

TENSILE PROPERTIES OF SINGLE CARBON FIBRES TESTED WITH AUTOMATED EQUIPMENT

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ABSTRACT

Extensive data on strength variability has been acquired for four carbon fibres (T700, 34-700, T300 and HS40) using automated single fibre testing equipment. Weibull distributions are identified based on samplings of 100 to 200 tests. According to the data sheet, the T700 and 34-700 carbon fibres have similar tensile strength, but measurements show a different Weibull modulus for these fibres. All fibres types stiffen with the applied strain but there is no correlation between the initial stiffness and the stiffness variation coefficient. Limitations in measuring properties of carbon fibres with a diameter of 5 μm (HS40) are identified.

1 INTRODUCTION

Modelling tools for composite materials require input properties to make reliable predictions. The accuracy of the predictions depends greatly on the input data used. Using the properties of the building blocks of the materials as input is better than using the properties of a sub-system that already depends on the interactions of the building blocks. As the fibres and matrix are the basic constituents of any fibre reinforced composite, acquiring their properties is fundamental to predict the mechanical behaviour of composite materials.

The failure of composite materials under tension usually coincides with the failure of the plies aligned with the loading direction. The failure of these plies depends ultimately on the accumulation of fibre breaks. Predicting the development of fibre breaks and the accurate determination of the failure strain requires knowing the strength and stiffness of the fibres. Fibres fail stochastically due to the presence of microscale defects. Fibre strength is therefore not represented by an average value but rather by a probability distribution, commonly a Weibull distribution [1].

Accurately representing the Weibull probability distribution for a fibre population requires gathering an extensive data set of individual fibre strengths. The number of individual fibre tests required depends on the Weibull modulus [2]. For carbon fibres, with a Weibull modulus typically between 5 and 8 [3,4], the number of the necessary tests was estimated as follows. Thomason reported that for a fibre with a Weibull modulus of 5.5, at least 80 fibres should be tested [5]. Swolfs et al. showed that to obtain less than 10% of variation in modelling predictions of composite strength, several hundreds of fibres should be tested to characterise the Weibull distribution [6]. While several authors used 20 to 50 fibres to represent it [4,7–11], both studies [5,6] prove that a higher number of individual fibre strengths need to be obtained to accurately represent a Weibull distribution.

The most common method to characterize the mechanical behaviour of fibres is the single fibre test [12]. Using this method, the fibre stiffness and strength can be directly obtained. Other methods as the fragmentation test or dry bundle test have the great advantage of consuming less time to obtain the same number of strength values but require data reduction. The drawback on the time consumption of single fibre tests has been now diminished with the creation of automated testing equipment by Dia-Stron Ltd, for example.

2 MATERIALS AND METHODS

2.1 Materials

Using the automated testing equipment, four different types of carbon fibres were tested. The properties of each fibre type according to the data sheets and the number of fibres tested are shown in Table 1.

Fibre type	Number of tests	Diameter [μm]	Stiffness [GPa]	Tensile strength [GPa]
T700 (Toray)	217	7	230	4.90
34-700 (Mitsubishi)	170	7	234	4.83
T300 (Toray)	105	7	230	3.53
HS40 (Mitsubishi)	98	5	425	4.61

Table.1 - Fibre types tested, data sheet properties and number of fibres tested.

The T700 and 34-700 fibres have similar stiffness and tensile strength but are produced by different manufacturers. The goal is to compare the properties of these two fibres and determine differences in their Weibull distributions that may lead to different composite behaviour. The T300 fibre has the same stiffness as the T700 but lower tensile strength. It's expected that the T300 fibre has a lower Weibull scale parameter but it's not straightforward to predict the variation in Weibull modulus. The HS40 fibre is almost twice as stiff as the other fibres but has lower strength than that of the T700 and 34-700 fibres. The Weibull scale parameter is therefore also expected to be lower for HS40 than for the T700 and 34-700 fibres.

A unidirectional composite panel made of HS40 fibres and ThinPregTM 736LT resin was manufactured to determine the fibre stiffness for this fibre type. The panel was manufactured by laying up prepregs and cured in an autoclave according to the manufacturers recommended cure cycle [13]. The 10 specimens produced were 0.5 mm thick, 15 mm wide and 250 mm long.

2.1 Methods

2.2.1 Single fibre testing

The single fibre tests were performed with the automated testing equipment developed by Dia-Stron Ltd. Each fibre is mounted onto two omnidirectional plastic tabs, where each tab holds one of the fibre ends. The tabs are placed in a cassette with slots for multiple fibres. The cassette helps in obtaining an accurate gauge length by providing the slots for the plastic tabs with a pre-defined distance between them (12 mm in this work). Each tab has a v-shaped slit that helps aligning the fibre. A droplet of an UV curing adhesive is then placed over each tab to fix the fibre and tab. The adhesive is cured by illuminating it with a UV lantern for 15 seconds. The entire fibre mounting procedure takes 1-2 minutes per fibre.

When all the slots in the cassette have been filled, the cassette is mounted on the testing equipment. From this moment on, the procedure is automated. Each fibre is automatically picked up from the cassette and placed on the tensile testing machine. The equipment is a combination of a laser diffraction system for the diameter measurement and a tensile tester (see Fig. 1). The diameter of each fibre is measured at one point in the fibre's axial direction for the several angles in the fibre radial direction. The fibre diameter used is the average of all measurements. The tensile tests were carried out with a cross-head displacement rate of 0.6 mm/min.

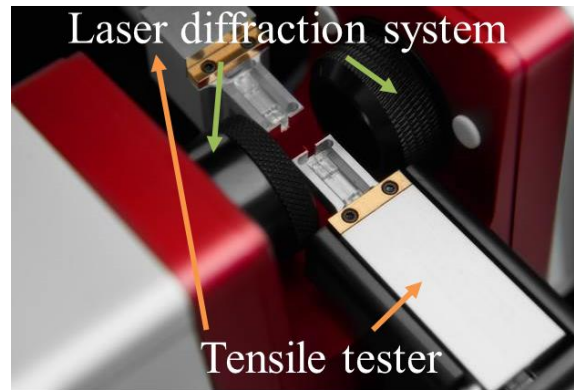


Figure 1 – Single fibre testing equipment consisting of a laser diffraction measurement system and a tensile tester.

In order to remove the machine compliance from the calculations, 15-20 fibres were tested with a gauge length of 4 and 20 mm. The machine compliance removal was done using the method described by ASTM C1557-14. By removing the system's compliance, the true stress-strain behaviour can be determined and hence the stiffness of the fibres calculated.

The fibre strength for each fibre was determined by extracting the maximum stress carried by the fibre prior to its failure. A two-parameter Weibull distribution was then fitted to the fibre strength values for each fibre type. The fitting was performed with Matlab 2018a function *wblfit*, which returns the maximum likelihood estimators for the Weibull modulus, m , and scale parameter, σ_0 . The same function also returns the 95% confidence intervals on the Weibull parameters.

2.2.2 Tensile testing composites

Tensile tests were performed on the UD composite specimens made of HS40 fibres and ThinPreg™ 736LT resin. The tensile testing machine used was a Zwick Roell Z100 equipped with a 100 kN load cell. The cross head displacement rate used was 1 mm/min. The strain was measured for the ten specimens using an optical extensometer incorporated in the tensile testing machine.

2.2.2 Matrix Burn-off tests

Matrix burn-off tests were performed according to ASTM D2584-02. The remains of each composite specimen were analysed separately, so that the fibre volume fraction for each specimen could be evaluated. The final goal was to back-calculate the fibre stiffness from the tensile stress-strain response of the composite specimens.

3 RESULTS AND DISCUSSION

3.1 Fibre diameter

The fibre diameter was measured for all the fibres tested using the laser diffraction system. In order to verify the accuracy of the method, a statistical study was performed on the fibre diameters for each fibre type. Figure 2 shows a histogram of the fibre diameters for each fibre type. Each histogram contains a line that represents the expected fibre diameter according to the data sheet.

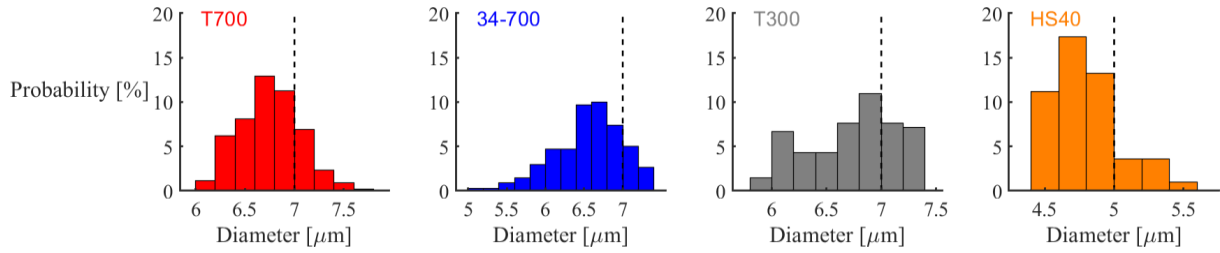


Figure 2 – Fibre diameter histogram and data sheet value represented by the dashed line for different fibre types.

All histograms show the peak for a lower value than the data sheet value. The difference between the two is lower than 0.5 μm for all cases, so it may be that the fibre manufacturers simply round up the value in the data sheet. During the experiments, the laser diffraction system failed to measure 32% of the HS40 fibres mounted. Those fibres were not taken into account for the results. Given that the histogram for this fibre does not show a low end tail, it is likely that the smaller fibre’s diameters were not measured due to a limitation in the measurement method. No correlation was found between the fibre diameter and fibre strength or stiffness for any fibre type, so the results gathered are believed to be not biased. Further work on the fibre diameter measurements should be done to confirm that the histogram is missing the left tail and this is not a real effect.

3.3 Fibre stiffness

The fibre stiffness was measured for all fibres as a function of applied strain. Figure 4 shows the stiffness-strain dependency for a representative fibre of each fibre type. The dashed line represents the linear fit for the stiffness-strain diagrams of all the fibres from the same fibre type. The stiffness-strain diagram for each fibre was measured in intervals of 0.1% with a 0.08% overlap. The first strain interval for each fibre was therefore from 0 to 0.1%, the second from 0.02% to 0.12% and so on. In the figure, ∇E is the stiffness-strain rate and E_0 is the initial stiffness, determined as the intersection of the fitted line with the Y axis.

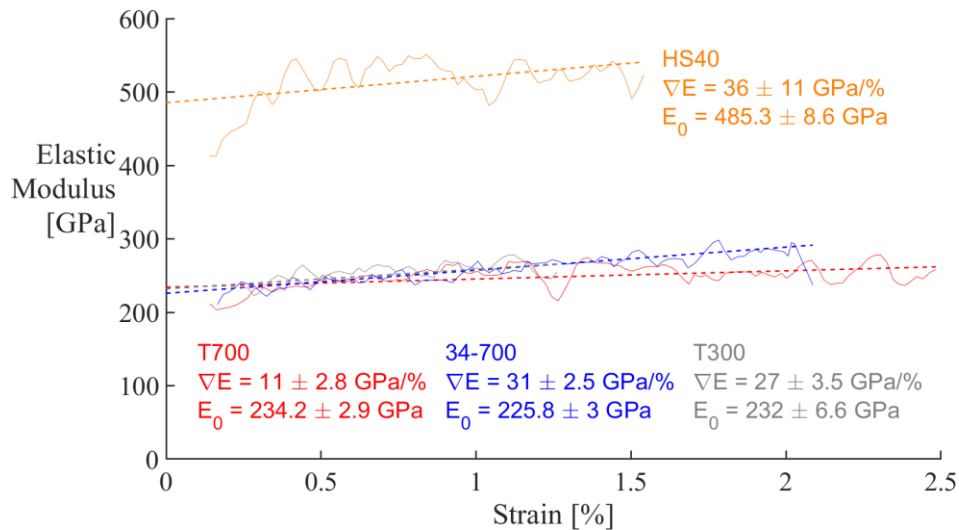


Figure 3 – Stiffness-strain diagram for a representative fibre of each fibre type. ∇E is the coefficient of variation of stiffness with strain and E_0 is the initial stiffness of the fibre

All fibre types show an increasing stiffness with the applied strain. This has been observed first by Curtis [14] and has been investigated further in literature. The data presented in this paper confirms the trends and provides further information on different fibre types. The T700, 34-700 and T300 fibre types show differences in the stiffness-strain rate although the initial stiffness is similar. The HS40

carbon fibre, despite having almost twice the initial stiffness, increases the stiffness at a similar rate as the 34-700 fibre. The stiffening of the carbon fibre does not show a correlation with the fibre's initial stiffness. The stiffening effect is therefore relevant and should be taken into account in modelling of composites.

The initial elastic modulus for the T700, 34-700 and T300 fibres were measured to have values in agreement with the data sheet values. The HS40 fibre appears to have a higher initial stiffness than the one mentioned in the data sheet. To verify this deviation, tensile tests were performed on ten UD composites specimens made with the HS40 fibres. Using the results from the matrix burn-off tests, the stress carried by the fibres was determined. Figure 4 shows the stress-strain diagrams calculated for 100% fibre volume fraction. The slope equivalent to the elastic modulus of the fibres reported in the manufacturer's data sheet is also represented with the dashed line. For the two diagrams with a load drop at approximately 0.6% strain, splitting occurred during the test.

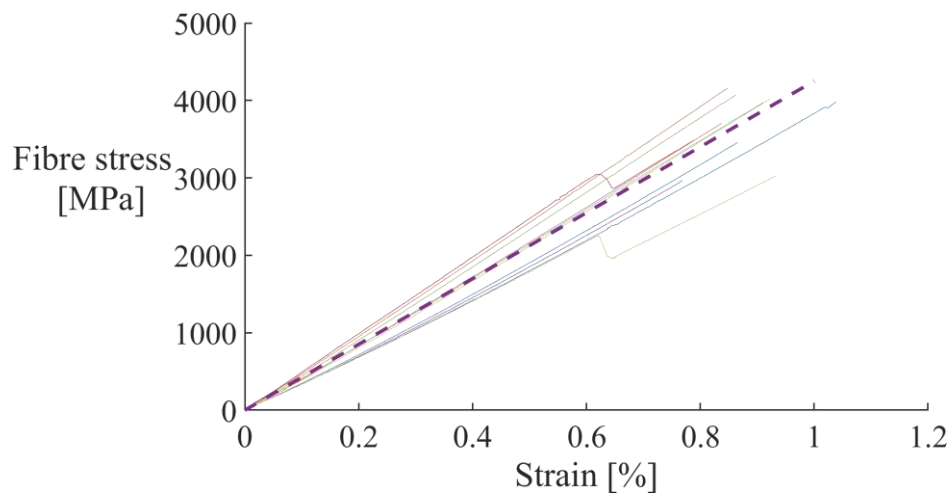


Figure 4 – Average stress strain response of the fibres in each tensile tested specimen (solid lines) and the manufacturer's datasheet stiffness (dashed line).

The stress-strain diagrams show that the back calculated elastic modulus of the fibres is significantly different between specimens. The manufacturer's data sheet value for fibre stiffness is well aligned with the average elastic modulus of the fibres determined over all the specimens tested. No information is provided by the manufacturer on the determination of the fibre properties but the most common method is the impregnated bundle test (ISO 10618:2004, for example). This methodology is equivalent to the tensile tests performed in the UD composite specimens, explaining the similarity in results between the back-calculated fibre stiffness and the manufacturer's data sheet value.

The high variability in the slope of the stress-strain diagrams shown in Figure 4 may indicate why the fibre stiffness determined by the single fibre tests differs from the average fibre stiffness determined by the tensile tests. The HS40 fibre is a high modulus fibre and data reported for other high modulus fibres shows that these fibres tend to be very anisotropic [15]. It is therefore possible that a small variation in fibre misalignment between specimens explains the variability in the fibre stiffness. The classical laminate theory was then used to determine the magnitude of the angle that a UD composite needs to be shifted from the loading direction to obtain the differences observed. An angle between 0° and 3° is sufficient to cause the decrease in fibre stiffness observed.

3.2 Fibre strength

Figure 5 shows the data points corresponding to the strength of each individual fibre and the line representing the Weibull fit for each fibre type. The Weibull parameters and corresponding 95% confidence intervals are also shown in the figure.

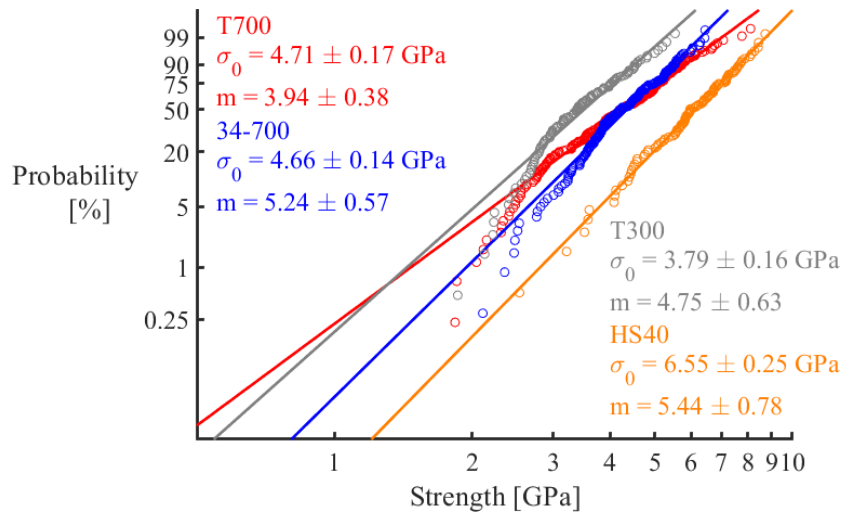


Figure 5 – Weibull fitting for the strength of each fibre type.

The T700 and 34-700 fibres have similar Weibull scale parameter, σ_0 , but the Weibull modulus, m , is significantly different. The lower Weibull modulus of the T700 fibres means that these fibres have more variable strength. When in a composite, more of the weaker fibres will tend to break at lower applied stresses but also more of the stronger fibres will prevent the failure of the composite. The T300 fibres have a lower σ_0 , which is expected as their stiffness is similar to the T700 fibre but they have a lower tensile strength. Although the HS40 fibres have twice the stiffness but lower tensile strength compared to the T700 and 34-700 fibres, their σ_0 is observed to be higher. This fibre has a higher Weibull modulus than the T700 and 34-700 fibre, which has an influence on the tensile strength of the composite, used to determine the fibre's tensile strength. The σ_0 therefore cannot be used to linearly scale the tensile strength of the composite.

4 CONCLUSION

The use of automated single fibre tensile testing was taken advantage of to acquire extensive single fibre properties samplings. Four different fibre types were analysed to verify the limitations of the automated testing machines and gain new insights into the properties of different carbon fibres.

The diameter measurements showed limitations in measuring the HS40 fibres, with a diameter according to the data sheet of 5 μm . The left tail of the diameter histogram is missing from the measurements and 32% of the fibres were not possible to measure, showing the limitations in the diameter measurement. The other fibre types showed that the measurements are consistent with the manufacturers data sheet, assuring the accuracy of the testing equipment for those fibre types.

All fibre types stiffen with the applied strain. The stiffness-strain rate differs between fibre types though. The HS40 fibre, despite having a higher initial stiffness, increases the stiffness at a similar rate as the 34-700 fibre type. The carbon fibres with similar initial stiffness also have different stiffness increase rates, showing that the stiffening does not correlate with the initial stiffness. The stiffness of the HS40 fibre was higher than expected. Tensile tests on UD composites made with this fibre type showed that the average back calculated stiffness matches with the manufacturers information, possibly due to fibre misalignment.

The T700 and 34-700 fibres, despite having similar stiffness and tensile strength, show different values of Weibull modulus. The Weibull modulus can have an influence in the strength of a composite material, making it important to know both Weibull parameters. The T300 fibre expectedly shows a lower Weibull scale parameter compared to the T700 and 34-700 fibre due to its lower tensile strength.

The HS40 fibre shows a higher Weibull scale parameter than the T700 or 34-700 but its Weibull modulus is higher, which has an influence on the composite's tensile strength.

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REFERENCES

- [1] W. Weibull, A statistical distribution function of wide applicability, *J. Appl. Mech.* 103 (1951) 293–297.
- [2] T.A. Parthasarathy, Extraction of Weibull parameters of fiber strength from means and standard deviations of failure loads and fiber diameters, *J. Am. Ceram. Soc.* 84 (2001) 588–592.
- [3] K. Naito, Y. Tanaka, J.M. Yang, Y. Kagawa, Tensile properties of ultrahigh strength PAN-based, ultrahigh modulus pitch-based and high ductility pitch-based carbon fibers, *Carbon N. Y.* 46 (2008) 189–195.
- [4] K. Naito, J.M. Yang, Y. Tanaka, Y. Kagawa, The effect of gauge length on tensile strength and Weibull modulus of polyacrylonitrile (PAN)- and pitch-based carbon fibers, *J. Mater. Sci.* 47 (2012) 632–642.
- [5] J.L. Thomason, On the application of Weibull analysis to experimentally determined single fibre strength distributions, *Compos. Sci. Technol.* 77 (2013) 74–80.
- [6] Y. Swolfs, I. Verpoest, L. Gorbatikh, Issues in strength models for unidirectional fibre-reinforced composites related to Weibull distributions, fibre packings and boundary effects, *Compos. Sci. Technol.* 114 (2015) 42–49.
- [7] Z. Chi, T.-W. Chou, G. Shen, Determination of single fibre strength distribution from fibre bundle testings, *J. Mater. Sci.* 19 (1984) 3319–3324.
- [8] K. Goda, H. Fukunaga, The evaluation of the strength distribution of silicon carbide and alumina fibres by a multi-modal Weibull distribution, *J. Mater. Sci.* 21 (1986) 4475–4480.
- [9] P. Zinck, E. Mader, J.F. Gerard, Role of silane coupling agent and polymeric film former for tailoring glass fiber sizings, *J. Mater. Sci.* 36 (2001) 5245–5252.
- [10] S. Pimenta, S.T. Pinho, The influence of micromechanical properties and reinforcement architecture on the mechanical response of recycled composites, *Compos. Part A Appl. Sci. Manuf.* 56 (2014) 213–225.
- [11] C.-T. Li, N.R. Langley, Improvement in fiber testing of high-modulus single-filament materials, *J. Am. Ceram. Soc.* 68 (1985) C-202-C-204.
- [12] Y. Swolfs, I. Verpoest, L. Gorbatikh, A review of input data and modelling assumptions in longitudinal strength models for unidirectional fibre-reinforced composites, *Compos. Struct.* 150 (2016) 153–172.
- [13] North Thin Ply Technology, ThinPreg Data sheet, www.thinplytechnology.com/assets/mesimages/NTPT-DS-ThinPreg-736LT-v2.pdf. (2016) 1–8.
- [14] G.J. Curtis, J.M. Milne, W.N. Reynolds, Non-hookean behaviour of strong carbon fibres, *Nature.* 220 (1968) 1024.
- [15] N. Baral, H. Guezenoc, P. Davies, C. Baley, High modulus carbon fibre composites: Correlation between transverse tensile and mode I interlaminar fracture properties, *Mater. Lett.* 62 (2008) 1096–1099.