TOWARDS ACCURATE AND EFFICIENT SINGLE FIBRE CHARACTERIZATION TO BETTER ASSESS FAILURE STRENGTH DISTRIBUTION

Faisal Islam[1], Sébastien Joannes[1], Steve Bucknell[2], Yann Leray[2], Anthony Bunsell[1], Lucien Laiarinandrasana[1]

[1]MINES ParisTech, PSL - Research University, MAT - Centre des Matériaux, CNRS UMR 7633 BP 87, 91003 Evry Cedex, France
Email: sebastien.joannes@mines-paristech.fr, Web Page: http://www.mat.mines-paristech.fr/
[2]Dia-Stron Ltd., 9 Focus Way, Andover SP10 5NY, UK
Email: yann.leray@diastron.com, Web Page: http://www.diastron.com/

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Abstract
Failure in unidirectional organic composites, loaded longitudinally, usually originates within fibres, which makes their failure kinetics a key parameter for failure modelling. Correctly capturing fibre strength statistics and failure kinetics is crucial to the success of composite stochastic strength models. The characterization of fibres is challenging, as the fibre diameter can be just a few microns. Nevertheless mechanical testing of single fibres is the only unambiguous means of characterising fibres or exploring their morphologies. Despite great progress in characterisation techniques, many obstacles remain to obtain accurate fibre strengths. Most studies have acknowledged the difficulty in obtaining fibre strength data which can accurately represent the entire fibre population in a given application. In this study an automated single fibre tensile testing machine has been used which overcomes most obstacles usually faced during fibre strength determination. Advanced statistical data processing techniques have been implemented to determine the reliability of the obtained results. Such analysis permits the calculation of confidence/accuracy associated with the generated fibre strength data. This may be used to extract more useful information from the limited resources available. The inadequacy of the popular statistical distributions for representing fibre strength has been highlighted. It is shown that inclusion of a truncation parameter in the Weibull distribution could help in estimating the strength behaviour of a given fibre population.

1. Introduction

Fibres are widely used in composite materials to make light-weight and high-strength products. Fibre reinforcement of polymeric matrices is found to bring about significant advancements in mechanical behaviour of polymers with added advantages of light weight, high strength to weight ratio, excellent weathering stabilities and enhanced dimensional stabilities. Owing to such wide use, it becomes important to determine the characteristics of individual constituents (fibres and matrix) and their interactions to fully understand their effect on the end-products. This advanced knowledge may empower us to engineer new and improved products.

For failure modelling and to simulate the effective properties of composite products, detailed information about fibre tensile strength distribution is required. The characterization of fibres is a difficult process mainly due to their small diameters. Despite great progress in characterisation techniques, many obstacles remain to obtain accurate fibre strengths. Many studies have acknowledged these difficulties.

The most popular methods to determine fibre strengths are the single fibre tests, fibre bundle tests and fragmentation tests. The single fibre test is the most straightforward but a laborious and time consuming process for testing fibres. With the fibre bundle method a bundle of fibres is tested by applying a load and observing the behaviour of the bundle as a whole. The single fibre fragmentation test embeds a fibre inside matrix and the composite is tested to determine the parameters for fibre strength distribution. Each study has its own limitations [1]. Contemplating the different limitations that each process contains, the classical single fibre testing process still appears to be one of the most reliable and unambiguous means of characterising fibres or exploring their morphologies. The different individual steps in single fibre testing, however, leave scope for improvement. If the existing problems could be solved, the efficiency of the processes could be significantly improved. One important factor that influences determination of fibre strengths is the fibre preselection effect due to which many fibres fail before testing and the obtained strength data set is not complete. Any misalignment in fibre positioning during testing develops unwanted bending stresses which may lead to the failure of fibre at clamping locations and provide incorrect strength data. Any error in measuring the cross sectional area will affect the accuracy of the calculated fibre strength which is vital in determining the true fibre strength [2].

Fibre strength information can be used for different purposes. One such application is to determine the reliability of a composite product. This is usually done by simulating the strength of the product using the known strengths of its constituents. Certain high-stake products such as composite pressure vessels require a certainty of zero failure. This requires strength information of constituents at a very high accuracy. Advanced statistical analysis should be conducted to find an accurate representative function of fibre strengths that could be used to obtain critical strength information especially at the limiting regions of the distribution.

2. Fibre strengths and Weibull distribution

The most common fibres used in composite materials are made of carbon or glass which are both brittle in nature. There is a significant scatter in properties of individual fibres due to the introduction of flaws during processing and handling. The strength of a single fibre cannot be captured in one single average value [2]. Single fibres normally exhibit a distribution of strengths that could be represented by using a probabilistic approach. The Weibull distribution is considered to be a good representation of the weakest link behaviour of brittle single fibres. This is based on the assumption that fibre failure is controlled by the random distribution of a single type of defect. The classical Weibull distribution function for fibre strengths is given by eq. 1.

$$P(\sigma_f) = 1 - \exp\left(-\left(\frac{L}{L_0}\right)\left(\frac{\sigma_f - \sigma_u}{\sigma_0}\right)^m \right) \tag{1}$$

where $P(\sigma_f)$ is probability of fibre failure, L being the characteristic gauge length, L_0 the reference gauge length, σ_f the fibre strength, σ_u the threshold strength, σ_0 the scale parameter, m the shape parameter or Weibull modulus. It is also commonly known as the 3-parameter Weibull distribution. The parameters σ_0 and m characterise the density and variability of flaws in the material. σ_u is the threshold stress below which the failure probability is zero. This means that the weakest fibre has a strength value of σ_u. The value for σ_u is usually set to be zero and the resultant is known as a 2-parameter distribution (Eq. 2). It is important to note that for most experimentally generated data sets for single fibres, a lower limit on fibre strengths is observed. However, this is probably due to experimental limitations [3]. It has become a very common practice to use the 2-parameter distribution to fit single fibre strengths and for deriving conclusions on the physical behaviour of fibres.

$$P(\sigma_f) = 1 - \exp\left(-\left(\frac{L}{L_0}\right)\left(\frac{\sigma_f}{\sigma_0}\right)^m\right) \tag{2}$$

Certain authors have speculated that multiple strength determining flaw populations may exist inside fibres and have recommended the use of a multimodal Weibull distributions to represent the strength of a fibre population [4]. Here $\sigma_{0,1}$ and $\sigma_{0,2}$ are the scale parameters, and m_1 and m_2 are the Weibull moduli for the first and second flaw population, respectively.

$$P(\sigma_f) = 1 - \exp\left(-\left(\frac{L}{L_0}\right)\left(\frac{\sigma_f}{\sigma_{0,1}}\right)^{m_1} - \left(\frac{L}{L_0}\right)\left(\frac{\sigma_f}{\sigma_{0,2}}\right)^{m_2}\right) \tag{3}$$

A power law accelerated Weibull distribution has also been proposed by some authors. It tries to represent the gauge length dependence of fibre strengths by including an additional parameter α in the distribution [5]. However, this is mainly based on curve fitting and does not have a strong physical explanation.

$$P(\sigma_f) = 1 - \exp\left(-\left(\frac{L}{L_0}\right)^{\alpha}\left(\frac{\sigma_f}{\sigma_0}\right)^m\right) \tag{4}$$

The most popular and effective distribution is still the classical two-parameter Weibull distribution. This could be used to fit experimentally observed fibre strengths. The validity and implications of using this distribution are discussed.

2.1. Single fibre strength

A sufficient number of single fibre breaking loads are required to obtain the parameters for the best fit of the statistical distribution that could represent the fibre strength variation. An improved and automated testing setup is required to increase the efficiency of the process and quality of the data generated. The automated setup developed at Dia-Stron Ltd. has been used to generate single fibre strength data using T700 12k carbon fibres. This automated system has several advantages over the conventional manual testing process. These include improved alignment, exact measurement of gauge length, reduced specimen preparation time, ability to test at shorter gauge lengths, reduction in the number of failed samples due to handling, automated and accurate measurement of the apparent diameter of each filament, and many more. Fibres are tested at different gauge lengths of 04mm, 20mm and 30mm. A Weibull cumulative density function and the corresponding logarithmic Weibull plot for a data set containing 30 fibre strength values obtained from testing fibres at a gauge length of 30mm are shown in figure 1.

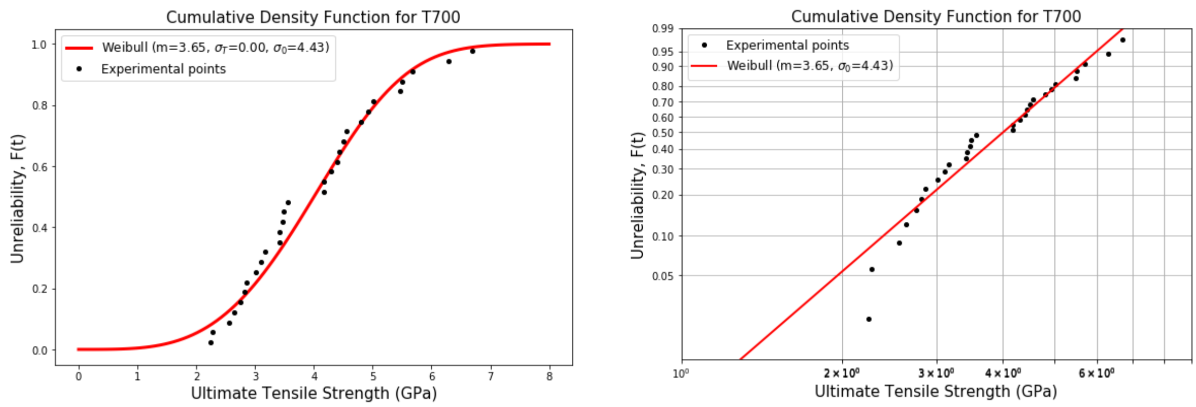

Figure 1. Cumulative density function and best fit Weibull plot for fibre strengths at 30mm

The tensile strength varies from 2 to 7 GPa with most fibres having a strength between 3-6 GPa for all gauge lengths. Slightly higher strength values were however observed in many specimens of 04 mm gauge length. This is in agreement with the weakest link theory according to which, fibres of short gauge length are comparatively stronger than fibres of longer gauge lengths [6]. The vertical axis in the logarithmic Weibull plot shows the 'unreliability' which represents the percentage of fibre population that is expected to fail. The horizontal axis represents corresponding strength and is known as B-life. The B-life is the age at which a certain percentage of the investigated fibre population is expected to fail. For example, the B50-life (corresponding to 50% unreliability, or median failure strength) for the 30mm fibre population can be read from the plot and is approximately 4.2 GPa. The Weibull distribution parameters for fibre strength distributions of different gauge lengths are given in Table 1. The shape parameter values for all gauge lengths are similar and falls between 3.49-3.65 GPa. The similar shape parameter values confirm the consistency of the test results. The scale parameters decrease with increasing gauge lengths. This is in compliance with the weakest link theory.

Table 1. Weibull parameters for fibre strengths at different gauge lengths

Gauge length (mm)	Shape parameter	Scale parameter (GPa)
04	3.65	5.84
20	3.49	5.16
30	3.63	4.61

2.2. Confidence Interval

Since the fibre strength behaviour for the entire bundle is determined by testing a comparatively small number of fibres, a confidence interval is calculated to obtain an indication of the confidence for the estimated B-life. Generally, confidence levels of 90% are used. A 90% confidence interval for a B-life has the following meaning: When the B-life would be estimated over and over again from samples similar to the original one, then the real, unknown B-life of the specimen population will, with a 90% frequency, be situated inside the confidence interval. There are different kinds of confidence intervals/bounds but Monte Carlo pivotal bounds are believed to be the best. The Weibull plot with Monte Carlo pivotal confidence bounds for a 90% confidence level calculated for fibre strengths at 30mm gauge length is shown in Fig. 2. The different simulated Weibull distributions are also shown.

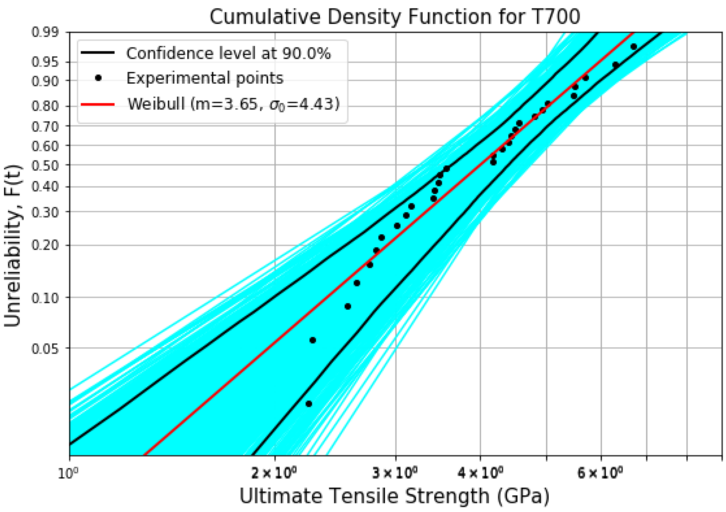

Figure 2. Weibull plot with Monte Carlo pivotal confidence bounds for a 90% confidence level for fibre strengths at 30mm gauge length.

The confidence intervals for any required B-life can now be read directly from the graph. For example, the confidence interval for B-50 life for fibres of gauge length 30mm is 3.73-4.49 GPa. This information can have very wide practical and commercial applications. The area contained between the two black curves represents an area of 90% confidence. If the analysis is repeated using another set of fibre strengths for the same type of fibres, it is seen that the new fibre strength data points (orange) also fall within these limits as shown in figure 3. However, even if both sets of fibre strength data points lie within the expected limits, in extreme cases there can still be large variations in their Weibull parameters, as shown in figure 4. This is mainly due to the very large size of the associated confidence bound. In order to reduce the confidence interval size, more fibres have to be tested. Using the automated testing method, it has been possible to obtain a large set of fibre strength data. Figure 5 shows a Weibull plot with about 150 data points. The confidence bound is seen to reduce and the confidence interval for B50 life narrows down to 4.04-4.39 GPa. However, using a large data set has also revealed a deviation of data points from the modelled Weibull plot. This deviation was not very prominent when a smaller data set of 30 fibre strengths was used. The main reasons for this observed deviation are the fibre preselection effect and use of an inappropriate distribution to represent fibre strengths.

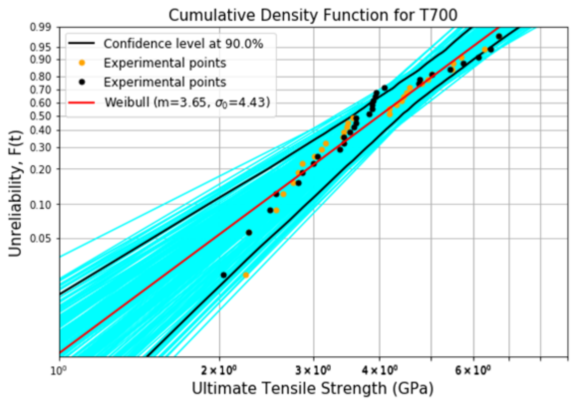

Figure 3. New set of experimental data points plotted on previous Weibull plot for comparison

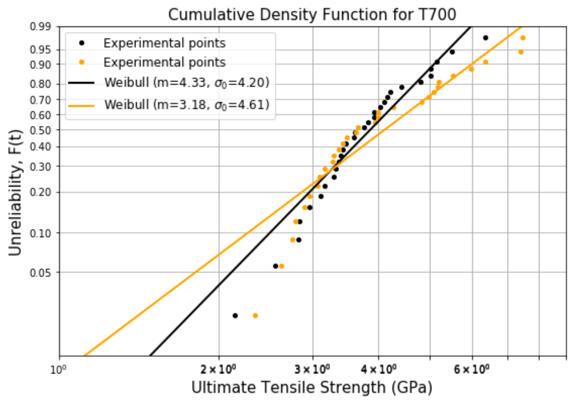

Figure 4. Two different sets of fibre strength data points obtained from fibres at same gauge length.

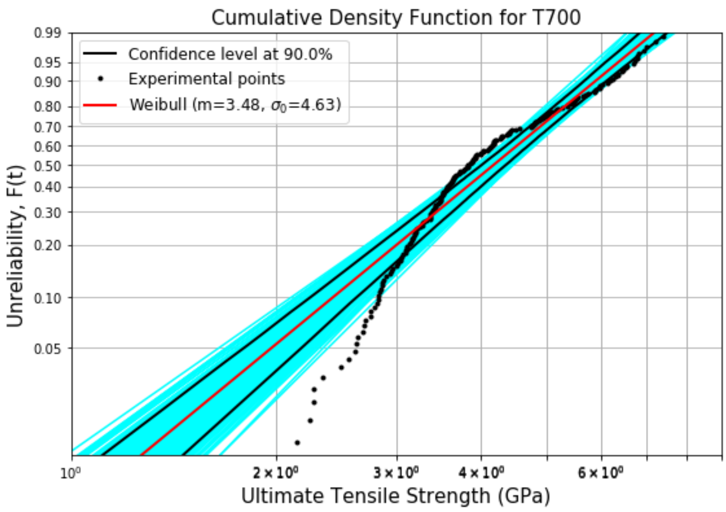

Figure 5. Weibull plot with 150 fibres at a gauge length of 30mm.

Fibre preselection: Since the diameters of carbon fibres are very small, they have very low breaking forces. Many weak fibres therefore break during the process of fibre extraction and preparation. The elimination of weak fibre strengths from the data set causes a deviation in linearity on the Weibull plot. To enhance the process of fibre extraction, it is recommended that sizing around fibre is removed using a suitable solvent. This dissolves the adhesive around fibres and facilitates easy fibre separation and extraction thereby allowing the extraction of weak fibres from the bundle with less breakage. To test this, a set of 50 T700 fibres of 30mm gauge length was used to measure tensile strength with fibres that were first treated with ethanol to remove or weaken the sizing for easy extraction. A considerable number of fibres were found to have strength values of less than 2GPa in the case when the sizing was removed prior to testing. These fibres may have probably failed prematurely if the size had not been removed prior to the extraction process. It is therefore better practice to treat the fibre bundle with a suitable solvent before proceeding with fibre strength tests. Doing so improves the representation of the fibre strength variation and slightly reduces this threshold value. However a significant portion of fibre strengths may still be inaccessible.

Inappropriate distribution: The classical 2-parameter Weibull distribution is not able to capture the non-linear behaviour of fibre strengths accurately as is evident from figure 5. A distribution that could better represent the fibre strengths is required. It may be tempting to use the 3-parameter Weibull distribution (Eq. 1) with the location parameter as it would be able to present a distribution that would fit the experimental data points very well. However, this distribution would assume that the minimum strength value in the data set represents the weakest fibre in the population which is obviously an incorrect assumption [3]. There is a need to analyse the data set statistically and find an appropriate distribution that could represent this left truncated data set. It may be useful to derive inspiration from Berger and Jeulin's work [7] who suggested the use of a truncation parameter to represent single fibre strength data set. This is explained in the following section.

2.3. Truncated Weibull distribution

Fibres need to have a minimum strength such that they can sustain the handling and unwanted external forces during specimen preparation. Weaker fibres usually break during the process and are not able to survive until final testing. Therefore, in most experimentally generated fibre strength data set obtained from single fibre tests, there is a small range of fibre strengths that is not represented. In statistical terms, such a data set is known as a truncated data set. Since it is always the weaker portion of fibre strength data that is not available, the fibre strength data set is left truncated. The limit below which data is not available is known as the point of truncation or truncation limit and can be represented as **t**. It is important to note that this parameter is different from the location parameter σ_u that appears in a 3-parameter Weibull distribution. The location parameter for the present case is still zero, however we do not have access to the data below the truncation limit. Equation 5 represents the 2-parameter Weibull distribution after incorporating the truncation limit **t** [7].

$$P(\sigma_f) = 1 - \exp\left(- \left(\frac{L}{L_0}\right)\left(\frac{\sigma_f^m - t^m}{\sigma_0^m}\right)\right) \tag{5}$$

Fibre strength data for 30mm fibres is used to generate the truncated Weibull cumulative density function and truncated logarithmic Weibull plot, shown in fig 6. The corresponding 2 and 3-parameter classical Weibull plots are also shown for comparison. The truncation limit is fixed at 2GPa which is about the same as the smallest measured strength value. It can be seen that the truncated Weibull distribution fits very well with the experimental data set. The maximum likelihood Weibull parameters for the different distributions are given in table 2.

It is important to note that the truncated Weibull distribution is obtained by the addition of a constant truncation limit to the classical 2-parameter distribution and so the parameters still hold the same physical meanings, unlike the 3-parameter distribution for which the parameters have a different physical

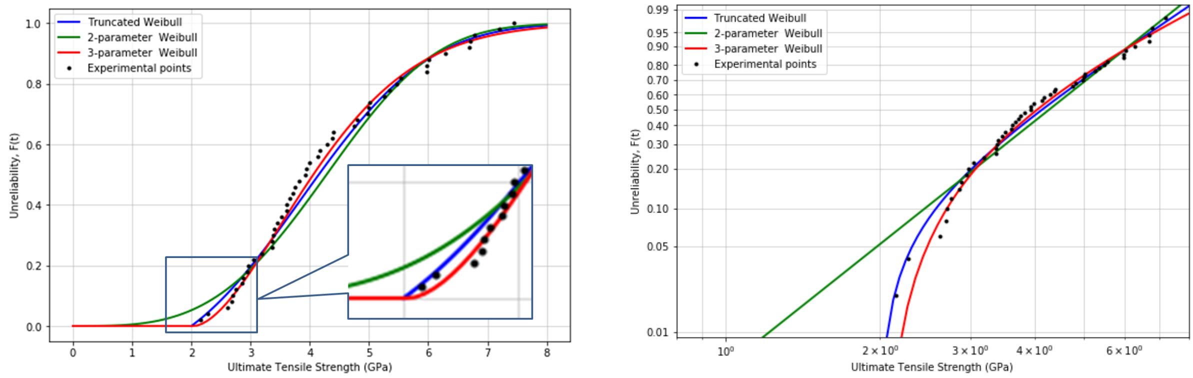

Figure 6. Comparison of cumulative density functions and Weibull plots for fibre strengths at 30mm

Table 2. Weibull parameters for different distributions

Distribution	Shape parameter	Location parameter (GPa)	Scale parameter (GPa)	Truncation limit (GPa)
Truncated Weibull	2.90	0	4.35	2
2-parameter Weibull	3.48	0	4.61	-
3-parameter Weibull	1.91	1.83	2.61	-

significance. Adding a truncation just provides additional information that the data set is incomplete. This does not alter the characteristics of the other parameters. Due to the preselection effect, the experimental data is spread over a slightly narrower range than what it should have. When the 2-parameter distribution is used for modelling this experimental data, the resulting distribution is slightly narrower and shifted to the right as compared to what it would have been if the entire fibre strength data set was available including the weaker strength points. This can be observed by comparing the shape and scale parameter values from Table 2. The larger shape parameter value for the 2-parameter distribution represents a narrower distribution as compared to the truncated case. Similarly, the larger scale parameter value shows that the 2-parameter distribution is more right shifted.

The truncated Weibull distribution can be used to obtain a more accurate representation of the fibre population. This is because it contemplates the incompleteness of the data set during analysis. If the truncation limit is disregarded, after calculation, the remaining shape and scale parameters would represent the Weibull distribution which would have been obtained if the entire fibre strength data set was accessible. This predicted distribution is shown in figure 7.

3. Conclusion

The single fibre testing method leaves a large scope for improvement. Different problems and limitations associated with the method have been identified and discussed. This allows necessary actions for process improvement to be taken. The automated single fibre testing setup developed at Dia-Stron Ltd. helps in overcoming many existing problems. It has also helped in highlighting some issues that were unknown before but are critical for accurate determination of the fibre strength. Implementation of new statistical techniques has allowed extraction of accurate and useful information from the fibre strength distribution. Fibre strength data generated from the automated setup has helped in confirming the reliability of these estimations. The present research has also identified the inadequacy of available distributions for rep-

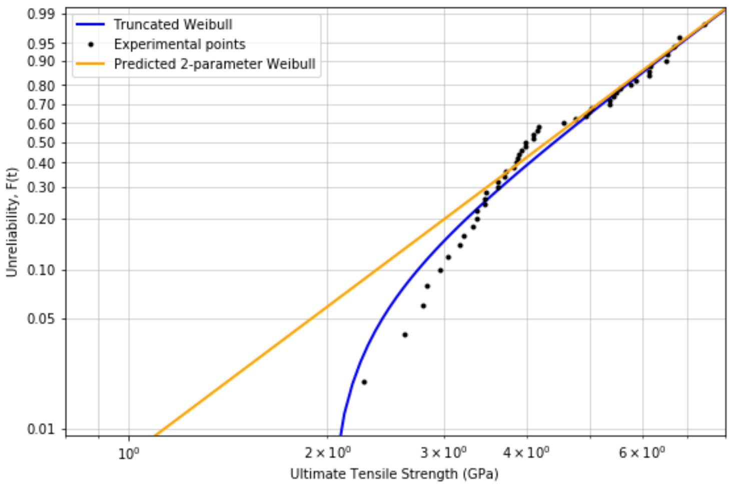

Figure 7. Predicted Weibull distribution from the truncated distribution

resenting a large fibre population. Inclusion of a new threshold parameter in the distribution has been shown to be a more accurate representation of the fibre population.

Acknowledgments

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