. Load–displacement plots were recorded in each case to analyse the fracture behaviour. The properties of the specimens were analysed in terms of tensile strength, fracture energy and Weibull modulus. Whereas the tensile strength indicates the ultimate strength of the composites under tension, the fracture energy provides an indirect estimate/index of the damage tolerance of the composite. Weibull modulus (m) reflects the degree of variability in the strength values, where a higher number indicates less scatter in data.

Results and discussion

Carbon mono-filament

Figure 1 shows the typical variation of load with elongation for the carbon mono-filament specimen. Though the load values encountered are extremely small (of the order of 0.2 N), they are fairly consistent as all the three specimens have failed at a peak load that was found to vary within a narrow range of 0.1–0.12 N. The data in Fig. 1 clearly show that the mono-filaments exhibit continuous increase in load with elongation. Hence, it is possible to calculate the failure stress and the resilience properties by converting the load–displacement data in Fig. 1 to the corresponding engineering stress–engineering strain plots, by taking the cross sectional area of the fibre considering the average diameter, 6 µm (Fig. 2). It is to be noted here that the experimentally determined variation in the fibre diameter is negligibly small (<0.1 µm). For the sake of clarity, the derived values of tensile properties (average of a minimum of three tests) are given in Table 1. Exact value

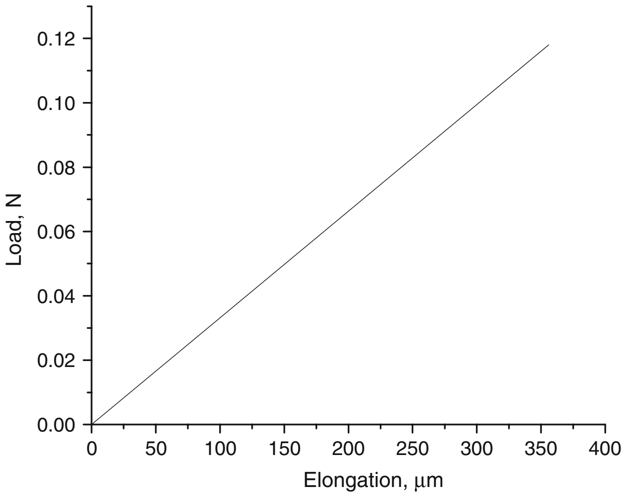


Fig. 1 Load–elongation plot for carbon mono-filament tested under tension

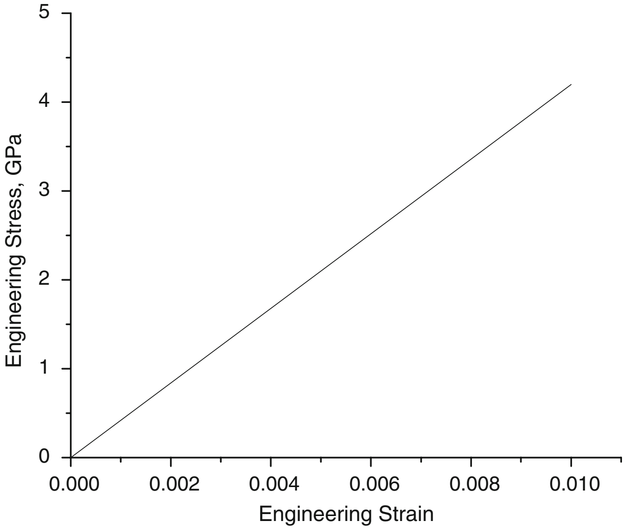


Fig. 2 Stress–strain plot for carbon mono-filament tested under tension

Table 1 Tensile properties of carbon mono and multi-filament windows (average of a minimum of three specimens)

Number of fibres	Peak tensile strength (GPa)		Resilience or energy to failure (MJ/m^3)		Weibull modulus
	Based on normal stress–strain data	Based on corrected stress–strain data	Based on normal stress–strain data	Based on corrected stress–strain data	
Single fibre	3.8	3.8	19	19	–
Two fibres	4.2	5.8	28	35	–
Four fibres	4.3	6.0	35	50	–
Virgin carbon tow[a]	0.94–1.17 (1.13)	–	9.1–10.9 (9.9)	–	10.5

[a] Range and average (values in parenthesis) correspond to a minimum of ten specimens

of young's modulus of the fibre specimens could not be determined as it was not possible to use any strain gauge in this study. However, the superimposition of data from more number of tensile tests showed that the modulus values are similar and do not vary significantly. A mean value as modulus of 384 GPa was found to be a reasonable estimate.

Two fibre multi-filaments

There had been attempts in the past to derive the tensile properties of carbon mono- and multi-filaments from the data obtained from the tensile testing of tows and then subjecting the data obtained from tows to statistical analyses. Though such procedure provides data of mono- and multi-filaments, there are no attempts made to verify these calculated values with the actual values. Hence mono- and multi-filaments having different number of filaments in a single test coupon/window are tested to obtain actual data. The load-elongation plot for the two filament sample mounted on a single window is shown in Fig. 3. It is clearly seen in Fig. 3 that the two filaments window undergoes two steps of failure. The data in Fig. 3 show that the two fibres sample undergo failure at point A with a sudden drop in load/stress value as indicated by AB. It is logical to assume here that one of the two filaments would fail first and the entire residual load/stress is borne by the second filament that has remained intact. A normal data analysis would suggest that the two filaments window could give a maximum failure stress of 4.2 GPa and the corresponding resilience value would be 26.7 MJ/m^3. This resilience value corresponds to the area under the normal stress–strain curve that can be obtained from the load–elongation data of Fig. 3.

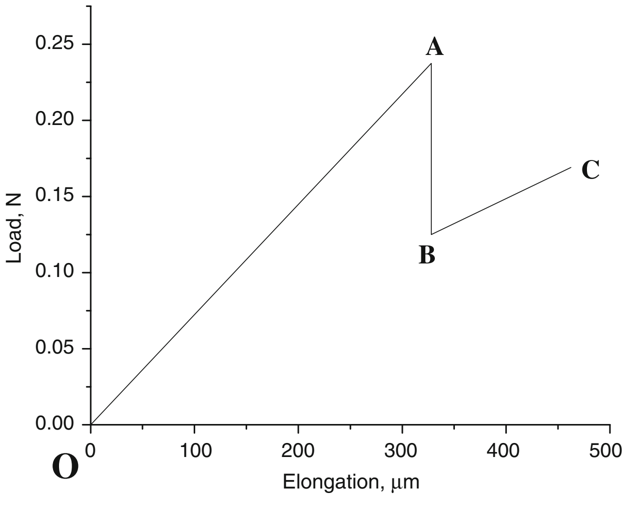


Fig. 3 Load–elongation plot for two carbon fibres specimen tested under tension

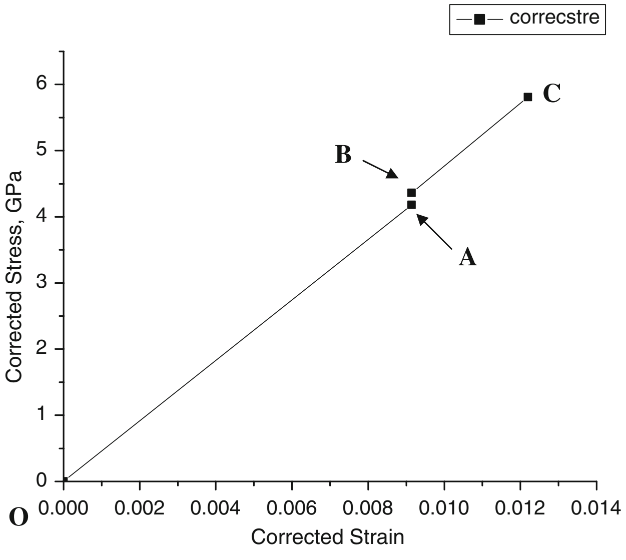


Fig. 4 Corrected stress–corrected strain plot for two fibre specimen

Alternatively, it is possible to conduct data analysis incorporating progressive fracture of filament failure. As can be seen from Fig. 3, the load in the initial portion of tensile stress–strain data from point 'O' to point 'A' is borne by both the filaments of the window. Where as, load from point 'B' to point 'C' in Fig. 3 is borne by one of the filaments that remained intact. This is because at point 'A' one of the two filaments is assumed to have failed which is logical as the nominal slope of two filaments remains similar as that of the nominal slope of the remaining filament. The slope of load–elongation curve from point B to point C is nearly half of the slope of the curve from point O to point A. However, the data in Fig. 3 in the two regions of O to A and B to C when interpolated, do not pass through the origin O. This points to the fact that the fibres in the multiple mini-composite are of slightly varied elastic modulus and cross-sectional area (fibre diameter). Considering these observations, we redrew the stress–strain data in Fig. 4 as corrected stress–strain data by considering the instant cross sectional area of the fibre(s) that are intact. It is seen from Fig. 4 that there is a slight increase in the stress value at the failure event of the first of the two fibres in the two filaments window. This is because of the arrest stress at point B, i.e., 4.25 GPa, is further increased as the second filament is loaded now to the fullest capacity and this fibre will now bear the full load. Comparison of data in Fig. 3 and corrected stress–strain curve given in Fig. 4 indicates that the maximum corrected stress (5.8 GPa at point C) is nearly 38% higher than the corresponding engineering stress (stress at point B; see the data included in Table 1). Similarly, the resilience calculated based on corrected stress–strain data (35 MJ/m^3) is nearly 20% higher than the corresponding resilience values obtained based on the load–elongation plot (see Table 1).

Four fibre multi-filaments

Figure 5 provides the load-elongation data obtained from the tensile test of four fibre window. The data in Fig. 5 clearly show that the four fibre window fractures progressively, thus resulting in not only a controlled failure but also significant increase in the resilience. Adopting the procedure, outlined for the two fibre filament train, the corrected stress–strain curve has been constructed and the same is shown in Fig. 6. The value of resilience (51.0 MJ/m^3) obtained in this case is nearly 175% higher as compared to the single fibre and nearly 50% higher as compared to the two fibre failure (see the data included in Table 1). The value of peak tensile stress obtained in case of four fibres is nearly same as of two fibres and considerably less as compared to the single fibre.

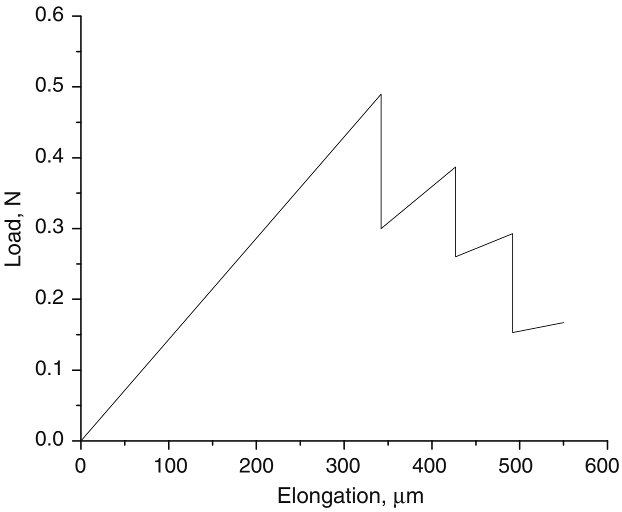


Fig. 5 Load–elongation plot for four carbon fibres specimen tested under tension

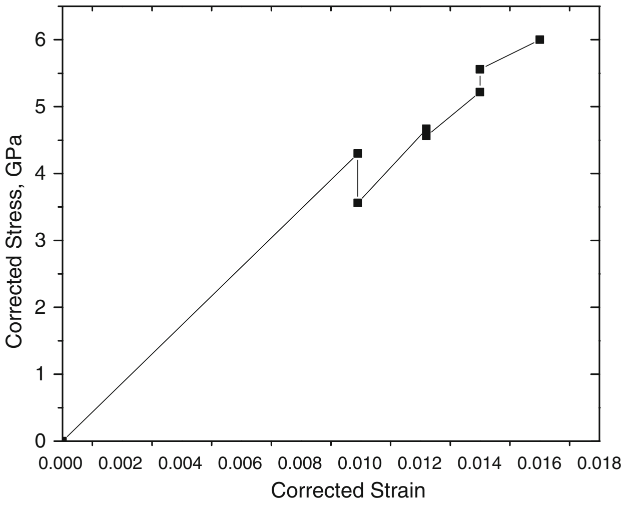


Fig. 6 Corrected stress–strain curve for the four filament fibre specimen

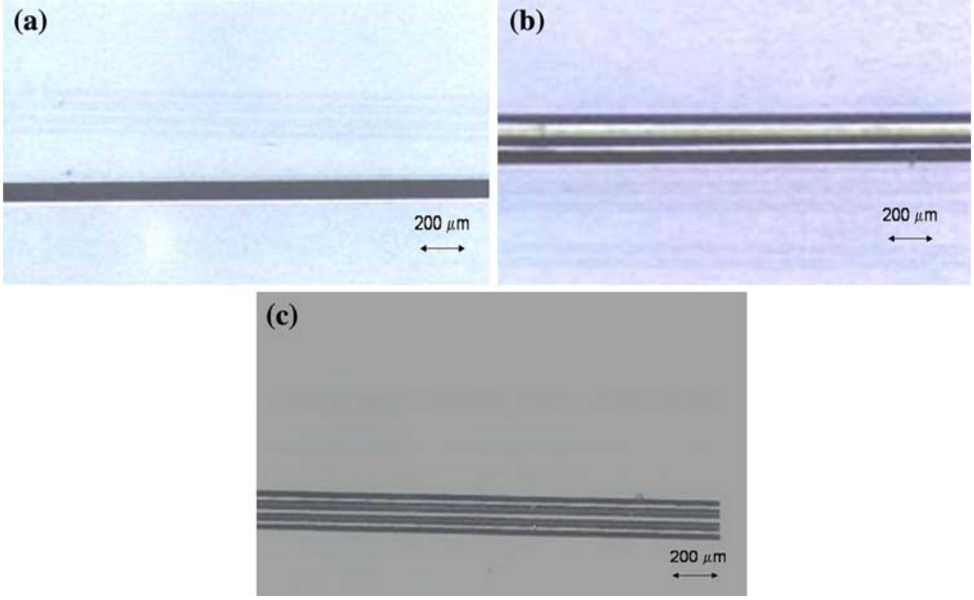
Fracture behaviour of mono- and multi-filaments

Optical micrographs of specimens before subjecting them to tensile loading have been taken and are shown in Fig. 7a–c, respectively for single fibre, two fibre and four fibre windows. The figure clearly shows that all the fibres in mono- and multi-filament specimens are damage free and could provide characteristic tensile properties. The fractured mono-filaments window after tensile loading has been again examined under optical microscope to study the fracture mode. The two fractured single carbon fibres have shown that, when observed for the nature of fracture, the fracture is nominal without any vertical splitting resulting in pure tensile failure of the specimen. This type of failure ultimately results into higher values of the tensile strength and resilience.

Virgin carbon tow

Figure 8 shows the typical stress–strain behaviour of a virgin carbon tow sample tested at ambient temperatures in air atmosphere. The stress values in Fig. 8 are derived from the experimentally obtained load values by considering the net cross sectional area of the tow (No. of fibres in the tow × Cross sectional area of single fibre based on the experimentally obtained average diameter). It is clearly seen from the data in Fig. 9 that multi-filament virgin

Fig. 7 Optical micrographs of **a** one, **b** two and **c** four fibre multi-filaments



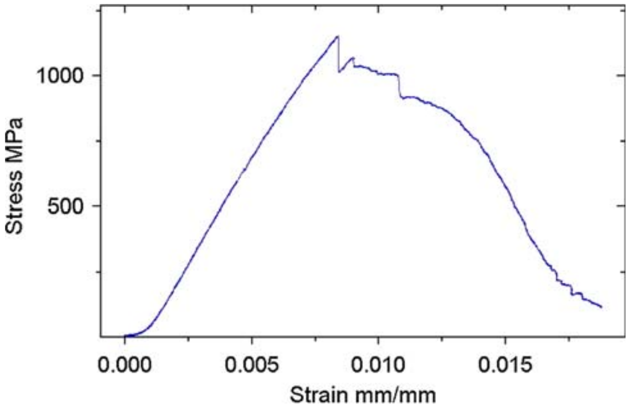


Fig. 8 Typical stress–strain plot of virgin carbon tow tested at room temperature

carbon tows exhibit a nonlinear tensile behaviour as a result of individual fibre breakage that does not cause complete tow failure. The stress value initially increases linearly with increase in the strain, followed by limited extent of non-linearity with increase in the strain until the attainment of peak load. Thereafter, the load drops gradually with increase in further strain due to fractional individual fibre breakage. The later stage of tensile fracture denotes controlled failure. Ten specimens have been tested under identical loading conditions to obtain the statistical distribution parameters, namely the Weibull modulus (m). Table 1 includes the data and its scatter corresponding to various tow specimens tested at room temperature. The degree of scatter in the tow strength is found to be significant. Concurring with the observation of reasonably high tensile properties, the virgin carbon tow shows extensive fibre movement before the onset of fibre fracture (Fig. 9).

Weibull distribution characteristics

For materials that fail by the weakest link theory, the Weibull modulus is the most widely used scientific index for the property variation, scatter and also, distribution of flaws in a given material. In ceramic-matrix, ceramic-reinforced composites, both the constituents i.e.,, matrix and fibre are essentially brittle and cracking involves defect-induced random failures. The statistical distribution of strength data can be described using a general form of the Weibull equation [16]:

$$P_s(V) = \exp\left[-V\left(\frac{\sigma - \sigma_u}{\sigma_o}\right)^m\right] \tag{1}$$

where, σ_u is the stress below which fracture is assumed to have zero probability, implying an upper limit to the flaw size (in many cases σ_u can conveniently be taken as zero); σ_o is a normalizing parameter of no physical significance; and m is a number, usually referred to as the Weibull modulus, which reflects the degree of variability in strength. The Weibull parameters pertinent to the fibre (m_f and σ_{of}) are usually derived from tensile tests performed on either single filaments or bundles [17]. Since the gauge length remained constant, for the purpose of evaluating the Weibull parameters, the above Eq. 1 can be linearized and written as:

$$\ln\ln(1/P_s) = \ln V + m \cdot \ln(\sigma - \sigma_u) - m \ln \sigma_o, \tag{2}$$

where, P_s is the survival probability, if the data follows Weibull distribution, a plot of ln $(-\ln(P_s))$ vs. $\ln(\sigma)$ assuming $\sigma_u = 0$, using a least square analysis, will be a straight line, having a slope equal to 'm', which parameter is known as Weibull modulus. A higher value of 'm'

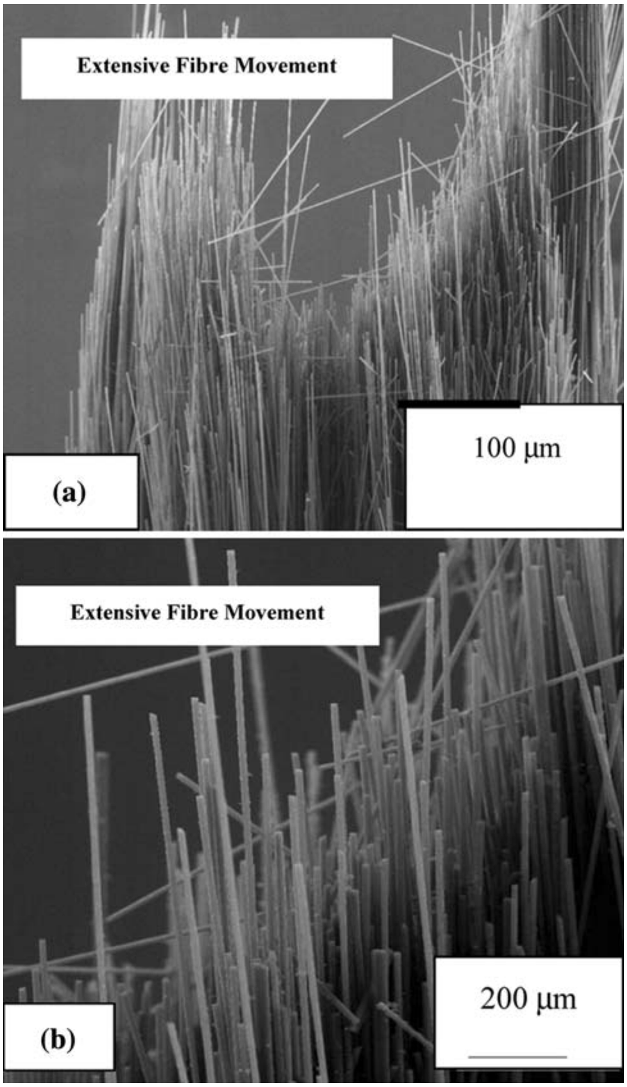


Fig. 9 SEM fractographs of the virgin carbon tow, showing extensive fibre movement/displacement before final fractrue

denotes less scatter in data and also, generally a material which has higher damage tolerance or elastic or elastic–plastic strain energy to fracture. The plot for evaluating Weibull modulus of the tensile strength of virgin carbon tows is shown in Fig. 10. The values of Weibull modulus (*m*) thus derived for the virgin carbon tow is included in Table 1. A value of Weibull modulus in case of the virgin carbon tows (10.5) indicates moderate degree of scatter as against those values associated with composites, which range in the values of 5–20 [16, 17].

Technological implications

The summary of the results obtained from this study is shown in Fig. 11. While the data from the uncorrected and corrected stress–strain curves show a constantly increasing peak stress and energy to fracture/resilience (such an increase is more pronounced in case of the later) with number of filaments in mono- and multi-carbon filaments, the values corresponding to the virgin carbon tow are significantly lower. This is simply due to the fact that in case of the virgin carbon tow, because of the large number of constituting individual fibres, only a very small fraction of the fibres are loaded at a given time and the fraction of the fibres undergoing fracture too is small. This is clearly reflected in the lower strengths, albeit with large strains to fracture, and also the large extent of gradual fall in the stress with strain after attaining peak tensile stress and an overall lower energy to failure (see data in Figs. 8 and 11). These observations point to the fact that construction of structural parts from long fibre-reinforced composites cannot have full advantage of the reinforcing fibres (which would have been huge in terms of the realizable property values), but there is a cost to be paid in terms of significant loss in the achievable strength and the fracture toughness. Similar set of results on the mini-composites based on the carbon fibre tows, reported simultaneously [18], points to additional loss in properties (both tensile strength and fracture energy) with further rigidization that occurs with matrix infiltration. However, it is interesting to note that despite such large losses in potential property improvements, carbon fibre-reinforced composites provide such property levels as compared to the other candidate materials that they are still attractive materials for ultrahigh temperature structural applications.

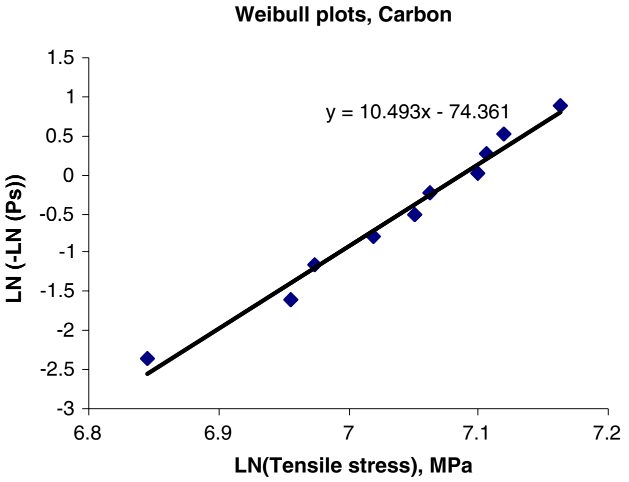


Fig. 10 Plot for evaluating the Weibull modulus of tensile strength of virgin carbon tows

Further, the data extrapolations in Fig. 11 from the observed behaviour of 1–4 fibres and a tow consisting of 12,000 fibres are massive and aforementioned discussion on the technological implication should be treated with caution. Extensive study needs to be conducted involving

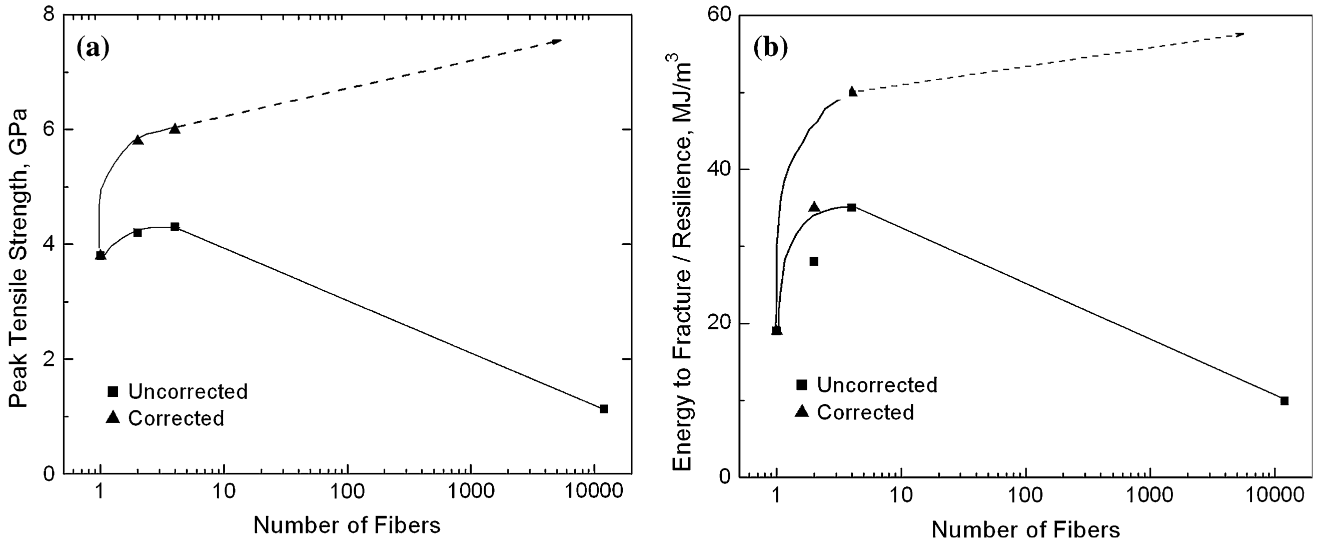


Fig. 11 Variation of **a** peak tensile strength and **b** resilience/energy to fracture with number of fibres based on the analysis of the uncorrected load–elongation data and corrected stress–strain data

the determination of tensile properties of multi-filament compartments covering the entire range of untested 4–12,000 fibres, to authenticate the above discussion. Till then, the conclusions thus drawn should be treated as indicative, if not illogical.

Summary and conclusions

Tensile properties, in terms of peak tensile strength and resilience values, are evaluated and reported for carbon mono- and multi-filaments as well as virgin carbon tow. Rationale has been provided for the progressive deformation fracture of these filament trains. In addition, the following conclusions are noteworthy:

1. The peak tensile strength of the single carbon filament tested under tension at ambient temperature is found to be 3.8 GPa.
2. The value of resilience determined from area under the stress–strain plot derived from load–displacement data of single fibre tested under tensile loading is found to be 19 MJ/m^3.
3. The value of peak tensile strength obtained in case of two fibres and four fibres is nearly the same, which is 50–60% higher as compared to the value of the mono fibre filament. On the other hand, the value of resilience was found to increase constantly with the number of fibres as a result of progressive nature of failure in case of multiple fibres.
4. The nature of failure is in the form of steps resulting into progressive failure of multiple fibre specimens. This ultimately results into graceful failure, and hence, increases the resilience property of the multiple fibre specimens. The resilience value obtained in the case of four fibre window is nearly 175% as high as that compared to single fibre window, and nearly 50% as high as that compared to two fibre window.
5. Significant fibre pull-out is observed during the tensile testing of virgin carbon tow. The tows exhibit graceful fracture with fracture stress of 1.13 GPa and an energy to failure of 9.9 MJ/m^3.

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