Arch. Mech., **65**, 1, pp. 27–43, Warszawa 2013

Axial compressive strength testing of single carbon fibres

I. P. KUMAR, P. M. MOHITE, S. KAMLE

Department of Aerospace Engineering
Indian Institute of Technology
Kanpur, UP 208016, India
e-mail: mohite@iitk.ac.in

In this study the tensile recoil test – an experimental technique to find the axial compressive strength of single fibres is discussed and the axial compressive strength of single carbon fibres has been determined for a fixed gauge length. Several fibres were tested at different stress levels and the data obtained regarding the failure of the fibre due to initiation of recoil compressive stress was used to determine the strength in compression. The recoil test data was evaluated using usual statistical and probabilistic models. Probabilistic Weibull and logistic models were used to evaluate the compressive strength distribution obtained from the recoil test. The axial compressive strengths of single carbon fibre predicted by all models are in good agreement with each other and the probabilistic models are very close to each other. The compressive strength of carbon fibres, is calculated as 869 MPa, which is very close to the value reported in the literature obtained by this method.

Key words: carbon fibres, axial compressive strength, tensile recoil test, experimental mechanics.

1. Introduction

The fibres are principal load carrying constituents in a fibrous composite. The mechanical properties of a fibrous composite are, in general, dominated by the properties of fibre. In a micromechanical modeling or analysis of such composites for the evaluation of effective composite properties and damage initiation and propagation [1, 2], the properties of individual constituents are one of the important factors. Thus, it becomes inevitable to determine the various mechanical properties and damage or strength parameters of a fibre. For a complete micromechanical analysis a number of such properties and parameters are required. Further, in the past many researchers have attempted to study the effect of morphology on the compressive failure of single fibres (for example, see [3–5]).

Over the years, the carbon fibres have found a prominent place in high-performance advanced fibre composites applications. The unidirectional laminated composites of carbon fibres are used in critical applications due to their excellent properties. The axial tensile strength of the some of the carbon fibres can be as high as 6.37 GPa (see [6]) and the axial tensile strength of the unidirectional composite lamina made from these fibres achieve a value of 3.04 GPa. Certainly, these values are very high as compared to most of the fibres. However, it is shown that the axial compressive strength of unidirectional carbon fibre composites is relatively weak in comparison to their tensile strength. This is shown in Fig. 1, wherein the tensile and compressive strengths of unidirectional lamina made from various grades of carbon fibres are compared (for details see [6, 7]). From this figure it can be seen that the compressive strength of unidirectional laminae ranges from 20 to 60% of their tensile strength. This feature of the carbon fibre composites limits their usage in structural applications under compressive loads. Further, this has invoked the extensive study of failure mechanims [9, 10], their modeling (see comparison of models in [8]), etc. that limit the compressive strength of unidirectional composites. For over a half century, researchers are trying to understand the issue and improve the compressive properties. It is shown in the literature that the compressive strength of unidirectional lamina depends upon various factors and axial compressive strength of the fibre is one of the important factors (for example, see work at this laboratory [11, 12]). A detailed review of study related to these issues can be found in [13].

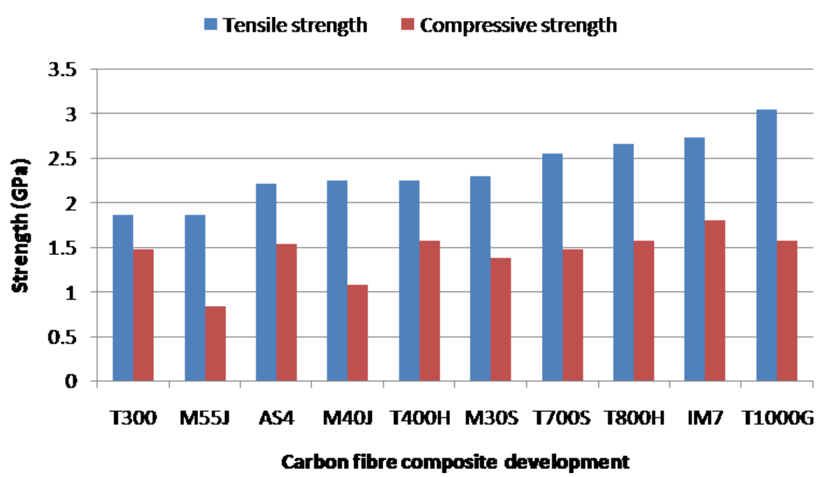

Fig. 1. Tensile and compressive strength of unidirectional carbon fibre composites [6, 7].

The longitudinal compressive properties of carbon fibers are not so well understood as the tensile properties are particularly due to the difficulties of carrying out compressive tests. Since the fiber diameter is small (5–10 μm), it is very difficult to apply a true axial compressive stress to a single fiber without causing buckling [14]. Since the 1950s, researchers have endeavored to find appropriate methods. These methods include: elastica loop [15–20], bending beam [20–22], single fibre composite [23, 24] and tensile recoil method [25–28]. However, all four have serious drawbacks because of their indirect interpretations of compressive properties. In methods such as the elastica loop and bending beam tests, the state of stress developed in the fibre is not purely axial compression (for example, see [15, 21]). Tests with single fiber in the matrix may provide better results. However, the residual stresses developed in the fibres during the fabrication by shrinkage action of the matrix affect the method significantly. Further, the alignment of the fibres is also an important issue (see Miwa *et al.* [29] as an example).

Recently, direct methods of compression of a single fibre were proposed (see [14, 30–37]). In [37] a comparison of axial tensile and compressive strength of carbon fibres was done. Tong et al [38] have used the nanoindentation technique for the measurement of compressive strength of basalt fibres. Together, these methods have provided much useful information for comparisons of fiber compressive properties. Some researchers have conducted tests on composite strands along with tests on single fibre for the evaluation of compressive properties (see [39]). Zu *et al.* [40] have recently used this technique for the measurement of axial compressive strength of carbon fibres fabricated from carbon nanotubes (CNT).

The aim of the present study is to explore the method of recoil by tension for the measurement of axial compressive strength of single carbon fibres. Further, the usual statistical methods and probabilistic models reported in literature are revisited and compared for compressive strength prediction. The present study is a part of the work carried out in our laboratory for the complete mechanical and damage characterization of fibres [41, 42] and the damage modeling of the composites [1, 2, 11, 12]. It was shown by the model proposed in [11, 12] that the axial compressive strength of the unidirectional lamina directly depends upon the compressive strength of the reinforcing fibres.

2. Experimental

2.1. Theory of the experiment

The tensile recoil method developed by Allen [25] in 1987 is preferred over other methods due to its simplistic procedure. In this method, the fiber specimen is stretched to a predetermined tensile stress level. Thus, the stretched fibre stores an equivalent amount of strain energy. A recoil effect is initiated in the fibre by cutting it either by an electrical discharge or a scissor. As the fiber is cut, the tensile stress goes to zero and the strain energy stored in the fibre gets converted into kinetic energy. This causes a stress wave front in the fibre which moves along the fiber towards the clamped ends. During the motion of wave front the additional strain energy is converted to kinetic energy. By the time the stress wave reaches the clamped ends, all the strain energy has been converted to kinetic energy. It is assumed that the clamp is rigid. Due to the rigid nature at the ends, the kinetic energy is converted back into strain energy. This initiates a compressive stress front and it propagates back down the fiber length. It is assumed that the compressive stress is equivalent to the released tensile stress. Thus, if the compressive stress exceeds the compressive strength of the fibre, then the fibre end fails by this compressive stress. For more details on the theory of the experiment, see [21, 30].

It should be noted that this compressive strength can not directly be evaluated by this test method. One needs to do testing on many specimens when the failure probability at different tensile stress levels is examined. Thus, at different recoil compressive stress levels the critical compressive strength can be determined. In general, this is given at 50% failure probability. The methods for the interpretation of the compressive strength are dealt with in a later section.

A number of researchers have used this method for the prediction of compressive strength. Like any other method, this method has also its own pros and cons. The method has serious flaws like indirect interpretation of compressive strength (see [25]), effect of dynamic loading (for details see [15]). Further, there are bending effects, particularly at lower end of the long specimen due to gravity (see [3, 4] for more details). This method makes assumptions that the fibre (1) obeys Hooke's law and the material is a linear elastic material, (2) is rigidly clamped at each end of the gauge length, (3) has a zero initial velocity and (4) has an initial uniform tensile stress along its length at failure with the exception that the stress is zero at the location of breaking (see [25] for more details).

In the recent years, the theory has been improved by many researchers to alleviate some of the issues and widely used in compressional behaviour studies of single fibres. Some researchers have suggested grease coating to reduce the recoil bending/buckling. This method has been further explored to investigate the mode of fibre fracture like shear failure, stepped fracture normal to fibre axis, kink-band formation, etc. in carbon fibres (see Dobb *et al.* [4] for more details). This provides important information for the desired morphology to improve the compressive strength. Further, some researchers have suggested improvements over interpretation of compressive strength with better models (see [4, 43–46]). The effect of non-uniform tensile strength along the fibre is also studied using this method by Miwa *et al.* [29].

2.2. Measurement of fibre diameter

Measurement of diameter to study the scatter in diameter of fiber specimen was obtained with an advanced optical microscope (model ZIESS AXIO Imager.M1m, with maximum possible magnification up to 1000X), which was used to measure diameter carbon fibers. A total of five fibers of each type were measured. Four measuring points were taken along the length of each fiber specimen. The variation in the diameter along the length of the fiber was found to be negligible and so the mean diameter of each specimen was taken out as the average of diameters at those two measuring points. A significant scatter in the diameter of different fiber specimens was observed. The mean diameter was determined and used for all the calculations.

2.3. Specimen preparation and testing

The tab for mounting the specimen in the machine is prepared out of thick paper. A slot of length equal to the gage length of 45 mm is cut out in the middle of the tab. A single filament is randomly chosen from the fiber bundle and pasted at both ends of the slot in the paper tab using suitable adhesive.

The specimens were tested in an INSTRON TT-CML machine (5 ton capacity), with a load cell of 10 g. The specimens were mounted similar to the tensile test. Immense care was taken to ensure the axis of the fiber was aligned in-line with the axis of the cross-head in order to simulate a uniform stress condition over the cross-section of the fiber. The full-scale load was set to 10 g and the cross-head and chart speeds were set to 0.2 mm/min and 50 mm/min, respectively.

Without disturbing the set-up, both sides of the tabs were cut carefully at the mid-gauge length. The specimen was then tensioned to a pre-determined stress levels or loads. Then without adding any load, the fiber was severed using a surgical scissor, holding the scissor parallel and using only the tip of the scissor to cut. Each tab with its remaining fiber segment was then carefully collected from the clamps for examination to ascertain whether or not fracture had occurred during recoil, and the event at each tab was recorded accordingly. Details of the test scheme are shown in Fig. 2. Five filaments (10 halves) were tested at each applied stress level. The tension was increased in steps of 0.1 g force (equal to 26.94 MPa stress), from 2.6 g to 4 g. It should be noted that nearly all recoil failures occurred in the region close to the tab ends.

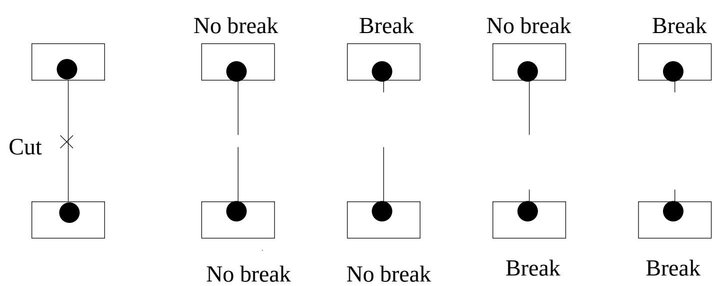

Fig. 2. Tensile recoil method and various possible fibre ends failure scenarios.

3. Calculation of compressive strength of the fibres

Unlike tensile testing, tensile recoil test does not provide exact compressive strength of a filament. The failure of the fibre is artificially introduced in this process. The recoil compressive wave is significantly above the intrinsic compressive strength of the fiber for failure and if it is below then there will not be any failure. When the fiber ends undergoing a secondary failure, it is difficult to estimate how much stress above the intrinsic compressive strength of the fibre the recoil wave has imparted to the fiber. Similarly, when the fibre survive at both ends one can not estimate how much the intrinsic compressive strength of the fiber exceeded the stress applied by the recoil compressive wave. Hence, in the literature several statistical methods have been proposed to evaluate the recoil test data. In the following, four methods have been briefly explained.

3.1. Model 1

In this method the data are arranged in ascending order of load. A range of stresses is identified over which the observations change from 100% survival to 100% failure (that is, 0% survival). Then, the recoil compressive strength is calculated as the average of the two endpoints of this range. For more details, see Allen [25] and Wang *et al.* [27].

3.2. Model 2

In this method (as described by Hayes *et al.* [3] and Park [28]) the data are arranged in groups by stress ranges. Then, for each stress range the percentage of fibre halves that survive the test is calculated. Then a plot is constructed for the percent survival of the fibre halves as a function of the applied stress. Here an applied stress range is represented as the numerical average of the highest and lowest stress values of that range. Finally, the sample recoil compressive strength is estimated by calculating the stress corresponding to 50% survival. It should be noted that the selection of the stress ranges is a subjective matter. Therefore, it is suggested that the smaller stress ranges to be used for more accurate results. The main assumption in the selection of stress range is that there is a smooth transition from 100% survival to 0% survival. This method presents a good graphical technique for presenting recoil compressive stress data. Further, this method is well suited where a wide stress range data are involved in the test.

3.3. Probabilistic models

Failure data do not conform well to any rigid statistical distributions. Thus, a model that could enable the shape of the distribution to be altered by the data itself is required. Thus, two models were adopted to represent the recoil compressive failure data. The Weibull model as proposed by Hayes et al. [3], for high performance fibers, is based on the weakest link theory. According to the model, the cumulative probability distribution, $F(\sigma) = \text{probability}\{\text{recoil strength} \leq \sigma\}$, where σ is the recoil stress and mean of the distribution $\bar{\sigma}$ of failure is given by

$$F(\sigma) = 1 - e^{[-L(\sigma/\sigma_0)^m]} \qquad \text{and} \qquad \bar{\sigma} = \frac{\sigma_0}{L^{(1/m)}} \Gamma\left(1 + \frac{1}{m}\right), \tag{3.1}$$

where L is gauge length of the fibers in m, σ_0 is the scale factor corresponding to severity of flaws, m is the shape factor relating to kinks within the fiber and Γ is the statistical gamma function.

The logistic model was also used by JIANG *et al.* [26] to predict the compressive strength of the fiber by fitting a logistic curve for the recoil test data. According to the model, the cumulative probability distribution $F(\sigma)$ is given by

$$F(\sigma) = \frac{e^{[a+b\sigma]}}{1 + e^{[a+b\sigma]}}, \tag{3.2}$$

where a is the intercept of a logistic curve and b is the regression coefficient of the recoil stress.

In this study the fraction of specimens that fail means that under a given recoil stress this fraction of specimen has a recoil strength less than the applied stress. As we know, in this test procedure when the fibre is cut at mid-gauge length, there will be two separate recoil processes in the upper and lower fibre segments. It is assumed that the recoil failure at either end is an independent process. Further, this process is assumed to be governed by the same probability distribution. Then the various probabilities of end failures are given as below.

The probability that only one end fails $g_1\{\sigma\}$ is given by

$$g_1\{\sigma\} = 2F\{\sigma\}[1 - F\{\sigma\}]. \tag{3.3}$$

The probability that both end fails, $g_2\{\sigma\}$ is then given by

$$g_2\{\sigma\} = F\{\sigma\}F\{\sigma\}. \tag{3.4}$$

The more details about these models can be found in [26] and [43–45].

REMARK. In this study the gauge length is kept fixed. One could have studied the effect of gauge length on compressive strength of the fibre (for example, see [26]). However, the effect of gauge length on the axial compressive strength of the fibre is not studied here.

4. Results and discussion

As we know, for the test method – tensile recoil, adopted for the measurement of compressive strength of single fibres the compressive strength of the fibre tested must be less than its axial tensile strength. As mentioned earlier this study is a part of the work carried out at this laboratory [41, 42]. The other properties measured for this carbon fibre are reported in Table 1.

Table 1. Axial properties of carbon fibre measured at room temperature for a 25 mm gauge length [41, 42].

Diameter μm	6.83
Tensile modulus (GPa)	246.72
Tensile strength (GPa)	3.02
Torsional modulus (GPa)	18.25

A set of five experiments was conducted on several carbon fibers as per the procedure given in Section 2.3. A set of five experiments was carried out according to this procedure. The calculation of the compressive strength from the experimental data by the four models used is given in the following paragraphs.

4.1. Compressive strength in Model 1 and Model 2

As discussed in Section 3.1 the range of recoil stresses and corresponding failure data for upper and lower end of the specimen are recorded. Such a sample data for the first experiment is given in Table 2. From this table the range of

Table 2. Failure data for experiment 1 (NF – not failed, F – failed).

Stress (MPa)	Top end	Bottom end
700.33	NF	NF
727.27	NF	NF
754.20	NF	NF
781.14	NF	F
808.07	F	NF
835.01	NF	NF
861.94	NF	F
888.88	F	F
915.82	F	NF
942.75	F	F
969.69	F	F
996.62	NF	F
1023.56	F	F
1050.49	F	F
1077.43	F	F

recoil stresses for which there is 100% survial to 0% survival of the specimen is identified. This data is reported in Table 3. The compressive strength for this specimen according to this model is 861.94 MPa. Similar procedure is carried out for remaining four experiments. The corresponding recoil stress ranges and compressive strengths are reported in Table 3.

Table 3. Compressive strength in Model 1.

Experiment number	Description	Mid-recoil stress (MPa)
1	100% survival	835
	100% failure	889
	compressive strength	862
2	100% survival	835
	100% failure	889
	compressive strength	862
3	100% survival	862
	100% failure	943
	compressive strength	902
4	100% survival	808
	100% failure	889
	compressive strength	848
5	100% survival	862
	100% failure	889
	compressive strength	875

For Model 2, the percentage survival and failure against the stress levels in ascending order of stress levels is arranged for each experimental data. The sample data for the first experiment is given in Table 4. This data is fitted with a linear fit (using least squares method with 95% confidence in MATLAB) and the stress corresponding to 50% survival is taken as the compressive strength.

Table 4. Percentage survival of the fibre segments for experiment 1.

Stress level (MPa)	Mid-recoil stress (MPa)	% Survival
700–750	725	100
750–800	775	75
800–850	825	75
850–900	875	25
900–950	925	25
950–1000	975	0
1000–1050	1025	25
1050–1100	1075	0

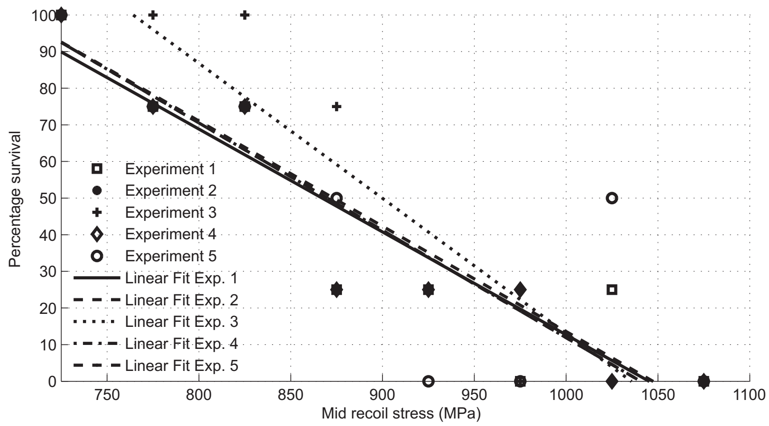

Fig. 3. Percentage survival of the fibre segments vs mid-recoil stress.

The plots for all the sets of experiments have been given in Fig. 3. The strengths, thus evaluated for all the experiments are 869, 869, 900, 870 and 879 MPa, respectively.

4.2. Probabilistic models for compressive strength

Two stress distribution models were fitted for the entire recoil test data (of all five experiments) to determine the compressive strength of carbon fiber using probabilistic approach. The probability of failure or fraction of fibers failed under recoil compression at each stress level from all the fibres tested is calculated. Using this data the compressive strength is calculated according to the procedure given in Section 3.3.

The value of $F(\sigma)$ for both the models, is obtained from the recoil test data, by calculating the fraction of fibers failed within a recoil stress range. The fibers were tested at eight different stress levels ranging from 700 to 1100 MPa, in steps of 50 MPa. The calculated $F(\sigma)$ is plotted against the midpoint of corresponding stress level and the both models are fitted for this plot to obtain the values of the unknowns. Curve fitting tool in MATLAB was used to fit the models to the experimental data. Using the values of the two parameters, σ_0 and m in Eq. (3.1), the mean recoil compressive strength of the fiber was determined for the Weibull model. In case of the logistic model, the values of a and b are used in Eq. (3.2), to determine the value of σ at 0.5 probability of failure, which gives the compressive strength of the fiber.

4.2.1. Weibull model. The failure data obtained from all five experiments are plotted against the recoil stress levels using a Weibull two-parameter fit governed by Eq. (3.1). From the fit, the values of scale and shape parameter are determined as

$$\sigma_0 = 701.1 \qquad \text{and} \qquad m = 12.61. \tag{4.1}$$

Further, the experimental points and fitted model are shown in Fig. 4. The mean of this distribution gives the compressive strength of the fibers as

$$\bar{\sigma} = \frac{\sigma_0}{L^{(1/m)}}\Gamma\left(1+\frac{1}{m}\right) = \frac{701.1}{0.045^{(1/12.61)}}\Gamma\left(1+\frac{1}{12.61}\right) = 860 \text{ MPa}. \tag{4.2}$$

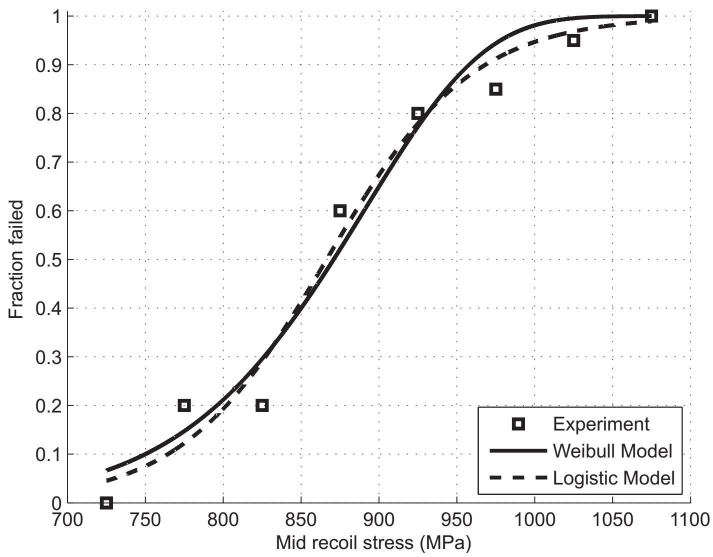

FIG. 4. Probability of fibre segment failure vs mid-recoil stress with Weibull and logistic models.

4.2.2. Logistic model. The failure data obtained from all the 5 experiments are plotted against the recoil stress levels using a logistic fit. The fitting equation for this method is given by Eq. (3.2). From the fit, the values of the intercept and regression coefficient obtained are given as

$$a = -18.68 \qquad \text{and} \qquad b = 0.02156. \tag{4.3}$$

The experimental points and the fitted model are shown in Fig. 4. The compressive strength of the fibres is calculated as the recoil stress at 50% failure. Thus, solving Eq. (3.2) with $F(\sigma) = 0.5$ we get the following equation for compressive strength:

$$e^{[-18.68+0.02156(\sigma)]} = 1. \tag{4.4}$$

Thus, the solution of Eq. (4.4) gives the compressive strength $\sigma = 866$ MPa.

The Weibull and logistic models are compared to each other along with the experimental results obtained from the tensile recoil tests. This comparison is shown in Fig. 4. We can see that both models agree with the experimental data very well. Further, there is not much difference in these two curves. Also, the compressive strengths obtained from these two fits are very close.

The assumption of independent failure processes at the two tab ends as in Section 3.3 is justified by the experimental results. Figure 5a shows the failure at only one end and Fig. 5b shows the failure at both ends with increasing

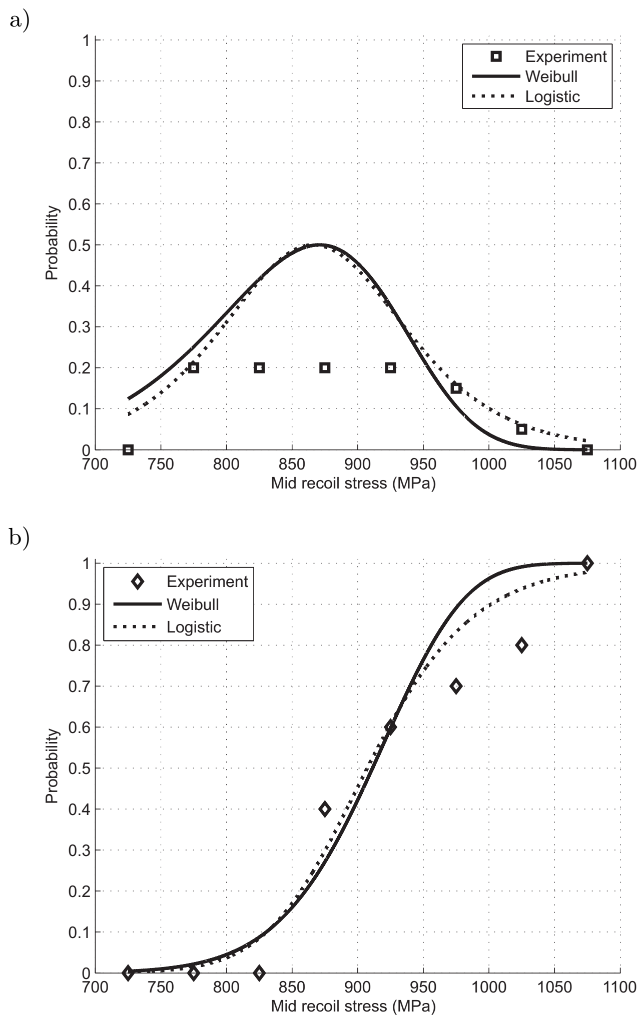

Fig. 5. Probability of (a) one end/segment failure, (b) both end/segment failure vs mid-recoil stress.

recoil stress. As expected, the probability that only one end fails increases with increasing recoil stress, reaches a maximum and then gradually decreases to zero. Similarly, the probability that both ends fail increases from zero to one monotonically. Further, these figures show the prediction of probabilities $g_1\{\sigma\}$ and $g_2\{\sigma\}$ by Weibull and logistic models used. From Fig. 5b it can be seen that the experimental and computed distributions using Weibull and logistic models agree well. However, for the failures at one end alone, as shown in Fig. 5a, there is a deviation of predicted values from the measured one. The Weibull and logistic models used assume that the axial compressive failure is due to pure tensile recoil stress. However, at the lower end due to gravity the bending effect can be significant. This leads to failure which is not due to purely compressive stress. Further, it adds the complexity of accounting for the failure through the models used (for example, one can see [26]).

4.2.3. Summary of all models. The compressive strength obtained by different approaches adopted is tabulated in Table 5. Since the failure is artificially introduced in recoil test, it is mandatory to evaluate the results obtained using various approaches. It can be observed that the results obtained by these approaches are almost consistent.

Table 5. Summary of compressive strengths (in MPa) measured by all models.

<table>
<tr><th>Experiment number</th><th>Model 1</th><th>Model 2</th><th>Weibull model</th><th>Logistic model</th></tr>
<tr><td>1</td><td>862</td><td>869</td><td rowspan="6">861</td><td rowspan="6">866</td></tr>
<tr><td>2</td><td>862</td><td>869</td></tr>
<tr><td>3</td><td>902</td><td>900</td></tr>
<tr><td>4</td><td>848</td><td>870</td></tr>
<tr><td>5</td><td>875</td><td>879</td></tr>
<tr><td>Mean</td><td>870</td><td>877</td></tr>
<tr><td>Mean of all models</td><td colspan="4">869</td></tr>
</table>

The compressive strength of fibers determined using recoil test is significantly lower than that obtained using other methods [26, 30, 39]. This may be due to several reasons and uncertainties that are not accounted for while performing this test. The first reason can be that the failure is artificially introduced. The method of initiation plays a vital role in this test. Non-uniform initiation of recoil stress will result in erroneous results. And the second reason can be that the fiber is assumed to fail under uniform compressive stress. But as the diameter of fiber is very small compared with the gauge length, there are chances of failure due to buckling, which has not been considered in this method. This fact can be substantiated with the experimental results as shown in Fig. 6. The gauge

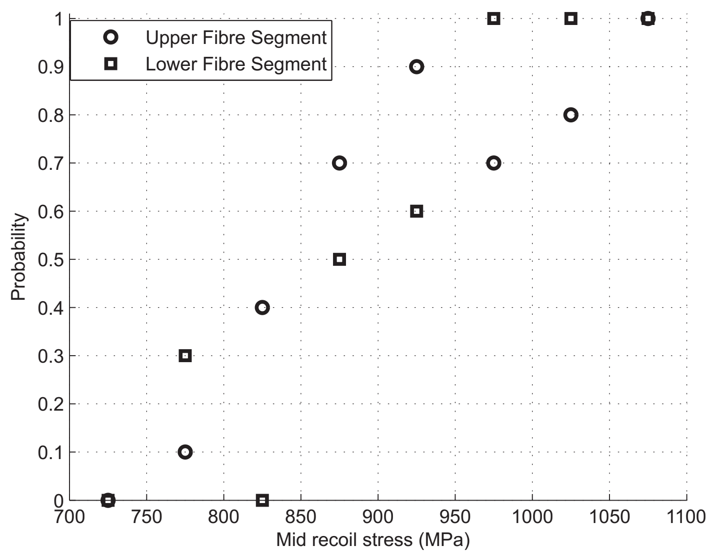

Fig. 6. Probability of upper and lower segment failure vs mid-recoil stress.

length of the specimens tested for the current study is much larger compared to its diameter. For such gauge lengths the upper end is prone to failure by recoils stress whereas the lower end is more prone to failure occurring by bending action due to gravity as observed during the tests (also see [26]).

The result for compressive strength of carbon fiber obtained from this test agrees with the values reported in literature obtained using the same technique [3, 25, 28]. Further, it can be seen from Table 1 that the compressive strength of the fibre is much less than its axial tensile strength.

Remark. The exact specifications like pre-cursor material, elemental constitution, manufacturer, etc. of the carbon fibres tested are not known to the authors.

5. Conclusion

In the present study an attempt has been made to measure the axial compressive strength of single carbon fibres by the method of tensile recoil test. Five different sets of experiments were carried out. The compressive strength was evaluated from the experimental data by four different models. The two of the models are statistics-based models and the other two methods – Weibull and logistic are probability-based models. Following are the conclusions that can be drawn from this study.

1. The compressive strength evaluated by all the models are in good agreement with each other.

2. Any of Weibull or logistic models can be used to fit the recoil stress distribution, as both models show very less variation.
3. The Weibull and logistic models predicted the probability of failure either at one end or both ends very well with the experimental data.
4. The probability of the upper and lower end failure increases with increase in applied recoil stress.
5. The compressive strength of carbon fibres measured by tensile recoil method is 869 MPa (average of all models studied) and agrees well with the results reported in literature obtained using the same method.
6. The compressive strength of the fibres tested is much less than its axial tensile strength.

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Received September 6, 2012; revised version November 26, 2012.