UNIAXIAL STRENGTH OF SUPPORTED AND
UNSUPPORTED CARBON FIBRES

BY

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He who finds what he seeks makes, in general, a good school exercise; intent on what he wants, he often neglects the signs, sometimes minimal, which indicate something else than the object of his attention. The real researcher must pay attention to signs which will reveal the existence of an unexpected phenomenon.

Des Atomes et des Hommes 11, Fayard, Paris
Louis Leprince-Ringuet, 1901-
SUMMARY

A comprehensive investigation of the strength distribution of unsupported single fibres (in air) and supported single fibres (embedded in epoxy-resin) in the context of the weak-link theory was conducted. Strength measurements of impregnated bundles were also carried out in order to compare the strength data with the unsupported and supported fibres. The entire experimental work was performed under similar experimental conditions, where a long length of a single fibre, or a bundle of fibres, was cut from the same spool of fibre (Celion fibre) into four specimens of 5, 12, 30 and 75mm gauge lengths respectively, thus ensuring a consistent base for the comparison. All the data produced were characterised using the Weibull model and other statistical tests, such as the non-parametric test, which can test the weak-link property without recourse to Weibull statistics.

Unsupported single fibres were found to deviate strongly from the Weibull model and the weak-link behaviour. This was due to the diameter variation found along the lengths of the tested specimens, which violated the implicit assumption in the Weibull model and the weak-link property that specimens should be similar in all respect except length. Another carbon fibre (XAS) was used to compare the Celion fibre data in relation to its behaviour to the Weibull model. The XAS fibre, however, was found to comply well to the Weibull model and the weak-link property, this was due to the mean diameter being uniform across the four gauge lengths.

The Celion fibre was shown to have an increasingly strong dependence of strength on the fibre diameter as the gauge length increases. This was due to the different effect that diameter produced on the four gauge lengths. The overall behaviour of the Celion fibre data was found to be complex due to the presence of two effects: firstly, the presence of two classes of failure flaws which may be represented by surface and volume flaws, and secondly, within the volume flaw
mode itself there was a strong diameter dependence.

The use of epoxy-resin to support the fibres provided a means to separate these two classes of flaws and to understand the complex behaviour of the unsupported fibres. There was an apparent diameter uniformity found in the supported fibres, and the weak-link property was restored by the use of the resin. The use of the resin has also increased the strength of the fibres. Failure of the fibres was found to be affected by length only, which explained the compliance of the data to the Weibull model. It was found that surface flaws were eliminated in all the gauge lengths allowing volume flaws to dominate the fibre’s failure.

Impregnated bundles were found to have similar mean diameters across the set of gauge lengths. The weak-link property was found to be present and the data were found to comply with the Weibull model. The strength of the impregnated bundles were found to be much stronger than even the supported single fibres. In fact their values for the four lengths were found to be relatively uniform suggesting that impregnated bundles must fail through the interaction of a group of fibres rather than an isolated first fibre failure.
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<td>CFRP</td>
<td>carbon-fibre reinforced plastic</td>
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<tr>
<td>ELS</td>
<td>equal load sharing</td>
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<tr>
<td>LLS</td>
<td>local load sharing</td>
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<td>MMW</td>
<td>multi-modal Weibull distribution</td>
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<td>PAN</td>
<td>polyacrylonitrile</td>
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<td>PAL</td>
<td>positively affected length</td>
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<tr>
<td>S.d.</td>
<td>standard deviation</td>
</tr>
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<td>S.E.M.</td>
<td>scanning electron microscope</td>
</tr>
<tr>
<td>W.I.S.E.</td>
<td>Watson image-shearing eyepiece</td>
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<tr>
<td>$P_f$</td>
<td>probability of failure</td>
</tr>
<tr>
<td>$P_s$</td>
<td>probability of survival</td>
</tr>
<tr>
<td>$\delta$</td>
<td>ineffective length of a fibre</td>
</tr>
<tr>
<td>$Q_i^*$</td>
<td>expected number of $i$ adjacent fibre breaks (Batdorf notation)</td>
</tr>
<tr>
<td>$K_r$</td>
<td>stress concentration factor</td>
</tr>
<tr>
<td>$D^*$</td>
<td>mid-value of diameter range</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>stress</td>
</tr>
<tr>
<td>$\phi$</td>
<td>fraction of stress recovered in a fibre</td>
</tr>
<tr>
<td>$L$</td>
<td>length</td>
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<tr>
<td>$\sigma_u$</td>
<td>lower threshold value in Weibull three-parameter distribution</td>
</tr>
<tr>
<td>$W$</td>
<td>Weibull modulus</td>
</tr>
<tr>
<td>$\sigma_0$</td>
<td>characteristic parameter in two or three-parameter distribution</td>
</tr>
<tr>
<td>$V$</td>
<td>volume</td>
</tr>
<tr>
<td>$j$</td>
<td>$j$th observation in series</td>
</tr>
<tr>
<td>$N$</td>
<td>number of data points in series</td>
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\( \Gamma \) gamma function
\( \bar{F}_L(\sigma) \) probability that a fibre of length \( L \) survives stress \( \sigma \)
\( \chi^2 \) Chi-square test
\( E_i \) expected frequency
\( O_i \) observed frequency
\( D \) diameter
\( E_f, E_m \) fibre, matrix moduli
\( G_m \) matrix shear modulus
\( x \) the measured distance from the central maximum to the \( n \)th order minima
\( s \) screen-to-fibre distance
\( \lambda \) laser wavelength
\( f \) focal length of lens
\( \alpha \) a constant representing the characteristic stress for unit length (1mm)
\( L_1 \) 75mm gauge length
\( L_2 \) 30mm gauge length
\( L_3 \) 12mm gauge length
\( L_4 \) 5mm gauge length
\( K \) length enhancing factor
\( E \) Young's modulus
\( i \) number of adjacent fibre failure
\( A \) cross-sectional area of a fibre
\( \chi \) XAS fibre.
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CHAPTER ONE

1.0 Introduction

Composite materials can be considered to be materials where one or more phases are dispersed within another phase, the matrix. They are often traced back to the biblical reference of straw-reinforced mud bricks. Medieval archers used bows made from laminated strips of wood, horn, tendons and silk to provide stiffness, and hence power. All these early simple composite systems were made of existing natural materials. Today, however, with the recent major advances in polymer chemistry, fibre manufacture and metallurgy, it is possible to use a wide variety of man-made materials to design advanced composite systems.

Modern engineering composites as understood today saw their beginning in the 1930’s with the commercial development of glass fibres and the production of unsaturated polyester resin in the 1940’s. Advanced composites stem from the development of synthetic fibres such as boron and carbon which began in the 1950’s; these fibres can possess a Young’s modulus two to four times that of steel, combined with much lower densities (1).

To utilise the high stiffness and strength that modern fibres exhibit, it is necessary to combine them with a matrix material that bonds well to the fibre surface which transfers stress efficiently to the fibres, enables the material to be formed into the desired shape. The wide range of fibres available (carbon, glass, boron, aramid and silicon carbide) and matrices (polymers, ceramics and metals) enable composites to be used in applications that are very diverse. Most composites in
use today are polymer based matrices with a fibre reinforcement, such as carbon-fibre reinforced plastic (CFRP). The use of these materials has an influence on the daily lives of most of us. The application of these materials generally falls into three broad categories: aerospace (aircraft, space and satellite structures), sports goods and industrial equipment. A review of some of the applications of CFRP and other advanced composites in sports goods and industrial equipment is given by Labin and Donohue (2), and the aerospace industry is extensively reviewed by Zweben (3).

Improvements in mechanical properties and weight reduction are the overriding purpose of substituting metals with advanced composites. However, they are generally more expensive than their metallic equivalent. Although there is a great incentive for using advanced composites from an engineering point of view, the economic consequences need to be realistically assessed with respect to the cost of the final product.

Clearly, with an increasing number of applications of advanced composite materials, more information is required about their performance in order to utilise them more efficiently. Since these materials are subjected to variable loads during their service lives, it is important to recognise that they are also subjected to damage development sooner or later. The damage could be manifested as localised fibre fracture which is relatively benign, or if sufficient damage is introduced, the whole system could fail catastrophically. It is, therefore, important to have an understanding of their fundamental behaviour under load in order to appreciate the failure mechanisms in general, and the way in which fibres behave and interact in particular.
An important feature of brittle materials under load is that their strength distribution tends to vary greatly about their mean value. A consequence of such a wide variability is that the usefulness of the strength mean value as a design parameter is limited. Rather, the failure events are dominated by the scatter which becomes a key parameter of the process. The reason for the scatter observed in the strength of brittle materials is due to the inherent distribution of flaws in the material. Attempts to understand this behaviour have led to the "weak-link" theory whereby strength depends on the probability of finding a large flaw in a given length.

The aim of this project is to improve the understanding of the fracture of fibres in the context of the weak-link theory. The work involved examining and comparing the strength distribution of individual fibres in two different environments: in air (unsupported fibres) and embedded in epoxy-resin matrix (supported fibres). The work also involved measuring the strength of impregnated bundles of fibres for comparison purposes.

A special experimental technique was developed for the work where a single long fibre (or a bundle of fibres) was selected and divided into four specimens of gauge lengths: 5, 12, 30 and 75mm. Large numbers of specimens were tested in order to enhance the quality of the data so that a reasonable statistical interpretation could be obtained. The essence of this new technique is that it provides the best test yet devised for examining the weak-link property when comparing the strength of four different gauge lengths. By drawing all four groups of specimens from the same fibre (or bundle) and then analysing each group as a separate entity, differences in behaviour due to variations in fibre diameter, for example, can be investigated.
CHAPTER TWO

2.0 Literature review

2.1 Statistical aspects of fibre strength

All materials will fracture if they are stressed severely enough. Some materials fracture without any appreciable plastic deformation, and are said to be "brittle". Most brittle solids have mixed covalent-ionic bonding (glasses and ceramics). By contrast, metals and polymers will deform plastically; a useful property for many general engineering applications. In many areas of technological development, the question of brittleness is a vitally important factor in design. Fibres such as glass and carbon have very good tensile properties and as a result they are the major load bearing components in practical composites. However, the strength of individual fibres shows a large variability and, it is possible that this will have a large influence on the composite strength. It is generally believed that it is necessary to understand the statistical behaviour of the strength of the fibre before examining the strength of composite.

In essence, the statistical models proposed in the study of brittle fracture take as a starting point Griffith's theory (4) which produced an explanation that the strength of a brittle material is several orders of magnitude less than that predicted for the energy required to break all the atomic bonds in a cross section of material. Griffith proposed that a body is only as strong as the strength of its weakest element and the presence of flaws reduces the strength. These flaws vary in magnitude throughout the body and are often considered to follow a Poisson distribution; a probability distribution applied to the number of occurrences of
particular events (magnitude of flaws) in a particular time period.

The other classic work of Griffith (5) and that of Inglis (6) examined the stress concentration effect of various shapes of internal and surface defects in material. They showed that internal and surface flaws of similar tip radius produce similar stress concentration effect, and that failure is controlled by internal voids and inclusions. In a publication by Johnson and Thorne (7), it was shown that in some carbon materials this situation may be reversed, the removal of severe internal defects increase the filament’s strength. Fracture is then attributed to surface flaws. A recent publication by Breedon-Jones et al (8) showed that for modern carbon fibres such as pitch-based filament, failure is still mainly due to surface defects and flaws, although these tend to be introduced by poor processing technique rather than an inherent part of the fibre structure. This publication also reviewed the work of a number of other authors on defects in modern non-PAN based carbon fibres.

To optimise the strength of brittle materials, it would appear best to produce the material with a low diameter filament. This reduces the number of inherent internal defects and gives a high surface area over a given length. Surface treatment is then carried out to minimise the effect of the remaining surface defects, which are normally accentuated by handling and processing, but maximises surface area per fibre bundle. The use of low diameter fibre in itself will obviously lower the likelihood of internal defects occurring in a given length, but this will also be influenced by the production route of the material. For a fibre such as glass which is normally produced by mechanical drawing from a melt, any serious flaws such as voids, bubbles, etc. are likely to cause fracture during production but are unlikely to be found in a large batch material. Carbon fibres are generally produced by pyrolysis of a polymer precursor fibre, and whilst this largely
determines the surface structure of the filament (generally very rough compared to other types of fibres) the internal structure is likely to be largely free of major defects owing to the drawing of the precursor.

For these brittle materials there exist a statistical distribution of strength dependent on both the distribution of flaw sizes and the spatial distribution of these defects within the material. On this basis, if a fibre contains a random distribution of flaws throughout its length it becomes clear that its strength can be quantitatively described by a finite probability function. For a brittle material, fracture will occur when the stress concentration around the most severe defect is sufficient enough to allow catastrophic crack growth. By increasing the volume of the material in the specimen, the number of flaws in the sample will also increase and hence with it the probability of finding an even more serious defect. Therefore, one would expect smaller specimens to be statistically stronger than larger ones.

One of the early workers to realise that the strength of the material under the Griffith theory (4) has a close connection with distribution of extreme values was Pierce (9) of the British Cotton Industry Research Association. Although the flaws may follow a Poisson distribution, as mentioned earlier, Pierce argued that only the most severe flaw determines the strength of the solid. The smallest or extreme value in a distribution of strength is the most relevant. He also proposed a model for a fibre, where a fibre is considered to consist of a series of links or segments like a chain, so that when one link fails the whole chain fails. In effect, Pierce described the strength of each link by a statistical function and the strength of the whole chain being equal to that of the weak link. This is commonly referred to as the weak-link concept or model of fibre behaviour.
Pierce's work coincided with the statistical work done by Fisher and Tippet (10), around the same time on extreme value distribution. They identified three types of extreme value distribution and described their limiting forms. The distribution they proposed was basically a normal or Gaussian type but with finite limits placed on it rather than extending from minus to plus infinity as the normal distribution does.

It was in the 1930's that the Swedish engineer Weibull (11, 12, 13) combined Pierce's (9) work with that of Fisher and Tippet (10) and the embryonic idea of Griffith (4) and proposed a general mathematical function for describing the best fit to experimental data for many brittle materials. The underlying basis of Weibull distribution function is the Fisher and Tippet type III distribution, which assumes the data sample is taken randomly from a large population distribution according to a single variable.

As his basic postulate, Weibull envisioned materials filled with small flaws with no physical description and classified each of these flaws by their tensile strength. The flaws were viewed as links of a chain with individual strengths which vary from one link to another. To predict the behaviour of this chain, consider n links connected in series.

For large n, the discrete distribution of strengths is replaced by a continuous distribution. The probability that a random link fails at or below a tensile stress (σ) is given by f(σ). For the chain to survive, each of the links must independently survive. Now if the probability of survival of a single link in a chain is $P_s$ then
Probability of survival of n links = \( P_s^n \) \[2.1\]

For convenience, however, it is better to consider the probability of failure, \( P_f \).

Therefore, equation 2.1 becomes

\[ P_f^n = 1 - P_s^n \] \[2.2\]

The above probability equation (2.2) is then written as a cumulative distribution function (CDF) in the form of

\[ F(\sigma) = 1 - \exp \left[ -n \left( \theta(\sigma) \right) \right] \] \[2.3\]

where \( \sigma \) is a variable describing some characteristic of the population, in this case stress, \( F(\sigma) \) is the probability of the variability being equal to or less than \( \sigma \), and \( \theta(\sigma) \) is a function of \( \sigma \). The use of this function in relation to the Weibull distribution to describe the strength of brittle fibres is discussed in detail in the following section.

### 2.1.1 Strength of a single fibre described by Weibull distribution

As discussed earlier, nominally identical specimen of brittle materials, e.g. carbon fibres, show a large variation of tensile fracture stresses. The material's strength, therefore, needs to be characterised. The cumulative distribution function (CDF) proposed by Weibull (11) is the most widely used expression. The Weibull function is also known to statisticians as the Fisher-Tippett type III distribution of smallest extreme value (14). It is important to note that although the Weibull statistics is based upon the "weak-link hypothesis", which means that only the
most serious flaw in the specimen will control the strength, the most serious flaw is not necessarily the largest one because its severity also depends on where it is situated. In other words, the flaw which is subjected to the highest stress intensity factor will be strength controlling. The defects initiating fracture can conveniently be classified as fracture data originating mainly from a single type of defect. It is also possible to use a modified form of the function to describe data originating from a number of distinctly different defects sites. This modified function is referred to as a multi-model Weibull (MMW) function as opposed to the more widely applied single-model (15) function. Nelson (16) described in detail the statistical properties and uses of the Weibull distribution and other functions based on weak-link theories.

2.1.2 Single-mode Weibull distribution characterisations

Weibull set certain conditions on the function \( \theta(\sigma) \) so that it must be positive, non-decreasing and becomes zero at a value of \( \sigma \) greater than or equal to zero. The condition that fulfils these requirements is:

\[
\theta(\sigma) = \left[ \frac{\sigma - \sigma_u}{\sigma_0} \right]^W
\]

substituting this equation (2.4) into equation 2.3 gives the general expression for the Weibull distribution.
L is used here in accordance to Griffith's theory who considered that the strength of brittle materials was determined by flaws, so that a brittle fibre is considered as divided into lengths of varying flaw severity and so the fibre can be considered to have failed if one of its segments has failed, i.e. if a failure has occurred anywhere along its length. The above equation (2.5) is called the Weibull “three-parameter” cumulative distribution function. Where (σ) is normally fracture stress or strain and L is the length of filament at which W, σ₀, and σ_u have been determined. The parameter σ_u is a threshold level; it is normally very small for brittle materials and thus is set to equal zero. W is the Weibull exponent or scale (sometimes referred to as shape parameter) parameter, it gives a measure of the material homogeneity. The larger the value for W the smaller the dispersion or variability within the data set (17). σ₀ is a location parameter sometimes referred to as characteristic strength, is given by the 63.2th per centile of the distribution since, σ=σ₀; and

\[ F(\sigma) = 1 - \exp \left( - \frac{\sigma - \sigma_u}{\sigma_0} W \right) \]

\[ F(\sigma) = 1 - \exp (-1) = 0.632 \]

\[ \ln \ln \left( \frac{1}{1 - P_d} \right) = 0 \]

It is important to note that L, the length of the filament was used here instead of
the accurate measurement of \( V \) (volume), since most filamentary materials appear to have approximately constant cross-section. Any systematic variation in fibre diameters must be allowed since this has a direct effect on the number of possible internal defects as well as the fibre surface area and the number of surface flaws.

When setting \( \sigma_u \) to equal zero, the two-parameter Weibull distribution is produced, which is the most practicable form of the distribution.

\[
F(o) = 1 - \exp \left[ -L \left( \frac{o}{\sigma_o} \right)^w \right]
\]

The form of the Weibull distribution is shown graphically in Figure 2.1 for lengths 1, 10 and 100 units using Weibull data (13). Equation (2.7) could alternatively be expressed in terms of strain (by replacing stress, characteristic strength and \( \sigma_u \) respectively with strain).

The evaluation of \( W, \sigma_o \) and \( \sigma_u \) for a set of data can be produced by using different graphical and analytical methods. The next section will consider the graphical methods used in this work. Analytical methods are not given here, since they are not used. However, methods, e.g. based on the Newton-Raphson iterative technique, have also been used to calculate Weibull parameters. A full discussion of these technique is provided in reference (16).
2.1.3 Weibull parameters estimation using graphical methods

Many workers have examined the various graphical methods available in calculating the three Weibull parameter distribution, namely $W$, $\sigma_o$, and $\sigma_u$ for a data set. Braiden (18) presented a general review of Weibull statistics and discussed two graphical methods, one based on equation 2.7 and the other on equation 2.14 (which will be discussed later). The first graphical method requires equation 2.7 to be rearranged so that the Weibull cumulative distribution function gives a linear plot. By taking the natural logarithms of equation 2.7 twice will result in:

$$\ln \ln \left( \frac{1}{(1-P_f)} \right) = W \ln (\sigma - \sigma_u) - W \ln \sigma_o + \ln (L)$$

This reduces the Weibull equation to its linear form. A plot of $\ln (\sigma)$ against $\ln \ln \left( \frac{1}{(1-P_f)} \right)$ become straight lines of gradient $W$. The gradient of the line represents $W$ in this plot, which is commonly referred to as a “Weibull probability plot”. The characteristic strength, $\sigma_o$, of $\sigma$ is the intercept at

$$\ln \ln \left[ \frac{1}{(1-P_f)} \right] = 0 \quad 2.8$$

or

$$P_f = 0.632 \quad 2.9$$

In this plot both $W$ and $\sigma_o$ are calculated by assuming $\sigma_u = 0$. 


The plot is done by ranking the observations into ascending order and then assigning a linearly distributed $P_f$ value between 0 and 1.0 to each point. This gives a measure of the probability of failure at a given value of stress. An arbitrary procedure and the equation for assigning $P_f$ values for a series of observations are generally termed estimators. Trustrum and Jayatilaka (19) carried out an extensive study of the probability models for the failure of fibres and discussed the estimator functions adopted for the Weibull analysis. The following two estimators have been used by these authors to calculate $P_f$ values.

\[
P_f = \frac{j}{N+1} \quad \text{(2.10)}
\]

\[
P_f = \frac{j-0.5}{N} \quad \text{(2.11)}
\]

Where $j$ is the rank and $N$ is the number of data points.

Trustrum and Jayatilaka (19) recommended that for sample sizes less than 50 specimens, equation 2.10 gives a more biased estimate than equation 2.11. The latter estimator therefore is to be preferred, a conclusion which was also drawn by Johnson (20). Among other estimators discussed by Johnson is the medium rank value which can be approximated by

\[
P_f = \frac{j-0.3}{N+0.4} \quad \text{(2.12)}
\]
A further example is:

\[ P_t = \frac{j^{3/8}}{N^{1/4}} \]  

2.13

As cited by Johnson (20) the study of samples of size 6, applied to equation 2.13 gave the least biased estimate and the most biased estimate resulted from equation 2.10, while equations 2.12 and 2.13 gave approximately equivalent estimates for \( W \). Other workers, such as Bergman (21) in a review of this technique, has recommended equations 2.11 for sample size larger than 20 and 2.13 for sample less than 20. He also studied other estimators and concluded that equations 2.10 and 2.12 have inferior statistical properties.

The second graphical technique is also applicable to the two-parameter distribution. It does not require the use of estimators and is based on equation 2.14, the calculation of the Weibull mean. Nelson (16) gave the following strength equation.

\[ \bar{\sigma} = \sigma_0 L^{-1/\gamma} \Gamma(1+1/\gamma) \]  

2.14

This corresponds to the arithmetic mean strength in a population. \( \Gamma \) is the gamma function. Taking the natural logarithm of the mean strength equation results in
A plot of ln σ against ln (length) gives a straight line with gradient (-1/W). This plot is often referred to as the Weibull plot of the second type. This graphical method indicates that fibre length does not affect the variability in the strength of the material, but it affects the mean and characteristic strength values. This suggests that long fibres tend to have lower characteristic strength than short fibres although in ideal situations they should show identical variation in W values.

There is some dispute as to which of the two Weibull distributions (the first type or the second type) is the best method. Clarke (22) suggested that the former method is more affected by errors in testing but these may be discarded leaving a more accurate determination of the Weibull parameters. Asloun et al (23) pointed out that the latter plot provides a simple method of extrapolating to short lengths of fibre.

The calculation of Weibull parameters by graphical methods have been studied by various workers notably Braiden (18), who presented a general review of Weibull statistics, Heavens and Mugatroyd (24), Trustrum and Jayatilaka (19) and Bergman (21, 25). The conclusion of all these authors is that provided that the sample population is large (i.e. greater than 40) the best graphical system for calculating the constant, W and \( \sigma_0 \), for the two parameter distribution is the Weibull plot of the first type.
2.1.4 Weak-link scaling

One of the most interesting aspects of the failure of brittle materials, such as carbon fibres, is that the effect of longer lengths of fibres (or larger volumes) on the measured strength distribution can be predicted using Weibull statistics. If the flaw distribution in a brittle material is considered completely random then the volume (length) parameter in the Weibull equation can be used

\[ P_f = 1 - \exp \left( -\left( \frac{\sigma - \sigma_u}{\sigma_o} \right)^w \right) \]

2.16

Weibull expression, equation 2.5 allows the prediction of the strength of a long fibre from experimental data obtained at shorter gauge lengths or vice versa. It was found experimentally that a longer fibre is likely to have a lower strength than a shorter fibre. This can be explained by arguing that there is more chance of finding a large defect (flaw) in a longer fibre. Since a larger flaw will mean that the fibre can withstand a lower stress before failure, longer fibres are more likely to have lower strengths. Weak-link scaling can be modelled using the Weibull expressions in the following way.

Consider two lengths, \( L' \) and \( L'' \) which have probabilities of survival, \( P_S \) (where \( P_S = 1 - P_f \)), of \( P_S(L') \) and \( P_S(L'') \). If these two fibres are joined to make length \( L' + L'' \), then the probability of survival of this length at a given applied stress is the product of the probabilities for individual lengths, i.e.
\[ P_S (L' + L'') = P_S (L') \times P_S (L'') \]  \hspace{1cm} 2.17

Therefore, for a chain of length, \( L \), consisting of \( k \) links, each of length \( L_b \),

\[ P_s (L) = [P_s (L)]^k \]  \hspace{1cm} 2.18

where \( k = \frac{L}{L_b} \).

If for each link of the chain, the probability of failure is given by:

\[ P_f = 1 - \exp \left( \frac{\sigma}{\sigma_o} \right)^w \]  \hspace{1cm} 2.19

then the probability of survival of each link is:

\[ P_s = \exp \left( \frac{\sigma}{\sigma_o} \right)^w \]  \hspace{1cm} 2.20

Thus for the chain consisting of \( k \) links, each of the length \( L_o \), the probability of survival is:
or, the probability of failure of the chain is given by:

\[ P_s = \exp \left[ -k \left( \frac{\sigma}{\sigma_0} \right)^W \right] \]

\[ 2.21 \]

\[ P_{1(L)} = 1 - \exp \left[ -k \left( \frac{\sigma}{\sigma_0} \right)^W \right] \]

\[ 2.22 \]

The same argument applies when discussing the probability of failure of a certain volume, \( V \), consisting of \( k \) units of volume \( V_0 \), in that case:

\[ k = \frac{V}{V_0} \]

\[ 2.23 \]

Weibull data (13) fit these distribution very well, Figure 2.2. This Figure shows strength values for a failure of a fibre having gauge lengths between 1 and 10 units. As the tested fibre length increases, the characteristic strength decreases but the Weibull modulus remains constant. Using this analysis it is also possible to predict the characteristic strength of the distribution for any fibre length. This is given by:

\[ \sigma_0 (L) = \sigma_0 k^{-1/W} \]

\[ 2.24 \]
where $\sigma_0$ is the experimentally determined characteristic strength for a length $L_0$, and $k$ is given by:

$$k = \frac{L}{L_0}$$  \hspace{1cm} (2.25)

where $L_0$ is the length at which the parameter estimates are made and $L$ is the new length at which it is required to “predict” the fibre strength.

Another way of writing equation 2.24 is as follows:

$$\sigma_{0(2)} = \sigma_{0(1)} \left( \frac{L_1}{L_2} \right)^{1/w}$$  \hspace{1cm} (2.26)

where $\sigma_{0(1)}$ is the strength of the fibre at length $L_1$, and $\sigma_{0(2)}$ is the strength of a fibre of length $L_2$. Equation 2.26 is commonly called the weak-link scaling equation. If $\alpha$ is a constant representing the characteristic stress for unit length (1 mm) of a fibre then the weak-link scaling can be represented in another form:

$$\sigma_{0(L)} = \alpha L^{-1/w}$$  \hspace{1cm} (2.27)

Priest (26) conducted tests on carbon fibres at four different gauge lengths in the range of 1-50 mm. All the fibres tested came from the same length of carbon tow containing 1000 filaments, but all specimens tested were from different filaments. Priest found that it possible to predict strength distribution by weak-link scaling to other gauge lengths within 5% suggesting behaviour is reasonably obeyed for single carbon fibres.

Watson and Smith (27) subsequently conducted a likelihood ratio analysis on
Priest's data to investigate the hypothesis that the strength distributions at different lengths have the same Weibull parameters. They found, however, different Weibull parameter values for the different gauge lengths. They conjectured that variation in fibre diameter, which can be appreciable, is an important factor and demonstrated how a model which takes this factor into account could be of the form:

$$
\bar{F}_L(\sigma) = \exp \left[ -L^c (\sigma/\sigma_o)^w \right]
$$

where $\bar{F}_L(\sigma)$ is the probability that a fibre of length $L$ survives stress $\sigma$. They estimated the parameter $c$ to be 0.90, but concluded that this was not a significant improvement on the case $c=1$.

Departure from the Weibull model is not a test of departure from the weak-link concept. This latter property is a fundamental one, although until now there has been no experimental work designed specifically to test this property. A simple test of the weak-link concept has been designed by Wolstenholme (28). This test is referred to as the non-parametric test and is outlined in detail in the next section.

### 2.1.5 Non-parametric test

This approach is applied to individual fibres, without making any assumptions about the strength distribution. Supposing a fibre of length $L$ is cut to lengths $L_1$, $L_2$, $L_3$, $L_4$, $L_5$, and $L_6$, the test statistic is given by:

$$
S = \frac{1}{6} \left( \frac{1}{L_1} + \frac{1}{L_2} + \frac{1}{L_3} + \frac{1}{L_4} + \frac{1}{L_5} + \frac{1}{L_6} \right)
$$

If the weak-link concept holds, the distribution of the lengths $L_1, L_2, L_3, L_4, L_5, L_6$ will be independent and identically distributed (i.i.d.) with a uniform distribution on the interval $[0, L]$. The expected value of $S$ is $1/6$, and the variance is $1/(12L^2)$. Therefore, the observed value of $S$ can be compared to the expected value and variance to test the weak-link concept.
where \( L_4 < L_3 < L_2 < L_1 \) and the failure stress of each length is noted and ranked 1, 2, 3, 4 in increasing order. There are four different gauge lengths, therefore, there are 24 different possible ways these ranking could occur \((4! = 24)\) and the probability of each ordering would depend on assumptions made about the underlaying statistics. Under the weak-link hypothesis, at any point in the loading process every unit length element has an equal chance, say \( P_x \), of failing next, regardless of which gauge length it is contained within.

If \( L_1, L_2, L_3, L_4 \) are to be the lengths of the sections failing 1st, 2nd, 3rd and 4th respectively then a total length \( L - L_1 \), is left after length \( L_1 \) fails and the probability that length \( L_2 \) is next to fail is \( L_2/(L-L_1) \), and so on. Then the probability of obtaining the order \( L_1, L_2, L_3, L_4 \) is given by

\[
\text{Prob (order is } L_1, L_2, L_3, L_4 \text{)} = \frac{L_1}{L} + \frac{L_2}{L-L_1} + \frac{L_3}{L-L_1-L_2} \tag{2.29}
\]

Similarly the probability for all other possible orderings may be calculated. The null hypothesis (the test of the hypothesis) dictates that longer fibres are on the average weaker than fibres which are shorter, i.e. the probability of encountering a fatal flaw is higher. Therefore, the expected order of failure for the four different gauge lengths is \( L_1, L_2, L_3, L_4 \). In this work, the gauge lengths are \( L_1 = 75\text{mm} \), \( L_2 = 30\text{mm} \), \( L_3 = 12\text{mm} \), \( L_4 = 5\text{mm} \), the probability of the most likely order of failure is:

\[
\frac{L_1}{L} \times \frac{L_2}{(L_2 + L_3 + L_4)} \times \frac{L_3}{(L_3 + L_4)} \times \frac{L_4}{L_4} \tag{2.30}
\]
\[
\frac{75}{122} \times \frac{30}{47} \times \frac{12}{17} \times \frac{1}{1} = 0.2771
\]

(N.B.: \( L = L_1 + L_2 + L_3 + L_4 = 122\text{mm} \))

Some of the 24 possible order of failure and their probability are listed below:

<table>
<thead>
<tr>
<th>Event</th>
<th>Probability</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 2 3 4</td>
<td>0.2771</td>
</tr>
<tr>
<td>2 1 3 4</td>
<td>0.1415</td>
</tr>
<tr>
<td>1 3 2 4</td>
<td>0.1345</td>
</tr>
<tr>
<td>1 2 4 3</td>
<td>0.1154</td>
</tr>
<tr>
<td>2 1 4 3</td>
<td>0.0589</td>
</tr>
<tr>
<td>3 1 2 4</td>
<td>0.0575</td>
</tr>
<tr>
<td>2 3 1 4</td>
<td>0.0300</td>
</tr>
<tr>
<td>3 2 1 4</td>
<td>0.0250</td>
</tr>
<tr>
<td>1 4 2 3</td>
<td>0.0467</td>
</tr>
<tr>
<td>1 4 3 2</td>
<td>0.0187</td>
</tr>
<tr>
<td>1 3 4 2</td>
<td>0.0224</td>
</tr>
<tr>
<td>4 2 3 1</td>
<td>0.0724</td>
</tr>
<tr>
<td>2 4 3 1</td>
<td>0.0724</td>
</tr>
<tr>
<td>2 3 4 1</td>
<td>0.0724</td>
</tr>
<tr>
<td>3 1 4 2</td>
<td>0.0187</td>
</tr>
<tr>
<td>2 4 1 3</td>
<td>0.0224</td>
</tr>
<tr>
<td>3 4 2 1</td>
<td>0.0224</td>
</tr>
</tbody>
</table>

+ 7 other possibilities

In practice, one can compare the expected and observed frequencies by using the Chi-squared test, assuming that the null hypothesis is true.
In the Chi-squared test, the difference found is between the corresponding expected, $E_i$, and observed, $O_i$, frequencies. The calculation is expressed in the formula:

$$
\chi^2 = \sum \left( \frac{(O_i - E_i)^2}{E_i} \right)
$$

2.31

If the observed and expected frequencies exactly agree, $\chi^2$, to be zero so that the method can test whether the differences between the frequencies of occurrence to be so large, thus it is unlikely to have occurred by chance.

2.1.6 Multi-modal Weibull distribution

Not all brittle materials conform to a single-mode Weibull distribution. The presence of several modes in the strength distribution implies the existence of several distinct types of strength-limiting defects in the fibre structure.

Fukunaga et al (29, 30, 31, 32) examined the strength of fibres in relation to the fractured surface of each specimen to try to determine the defect type which initiated failure. They argued that the strength of the fibre can be defined in terms of the strength of each subpopulation of flaws.

Generally, the analysis of a bi-modal mixture of Weibull distributions is difficult, if not impossible, to carry out graphically, since in most cases the different subpopulation of flaws tends to overlap with each other. More complicated analyses, such as the maximum likelihood technique, are used for
determining the most probable set of parameters for this mixed distribution, by assuming that each subpopulation is independent of all the others. In this work, a graphical approach is used to try to disentangle distributions related to volume and length effects.

2.2 Weibull results for single fibre strength data

Many workers have examined the characterisation of single carbon fibres. As mentioned earlier, the majority of brittle filament failure tends to originate at defects, in the form of flaws and voids, on the fibre surface. The Weibull distribution method can then be used accurately to describe fibre strength. Kasai and Saito (33) examined the applicability of the Weibull distribution with respect to strength for a range of filamentary materials, including carbon, glass, silicon carbide and boron. For carbon fibres they presented the results from the work of six authors. Kasai and Saito (33) also calculated the best value of the Weibull exponent from the raw data, assuming a three-parameter distribution. They applied a maximum likelihood technique to this data which showed that although there was a wide variation in the scale parameter ($\sigma_0$) for the different materials the shape parameters ($W$) were remarkably constant being in the range of 2-4 for all four fibre types. They concluded that failure strengths of the fibres were governed by similar distributions of defects. These flaws which are almost certainly surface defects can be inferred from many observations on the strength increase of many fibre types after surface etching. Griffith (4) and more recently Proctor (34), Johnson (35) and Thorne et al (36) have also showed similar effects on carbon and glass fibres after surface etching.

Barry (37) reported an extensive testing programme on three types of carbon
fibres. Seventeen hundred strength and Young's modulus measurements were made. Barry's work, as well as the recently by Priest (26) show that the strengths of carbon fibres fit well to a two-parameter Weibull distribution. By contrast Martineau et al (38) suggested that silicon carbide with a tungsten core may exhibit a bi-modal strength distribution with low strength failures attributable to surface initiated fracture and high strength fracture originating in or around the tungsten core of the material.

Metcalfe and Schmitz (39) who worked on glass fibres and the work of Hitchon and Phillips (40) on carbon fibres, both have used the Weibull \( \ln(1)/\ln(\sigma) \) plot and concluded that fibres with gauge lengths of less than 5mm are shown to be much stronger than expected. These authors attributed the behaviour to carbon fibres having a second defect population due to local microstructural imperfection, arguing that in short length of fibres surface damage due to handling is less likely to be found. These observations subsequently became the subject of research by many workers, notably Larder and Beadle (41), who proposed a mathematical model based on fibre strength following a Gaussian distribution. They assumed strength to be determined by flaws of varying severity along the fibre lengths following a Poisson distribution. They argued that since the spread in strength for very short fibre lengths is likely to be quite large the use of a Gaussian rather than a Weibull distribution for strength is not unreasonable. Fibre strength was then estimated by notionally dividing the length into short links, assigning a strength to each link using the Gaussian distribution and then superimposing a random spatial flaw distribution on this as the strength limiting mechanism. This method, however, is found to give very unreliable results.

Recent work by Wagner (42) who studied the strength of single fibres and concluded that for size effect two defect populations needed to considered.
Firstly, if the potential weak sites are distributed throughout the entire volume of the specimen, then owing to the cylindrical shape of the filament \( V = (\pi/4) D^2 L \), where \( D \) and \( L \) are the fibre diameter and length respectively. If the Weibull model is applicable, a length effect will translate as a straight line of slope \(-1/w\) in a log \( \sigma_0 \) versus log \( L \) plot, whereas a diameter effect will translate as a straight line of slope \(-2/W\) in a log \( \sigma_0 \) versus log \( D \) plot. Secondly, if the potential weak sites are distributed mainly on the specimen surface rather than in the bulk, then \( A = \pi DL \). Hence if the Weibull model is applicable, both the length and diameter size effect will translate as straight lines with slope \(-h/W\) in log \( \sigma_0 \) versus Log \( L \) and versus log \( D \) plots respectively.

In summary, the Weibull weak-link model for strength can provide a simple interpretation of size effects both in the case of surface and volume defect population.

2.3 Single fibres in a matrix

The presence of a matrix around a single fibre provides a mechanism for the transfer of load back into the fibre away from the break. This is achieved over a length of fibre commonly called the “ineffective length”. The load that is carried by the failed length of the fibre is supported by the surrounding matrix until the fibre is again capable of supporting its portion of the load. An understanding of ineffective length in a fibre and load redistribution are important in studying the mechanism of fibre/matrix fracture. Single fibres embedded (supported) in resin provide a technique to investigate these phenomena.
2.3.1 The ineffective length

The ineffective length is an important concept in considering short fibre composites or continuous fibres which fracture. When an aligned composite consisting of a short fibre embedded in a matrix is axially stressed, the matrix being more compliant than the fibre tends to undergo a larger axial deformation. The longitudinal tensile stress in the fibre then becomes maximum in the middle (neglecting the effect of adjacent fibre ends) and minimum at the ends. The region near the end of the fibre over which the longitudinal stress is smaller than the maximum is called the ineffective length or transfer length and corresponds to the length over which the stress is some particular fraction (half, 0.9, etc.) of the maximum longitudinal stress in the fibre (Figure 2.3).

The notion of an ineffective length is unique to composite materials. It is a very difficult parameter to measure accurately in practice and is influenced by many variables which make its characterisation difficult. Rosen (43) looking at fibre fracture in continuous fibres used a shear-lag type of analysis, (e.g. Cox (15)), to calculate the distance from a fibre break to where the stresses in the fibre reach their undisturbed value (Figure 2.3(a)). He found that it was quite rapid and localised, but asymptotic in nature. So the ineffective length was defined by specifying some fraction $\phi$, between 0 to 0.9. He marked this parameter as $\delta$ and defined it as the length of the filament either side of the break where the tensile stress is less than the fraction $\phi$ below the undisturbed stress. It is important to highlight that the length $\delta$ is unloaded on both sides of the failure and the total affected length is $2\delta$. Using this model, Rosen (43) has showed that the ineffective length tends to increase with increasing modulus ratio, $E_t/G_m$, with decreasing fibre volume fraction and increasing fibre diameter. The ineffective
length, $\delta$, value will depend on the extent of matrix damage around a fracture. For example, the value of $\delta$ will increase if debonding occurs, particularly if the debond region extends with further loading.

Cox (15) and Dow (44) derived similar shear-lag models for estimating the stress distribution at a fibre end. Dow (44) made the assumption that a series of straight radial lines in the fibre and the matrix remains straight during loading. Cox, on the other hand, assumed that the interfacial shear stress is proportional to the effective relative displacement of fibre and matrix. There are a number of assumptions common to both models:

1. There is no fibre end adhesion to the matrix, but there is perfect bonding along the cylindrical interface between fibre and matrix.

2. The fibre and matrix are both completely elastic.

3. Interfacial shear stress is proportional to the difference in the relative displacement of fibre and matrix when loaded as a bonded composite and as two separate components.

4. The fibre only carries axial tensile stress.

5. The matrix only carries shear stresses.

6. There is no stress concentration around the fibre ends.

Figure 2.4 shows the type of stress distribution for the interfacial shear stress and
fibre tensile stress using the Cox model. Figure 2.4 shows the general form of the distribution of the tensile stress in the fibre and the shear stress at the interface. The rate of the stress transfer is determined by the ratio of the fibre tensile modulus to the shear modulus of the matrix, \( E_f/G_m \). The higher this ratio is the longer the transfer length, \( \delta \), and the lower the maximum shear stress at the fibre end. From the above common assumptions made for both models, it can be said that 1, 2, 4 and 5 are reasonably realistic for a broken continuous fibre, which has a very high modulus compared to the matrix. The final assumption made simplifies the model, although it is extremely unrealistic. Tyson and Davies (45) and Allison and Hollaway (46) studied this phenomenon by using photoelastic experiments. They concluded that the stress field away from the fibre ends is basically non-uniform, and that the maximum shear stress as well as the maximum fibre load occur away from the fibre end. They also reported the shear stress along the interface within one or two diameters of a fibre end is about five times greater than values estimated by Cox's method. Recent studies by Galiotis et al (47) examined the strain in single polydiacetylene single crystal fibres embedded in epoxy-resin. The tensile strain resulting in the fibre from the applied load was measured using resonance Raman spectroscopy. Their findings agreed with Cox's analysis and they reported that for axial strains of greater than 1%, Cox's model appears to give a good qualitative description of the fibre stresses.

First strain at failure breaks for each individual specimen was measured in this work (assumed to be the weak-link) and its corresponding stress values was calculated using a nominal mean fibre modulus supplied by the fibre manufacturer's Weibull statistics were applied to the data and the results were compared to the unsupported fibres.
2.4 Bundles of fibres in a matrix failure mechanism

A single fibre embedded in a resin matrix has been mentioned in detail in the previous sections. This is commonly regarded as the basic building block for impregnated bundles of fibres in a matrix. A typical uniaxially reinforced composite is constructed of many parallel fibres having high tensile strength and high modulus held together by a relatively weak low modulus matrix material, for example epoxy-resins. For the following analysis it is assumed that the matrix is a homogeneous solid of negligible tensile modulus and a high strain to fracture. In examining the failure strength characteristics of a bundle of fibres in a matrix, it is also assumed that the matrix bonds well to all the fibre surfaces and to be capable of transferring only a shear force between the fibres. The mechanism governing the failure of strong fibres embedded in a resin matrix is essentially of three factors:

(i) the inherent strength of individual fibres;
(ii) the ability of the matrix to transfer load between fibres; and
(iii) the extent to which the fibre becomes unloaded in the region of the break.

When a uniaxial reinforced composite is extended monotonically in the fibre's direction, sporadic fibre failure occurs at the weakest point of the fibres. Eventually a catastrophic failure process develops from the individual fibre breaks. Many theories of tensile strength of uniaxial fibre reinforced composite have been proposed over the years.

Daniels (48), was the originator of the equal load sharing rule (ELS) for a bundle of fibres. Here a progressive failure mechanism occurs, until a sufficient number of threads or fibres have failed when the remaining intact fibres are unable to
sustain the applied load. The magnitude of the applied load and the variation in
the strength of the fibres will determine the actual number of failed fibres in the
bundles prior to catastrophic fracture. The undue simplification in this model lies
in the fact that there is no transfer of load between individual fibres. It is important
to note, however, that in practice frictional coupling and non-ideal loading will
produce localised stress concentrations.

Queer and Gurland (17) were the first to devise a model for homogeneous
materials. They have combined Pierce’s (9) weak-link statistics with Daniel’s
bundle statistics and indicated the valid applicability of the model to the uniaxial
fibre reinforced material, (Figure 2.5). They conceived a composite as tension-
loaded cylinder to be cut up into a large number of cross-sectional slices of equal
thickness. Each of these layers is composed of identically shaped, independent
cylindrical elements, and the probability distribution of tensile strength for these is
assumed to be known. The individual elements fracture independently of each
other. Queer and Gurland investigated two separate fracture modes. First, the
whole cylindrical structure fractures even if only one individual element fractures.
Second, the whole cylinder fractures if an individual layer fractures (weak-link
statistics). An individual layer fractures if the number of its individual elements
that are broken when the load on the remaining elements in this layer is
increased to such an extent that the whole bundle of the elements in the layer
fractures (bundle statistics). Thus, the elements of this layer fracture
independently of each other and there is no mutual influence between them; they
all have to carry the same load. Statistically, this model is very conservative when
compared to experimental data, although it gives a lower bound on composite
strength. Hitchon and Phillips (49) observed that this model appear to predict
accurately the size effect for large composites. This model neglects local
increases in stress intensity in the vicinity of a broken element and the possibility
of failure involving more than one bundle. It also “over predicts” composite strength, although it accurately predicts a shape effect on composite strength and a decrease in strength with absolute volume. Gucer and Garland, however, failed to present a rationale for determining the slice thickness in their model.

Rosen (43) was the first to consider a chain of micro-bundles model in which layer thickness is specifically equated to the ineffective length. He examined sequential failure of glass fibres in an epoxy-resin, using transmitted polarised light (photoelastic technique) and observed that at less than 50% of the ultimate load, random breaks occurred in the fibres throughout the sample; the number of breaks increased with the applied load. In his model the composite is split into lengths along the fibre direction with each length considered to be a link in a “chain of bundles”. The length of each segment is described by an effective length, specified as the distance from a broken fibre end to where the stress is below a specified fraction of the undisturbed fibre stress. The stress build-up from the broken fibre ends is calculated using shear-lag analysis. Rosen considered that as the load increased, fibres would fail sporadically at various stresses, and the whole composite would fail when the remaining unbroken fibres in a particular segment were unable to carry the applied load. A schematic diagram of tensile stress distribution carried by a filament containing a single fracture, while its adjacent neighbour is intact is shown in Figure 2.3. A strength distribution for each segment is described by a cumulative distribution function (CDF) and an application of the weak-link concept is made since all the mini-bundles (segment) are connected in series. This is analogous to the weak-link concept in the Weibull model for a single fibre. Rosen’s model considers tensile stress to act only on the fibres and shear stress is considered to be confined to the matrix.
The ineffective length, $\delta$, is required to be estimated for this model. This is, in fact, wrongly defined as the ineffective length by Rosen and by subsequent workers using this model, since what is really required is the affected length of the neighbouring intact fibre for which the stress concentration factor is greater than 1. A value of 10 fibres diameter is used by Hedgepeth (50), for example, based on a shear-lag model.

Rosen (43) for his model did not take into account fibre strength variability or consider local stress concentrations in fibres adjacent to the failed fibres. Zweben (51) took the geometrical model of Rosen and studied the influence of load concentrations caused by fibre breaks on the strength of two dimensional composites, with special regard to the local stress. In effect he introduced a rather more severe load distribution rule, which is local load sharing (LLS). This enabled the expected number of groups of $i$ adjacent fibre breaks as a function of stress to be calculated, although it became more difficult as $i$ increases as a conservative failure criterion. Zweben suggested taking the stress at which the first multiple fibre break was expected. This model gives much lower predicted tensile strengths than the Rosen model, and measured strengths are often found to lie between the bounds of the models. The effect of stress concentration was also able to explain in principle the catastrophic failure normally observed in tension. Zweben quoted data from Gatti et al (52) on boron fibres in an epoxy-resin matrix. Zweben found that two failure mechanisms can be used depending on the strength of the interface. He concluded that when the interface is strong, his chain of bundles model could be applied to the weak-link failure mechanism. However, when the interface was poor the cumulative failure mechanism could be used instead.

Harlow and Phoenix (53) studied the chain of bundle model using the weak-link
approach. In order to avoid the complication of edges they assumed a circular, uniformly spaced array of fibres and obtained an approximate general expression for load concentration. They calculated the cumulative distribution function for bundle strength using computer simulation to generate all states of failed and surviving fibres of which there are $2^n$ combinations ($n$ is the number of fibres). All possible failure sequences were then generated and the probabilities for each of these were calculated. Although their method was mathematically precise, the simplification involved gave rise to lower values for composite strength than those observed experimentally.

Batdorf (54) abandoned the chain of the bundle model and concentrated on the formation and growth of multiple fractures. He used the weak-link theory to determine the number of isolated fibre fractures (singlet, double fracture (diplet) and multiplets) of arbitrary order as a function of stress. Batdorf showed that at a certain stress, due to the effect of stress concentrations there is a critical i-plet size beyond which unstable failure occurs. He proposed the following sequence of events for final failure of the composite. When the first sporadic fibre break occurs the surviving neighbours will experience a stress concentration. This will increase their probability of failure. Eventually, under a monotonically increasing stress concentrations on the surviving fibres around a single fibre break, or singlet, will cause a second fibre to fail and form a diplet. The resulting stress concentration around a diplet will be more severe than that associated with a singlet, so the probability of further failures to form i-plets of higher order will be increased. Eventually an i-plet of critical order will be formed, at which point the failure will spread from one fibre to another with no further increase in stress being required to sustain the process. This is the final event. The sequence described above is illustrated in Figure 2.6.
Overall failure can be predicted from the stress at which the first critical i-plet forms. An important point about this model is that different lengths of affected adjacent fibres for different i-plet sizes can be incorporated. This means that the final failure is not confined to a single short segment of thickness equal to the ineffective length. If the fracture of individual fibres obeys the Weibull distribution, a plot of \( \ln(Q_j) \) versus \( \ln(\sigma) \) will be a straight line of slope \( W_j \). Here \( \sigma \) is the applied stress, \( Q_j \) is the number of i-plets formed during loading to stress \( \sigma \), and \( W \) is the Weibull modulus. The envelope of the \( Q_j \) curves serves as a failure line (Figure 2.7) the use of the failure line leads directly to a rational failure criterion based on a Griffith-type instability. Figure 2.7 shows the line for each order of i-plet as a straight line of gradient \( W_j \). For the stress range within which an i-plet lies on the envelope, it is unstable, as it immediately becomes an \((i+1)\)-plet which in turn becomes an \((i+2)\)-plet and so on, resulting in composite failure. Therefore, the failure stress of the composite is the stress at which any unstable i-plet is present and can be found where the failure envelope intersects \( Q=1 \). The size effect of the composite is also predicted from this model, with the failure stress decreasing as the length of the fibre increases, but with less influence at higher lengths due to the requirements of a higher order i-plet for failure. The Weibull modulus is also predicted to change with increasing fibre length. The Weibull modulus of the composite is predicted to be \( W_{\text{composite}} = nW_{\text{fibre}} \). Where \( W \) is the Weibull modulus and \( n \) is the number of the broken fibres required to initiate failure.

### 2.4.1 The positively affected length of a fibre

Rosen (43) in his chain-of-bundle model introduced the concept of a positively
affected length (PAL) and calculated it using the ineffective length, \( \delta \), from a shear-lag analysis. He defined this to be the link length in his model. Although there is a limitation to his approach where the ineffective length increases with modulus ratio, \( E_f/G_m \), Rosen's definition was accepted by many workers (55, 56, 57, 58, 59, 60, 61) in their work of composite failure analysis.

Fichter (62), however, argued that the value of the ineffective length varies between fibres. He concluded that the central fibre in a bundle tends to have a longer value for \( \delta \) and that as the number of failed fibres increases, the value of the ineffective length increases too. He suggested a value of 17 fibre diameters, for a single fibre failure in carbon/epoxy composite.

Barry (63) produced a computer model predicting the expected range of composite strength by adopting Rosen's chain-of-bundle approach, and introducing the PAL instead of the ineffective length. Barry defined the PAL as the length over which a fibre is subjected to an increase in stress when adjacent fibre fails. This, he argued, corresponds to a distance of twice the ineffective length in the failed fibre. He determined the "slice" thickness by a stress analysis and found it to be dependent on the fibre/matrix modulus ratio, fibre spacing and debond lengths. For a single fibre failure, he found the PAL to equal 8-10 fibre diameter with no debonding. He assumed that his approach for a large composite system the fibre strength can be described by Gaussian function rather than the previously thought Weibull. Barry's approach of PAL was adopted by Batdorf (54) who considered the variation of its value with a varying number of breaks in a particular group. This approach, however, gave values which were one order of magnitude higher for strength, for \( \delta \), than experimentally derived by Manders and Bader (64). This he considered to be due to all the fibres adjacent
to a crack being allocated the same stress concentration factors, and PAL is independent of the crack size. Batdorf and Ghaffarian (65) in a later paper modified the theory to take into account the discrepancy in the value of PAL. In this modified version, they assumed that each fibre will not necessarily have the same ineffective length even though it is adjacent to a break and there will be one neighbouring fibre that will experience a greater overload than the other fibres. This increases the stress concentration factor and reduces the ineffective length to try to establish agreement between theory and experiment.

Harlow and Phoenix (66) investigated the problem of what value the PAL should take. They agreed that it must reflect fibre/matrix interactions and typically will be of a few fibre diameters but they concluded that the chain-of-bundles is fairly insensitive to the magnitude of δ.

2.4.2 Fibre stress concentration

In order to predict the probabilistic strength of a composite material, it is necessary to know the detailed stress state. It has been discussed in the previous section that a broken fibre is unloaded over a short distance on each side of the break. To preserve equilibrium, the load that is originally carried by the fibre over the ineffective length must be redistributed into the remaining composite. Daniels (48) assumed that in an event of one fibre failure in a loose bundle of fibre held in parallel, there will be a load redistribution equally over all the surviving fibre. Rosen (43), assumed that the composite material consists of a chain of bundles of fibre links and that the links break statistically. In the real failure process, when some fibres are broken the adjacent fibres will have to sustain more stress than the average fibre stress. This redistributed load results in
a stress concentration in that fibre over the positively affected length, which subsequently increases its probability of failure. The ratio of the stress in a fibre adjacent to r broken fibres to the fibre stress at infinity is called the "stress concentration factor, $K_r"."

In a two dimensional composite stress concentration factors obtained by the local load sharing rule as follows:

$$K = 1 + l/2$$

where $K$ is the stress concentration factor and $l$ is the number of adjacent fibre failures.

Hedgepeth and Van Dyke (67) calculated stress concentration values using shear-lag model and obtained value of 1.33 for a fibre neighbouring a single fibre break, increasing up to 2.216 for a fibre neighbouring five fibre breaks. Watson and Smith (27) studied an array of parallel fibres and assumed it to have the cross-sectioned of a square lattice (Figure 2.8). They assumed that this array of fibres exhibits a local stress concentration, which means that fibre failure is restricted to small lengths of the composite, measured in the direction of the fibre axis. In the event of fibre failure, the stress redistribution among neighbouring fibres will depend on the overall arrangement of the fibres, and the distance between the failed fibres and the surviving neighbours. The matrix will absorb extra load which has not been redistributed.

Many researchers have worked on the effect of stress concentrations on various composite materials. Among the earliest workers were Schuster and Scala (68) who have reported that although stress concentration at a break is greater than at a whisker end, they found that for multi-whisker sapphire composites staggering
the ends of short filaments tend to reduce the stress concentration to levels below those of isolated whiskers. Schuster and Scala also reported that at a spacing of 5-6 whisker diameters, there is little, if any, interaction between the stress fields of each filament.

Since fibre spacing in a composite tend to effect the overall stress concentration. MaClaughlin (69) reported that the increase of fibre spacing tend to have the effect of reducing fibre volume fraction. This in effect increases the severity of stress raisers and hence increases stress concentration in adjacent fibres.

The effect of interfacial bond strength on stress concentration factors has been studied by many workers. Reedy (70) found that in a perfectly bonded elastic composite system stress concentration does not depend on the material properties. He also found that when the interface debonds a residual frictional shear stress builds, and as debonding increases the length of fibre affected increases, the redistributed stress remains constant which subsequently decreases the concentration factor in neighbouring fibres.

Fichter (62) used Hedgepeth's original idea of analysing $K_C$ values using shear-lag analysis, of co-linear cracks. He concluded from his results that the influence of one crack extends no more than a distance on either side of the crack equivalent to the width of the crack. The effect of crack orientation on the load concentration factor in a two dimensional composite plate has been studied by Ko et al (71). They found that as the angle of crack to the fibre direction increases, $K_r$ values rises.

Armenakas and Sciammarella (72) applied Hedgepeth's two dimensional model, to their experimental data. They used a semi-empirical method and computed
values for stress distributions around fibre breaks from strain distribution at the surface of the specimen indicated by a pattern technique. They concluded that there exists a discrepancy of 10% between the computed experimental data and the stress concentration factors obtained by Hedgepeth.

Fukada and Kawata (61) applied a force balance to a section of a composite plate which already contains one or more broken fibres. Two assumptions were made, namely that plane stress is applicable and that the gap between the broken fibre ends is filled with matrix material. Manders and Bader (64) argued that there is no sufficient homogeneous material in the case, and therefore the former assumption of Fukada and Kawata is not applicable. Fukada and Kawata found that there is a rapid rise in the interfacial shear stress along the broken fibre interface near the crack. This results in a stress concentration factor for a single fibre break of 1.13 in the adjacent fibre, decaying to 1.0 for the fourth fibre away from the break. They claimed good agreement between their analysis and the experimental results of Armenakas and Sciammarella (72) (but there was a difference in the fibre volume fractions of about 15%). For the multiple fibre breaks the stress concentration factors were less than those of Hedgepeth (50).

Stress concentration values were calculated for three dimensional arrays using analytical and finite element methods by many workers. These calculated values were reported by Pitkethly (73). They all tended to be low because the load was considered to be shared beyond the nearest neighbours. Calculated stress concentration values for a single fibre break tend to lie between 1.02 and 1.2, values for neighbouring fibre breaks lie between 1.032 and 1.4, and values for five neighbouring fibre breaks have been calculated to lie between 1.05 and 1.75.
2.4.3 Failure modes

The fracture surface of uniaxial reinforced composites exhibits two types of topography which imply two different modes of failure. The first mode of failure can be characterised by a relatively smooth fracture characteristic of brittle fracture. The second mode of failure can be characterised by brush like or fibrillar fracture surface. Composite with the comparatively strong interface fail by propagation of a single crack across the section, with little or no longitudinal splitting. This type of failure is often considered to be brittle because it is associated with materials of low impact strength, whereas the brush failure is associated with materials of greater energy absorbing potential. In reality, both matrix and fibre are often brittle and all failure processes are brittle. Energy is absorbed by the formation of large fracture surfaces and by mechanical dissipative processes such as friction and fibre pull-out. The brittle mode occurs in systems with a high resistance to debonding. This would be expected when the interface bond is strong and the matrix has a high shear strength. Failure develops from a simple critical initiation point, an i-plet, and propagates across the entire cross-section. Little splitting would proceed failure but in the splitting mode, sporadic fracture initiation occurs but each crack is arrested when debondment decouple it from the remaining section. As the section becomes cumulatively weakened by these failures the process becomes unstable and a sequence of i-plet formation and splitting leads to final separation and failure. Further splits probably occur after separation due to the large amount of elastic strain energy released. Examination on the broken specimen would prove to be impossible to identify the failure initiated, or the exact path of the fatal crack. This behaviour would be expected of composites with weak interfaces and brittle resins of low shear strength. Idealised sketches of the two types of failures are shown in Figure 2.9. The implication of the model is that the first critical i-plet will
lead to total failure. In fact, it is usual to observe splitting, parallel to the fibres, during tensile testing. Bader (74) suggested this is due to shear cracks initiating from growing i-plets. This may happen in either sub-critical or post-critical phases of developments. This process decouples potentially critical i-plet from the remaining section for further i-plets and hence decoupling to occur prior to failure. Thus, a brittle type failure is delayed by effectively the tortuosity of the failure crack path.

Any variable which increases the stress transfer zone around a fibre break will tend to reduce the tensile strength and to increase the likelihood of splitting type failures. The relevant variables are lower shear modulus, lower shear strength, and reduced interface strength.

### 2.5 Conclusions

The literature indicates that the strength of brittle materials, such as carbon fibres, depends on the volume of stressed materials and the nature of the stress distribution. Both of these effects arise because brittle materials are flaw-sensitive. Flaw severity and distribution are generally statistical in nature. As the probability of finding a serious flaw increases with increasing material volume, large brittle materials tend to fail at lower stress level than smaller ones when subjected to the same tensile load, for example. This is the weak-link theory.

The broad stress distribution exhibited by these fibres is attributed to the pre-existing flaws in the bulk (volume) of the fibre, and on their surfaces. There is some work reported in the literature which aims to improve the understanding of the failure mode of fibres in the context of the weak-link theory. However, no work
up to date have incorporated this study using actual individual diameter for each specimen, rather than nominal values. Additionally, no reported work has specifically tested the weak-link property independent of the Weibull distribution (which is normally used to characterise the weak-link property).

This work attempts to study the effect of length and diameter (of four different gauge lengths specimens in three different environments) on tensile strength. The tensile strengths are compared and the weak-link theory is thoroughly examined using the Weibull distribution and the non-parametric test. The work also attempts to study the effect of length and diameter on the two classes of flaws: namely volume and surface.
Figure 2.1 Probability of failure of a fibre ($P_f$) versus stress showing the effect of fibre length, taken from Weibull (13).

Figure 2.2 Weibull plot showing the effect of changing the fibre length.
Figure 2.3 The ineffective length ($\delta$) of a broken fibre (a) and the length affected (PAL) in the neighbouring fibre (b).
Figure 2.4 Interfacial shear stress and fibre tensile stress distributions. Calculated using Cox's analysis for a single fibre in a matrix under an applied tensile load.

Figure 2.5 Gucer and Gurland's model for fibre-reinforced materials.
Figure 2.6 Brittle failure sequence (schematic). Failure sequence for brittle fracture mode. A number of sporadic fibre breaks form singlets (a), as the stress is increased (b), further singlets form and some grow into higher order i-plets. Eventually (c), one i-plet attains critical size and propagates across the section with no further increase in the applied stress.
Figure 2.7 Failure envelope (Batdorf (54)) obtained by plotting the natural log of $Q_i$ versus the natural log of stress. $Q_i$ is the number of cracks consisting of $i$ neighbouring fibre breaks.

Figure 2.8 Square lattice configuration.
Figure 2.9 Failure of a uniaxial laminate in tension. In the brittle mode (b) the fatal crack propagates completely across the section, with minimal longitudinal splitting. The alternative mode (c) is characterised by extensive splitting so that it is not possible to identify the path of the crack. Failure is by random fracture of the ligaments isolated by the splitting.
3.0 Experimental programme

The experimental programme for this work can be divided into three different sections. Firstly, the determination of the strength distribution of single carbon fibres in air (unsupported fibres) for which a single long length of fibre was selected from a bundle and divided into four different test specimens of gauge lengths. The work involved measuring the mean fibre diameter of each gauge length at three different points along its length and obtaining a mean value for each specimen. Each individual specimen was then tested under tension in order to determine its breaking stress. Secondly, the determination of the strength of single carbon fibres supported in epoxy-resin (embedded fibres) with the same gauge lengths as the unsupported fibres. The mean diameter of each specimen was derived from measurements along the length of the fibre, together with the strain to failure. Thirdly, in order to compare the strength values obtained for the unsupported and the supported fibres, impregnated bundles were tested under similar experimental conditions as used in the tests on the unsupported and supported fibres.

The techniques involved in these experiments are described below.
3.1 Materials

3.1.1 Fibres

The carbon fibre used throughout this work was obtained from a single 1/2 kg spool of high strength 'Celion 1000' in which there were 1000 filaments in a bundle. The fibre was manufactured by the Celanese Corporation Ltd. According to the manufacturer's published data, the fibre was sized and treated, and the tensile strength and tensile modulus are 3.1 GPa and 228 GPa, respectively, measured by the impregnated strand test method. The filament nominal diameter was 7.00μm with a circular filament shape.

A second fibre used for tests on unsupported single filaments was the Graf II As PAN precursor based carbon fibre manufactured by Courtaulds. According to the manufacturer's specifications, the fibre was treated and sized with a tensile strength of 4.2 GPa. Its nominal diameter is 7.5μm with a circular filament shape.

3.1.2 Matrix

All the matrix based specimens in this work involved the use of resin and associated curing agents obtained from Shell Chemicals. The formulations and cure schedule are recommended for a general purpose wet lay-up application. The base resin is a Bisphenol-A-based epoxy (commercially known as Shell Epikote 828) cured with Nadic Methyl Anhydride (commercially referred to as Shell Epicure N.M.A.) and an amine catalyst (Shell Epicure K61B). The formulation details and cure temperatures are given in Table 3.1.
3.2 Preparation of test specimens

3.2.1 Removal (extraction) of a single fibre from a tow

The following procedure was followed meticulously and consistently for the preparation of single fibres for testing, whether tested in air or embedded in an epoxy-resin matrix.

A single 300mm long tow or bundle of fibres was cut from either a spool of Celion 1000 or XTN carbon fibre and carefully submerged in a glass bath half-filled with acetone for one hour. The glass bath measured 500 x 200 x 300mm in relation to its length, width and depth, respectively. A glass rod was used to stir gently the acetone in the glass bath without making any contact with the fibres. This gentle motion assisted in removing the epoxy size and separated the fibres from the bundle.

3.2.2 Unsupported single fibre specimen preparation

A single long fibre was selected from the tow with careful use of a pair of tweezers and mounted on a multi-window card (Figure 3.1). The multi-window card was specially designed for this work (75) at the Materials Science and Engineering Department, University of Surrey. This technique differs from the usual way of testing where a single fibre is first drawn from a tow and later mounted on a card with one window (the likelihood then is that every test is on a different fibre).

The new multi-window card technique consisted of four windows of 5, 12, 30 and 75mm gauge lengths, where a single long fibre provided four different test
specimens, thus ensuring that any difference in behaviour due to variations in fibre diameter, for example, can be investigated. This technique allows the weak-link principle to be thoroughly examined when comparing the strength data for the four specimens. The four gauge lengths were selected for testing so that they fall at approximately equal intervals on a ln (length) plot. The extracted long fibre was carefully placed along the axis of the card and glued in position with a quick setting cyano-acrylate adhesive applied at the points indicated in Figure 3.1. The multi-window card was then cut with a pair of scissors on the dotted lines, into four separate test cards providing the required four gauge length specimens. All the specimens were carefully labelled and stored away separately according to lengths in a compartmentalised box ready for testing.

It is important to note that some fibres broke between their extraction from the acetone bath and testing. This problem was difficult to avoid given the fact that single filaments are very brittle with small diameters (6-10 µm). Only those fibres that were successfully extracted and glued on the cards were used for testing. The unsuccessful fibres, i.e. those that broke before testing were recorded as having zero strength.

3.2.3 Supported (embedded) single fibre specimen preparation

In order to prepare these specimens, a rectangular multi-cavity silicon rubber mould having the four required gauge lengths was made from a master steel template cast. A silicon mould release agent was sprayed on the master steel cast and 100g of Dow Corning 3110 RTV silicon rubber was mixed with 1ml of catalyst 4 (a curing agent of Tin (11) Octoate) in a beaker. The mixture was degassed and poured quickly into the master steel cast. The silicon rubber
mixture was allowed to cure for two hours at room temperature before the silicon rubber mould is removed from the cast. Each silicon rubber mould comprised four different cavities for the four gauge lengths required, Figure 3.2. Approximately twenty rubber moulds were prepared this way.

To make the supported single fibre specimens, a long length of fibre (300mm) was aligned along the axis of the prepared silicon rubber mould after its removal from the acetone bath. This was achieved by use of a small metal frame to which the fibre was attached by a quick setting cyano-acrylate glue. The frame was lowered into the mould and the fibre bonded to two raised stubs of rubber at either end. These stubs were designed to be exactly in the centre of the mould. The fibre was then cut beyond the stubs and the frame removed. In this way the fibre is firmly gripped under a light tension.

The resin was weighed and mixed in accordance with the formulation details shown in Table 3.1. The resin was degassed at 70°C for 15 minutes and was introduced carefully into the mould from either end of each cavity using a syringe. The resin flowed easily into the rubber mould cavities displacing air. Excess resin was removed from the ends with an absorbant tissue. The entire rubber mould was then placed in a preheated oven and left at 100°C to cure for three hours. After curing, the coupons were removed from the moulds and the ends of the coupons were trimmed with a diamond cutting wheel to produce the four different gauge length specimens. The moulds could be re-used up to five times. The specimens were finally post-cured for three hours at 150°C under a small weight. This was to ensure that they remained flat. All the prepared specimens were carefully labelled and safely stored away in a box until required for testing.
3.3 Diameter measurements of single fibres

Diameter measurements were made prior to examining the tensile strength of each specimen. A circular fibre cross-section was assumed based on the manufacturer's literature and only the fibre diameter need, therefore, to be determined. Localised variation in the fibre diameter along the fibre length could lead to changes in measured strength distributions and it was decided to make a systematic examination of fibre diameter along the length of each specimen. In works by other authors (26, 73, 76) a nominal fibre diameter is usually used for all specimens, rather than the measured values which are used in this work.

Two techniques were used to measure the fibre diameter. A laser diffraction method (77, 78) was used to measure the diameters of the unsupported single fibre specimens. This involved the interpretation of a diffraction pattern from an incident laser beam on the fibre. For supported fibres, a pre-calibrated Watson Image-shearing eyepiece (W.I.S.E.) was used on an optical microscope for direct measurements of diameter. Both methods allow a rapid determination of the mean diameter for each specimen.

The diffraction technique was initially tried for measuring the diameter of the supported fibres before casting them in an epoxy-resin. However, this was found to be difficult, since the fibres were first required to be glued onto the multi-window cards for the diameter measurements to be made then "unglued" so that they can be embedded in the epoxy-resin. This procedure was found to be very difficult and led to many fibres breaking at the stage of ungluing. In order to avoid this, it was important to use a technique where the diameters could be measured after the fibres were cast in the epoxy-resin (i.e. after the specimens were made). Hence the use of the W.I.S.E technique.
3.3.1 Diameter measurements of unsupported single fibres

Laser diffraction is an established technique for measuring small diameter filaments, such as single carbon fibres. The interpretation of the laser diffraction pattern of a fibre is as follows. The pattern created by an infinitely thin fibre of width, \( d \), is the same as that of a slit of the same width. It is a series of maxima and minima on either side of a central, or zero, maximum as indicated in Figure 3.3. The separation, \( x \), between the first corresponding pair of minima is given in the Fraunhofer approximation (79) by the relation

\[
x = \frac{2\lambda s}{d}
\] 3.1

where \( s \) is the screen-to-fibre distance, \( \lambda \) is the laser wavelength (633\( \mu \)m) and \( d \) is the fibre diameter.

In practice, to measure the diameter of a fibre, a convex lens of a specified focal length is needed so that the laser beam (1mm diameter) can be focussed on the screen. The fibre is mounted in the beam at some arbitrary distance from the lens, Figure 3.4. The fibre diffracts the light into a series of narrow bright and dark fringes. The diameter of the fibre is given by (80).

\[
d = \frac{n\lambda f}{x}
\] 3.2

where \( d \) = the fibre diameter
\( \lambda \) = the laser wavelength
\( f \) = focal length of the lens (i.e screen-to lens distance)
\[ x = \text{the measured distance from the central maximum to the nth order minima.} \]

It is more accurate to measure \( x \) the distance corresponding to the fourth minima by measuring the distance corresponding the dark fringes on either side of the direct beam and dividing by two.

A disadvantage in using this technique arose because the diameter of the laser beam is much larger than the diameter of the fibre. In fact it is approximately 130 fibre diameters. This means the laser beam diameter will mask the true fibre diameters with the effect of the measured fibre diameter being averaged out over the length of 1 mm. Ideally, the use of a smaller laser diameter would be preferred which would render the technique as a "point probe" method, with no averaging effect, but this was not available.

There is no known method to date in the literature which attempts to use a smaller laser beam than 1 mm in the measurement of fibre diameter. Previous workers have overlooked the need for this (in the context of the problem associated with the averaging effect that a large beam has on the measured fibre diameter). Many, if not all, Helium-Neon laser sources in research laboratories have a minimum beam diameter of 1 mm.

A modification to the method illustrated in Figure 3.4 has been devised for this work. This involved the incorporation of an adjustable slit, specifically designed to reduce the diameter of the laser beam. The principle behind the technique is illustrated in Figure 3.5. A 2 mW Helium-Neon laser with a beam of 1 mm diameter was mounted on an optical bench and aligned with an adjustable slit and with a white screen placed 100 cm away from a convex lens (100 cm focal length),
Figure 3.4. The aperture of the slit was reduced in size until it was virtually closed to ensure a minimum beam to pass through it. A Fraunhofer diffraction pattern was produced on the screen comprising maxima and minima. The fourth minima on either side were measured from the central, or zero, maximum, and the diameter of the slit was calculated using equation 3.2. The value obtained was 0.08mm which was the reduced laser beam diameter used to measure the fibre diameters. A fibre specimen was mounted horizontally on the optical bench between the lens and the adjustable slit and a vertical diffraction pattern was produced on the screen, Figure 3.5. The fourth minima of the fibre fringe diffraction pattern was measured from the central maximum and the fibre diameter was calculated using equation 3.2. Each fibre specimen was measured at three points along its length (top, bottom and middle) and the mean value was calculated from these three measurements.

3.3.2 Diameter measurements of supported single fibres

Optical microscopy using a shearing eyepiece was used for single fibres embedded in epoxy-resin matrix. The technique was chosen for embedded specimens since diameter measurements can be made on the fibre that was to be tested for mechanical properties without having any effect on the specimen.

The image shearing is achieved by means of a dichroic beam splitter giving colour differentiation of the beam, Figure 3.6. The shear is proportional to the movement of the mirror and rotation of the setting head. The action of image shearing involves both components of the sheared image moving away simultaneously and by an equal amount from the point of zero shear where a normal image is seen. Rotation of the setting head produces two images of the
specimen - one red, the other green, Figure 3.7. The relative position of the two images is controlled using a vernier screw which adjusts the position of the beam splitter. The technique was calibrated using polystyrene spheres prepared under conditions of zero gravity in the space shuttle Columbia. Certified by the National Bureau of Standards, they possess a uniformity of roundness and diameter, 9.89\(\mu\text{m}\), with a standard deviation of only 0.9%.

The specimen diameter was measured by shearing its images until the edges of the two components images just “touch” each other, the extent of the setting to achieve this was then noted. The fibre diameter is then calculated from the difference of the two vernier readings (81). Three diameter readings were taken at different points along the length of each specimen (similar to the unsupported fibres) and the mean value was calculated.

3.4 Strength measurements of single fibres

The breaking loads of the unsupported and supported fibres were measured in the following manner.

3.4.1 Strength measurement of unsupported single fibres

A TTM bench top Instron tensile testing machine was used, fitted with a type A load cell. Each fibre in the window card was mounted carefully in the grips of the machine. The lower grip was the standard, light weight pneumatic grip and the upper grip was specially designed to be of low mass and to be able to accommodate realignment on fibre tensioning. With the specimen held firmly in
the grips, the sides of the card were cut using a pair of scissors, disturbing the
card as little as possible and making the fibre into the only load-bearing element.

The fibre was tested at an extension rate of 0.1 mm/minute, using a 50g load cell.
The ultimate load and extension to failure were recorded autographically. Each
specimen’s failure stress was calculated from its maximum load on the
load/extension plot and its cross-sectional area, based on its measured mean
diameter.

The testing machine was calibrated using a series of gram weights, and the
estimated error in load measurement was 0.9%.

3.4.2 Strength measurements of supported single fibres

When tested as an unsupported single filament, a fibre is effectively loaded
directly in tension. Once embedded in a matrix, however, it is loaded indirectly by
shear stress transferred at the interface. As a consequence, if the fibre is well-
bonded to the matrix then only a small length of fibre adjacent to the break is
unloaded and further fractures may occur in other parts of the fibre at higher
applied load. In this work, only the first fracture was measured, since this may be
regarded as the weak-link of the fibre. The strengths corresponding to these first
failures in embedded specimens can be compared directly to the strength data of
unsupported fibres.

First fractures for the embedded specimens were obtained by loading each
sample in a conventional Instron testing machine. The specimens were loaded in
an Instron 1195 screw-driven machine fitted with standard wedge action grips. A
cross-head speed of 0.1 mm/minute was applied to give a similar strain rate to that used for the unsupported single fibre tests and the experimental set up is illustrated in Figure 3.8.

The strain on the sample was measured using an electrical resistance strain gauge bonded to the specimen's surface and linked to a “Vishay” strain gauge indicator. Specimens were illuminated from behind using a polarised light source and observed with a camera fitted with a polarising filter (acting as an analyser for the illumination system) and a photograph was taken of the first fracture of each specimen tested. The stress distribution pattern in the fracture zone showed up as a birefringent pattern due to the photoelastic behaviour of the resin (Figure 3.9).

3.5 Impregnated tows

Resin-impregnated tows of Celion fibre were produced using an improved in-house designed equipment where motor driven rollers gently pull the tow from the spool (which rests freely on a specially designed “cradle”) through a continuous impregnating and curing process (Figure 3.10). The aim of the technique is to produce a partially cured, rigid impregnated tow on a continuous basis, so that a large quantity of good and consistent quality material can be made. The final product was cut and prepared to the required gauge lengths.

The tow passes through a resin impregnating bath which was previously prepared in accordance with the formulation details shown in Table 3.1. The temperature of the resin in the bath was maintained by using a large coiled heater around the resin impregnating bath. The tow emerged out of the bath and
through a PTFE die (0.68mm diameter), which was also heated to maintain the same resin viscosity and to ensure that the resin-impregnated tow could be drawn through the aperture of the die without the resin cooling. The impregnated tow then continued through a vertical oven (1m long), where it was cured continually (at approximately 100°C) beyond its gel-point. The electrical coils around the vertical oven were specifically adjusted to improve the temperature uniformity within the oven. A continuous flow of a rigid impregnated tow emerged from the top of the vertical oven using pulling rollers and the tow was cut into approximately metre lengths. The speed at which the impregnated tow travelled through the vertical oven was 2mm/minute. This speed was found to be adequate to ensure that the resin gel-point was reached in the time it took the impregnated tow to travel along the length of the oven. It was also found that this speed ensured improved dimension control, due to the resin having less time to flow out of the tow at the curing temperature.

The 1m long impregnated tow specimens were measured and cut to the required lengths for tensile testing. Each cut was made longer than the gauge length required so that a braided glass-fibre/epoxy-resin end tags could be fitted. In early experiments, end tags were made by soaking glass fibre sleeves in epoxy-resin and sliding them onto the ends of the specimens. The end tags were cured in an oven using moulds to shape the resin at the end of the tag nearest to the gauge length. However, these specimens were found to fail at the region where the end tag joined the tow. To overcome this difficulty the end-tagging method to be found in the British Tensile Standard (number BS 2782 Part 3 Method 320A) was used as a guide to the geometry needed to redistribute the stress so that failures occur within the gauge length. This involved the use of braided glass-fibre end tags and a resin filet to distribute the stress at the tag entry point. Figure
3.11 shows a schematic diagram of the impregnated tow specimen. All specimens were finally cured and post-cured according to the cure schedule indicated in Table 3.1. The mean diameter of each specimen was measured using a travelling microscope.

Impregnated tows were tested on an Instron 1175 tensile testing machine, at a cross-head speed of 0.5mm/minute. Load/extension data were graphically recorded as part of the test for each specimen. Comments on any notable failure or events were recorded for each test.

3.6 Results

3.6.1 Results for the unsupported single fibres

All the Celion fibre diameter measurements with respect to their standard deviations are shown on Table 4.1. It shows five different data sets, where each data set came from one single tow that is originally cut from the fibre spool. Therefore, the five different data set of fibres come from five different tows. Table 4.2, on the other hand, shows the diameter measurements for the XAK fibre with respect to their standard deviations. Only one tow of fibre was cut for testing and is shown as one data set, Table 4.2.

3.6.2 Results for the supported single fibres

Table 5.1 shows the mean diameters and standard deviations of two data sets and when combined become one global data set. A data set consists of a
collections of single fibres extracted from a single tow of fibres during the preparation campaign.

3.6.3 Results for the impregnated tows

Table 6.2 shows the mean diameters and the standard deviations for all the gauge length specimens as one global data set. The failure of all the specimens was instantaneous and occurred without any warning. There was no sign of damage prior to failure either in the load/extension curve, visibly or audibly in the specimen. There was little splitting of the impregnated tows observed at failure.
Table 3.1 Resin formulation and cure.

- **Resin type**: Shell Epikote 828 epoxy
- **Cure regent**: Shell Epicure N.M.A.
- **Accelerator**: Shell K61B

**Formulation**: Resin 100 parts vol.
- N.M.A. 60 parts vol.
- K61B 4 parts vol.

**Cure schedule**: Degassed at 70°C for 15 min.
- Cured at 100°C for 3 hours
- Post-cured at 150°C for 3 hours.
Figure 3.1 Schematic diagram of the four-window card used for mounting the single fibre specimens. The card is cut at the dotted lines to form the four gauge length test specimens.
Figure 3.2 A diagram of the multi-cavity silicon rubber mould used for the manufacture of supported (embedded) single fibres specimens.
Figure 3.3 The diffraction pattern of a laser in TM mode by a fibre, taken from Perry (79).

Figure 3.4 A schematic of a laser diffraction technique for measuring the diameter of a single carbon fibre specimen mounted on a window card.
Figure 3.5 A modified laser diffraction technique using an adjustable slit specifically designed to reduce the laser beam. The technique follows two steps: a) reducing the laser beam and measuring its diameter; and b) the incorporation of the fibre specimen to measure its diameter using a), the reduced beam diameter.
Figure 3.6 Watson image-shearing eyepiece.
Figure 3.7 The principle of image-shearing from zero shear (a) through half shear (b) to full shear (c).
Figure 3.8 The apparatus used for lighting and photographic system for embedded single fibre tensile testing.
Figure 3.9 A typical photoelastic effect showing the birefringence pattern around the broken ends of a fibre.
Figure 3.10 Apparatus for the continuous impregnation of tow.
Figure 3.11 Sample schematic for a typical impregnated tow test-piece.
CHAPTER FOUR

4.0 Strength of unsupported single fibres

4.1 Introduction

The fundamental assumption made in the statistical analysis of the strength of brittle solids, such as carbon fibres, is the weak-link principle (section 2.1.4). This long held hypothesis assumes the strength of materials under load to be determined by the weakest elements of the material; all elements act independently and equally likely to be the cause of failure under a specific load. Therefore, it follows from this assumption that a random (Poisson) distribution of flaws of variable intensity are distributed on the fibre surface or throughout its entire volume. The Weibull distribution is commonly used in the analysis of experimental data because it embodies the weak-link principle. The distribution describes the surface area or volume effect on the fibre's breaking stress. It also carries the implication of a strength-to-length relationship quantified by the weak-link scaling, where a shorter length of fibre will be statistically stronger than a longer length. It is normally assumed that the effect of diameter variability on the breaking stress is negligible and therefore, a nominal failure stress is often calculated using the mean diameter of the fibre (26, 73, 76).

A new experimental procedure (section 3.2.2) was devised specifically to test the weak-link property on two different types of carbon fibre under tension. The experimental programme involved extensive measurements of individual fibre diameter with different gauge lengths prior to testing. This procedure allows the study of the diameter effect as well as the length on the breaking stress of fibres.
Hence the important concept of size effect.

The experiment enables the use of the Weibull model to assess the weak-link principle. By obtaining a much better value of the actual diameter of the failed fibre, the true stress can be calculated and the influence of surface area and/or volume on the Weibull statistics can be discovered. Furthermore, the procedure permits the use of new statistical models to assess the agreement with this principle without recourse to Weibull statistics.

4.2 Results

A batch of data in this work is defined as a set of observations (comprising four gauge lengths) made from a collection of single fibres originating from a single tow prior to testing. The experimental preparation of these fibres is outlined in sections 3.2.1 and 3.2.2. The batch includes those fibres that broke before testing, although for these zero strength is recorded.

The work in this chapter involved the use of two different carbon fibres. In total five different batches of data were collected for the Celion fibre and only one batch for the XM fibre. Therefore, the number of observations for the Celion fibre far exceeded those made for the XM fibre. This is because the original intention was to work on the Celion fibre only. However, early analysis of the data produced surprising results that were powerful enough to warrant new experiments to be performed in order to obtain satisfactory explanation for their behaviour. The direction of the work is outlined below.

Three batches of data were originally tested for the Celion fibre. Early analysis of
the data collected for these batches indicated a strong deviation from the Weibull model and a departure from weak-link behaviour (section 4.2.4). However, the question of bias, due to the order of testing the specimens was raised, since all three batches were tested in a defined order of gauge length increase. In order to answer this question of bias, it is important to test a randomised batch of fibre under the same experimental procedure to see if the results were reproducible. The data collected as will be shown in section 4.2.4, referred to as the random '88 (collected and tested in the year 1988) were analysed and produced similar results to the three non-random batches tested earlier in that same year.

Another batch of non-random fibre was tested again in a random order (random '92) after a period of four years. This was important in order to address the question of time dependence. The results as will be presented in section 4.2.4, produced similar behaviour to those early ones.

Regardless of the tests of bias and time dependence, the results obtained remained similar that they consistently deviated from the Weibull model. It was then decided to test the experimental technique employed in the Celion programme against the standard fibre (Courtauld, XÅ) then in use in the department. The results produced for the XÅ fibre (section 4.2.4) conformed well to the Weibull model giving good compliance to the weak-link principle and hence eliminated the question of technique dependence.

4.2.1 Fibre diameter

Diameter measurements for five different batches of data sets, performed on the Celion fibre at the four gauge lengths of 5, 12, 30 and 75mm respectively,
including their combined data sets as one global data set, are shown in Table 4.1 together with their means and standard deviations. Similar observations for the XAs fibre are presented in Table 4.2.

The global (total) number of observations obtained for the Celion fibre, indicated in Table 4.1, is 160, 161, 147 and 148 tests for lengths 5, 12, 30 and 75mm respectively where each of these four gauge lengths was cut from a single long length of fibre (without breaking any of these lengths during the preparation procedure). In other words, the difference in the total observations obtained for each gauge length was due to some specimens breaking prior to testing. This is important since the fragile nature of the fibres meant that some of the gauge lengths break before testing. Similarly, Table 4.2 indicates the number of observations made for the XAs fibres comprising 24, 25, 24 and 23 for each respective gauge length.

Table 4.1 indicates the standard deviations about the mean diameters for a collection of n observations per gauge length within a particular data set. Three observations can be drawn from the Table. First, a similar mean diameter value is observed for all the data sets irrespective of the gauge lengths. Second, the standard deviation within each gauge length points to a substantial variation in the mean diameter. This behaviour is observed in all the five data sets for all the gauge lengths. The results, therefore, suggest that the diameter of individual specimen (irrespective of gauge length) cannot be neglected. As this varies considerably and may be relevant in terms of the real applied stresses. On the other hand, diameter variation of individual specimen were not previously considered (26, 73, 76). Third, the standard deviation of their respective mean diameter increases from one gauge length to the next for each data set. This behaviour is common to all data sets and suggests a strong effect on the
variability of the measurement of diameter as the gauge length increases. In other words, as the fibre length increases, the measured variability of the diameter becomes more pronounced. This is an important finding since the experimental technique of selecting a long length of fibre and dividing it into four separate entities of four different gauge lengths imply that these four specimens ought to be truly similar in all respects, apart from length.

Table 4.2 indicates the standard deviations about the mean diameters of the four gauge lengths for the XAs fibre. Here the variation of diameters between fibres is much lower than those found for the Celion fibre. Moreover, the variation remains the same as the gauge length increases, resulting in the uniformity of diameter, which does not enable its effect to be tested. Therefore, the assumption of uniform diameter for each long length of fibre may be relevant for the XAs fibre.

4.2.2 Fibre strength

Fracture stresses, \( \sigma \), were calculated from the maximum load on the load/extension plot for each single specimen, using its own unique mean diameter value, and hence cross sectional area. The latter was derived using the assumption that a fibre has a circular cross section. Stress values were thus calculated for each specimen at every gauge length for each batch of data set for both Celion and XAs fibres.

4.2.3 Influence of diameter

Figure 4.1 shows a representative plot of diameter versus stress at failure for the
Celion fibre. This plot clearly indicates a strong tendency for higher breaking stresses to be associated with thinner fibres; a tendency which decreases with increasing fibre length. It is clear from this plot that shorter fibre lengths have higher fibre breaking stress, whereas higher lengths do not show strong diameter dependence. This behaviour is reinforced in Figure 4.2 for all the five data sets. Wolstenholme (28) analysed a subset of these data but failed to identify this behaviour.

4.2.4 Influence of gauge length/Weibull analysis

In section 2.1.3, it was mentioned that the strength distribution of fibres is assumed to comply with the two-parameter Weibull model (equation 2.7a) to generate a characteristic stress, $\sigma_0$, and a Weibull modulus, $W$, values for each data set.

Weibull plots of $\ln \ln (1/P_f)$ versus $\ln (\sigma)$ were constructed for all the data sets of both types of fibre, and a linear regression was carried out to fit a straight line to the data. The plot should give a straight line of slope $W$, the Weibull modulus, and intercept $\sigma_0$, at $\ln \ln (1/P_f) = 0$, if the Weibull expression is obeyed. This plot is known as a Weibull plot of the first type.

The principal interest in this plot is to see whether the weak-link hypothesis holds for the data under the assumption of a random distribution of flaws of variable intensity. If this assumption is correct, one would expect the parameter $W$ to be the same for each data set but $\sigma_0$ should vary for each according to the weak-link scaling equation:
\[ \sigma_o = \alpha L^{-1/\nu} \quad 4.1 \]

where \( \alpha \) is a constant representing the characteristic stress for unit length \((1 \text{ mm})\) and \( L \) is the gauge length in millimetres. The derived value of \( \alpha \) should, therefore, permit the estimation of the characteristic stress from the experimental data collected at different gauge lengths.

The plots of this type were constructed for the five different data sets of Celion fibre and are shown in Figures 4.3 and 4.4. Figure 4.3 represents the three non-random data sets; Figure 4.4 is the Weibull plots for random '88 and random '92 respectively. Figure 4.5 shows the global Weibull plot for all these five data sets and its parameters estimates are shown in Table 4.3.

The results clearly indicate no significant difference between the data sets. However, none of the data sets are in accord with the Weibull model. This conclusion is reinforced in the global plot of Figure 4.5 and in Table 4.3. However, the parameters estimates presented in Table 4.3 do not fit equation 4.1.

A similar Weibull analysis for \( X \) fibre is shown in Figure 4.6 and Table 4.4. However, this fibre shows a very good compliance with the Weibull model and fits equation 4.1. Therefore, the assumption of variable intensity flaws with a random distribution is valid here. This may be because the range of values of diameter within the \( X \) set is so small, that the only effect is length. Celion fibre, on the other hand, may not fit the Weibull model because there are both diameter and length effects.

Cumulative distribution Weibull plots were constructed to give at a glance the
range of breaking stresses and the distribution of values as well as their mean and median values for all the five Celion data sets. These plots were constructed by a computer programme in which stress was determined for each individual specimen from the force measured by inputting the diameter (mean of three readings) for each fibre in turn. The values were then ranked and assigned a probability of failure. Figures 4.7 and 4.8 show the combined data for both random and non-random data sets respectively, whereas Figure 4.9 shows the distribution of all data sets combined together. These plots indicate no significant difference between the distribution of the random data to that of the non-random ones. Therefore, the order of testing the specimens is clearly not an important parameter in influencing fibre failure. The fact two random data sets were identical in distribution and performed under similar conditions, separated by a four year period (Figure 4.4), indicate that the fibre has remained resistant to any possible oxidation or degradation effect on its strength.

Since the experimental technique used in testing both fibres is the same and the fact that Celion fibre consistently deviates from the Weibull model in comparison to the XAr fibre which complies with it, proves that any doubt in the experimental technique used can be eliminated.

### 4.3 Discussion

Previous workers tested fibres by selecting a fibre from the tow and arbitrarily allocating the gauge length. The likelihood of this is that every test in the set is on a different fibre. However, this work was performed under a new experimental technique specifically designed to examine the diameter effect on the strength of fibres.
In Table 4.1, the values of the diameter ranges widens as the gauge length increases, which is displayed by the standard deviations. The same Table also indicates that all the data sets have a similar mean diameter, but the standard deviation of the first data set was found to be comparatively higher than the others. This was the first data set tested and hence some experimental error may have been introduced in the process of perfecting the laser diffraction technique when measuring fibre diameters. Other data sets of the Celion fibre indicate no significant differences with regard to their mean diameters and standard deviations.

Table 4.2 indicates the values of the mean diameter range being the same regardless of the gauge length as shown by the uniformity of the standard deviations. The XA fibre has a slightly lower diameter variation than the Celion fibre. This confirms the manufacturer's published data (section 3.1.1).

Ideally, the diameters of individual fibres from the same spool should have similar standard deviations. However, the large diameter variation of individual specimens displayed by the fibres, i.e. the diameter from fibre-to-fibre, could be due to the nature of the fibres. At a manufacturing level, carbon fibres are made from tows of 1000-12000 PAN filaments and converted to graphite by thermo-chemical-mechanical treatments. They are then surface treated and sized with a thin coating of resin. Therefore, one can expect the fibres to contain flaws originating from the impurities or Imperfection in the precursor and from abrasion during the processing cycle. Both the Celion and the XA fibres were received from their respective manufactures as 100% surface treated and sized (section 3.1.1).

The large diameter variations observed between fibres could probably originate
from diameter variation in the precursor fibre due to difference in hole sizes in the spinnerets, and possibly also from the difference in the degree of chemical conversion between fibres which lie on the outside of the tow, and are thus more exposed to the oxidising atmosphere in the first stage of conversion than those in the middle of the tow. It is also likely that the degree of abrasion damage will vary from fibre-to-fibre within the tow. All these factors will increase the intrinsic strength variability of the fibre.

Figure 4.2 indicates that there is a tendency for higher breaking stresses to be associated with thinner fibres, a tendency which decreases with increasing fibre length. Stress being force per unit area tends to imply independence of diameter, but diameter has been shown not to be eliminated in this way. Since a larger diameter means a larger stressed volume, thus an increased probability of encountering a more severe flaw, the volume effect was the first to be examined. Consideration of the data shows that the volume effect is not straightforward; the diameter is much more important for the short fibre strengths than for the long fibre ones.

Figures 4.5 and 4.6 respectively show the Weibull plots for Celion and XAs fibres. It is clear from these plots that Celion fibre does not conform to the Weibull model but the XAs fibre complies very well. These conflicting findings for the two types of fibres pose a challenging question as to why carbon fibre should behave so differently under identical experimental conditions. The findings suggest that their failure mechanisms may be different. The Weibull model used in analysing failure mechanisms has the implicit assumption that the cause of failure arises from one essential flaw (i.e. the weak-link). This model could be applied to the XAs fibre, but the Celion fibre clearly shows a deviation from this assumption and requires further analysis.
A close examination of Figure 4.5 shows that the 12mm gauge lengths behave in a pronounced bi-modal behaviour. The high stresses shown in the upper part of the curve behave like the 5mm gauge length and the low stresses displayed in the lower region behave similar to the 30mm length. This implies that 12mm is the critical gauge length at which diameter becomes important. Therefore, the longer lengths (30 and 75mm) indicate failure to be length dependent and the 5 and 12mm gauge lengths are both diameter and length dependent. These two classes of behaviour need to be noted and will be discussed in detail later.

An alternative Weibull analysis, commonly known as a Weibull plot of the second type, was performed on the Celion fibre in order to obtain an independent measure of the Weibull modulus. This analysis is based on a plot of ln (mean strength) against ln (gauge length). Mean strength, as mentioned earlier in section 2.1.3, can be calculated in many ways. Equation 2.14 indicates the relationship between mean strength and the characteristic strength multiplied by a Weibull dependent length and a gamma function. The gamma function will be constant, if ln (σ_L^{-1/W}) is plotted against lnL, the gradient should be -1/W, hence allowing the weak-link estimation to be made directly from the plot.

It is worth noting that the parameter σ_L generated in the Weibull plot of the first type can be directly used for these plots, since they are in fact the length dependent σ_L . L^{-1/W}. This was completed for both Celion and XAS fibres. In theory the estimate of W by both the first and the second type Weibull plots should be the same.

Plot of the second type Weibull distribution for Celion and XAS fibres are shown in Figure 4.10, which yields W to be 3.91, for Celion fibre, compared with 4.75-9.72
as determined from the conventional plot. This suggests that the fibre strength cannot be accurately described using the single-mode Weibull distribution. The X4 fibre, on the other hand, yields \( \lambda = 4.71 \), which is in close agreement to the values, 5.05-5.36, derived from the conventional plot, suggesting compliance with single-mode failure.

### 4.3.1 Further analysis and discussion of the Celion fibre results

It is clear from the Weibull analyses performed so far on this fibre, that the Weibull model is not obeyed. This is not surprising since the Weibull model will only be obeyed correctly if all the specimens are geometrically identical, precisely because size effects are known to operate as discussed in section 2.1.4. The analysis in section 4.3 points to diameter being an important covariate in so far as affecting breaking stress of a fibre. This effect of diameter may be bi-modal, possibly due to its wide variability from one gauge length to another. It will be valuable to investigate diameter variability further in order to understand its effect on breaking stress for each gauge length, using a modified approach of the Weibull model.

In order to address the effect of diameter, a global plot of frequency distribution for the diameters of all five data sets is performed as shown in Figure 4.11. It is possible that the whole data set can be represented by four normal distributions. These four distributions cover four different mid-values of four diameter ranges, \( D^* \), namely 6.00 <-> 7.25\( \mu \)m; 7.26 <-> 7.75\( \mu \)m; 7.76 <-> 8.75\( \mu \)m; and 8.76 <-> 9.50\( \mu \)m.
If a plot is performed, for example, on the global data set pertaining to the 5mm gauge length using these four different mid-values of four diameter ranges the plot in Figure 4.12(a) is obtained. It can be clearly seen from this plot that the whole data set for this gauge length has been divided into four separate subsets of breaking stresses according to the four $D^*$. As a result, four different Weibull distributions are obtained. This procedure was completed for the other three gauge lengths, thus generating the rest of the plots shown in Figure 4.12.

Figure 4.12(a) indicates the characteristic strength as being dependent on diameter for the 5mm gauge length; since it shows four clear Weibull distributions the characteristic strength reducing significantly with the increasing of $D^*$. The effect of characteristic strength reduction with respect to $D^*$ is also observed for the 12mm gauge length (Figure 4.12(b)). However, the mid-value of diameter range at 7.76<-> 8.75μm indicates a clear double kink, thus implying a possible mixed effect. The effect of characteristic strength reduction on the Weibull distribution lines is significant for the 30mm length (Figure 4.12(c)), thus implying that diameter effect is not particularly important at this gauge length. The 75mm gauge length (Figure 4.12(d)), on the other hand, clearly shows the four characteristic strengths to be virtually the same, indicating very strongly that diameter effect is not a factor of importance at this gauge length.

This analysis concludes that diameter produces different effects with respect to the different gauge lengths. Therefore, it is an important parameter when considering fibre breaking stress. Its importance will be discussed again in a later section.
4.3.2 Non-parametric test

Departure from the Weibull model is not necessarily a test of departure from the weak-link property, despite the former being commonly associated with the latter. It is important to mention that a weak-link behaviour does not require the Weibull distribution to hold. In other words, one can get a weak-link behaviour where the data can be consistent with another distribution other than Weibull. Wolstenholme (28) recognised the need to test this basic property and proposed a direct test of the weak-link principle based on a simple non-parametric test (section 2.1.5). She used a subset of earlier data pertaining to this work and examined the weak-link principle under this new test. On the other hand, in this work all sets of data for the Celion fibre were used to examine the non-parametric test. In addition, XAS fibre was used for comparison purposes.

The test is a distribution free method, which does not depend on the stress distribution of fibres but based on the order of failure of the four specimens of different gauge lengths cut from the same fibre and stressed under identical conditions. The basis of the test assumes that the fibres are similar in all respects except lengths. Table 4.1 shows that a complete data set (minimum successful tests) for data set one is 39, data set two is 33, data set three is 34, data set '88 is 18 and data set '92 is 19, which gives a global (total) of 143 as one complete data set. A complete data set is the minimum successful tests obtained for each gauge length. Similarly, Table 4.2, shows the complete data set for the XAS fibre is 23.

Table 4.5 shows the orders of failure for all probabilities together with theoretical and observed percentages. It is expected that long fibres are on average weaker than shorter ones, due to their higher probability of encountering a fatal flaw. Therefore, the most likely order of failure is 75, 30, 12 and 5mm respectively. It
can be seen from Table 4.5 that all the five Celion data sets demonstrate similar properties in failing well in excess of the theoretical value of 27.71% for this particular order. Even when one takes into account those fibres that failed prior to testing (Incomplete data set) and assumes their failure order is other than 1 2 3 4; the overall percentage failing in the order of 1 2 3 4 would still be 64%, which remains a long way away from the theoretical value. The evidence, therefore, is overwhelmingly against the weak-link principle. These findings have considerable implications to the important question of strength prediction.

A similar analysis to the above was applied to the XAS fibre, Table 4.6. This fibre conforms very well to the weak-link hypothesis. 23.81% of the fibres failed in the order of 75, 30, 12 and 5mm respectively. This is close to the predicted value of 27.71%.

It is not surprising, therefore, that the non-parametric test does not fit the Celion data, as the length/diameter plot of Figure 4.2 shown earlier gave evidence of a different distribution at low and high stresses due to the different effects of diameter on breaking stresses. It is important to mention that the non-parametric test uses only one-parameter, namely length, since it assumes no diameter effect. Therefore, since diameter is found to be relatively uniform for the XAS fibre, this test adequately represents this fibre. Strength for Celion fibre, on the other hand, is shown to be influenced by two parameters, i.e. diameter and length, since both diameter and length are important in determining the breaking stress. However, Wolstenholme (28) who used a subset of data from this work could not derive from her observations that diameter is an important parameter.

Furthermore, the inadequacy of the weak-link model for this fibre raises serious doubt about the fundamental assumption made about the failure mode of this
fibre. The assumption of one essential flaw leading to failure, as dictated by the weak-link principle, may be unrealistic and a multiflaw failure could be relevant to this fibre.

Wolstenholme (28) again using the subset of data from this work shown that the non-parametric test may be applied to failure mechanisms which are not based on the simple weak-link property. The assumption here is the presence of multiple flaw types. This will only make a difference in the test if it results in the probability of unit element, $P$, being dependent on section length. At a given stress, therefore, unit elements will have unequal probability of failing next. The four gauge lengths used in this work are in approximately the same ratio, i.e. $L_1 : L_2 : L_3 : L_4$ are all about 1:2.5, where $L$ is the individual gauge length. If it is assumed that there is a constant scaling of length which enhances the probability of unit element failure, the factor $K L_1 / L_2$, where $K$ is a "length" enhancing factor, can be applied so that these probabilities are in the ratio $K^3 : K^2 : K : 1$ for $L_1$, $L_2$, $L_3$, $L_4$ respectively. Weak-link is obeyed when $K=1$, any value greater than 1 would make a shorter length stronger than predicted under the weak-link hypothesis.

The probability of $1, 2, 3, 4$ ordering becomes

$$\frac{75K^3 \times 30K^2 \times 12K}{\left(75K^3 + 30K^2 + 12K + 5\right)\left(30K^2 + 12K + 5\right)\left(12K + 5\right)} = P_1$$

Figure 4.13 shows the probability of failure for different values of $K$ for this order. Calculations for the Cellon fibre, in this work (using all the data sets) mentioned earlier, gave 79% of fibres failing in this order. This value from Figure 4.13
corresponds to K=5.

The alternative P2 model shown in the same Figure, follows from the work of Watson and Smith (27) (equation 2.29), where, \( L_i \) is replaced by \( L_i^K \). Here the probability of failure for the same ordering of 1, 2, 3 and 4 becomes:

\[
\frac{75^K \times 30^K \times 12^K}{(75^K + 30^K + 12^K + 5^K)^2 (30^K + 12^K + 5^K)^2 (12^K + 5^K)^2} = P2
\]

Figure 4.13 shows the K value corresponding to 79% failure under this model to be approximately 2.8.

The comparison between the two models (P1 and P2) thus suggests that failure for this fibre may be of a multiflail type, taking the form of surface-initiated failure and bulk-initiated failure. However, since the models do not assume diameter effect, then it can not be realistically applied in this circumstance. Wolstenholme (28) rejected both models when she applied it to a subset of the Celion fibre data, but again could not recognise the importance of diameter in assessing its breaking stress. A two-parameter model is likely to be more plausible.

All the statistical analyses performed, including the non-parametric test, jointly point to the effect of diameter being a significant parameter in the breaking stress of Celion fibre. In order to give some physical justifications to these statistical inferences, fractographical studies were performed on the fractured surface of the fibre.
4.3.3 Fractographical studies

Fractographical studies were conducted on the fractured surfaces of the fibres using a scanning electron microscope (S.E.M.). This analytical technique is widely used in failure analysis, primarily because of its combination of good depth of field and high resolution. The enhanced depth of field allows the edge-to-edge in-focus viewing of largely irregular fracture surfaces. The high resolution is necessary to observe very small fracture origins.

Figure 4.14(a) shows an S.E.M. micrograph of the Celion fibre fracture surface. The figure clearly indicates a large shear lip of approximately half the diameter of the fibre at $45^\circ$ to the length direction. Figure 4.14(b) shows a definite splintering or peeling of the fibre. These observations suggest that this fibre may intrinsically have some toughening characteristics. Hence its failure is not a simple brittle fracture. For comparative study, S.E.M. analysis was also extended to the XAS fibre, as shown in Figure 4.15. This fibre, on the other hand, indicates a smooth fracture surface suggesting a simple brittle fracture.

4.3.4 Co-operative flaws

A suggestion is made here in an attempt to describe the possible failure mechanism. It is possible that failure originates at two different sites on the fibre, shown pictorially on Figure 4.16. The stress applied could form a crack that propagates in a co-operative manner from one flaw-site to the other, representing the easiest energy dissipation route. Hence resulting in failure. This proposal would be in agreement with the finding that the weak-link notion of one essential type of flaw is unrealistic in determining the failure mode of Celion fibre. Further
work would be required to clarify the details of the failure mode and the effect on the statistics of failure.

4.3.5 Diameter effect revisited

A suggestion is proposed here in an attempt to explain the observed bi-modal effect that diameter produces on the different gauge lengths of the Celion fibre. This may be related to earlier results discussed in section 4.2.1. It was observed in that section as fibre lengths increase the variability of diameter or the standard deviation about the mean, also increases, Table 4.1. This effect may be explained in terms of the likelihood that at shorter lengths the point at which the reduced laser beam (0.08mm) focusses on the fibre, when measuring diameter (section 3.3.1), has a higher chance to coincide with the position of the fibre’s weak-link than the longer lengths of fibre. In other words, the diameter of 5 and 12mm lengths have a higher chance of being measured at their respective weak-links, with the likelihood being higher for the 5 than the 12mm length. However, the 30 and 75mm lengths are too long for the diameter to be measured at their weak-links. Therefore, at these long lengths, diameter effect is likely to be randomised with the consequence of missing the weak-links, with the likelihood being higher for the 75 than the 30mm length. Hence the overall observation is that diameter variation increases as the gauge length of the fibre increases.

The original data for the Celion fibre is not straightforward to understand and its mode of failure across the gauge lengths is very difficult to describe. However, all the analyses performed on the data suggest that this fibre does not fail by a single-mode of failure and that its failure is a complication that needs to be addressed. A recent set of papers dealing with the Weibull statistics found in the
testing of large ceramic specimens is useful in understanding the conclusions to be drawn from the Celion data at various points throughout this chapter. In these papers, Danzer (82, 83) explores the influence of the presence of distinct families of flaws on failure statistics.

Figure 4.17 is drawn from his work. Here he considers the case of two overlapping families of flaws, represented by the bi-modal distribution of Figure 4.17(a). The corresponding Weibull plot for a set of specimens which contain members of both families will then show the behaviour in Figure 4.17(b) where there is a crossover from one line to another. In section 4.3, it was shown that there are two classes of behaviour in the present results, diameter independence being shown by the longer fibres and diameter dependence in the shortest fibre. The 12mm fibres (Figure 4.5) appear to cross between these classes of behaviour in exactly the manner predicted by Danzer’s work in ceramics. It might be speculated that the longer lengths in this work, with lower characteristic strength, are dominated by a surface effect. Further evidence for this hypothesis is assembled in the next chapter. The 12mm fibre length shows a bi-modal behaviour and might fail through the action of a surface flaw or a volume flaw.

On the above basis the 5mm length should be the representative of the volume mode of failure, yet this too has a very low Weibull modulus. Danzer shows, Figure 4.18, that a low modulus will result from a multi-modal mix of flaws, in which the measured strengths straddle across a series of Weibull lines. This again is exactly the behaviour observed in the 5mm lengths, Figure 4.5, which could be following a series of parallel Weibull lines determined by a multi-modal diameter population, Figure 4.12.

Thus in summary, the complex behaviour seen in the original data for the Celion
fibre stems from two effects: firstly, the presence of two classes of failure which may be represented by surface and volume defects; and secondly, within the “volume” mode alone, a strong dependence on diameter. The origin of the diameter effect remains unclear. There is a discussion of volume and surface defects given by Wagner (42). The basis of his argument, mentioned in detail in section 2.2 depends on the way defects are distributed within, or on, the surface of the fibre. Characteristic strength is proportional to $dl$ (surface effects) or $d^2l$ (volume effects), where $d$ and $l$ are diameter and length respectively. The relationships describe the two possible defects distributions respectively representing surface and volume failure modes. In Figure 4.19, the data for the 5mm gauge length is tested by a plot of $\ln$ (strength) against $\ln$ (diameter). It is clear that the data does not fit a straight line: three points are close to $1/d$ dependence whilst the inclusion of the fourth yields a slope which is closer to $1/d^2$. A choice of mechanism cannot be made from this data alone. The answer to this important question of whether surface or volume flaws is more important will become clearer in the next chapter when the fibres are embedded in epoxy-resin and tested under identical conditions.
4.4 Conclusions

This chapter involved an extensive study of the breaking stresses of single unsupported fibres, with particular reference to the diameter and length of the fibre. Four different gauge lengths were cut from a long length of fibre. The entire experimental programme requires considerable dexterity and skill, since single filaments are prone to damage very easily due to their fragile nature.

The following points are considered to be the major conclusions after thorough analyses have been performed on the data.

1. Two carbon fibres were used, i.e. Celion and Xfasfibres. The data for the Celion fibre were shown to deviate strongly from the Weibull model and weak-link behaviour, this was thought to be due to diameter variation. The Xfas fibre, on the other hand, was shown to comply with both the Weibull model and the weak-link behaviour, due to its diameter uniformity.

2. The complex behaviour for the data of the Celion fibre was shown to stem from two effects: firstly, the presence of two classes of failure which may be represented by surface and volume defects, and secondly, within the “volume” mode itself, there was a strong dependence on diameter.

3. There was an increasingly strong dependence of strength on fibre diameter as the gauge length decreases.

4. Variation of diameters about their mean values across the set of gauge lengths were shown to vary in behaviour for the two carbon fibres. This was thought to
be due to their respective manufacturing processes involved in their production. However, diameter variability about the mean value increases with gauge length for the Celion fibre only. It is probably due to the weak-links being uniformly randomised as the length increases. Thus the probability of the reduced laser beam focusing on the fibre's weak-link (to measure the fibre's diameter) is higher for longer lengths than shorter ones. On the other hand, the X$_A$ fibre shows uniform diameter measurements irrespective of gauge length. This could be due to non-randomised weak-link distribution.

5. The assumption of using a nominal diameter, as used by many workers, was found to be questionable.

6. Diameter was found to produce different effects on the gauge lengths, in that it affects shorter fibres more than longer fibres. Longer lengths (75 and 30mm) showed failure to be length dependent and the shorter lengths (12 and 5mm) were found to be both diameter and length dependent.

7. The 12mm gauge length was shown to be the critical length, where diameter effect becomes important, since it clearly displayed a pronounced bi-modal behaviour of mixed effect. Its higher stresses behave like the 5mm length and the low stresses exhibit similar behaviour to the 30mm length.

8. A non-parametric test which has the advantage of not being reliant on the assumptions about possible different modes of failure or about the distribution strength, but aims to test independently the weak-link principle was applied to the data. It was clearly found that this test does not apply to the Celion fibre.
9. Other statistical models that assume multiple flaws failure were applied to the Celion fibre data, but they were rejected since they do not assume diameter effect.
Table 4.1 Mean diameter measurements with respect to their standard deviation per gauge length for the Celion fibre

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<td>7.75</td>
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Table 4.2 Mean diameter measurements with respect to their standard deviation for the XU fibre

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<td>S.d (µm)</td>
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Table 4.3 Regression estimates of the Weibull parameters for the Celion fibres

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<th>W</th>
<th>( \sigma_0 ) (L)</th>
<th>( \alpha )</th>
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Table 4.4 Regression estimates of the Weibull parameters for the XU fibre

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Table 4.5 Non-parametric test results for each data set of the Cellon fibre

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Table 4.6 Non-parametric test results for the Xas carbon fibre.

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</table>
Figure 4.1 A representative plot of diameter against breaking stress for the Celion fibre (data set two).

Figure 4.2 A global plot of diameter against breaking stress for all the five data sets of the Celion fibre.
Figure 4.3 A combined Weibull probability plot for the three non-random data sets of the Celion fibre.

Figure 4.4 A combined Weibull probability plot for the random data sets ('88 and '92) of the Celion fibre.
Figure 4.5 A global Weibull probability plot for all the five data sets of the Celion fibre.

Figure 4.6 A Weibull probability plot for the XAS fibre.
Figure 4.7 A plot of probability of failure ($P_f$) against breaking stress for the combined random data sets ('88 and '92) of the Celion fibre.

Figure 4.8 A plot of $P_f$ against breaking stress for the combined non-random data sets (one, two and three) of the Celion fibre.

Figure 4.9 A global plot of $P_f$ against breaking stress for all the data sets of the Celion fibre.
Figure 4.10 A global ln (strength) against ln (length) plot for a) the Celion fibre and b) the XAS fibre.
Figure 4.11 A global diameter frequency distribution plot for the Celion fibre.
Figure 4.12 A global Weibull probability plot for all the gauge lengths per mid-value diameter range.
N.B. (a), (b), (c) and (d) are 5, 12, 30 and 75mm gauge length respectively.
Figure 4.13 Plot of Pf per unit length against factor K.
Figure 4.14 S.E.M. micrograph showing the fractured surface of the Celion fibre.
Figure 4.15 S.E.M. micrograph showing the fractured surface of the X fibre.
Flaw one

Stress co-operating from one flaw to the other assuming the easiest route to initiate failure

Flaw two

Figure 4.16 Co-operative flaw model.
Figure 4.17 A plot showing the influence of distinct families of flaws for ceramic specimens. a) bi-modal distribution and; b) a corresponding Weibull plot containing both families and showing a cross-over from one Weibull line to another, taken from Danzer (82, 83).
Figure 4.18 A plot showing a low modulus resulting from a multimodal mix of flaws, in which the measured strength straddle across a series of Weibull lines, taken from Danzer (83).

Figure 4.19 A plot of ln (strength) against ln (diameter) for the 5mm gauge length of the Cellon fibre.
CHAPTER FIVE

5.0 Strength of supported (embedded) single fibres

5.1 Introduction

It was shown in the previous chapter that the breaking stresses of unsupported Celion fibres do not comply with the Weibull model. This was due to two effects. First, there were two classes of flaw, probably surface (long length) and volume (5mm length), and second, the short lengths showed a strong diameter effect.

This chapter follows on from this work to test the above findings on single fibres supported by an epoxy-resin matrix. It is commonly believed (84) that the surface flaw is eliminated by the action of the supporting matrix and this fact provides a means of separating the two classes of flaw. The experimental programme, therefore, mirrors the work on unsupported single fibres and is based on the study of the breaking stresses of single fibres from the same spool under similar conditions except this time the fibres were embedded in epoxy-resin. This involved casting a single fibre into a resin coupon so that it was aligned along the axis. The diameter of each specimen was carefully measured at three different positions along its length so that a mean value could be obtained. The specimen was then subjected to a monotonically increasing strain and the first failure break was recorded as this represents the weak-link of the fibre. Hence its strength. In this way, it was possible to determine the strain-to-failure of each specimen, from which breaking stresses were deduced using the mean fibre modulus. Subsequently, the produced data were characterised using the Weibull model and other statistical tests, as used in the previous chapter, in order to study the
size effect of this fibre in its new environment (epoxy-resin matrix).

The overwhelming importance in the analysis of the data is to establish whether a “weak-link behaviour” is restored in this new environment and whether the strength of the fibre for each gauge length is affected by the matrix when compared to the unsupported fibres.

5.2 Results

Two separate batches of embedded fibre coupons were tested comprising 26 and 28 long fibres. Each long length of fibre was cut into 5, 12, 30 and 75mm gauge length specimens; giving a total of 104 and 112 tests pieces for the two data sets respectively. Diameter and strain-to-failure measurements were performed on each specimen and their analyses are presented in sections 5.2.1 and 5.2.2 respectively.

5.2.1 Fibre diameter

Table 5.1 shows diameter measurements for the two data sets of the supported single fibres at 5, 12, 30 and 75mm gauge lengths with their respective mean diameters and standard deviations. The table also shows similar observations when the two data sets combined as one global data set. The number of observations made for each gauge length, indicated in Table 5.1, is 52, 54, 52 and 54 tests.

Table 5.1 indicates a very low range of diameter variations shown by the standard
deviations about the mean diameters when compared to the global data set of the unsupported Celion fibres shown in Table 4.1. These two tables are presented here again, in order to highlight at a glance the observations made on them. It is clear from both tables that the mean diameter is relatively the same for both supported and unsupported fibres. However, their respective standard deviations are different from one another leading to opposite assumptions being made about them. The supported fibres indicate diameter uniformity with increasing gauge length, whereas the unsupported fibres show large diameter variability with length. The question must be asked to why this fibre yields different diameter observations when tested in the two environments. The answer may stem from the two techniques employed in measuring diameter which is discussed in section 5.3.1.

Table 4.1 Mean diameter measurements with respect to their standard deviation (S.d) for the unsupported fibres.

<table>
<thead>
<tr>
<th>Global data set</th>
<th>160</th>
<th>161</th>
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<tr>
<td>n</td>
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<td>12</td>
<td>30</td>
<td>75</td>
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<tr>
<td>Gauge length (mm)</td>
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<td>7.74</td>
<td>7.71</td>
<td>7.80</td>
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<tr>
<td>Mean diameter (μm)</td>
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<td>0.50</td>
<td>0.63</td>
<td>0.86</td>
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</table>

Table 5.1 Mean diameter measurements with respect to their standard deviation (S.d) for the supported fibres.

<table>
<thead>
<tr>
<th>Global data set</th>
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<th>54</th>
<th>52</th>
<th>54</th>
</tr>
</thead>
<tbody>
<tr>
<td>n</td>
<td>5</td>
<td>12</td>
<td>30</td>
<td>75</td>
</tr>
<tr>
<td>Gauge length (mm)</td>
<td>7.73</td>
<td>7.77</td>
<td>7.76</td>
<td>7.76</td>
</tr>
<tr>
<td>Mean diameter (μm)</td>
<td>0.18</td>
<td>0.20</td>
<td>0.21</td>
<td>0.22</td>
</tr>
</tbody>
</table>
5.2.2 Fibre strength

The strain-to-failure for each specimen was measured because it was not possible to measure the fracture load of supported fibres directly. Thus breaking stresses were calculated using the mean Young’s modulus supplied by the manufacturer of the fibre. The value given is 215 GPa.

The specimen’s failure breaks were easily observed, since the test was performed using an optical microscope and viewed between cross-polarising filters. Figure 3.9 shows a typical photoelastic stress birefringence pattern surrounding the broken end of a fibre. This effect is due to shear deformation in the matrix.

5.2.3 Weibull analyses

A two-parameter Weibull plot defining a fibre’s failure due to a random distribution of a single flaw was applied to the data collected for these specimens in the same way as applied previously on the unsupported specimens. The Weibull analyses were first conducted on the two separate sets of data respectively, followed by a similar treatment on the two sets of data combined together to form a single global data set.

Cumulative distribution Weibull plots, where the fibre failure stress at the four gauge lengths tested, were plotted against failure probability for the first and second data sets and then on their global data set are shown in Figures 5.1, 5.2 and 5.3 respectively. Figures 5.1 and 5.2 clearly show both data sets have regular spacing between the 75, 30 and 12mm gauge length, as expected from
the strength/length effect, but this regularity does not extend to the 5mm length. Again this behaviour is demonstrated strongly in Figure 5.3 for the global data set.

Figures 5.4, 5.5 and 5.6 show Weibull probability plots for the three data sets. These figures show a very good agreement to a linear Weibull relationship for all the gauge lengths tested since each figure displays similar characteristics. In addition, the three parallel lines shown for each of the three figures are displaced from one another by an equal measured quantity. On the other hand, the 5mm line is closer to the 12mm line than expected.

Weibull parameters estimates were obtained for the global data set by performing a linear regression on the data, in a similar manner as performed on the data of the unsupported fibres. The Weibull parameters estimates generated from the data of the supported fibres are presented in Table 5.2. The Table strongly indicates that strength values per gauge length have increased in comparison to the strength values per gauge length for the unsupported fibres, shown in Table 4.3, with the exception of the 5mm length which has already been indicated to be less strong than expected. Table 5.2 also indicates the scatter of the data has been considerably reduced compared to Table 4.3 as this is represented by the higher and more consistent values obtained for the Weibull modulus for each gauge length tested.

The similarity of the parameters, $W$ and $\alpha$ (characteristic stress for unit length, 1mm) shown in Table 5.2 at the same time indicates compliance with the weak-link hypothesis. The assumption, therefore, of the existence of randomly distributed flaws may be valid for these fibres when supported in epoxy-resin.
matrix. The restoration of the weak-link model for supported Celion fibres is not surprising since the measured diameter ranges, shown in Table 5.1, are uniform across the four gauge lengths. Hence their effect on the deduced stress to failure of the fibres do not lead to the wide ranging stress values which were found in the unsupported fibres. The effect of length, on the other hand, may be the only important parameter in this situation. It is worth noting here that the measured diameter for the supported fibres is not the true diameter of the fibre. This will be discussed in section 5.3.1.

5.2.4 Non-parametric test

The results obtained in section 5.2.3 indicate very strongly that the weak-link scaling effect could be predicted from the Weibull analysis performed on the data. However, as an additional check the non-parametric test previously applied on the data of the unsupported fibres may now be applied on the supported fibres.

In order to apply the test again, only complete data sets can be used. Therefore, the two batches under study produced 24 and 27 specimens for the four gauge lengths as two complete data sets.

Table 5.3 shows the order of failure for all 24 probabilities together with theoretical and observed percentages pertaining to the two respective complete data sets. The most likely order of failure is 75, 30, 12 and 5mm gauge length respectively in accordance to the weak-link principle. The predicted probability percentage for that order is 27.71%. The two data sets tested yielded 25% and 25.93% respectively for that order of failure, Table 5.3, which conformed very well to the predicted probability percentage of 27.71%.
5.3 Discussion

A common assumption made in theoretical and experimental studies of fibrous materials is that the individual fibres from the same spool have the same diameter or perhaps, that some common average diameter may be used for all single filaments, in order to derive a value for breaking stress. If actual filament diameter variation occurs, then the failure of the fibre will be affected by these variations. This observation was found in the previous chapter for the unsupported fibres. The measured diameter for the supported (embedded) fibres was found to be uniform pointing to length as the only important parameter. It was shown in chapter Four that the unsupported fibres did not comply with the Weibull model, and this was due to the effect of two classes of flaw. Embedding the fibres in an epoxy-resin (84) is believed to eliminate surface flaws making them less likely to lead to failure. Hence volume flaws dominate. Consequently, the observation is that the supported fibres in resin appear to be stronger than the unsupported fibres.

5.3.1 Fibre diameter

Table 5.1 shows standard deviations for both data sets to be relatively uniform along the four gauge lengths tested. This is in contrast to observations made previously on the unsupported fibres. These conflicting results lead to the crux of the investigation as they throw a serious doubt to the assumption usually made that a nominal fibre diameter can be used in calculations.

Two different techniques were used for measuring diameter: in the previous chapter this parameter was measured using the laser diffraction method; in this...
chapter the Watson shearing eye-piece (W.I.S.E.) is used. The conflicting findings indicated on both Tables 4.1 and 5.1 may be explained from understanding the working principles behind these two techniques. The latter involved the use of a pre-calibrated eye-piece in conjunction with an optical microscope. The basic principle of a shearing eye-piece is to generate two identical images, one which is moved through a known distance to bring its edge into contact first with one side of the first image and then another with the second opposite side (Figure 3.6). The measurements are not affected by any movements of the image, since the edges of the fibre serve as the fiducial reference lines. The technique is mentioned in details in section 3.3.2. Watt et al (85) used this technique for measuring their fibre diameter quoting an accuracy of 0.1μm. Moreton (76) applied the same technique on his carbon fibres and also quoted the same accuracy.

The former method involved a laser beam focused on a screen by means of a convex lens placed at its focal length from the screen. A diffraction pattern was formed on the screen when a fibre was introduced into the beam. The fibre-to-screen distance was measured and assumed to follow a Fraunhofer line from which diameter was deduced. This technique is also discussed in detail in section 3.3.1. Perry and De Lamotte (86) compared carbon fibre diameter measured by laser diffraction against values obtained optically and found their values to be too large consistently by about 1/3μm. In this work the mean diameters are found to be the same but the standard deviation for the laser method is larger by a factor of almost four.

The procedures mentioned may attempt to explain the observations shown in both Tables 4.1 and 5.1. In measuring the diameter, the shearing eye-piece forms
the fiducial reference sheared line along the points undergoing measurements. The result of this effect is that each of these three readings have actually been averaged out by virtue of the technique itself. In other words, when a mean diameter value for each specimen is calculated, what is derived, in fact, is a value which has already been averaged out by the shear line. A typical photograph of the field of view for a supported fibre under observation using the W.I.S.E. technique is shown in Figure 3.7. The diameter of the fibre was found to be uniform along the entire length of the field of view. Calculations based on this yield the length of the fibre under view to be approximately thirty times the fibre’s diameter. In fact the uniformity of the fibre diameters was found to extend to six times the field of view, before any re-adjustment of the setting was needed. This means that the averaging effect of this technique is of the order of 1.4mm (approximately 180 fibre diameters) rendering it to be a “line-probe” method. In addition, the fibre diameter measured by this technique leads to it been averaged out twice, once by the process itself and another by the researcher thus generating uniform diameter values.

The observed uniformity of the standard deviations across the set of gauge lengths may be explained in terms of the likelihood that the position of the fibre’s weak-link is masked by the sheared line. In other words, when measuring diameter by this technique the diameter of the fibre coinciding with the fibre’s weak-link is not measured due to the large averaging effect brought about by the long sheared line.

The laser diffraction technique, on the other hand, produces diameter values which are by comparison closer to the true values of the fibre diameter. This is because the reduced laser beam is only 10 fibre diameters resulting in a much reduced averaging effect on the measured fibre diameter. However, the effect of
the diameter variations on the different gauge lengths was found to be increasing with increasing gauge lengths. This was discussed in section 4.3.5.

In summary, it has been seen that the mean diameter for either the unsupported or the supported fibres has remained the same, Tables 4.1 and 5.1, irrespective of the techniques used for diameter measurements. However, it has also been observed that the standard deviations for both the unsupported and the supported fibres are contrastingly different. The direct effect of diameter variations for the unsupported fibres is that the short gauge lengths, and in particular the 5mm length, showed strong diameter dependence on strength, whereas the longer lengths (30 and 75mm) showed their failure to be independent of diameter, Figure 4.1. The effect of diameter on the supported fibres, on the other hand, is effectively negligible across the set of gauge lengths, this is because here the diameter has been uniformly randomised as shown by the uniformity of the standard deviations, Table 5.1. This means that the effect of diameter on strength disappears altogether and that the strength of fibres then becomes dependent on the effect of one class of flaw. Either surface or volume.

5.3.2 Weibull analyses

The uniformity of diameters for the supported fibres forms the basis for assumptions relating failure breaks to linear Weibull relationship, since the latter implicitly assumes a uniform diameter.

Figures 5.4 and 5.5 are the two respective data sets for the supported fibres plotted on the Weibull co-ordinates. From the observed linearity of these plots, it is clear that the Weibull model very adequately describes the fibre strength in this
environment over the gauge length range from 5 to 75mm. This observation also satisfies the premise that there is a strength/length dependency, which has been demonstrated by many workers (40, 60, 76). Figure 5.6 reinforces the above observation when the two data sets are combined to produce one global data set. Linear regression analysis is applied to the global data set, rather than to individual data sets, since it is important to use a large sample size in order that a realistic Weibull parameters estimates may be obtained. The results of the analysis are indicated in Table 5.2, where all the four gauge lengths tested yield similar Weibull moduli (W). Therefore, the consistency in the value of W for all the gauge lengths supports very strongly the findings that this fibre when embedded in epoxy-resin complies with the notion that there is a direct correlation between strength and length. This consistency of the Weibull moduli values obtained for all the gauge lengths suggests that there is very little scatter or variability in their strength distribution and has the effect of eliminating diameter as a covariate parameter in influencing failure break in the fibre. This is in accordance with the earlier discussion of diameter, section 5.3.1, measured by the Watson shearing eye-piece. It was pointed out that the averaging effect of this method leads to the use of nominal diameters which will mask any effect of true diameters in the Weibull distribution.

It is worth noting that the mean value for the Weibull modulus obtained for these fibres is 15.52, which is approximately twice the mean value for the unsupported fibres, 7.81. This reinforces the view again that the observed uniformity of diameters in the supported fibres implies that length is the only parameter of dependence to describing failure breaks in this environment. Table 5.2 also indicates the strength/length relationship for the mean length dependent characteristic strength, $\alpha$, for all the gauge lengths, as they exhibit similar values, with the exception of the 5mm gauge length, which has a slightly lower value.
The plot of \( \ln(\text{strength}) \) against \( \ln(\text{length}) \) is shown in Figure 5.7. This type of plot should clearly show the weak-link scaling effect, since the data should fall in a straight line of slope \(-1/W\). A linear regression analysis was applied and the Weibull modulus was deduced from the slope of the line. The value obtained is 16.42 which is in close agreement to values generated by the earlier analysis shown in Table 5.2. Hence indicating conformity to the Weibull model once again.

The parameter estimates for the characteristic strength, \( \sigma_0(L) \), will be discussed separately in the next two sections.

### 5.3 Effect of resin

The use of resin to support fibres in this chapter has provided the expected effect of separating the two classes of flaw observed in section 4.3.5. It was shown in section 4.3.5 that higher lengths of the unsupported fibres (30 and 75mm) could be represented by only surface flaws, and the 5mm length by volume flaws and diameter effect. The 12mm gauge length exhibited a bi-modal behaviour, where it crossed the two classes of flaw.

The use of epoxy-resin on fibres has eliminated the family of flaws ascribed to the surface flaws, in all gauge lengths, Figure 5.6 compared to Figure 4.5, for the unsupported fibres. This effect is strongly demonstrated for the 12mm length where the cross-over of the Weibull line seen in Figure 4.5 has been eliminated in Figure 5.6.

Furthermore, embedding the fibres in epoxy-resin has led to an increase of characteristic strength values, Table 5.2, when compared to the unsupported fibres, Table 4.3. This effect by the resin strongly suggests that the surface flaws
has been eliminated and is in agreement with early workers (84). The 5mm length, however, show a reduction of strength, and may be due to the fibre being dependent on its diameter for its failure. The behaviour of this length will be dealt with in detail in the next chapter.

5.4 Fibre strength

The Weibull parameter estimates for the characteristic strength, $\sigma_0(L)$, are shown in Table 5.2, and indicate a tighter distribution of strength compared to the same parameter estimates derived from the unsupported fibres, Table 4.3. The $\sigma_0(L)$ estimate for the 5mm gauge length has actually reduced in value compared to the unsupported single fibres. The effect is also demonstrated clearly on the Weibull co-ordinate plots shown in Figures 5.4, 5.5 and 5.6, where it has shifted to the left. In the next chapter the comparative behaviour of the supported and the unsupported fibre is discussed in more detail.
5.5 Conclusions

The following points are the most important conclusions made from the analyses on the supported fibres data.

1. There was a marked increase in strength for every gauge length when compared to strength values obtained for the unsupported fibres, with the exception of the 5mm length which actually reduced in value.

2. The effect of resin has eliminated the surface flaws in all the gauge length and the volume flaws were believed to be the only dominant class of failure present.

3. The Weibull model conformed very well to the data obtained over the four gauge lengths tested.

4. The weak-link property was restored due to the apparent uniformity of the measured diameter, which effectively resulted in the use of a nominal value.

5. There was an apparent diameter uniformity with increasing gauge length.

6. The above observation was explained in terms of the technique employed (Watson shearing eye-piece) for measuring diameter, which is different from the technique used for the unsupported fibres (laser).

7. The effect of length was the only parameter affecting the failure of the fibre, since diameter in this environment was found to be uniformly randomised.
8. The application of the non-parametric test to the data overwhelmingly supported the restoration of the weak-link property.
Table 5.1 Mean diameter measurements with respect to their standard deviation (S.d) for the supported fibres.

### Data set one

<table>
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<td>Gauge length (mm)</td>
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<td>S.d (μm)</td>
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### Data set two

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<tr>
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### Global data set

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</tr>
<tr>
<td>Mean diameter (μm)</td>
<td>7.73</td>
<td>7.77</td>
<td>7.76</td>
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<tr>
<td>S.d (μm)</td>
<td>0.18</td>
<td>0.20</td>
<td>0.21</td>
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Table 5.2  Regression estimates of the Weibull parameters for the global data set of the supported fibres.

<table>
<thead>
<tr>
<th>Gauge length (mm)</th>
<th>W</th>
<th>$\sigma_0$(L)</th>
<th>$\alpha$</th>
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</thead>
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<tr>
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<td>3.78</td>
<td>4.19</td>
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<td>12</td>
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<td>30</td>
<td>15.41</td>
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<tr>
<td>75</td>
<td>15.29</td>
<td>3.24</td>
<td>4.30</td>
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</table>
Table 5.3 Non-parametric test results for the two data sets of the supported fibres.

**Data set one**

<table>
<thead>
<tr>
<th>Event of failure</th>
<th>Theoretical Prob. %</th>
<th>Expected %</th>
<th>Obser. No.</th>
<th>Obser. %</th>
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<td>2134 1324 1243</td>
<td>39.14</td>
<td>9.40</td>
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**Data set two**

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<td>33.16</td>
<td>8.95</td>
<td>3</td>
<td>18.52</td>
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</table>
Figure 5.1 A plot of probability of failure ($P_f$) against breaking stress for the first data set of the supported fibres.

Figure 5.2 A plot of probability of failure ($P_f$) against breaking stress for the second data set of the supported fibres.

Figure 5.3 A plot of probability of failure ($P_f$) against breaking stress for the global data set of the supported fibres.
Figure 5.4 A Weibull probability for the first data set of the supported fibres.

Figure 5.5 A Weibull probability for the second data set of the supported fibres.

Figure 5.6 A Weibull probability for the global data set of the supported fibres.
Figure 5.7 A global ln (strength) against ln (length) plot for the global data set of the supported fibres.
CHAPTER SIX

6.0 Discussion

6.1 Supported and unsupported single fibres

A commonly held view is that embedding fibres in resin will eliminate the effect of surface flaws but will have little effect on bulk or volume flaws, which are not in contact with the resin. If surface flaws account for the "weak-link" in the failure of fibres, embedding should lead to an increase in characteristic strength. For example, Clarke and Bader (84) measured the strength of single fibres using a nominal 100μm diameter silicon carbide both tested in air and supported in polymer matrix. They found that the strength had increased when the fibre was embedded in matrix. They explained their findings on the basis that failure may have been initiated by either surface or volume flaws, and suggested that embedding fibres in resin matrix may have the effect of "healing" some of the surface flaws and hence enhancing the fibre's strength.

However this bi-modal behaviour does not adequately explain the strength observations shown in this work. Examination of the data presented in chapters four and five shows a picture which is less straightforward. Embedding the fibres in an epoxy-resin has led to a reduction in the range of characteristic strength values for the set of lengths examined in this work, Table 6.1. This reduction arises mostly from an increase in the strength of the two longest lengths. The effect is quite dramatic: for the 75mm gauge length, there is an increase in strength from 2.26 to 3.24 GPa. This is the classic behaviour expected for the removal of surface flaws and one may ascribe this effect to this process.
However, the shorter lengths behave quite differently. There is hardly any change for the 12mm gauge length whilst the strength of the 5mm length is actually reduced.

It was shown in chapter four that diameter had an important influence on the fibre strength. This effect can be particularly well displayed when a Weibull probability plot of ln ln (1/1-Pf) versus ln (load) is performed with the effect of diameter removed from the analysis. An example of this plot is shown in Figure 6.1 for the global data set of load at failure. The characteristic strength values for both the 30 and 75mm lengths fall on the ln (stress) axis, Figure 4.5, at similar spacings to that found on the ln (load) axis, Figure 6.1. These lengths appear to be members of one family (which was referred to be surface flaw mode in chapter four), whilst the 5mm data is clearly far to the right and belongs to a different family (assumed to be volume flaw mode). The 12mm data still passes from one family to the other. This is the bi-modal behaviour discussed in section 4.3.5.

It is clear from Figure 6.1 that the 75, 30 and 12mm gauge lengths are independent of diameter effect and that the surface flaw mode may well be affected by length. The 5mm length, on the other hand, shows diameter dependence. The effect of diameter on this length can be seen by comparing the change of the slope of the Weibull line in Figures 4.5 and 6.1. The clear increase of the Weibull slope of Figure 6.1 compared to Figure 4.5, indicates that the data are more uniformly distributed for this length when diameter effect is removed from the analysis. The slope of the Weibull line is its Weibull modulus. The value obtained from the conventional Weibull probability plot of ln (stress), Figure 4.5, is 7.25 compared to the value 13.25 derived from the novel Weibull probability plot of ln (load), Figure 6.1. The increase of the Weibull modulus value, brought about by the removal of diameter from the analysis, indicates that the 5mm length data
are more uniformly distributed. Hence the likelihood of volume flaw mode being present increases.

In fact the value of the Weibull modulus for the 5mm length derived from the Weibull probability plot of ln (load), Figure 6.1, is in close proximity to the value obtained from the conventional Weibull probability plot for the same length when the fibre is supported in epoxy-resin, (15.52), Table 5.2. This suggests that the removal of diameter from the 5mm length data has the effect of aligning the distribution of the unsupported data to the supported data for this length. It was shown in chapter five that the measured diameter for the supported fibres were assumed to be nominal due to their apparent uniformity. The removal of diameter in the analysis of the 5mm length data can also be assumed to be nominal. Hence strength at this length can be attributed to volume flaw mode with diameter dependence.

It has been pointed out already that the diameters of fibres unsupported or supported are measured by different techniques. To minimise the influence of this the data of chapter four on unsupported fibres have been recalculated using the measured mean value of 7.75μm (obtained using the laser technique) as a nominal diameter which is normally done in measurements of fibre strength. This recalculation should have the effect of aligning the calculated strength values of unsupported and supported fibres. The recalculation gives the values listed in column 4 of Table 6.1. It still leaves the strength of the 5mm unsupported fibres higher than the equivalent resin-supported set. One can, however, understand the effect of resin support more clearly by comparison of the failure strengths as a function of diameter as well as length. This is displayed in Figure 6.2. The data for both the supported and unsupported fibres are shown clearly in this Figure. It is clear then that the exceptional strength of the 5mm unsupported fibres comes
from the behaviour of the subset having a small diameter. Apart from this the strength values for the two shorter fibres, whether supported or unsupported, lie close together and quite different to the values for the longer fibres. Since it was argued that the failure of the 30 and 75mm lengths was dominated by the surface flaw it follows that the shorter fibres, supported and unsupported, fail through the operation of the volume flaw, this having a higher characteristic strength.

The change of failure mode within the lengths used in this work reveals something about the distribution of the surface flaws. Failure due to the surface flaw is almost always encountered in the 30mm length but hardly found at all in the 5mm length. The behaviour of the 12mm samples is particularly informative because of the obvious change in pattern in the failure of these samples which is seen in the global Weibull plot, Figure 4.5, as a curve which straddles across from the 30mm data set to the 5mm data set. This suggests that some of the 12mm sample length will include a surface flaw and some will not and that the frequency of large surface flaws along the length of the fibre is of the order of one flaw per 12mm interval. The fractographical studies discussed in section 4.3.3 on the surface fracture of the Celion fibre, concerned the 12mm gauge length specimen, Figure 4.14(a and b). The Figure indicated that this fibre contains some toughening characteristics and that its failure is not a simple brittle fracture. Interestingly, if the fracture surface of several 12mm length specimens were tested, the chance of selecting a specimen with a flat surface of a simple brittle fracture attributed to surface flaw mode may increase and this would be in accordance to the bi-modal behaviour observed in the 12mm length, where some will include surface flaw and some will not.

When examined as a function of diameter the 12mm gauge length show a behaviour which crosses from the failure strength characteristic of the surface
flaw to that characteristic of the volume flaw a behaviour shown in Figure 6.2. However, since the longer lengths show a strength independent of diameter, and this strength is due to surface flaws, it is clear that the presence of surface flaws is not related strongly to diameter. This is the reason why the longer lengths do not show a diameter dependence. The shorter lengths on the other hand, and in particular the 5mm length, do show a diameter dependence. This may arise as a consequence of the core and skin structure of the fibre. The relationship of the fibre diameter to the Young's modulus is a complication which also needs to be addressed.

For the supported fibres, it is the strain at failure, rather than the load which is measured and the strength value then depends on the Young's modulus. An average value of the modulus, supplied by the manufacturer, has been used here. A lack of knowledge of individual fibre moduli makes it impossible to discover any influence of diameter on the Young's modulus from the data obtained. The effect of fibre diameter was found to be very significant for the failure stress observed in chapter four, where thinner fibres were shown to be stronger than thicker ones. It is highly probable that thinner fibres are also stiffer.

It is known that polyacrylonitrile (PAN) based carbon fibre have a skin, or sheath, and core arrangement of graphite-like structural elements or crystallites (87), the skin having a greater alignment of graphite planes along the fibre axis than the core. The different mechanical properties of the core and skin (owing to their morphology) greatly influence the final behaviour of the fibre, since the aligned skin is stiffer and stronger, than the relatively compliant core structure. The final strength and stiffness of the fibre are both influenced by the oxidative stabilisation process and the carbonisation heat treatment temperature. The process of producing PAN-based carbon fibres includes these two principal stages:
oxidative stabilisation followed by carbonisation and graphitisation (for higher modulus fibres). The degree of stabilisation determines the extent of the skin formed when subsequent carbonisation takes place. That is, the more extensive the stabilisation process the greater the depth of the skin. Carbonisation of the stabilised fibre results in the formation of graphite crystallites. The degree of crystallite alignment in the skin is dependent on the carbonisation temperature. The greater the temperature at which carbonisation takes place the greater the crystallite alignment in the skin. As the final heat treatment temperature is increased, the alignment of the basal planes of the crystallites parallel to the fibre axis also increases. Depending on the carbonisation temperature carbon fibres are classified either as high modulus (above 1600°C) or high strength (below 1600°C).

As mentioned above the two important factors that influence fibre diameter are the extent of oxidation treatment given to the fibre (which determines the extent of the skin) and the final heat treatment temperature (influencing the degree of orientation of the crystallite within the skin). If a rule of mixtures is applied then the proportion of the core diameter in thick fibres will be greater than in thin fibres (88). The carbonisation temperature is important in that the degree of orientation of the crystallite will also contribute to the final diameter of the fibre. A fibre carbonised at a higher temperature will have a smaller diameter than that of one heat treated to a lower temperature, due to the more highly oriented crystallite layer within the skin.

Jones and Duncan (89) also demonstrated that there is a consistent relationship between the diameter of PAN precursor graphite fibre and both Young's modulus and the tensile strength of the fibre. They observed that the thinner fibres exhibit
higher values of Young's modulus and tensile strength than thick fibres. This relationship was also observed by Buxton (90). Jones and Duncan (89) used the skin and core structure to explain their findings. It was pointed out that the Young's modulus of PAN fibres is controlled by the degree of orientation of the graphite basal plane parallel to the fibre axis (89). Crystallites close to the fibre's surface tend to be larger and better aligned, running parallel to the fibre axis, than crystallites found in the interior. Thus thin fibres will have greater crystallite orientation and, hence, a greater Young's modulus than thicker fibres. It is, therefore, very likely that when calculating the strength of the supported fibres from the measured strain at failure different Young's moduli values should have been used for large and small fibre diameters. The effect of using a nominal value is to produce lower strength values for the smaller diameters than if the anticipated high fibre modulus is assumed. Similarly the large diameters will produce higher values. The use of different Young's moduli values for small and large fibre diameters is particularly relevant to the 5mm gauge length, since this length shows strength to be strongly dependent on diameter. It has been pointed out already that the exceptional strength of this length in the unsupported environment comes from the behaviour of the subset having small diameters; it has been argued earlier also that the diameter measurements in this environment are likely to be accurate values. Hence the depressed strength results obtained for the supported fibres may be related to the nominal Young's modulus used in the calculation of strength. Using a higher value of Young's modulus would increase the supported strength to a higher value, relative to the unsupported strength and might bring the characteristic strengths into line.

In summary, it has been seen that the longer lengths of unsupported fibres show strength to be independent of diameter and that surface flaws appear to be dependent on fibre length and not on fibre diameter. The effect of these flaws
was effectively eliminated by supporting the fibres in epoxy-resin matrix and failure for the longer lengths must also be attributed to bulk flaws. The strength of shorter fibres has already been shown to be determined by bulk flaws. The overall effect of embedding the fibres in resin is that surface flaws are eliminated, with the consequence that the operation of bulk flaws affects the failure of all the gauge lengths tested, which helps to explain the compliance of the supported fibre data with the non-parametric test (this test assumes that fibres must be similar in all respects except length).
6.2 Comparison with Impregnated Bundles

In order to develop a better understanding on fibre strength, it is useful to take a step further and extend the study to the strength of impregnated bundles of the same fibre. Although it is known that the failure mechanism for impregnated bundles is quite different from the previous two systems studied (unsupported and supported fibres), the same theme is pursued here which only considers the tensile strengths of impregnated bundles. The data are investigated for compliance with the weak-link hypothesis, and the use of similar gauge lengths to the work already described (chapters four and five) allows a direct comparison between the three types of sample.

A large number of prepared specimens, each comprising 1000 single filaments, were loaded monotonically in tension to failure. A mean diameter, comprising three readings, was taken for each specimen and the tensile strength was calculated using this value. Failure of all the impregnated bundles was instantaneous and occurred without warning. There were no signs of damage prior to failure either in the load/displacement curve (which is linear to failure) or by audible acoustic emission from the specimen. There was little splitting of the bundles at failure.

Table 6.2 shows diameter measurements for all the impregnated bundles as single data set for all the specimens at the four gauge lengths. The measured mean diameter for each gauge length is also indicated. The table indicates similar mean diameters with respect to the four gauge lengths. This is shown by the uniformity of the standard deviations across the set of gauge lengths. The contribution of resin on the measured diameter, which binds together all the
filaments, is not negligible. The diameter of the 1000 fibre bundle used in this work, assuming fibre/fibre contact in close packing and based on a mean value of 7.75μm for the diameter of a single fibre, would be 0.28mm, (Appendix 1). The value of 0.28mm is the corrected bundle mean diameter which is very important since it will have a direct effect on the bundle strength. This will be discussed later in this section.

Fracture stresses were calculated for each specimen using their mean diameter and failure load. The strength distribution of the data is assumed to comply with 2-parameter Weibull as discussed in section 2.1.3.

Figure 6.3 displays the cumulative distribution function for each gauge length. The plot shows a regular spacing between all the gauge lengths as expected from the weak-link hypothesis. A Weibull probability plot is shown in Figure 6.4, giving a very good fit for all gauge lengths and Table 6.3 shows the Weibull parameters for the data. Comparing these results with those obtained earlier, at a fixed gauge length the characteristic strength of the impregnated bundles is much greater than that of the single fibres, either supported or unsupported. In fact the characteristic strengths for the fibre component of the bundles is even greater since diameter in this system, as mentioned earlier, includes a resin contribution which needs to be excluded if the cross section for the fibres alone is sought. Using the value for true diameter of the fibres alone, (i.e. excluding the resin) gives strength data presented in Table 6.4, which are approximately 15% stronger for each gauge length. It is clear that the strength data for the bundles shows a large increase for any gauge length from that of the equivalent unsupported or supported single fibres.

The Weibull moduli for the impregnated bundles (Table 6.3) are relatively uniform
for the four gauge lengths and very adequately satisfy the strength/length relationship implicit in the Weibull model. The high value of about 20 indicates a relative uniformity in strengths even better than the results for the supported single fibres.

Figure 6.5 confirms the conformity of these results with the Weibull model. It shows a plot of ln (strength) versus ln (length), from which the slope should be (-1/W). The Weibull modulus obtained from this plot is 17.36, which is in close agreement with the values for W shown in Table 6.3. In other words, the strength data for impregnated bundles conforms very well to the Weibull model.

The similarity of the parameters W and a (where a is the characteristic stress for unit length, 1 mm) shown in Table 6.3 also indicates compliance with the weak-link hypothesis. This hypothesis when tested using the non-parametric test for the expected order of gauge length failure also gives results that conforms very well to predictions. The order of failure for the 75, 30, 12 and 5 mm occurred in 25% of the tests compared to the predicted value of 27.71%. Weak-link behaviour appears to be clearly followed for the impregnated bundles of fibres.

The fact that impregnated bundles display relatively uniform strengths with a high characteristic strength suggests that an impregnated bundle does not fail as a result of an isolated first fibre failure. It is possible that bundle failure requires a group of interacting fibre breaks to initiate total failure. When the first fibre breaks, all the remaining fibres will be very close to their ultimate load, therefore, a small over-load will propagate a complete failure. Thus produce the observed "brittle" failure mode.

The mechanism involved may follow the Batdorf (54) model, section 2.4. After
the first sporadic fibre break occurs, its surviving neighbours experience a stress concentration brought about by local load sharing (LLS), which increases their probability of failure. Under increasing stress, the stress concentration around the first fracture (first i-plet or singlet), causes a second fibre failure to give a diplet. The stress concentration becomes more severe and the probability of forming a higher order i-plet increases. Eventually a critical i-plet forms and failure spreads from one fibre to another with no further increase in stress required resulting in catastrophic crack propagation. Finally the bundle fails. The process is diagrammatically represented by Figure 2.6.

A singlet grows to failure as the load reaches the value necessary to fail adjacent fibres. Examination of the data for single supported fibres shows that this is to far exceed the strength of weak-links found within a 5mm segment, Figure 5.7. On average, the fibre strength in the impregnated bundles is increased by about 47% compared to the single supported fibres. Interestingly, the length of single fibre which would have a characteristic strength equivalent to this value can be obtained by extrapolation of the data in Figure 5.7. A value of 0.014mm is obtained (at a stress of 262.43 GPa) and this is consistent with the flat topography of the brittle type of failure. This reinforces the argument that there is a strength/length relationship in the composite and that failure is dependent on that critical i-plet (0.44mm length) for the propagation of a crack that leads to catastrophic "brittle-mode" failure.

To summarise, impregnated bundles were found to have similar mean diameters with respect to the four gauge lengths. Their strength values were shown to be higher than even supported single fibres. Removal of resin contribution on the bundle mean diameter gave the corrected bundle mean diameter and increased the bundle strength by 15%. The strength values for the four gauge lengths were
found to be relatively uniform suggesting that impregnated bundles do not fail as a result of an isolated first fibre failure but rather due to the interaction of a group of fibres. Weibull statistics performed on the impregnated bundle data were shown to adequately satisfy the strength/length relationship implicit in the Weibull model. Weak-link behaviour was also shown to be followed.
Table 6.1 Characteristic strength values for the global data set of the unsupported and the supported fibres using their respective mean diameters compared to the global data of the unsupported fibre when using a mean diameter of $7.75 \mu m$ as a nominal value.

<table>
<thead>
<tr>
<th>Gauge length (mm)</th>
<th>$\sigma_0 (L)$ (unsupported fibre)</th>
<th>$\sigma_0 (L)$ (supported fibre)</th>
<th>$\sigma_0 (L)$ (unsupported fibre using a nominal diameter value)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>4.34</td>
<td>3.78</td>
<td>4.08</td>
</tr>
<tr>
<td>12</td>
<td>3.57</td>
<td>3.67</td>
<td>3.34</td>
</tr>
<tr>
<td>30</td>
<td>2.55</td>
<td>3.47</td>
<td>2.40</td>
</tr>
<tr>
<td>75</td>
<td>2.26</td>
<td>3.24</td>
<td>2.17</td>
</tr>
</tbody>
</table>
Table 6.2 Impregnated mean bundle diameters with respect to their standard deviations (S.d).

<table>
<thead>
<tr>
<th>Gauge length (mm)</th>
<th>n</th>
<th>Mean diameter (mm)</th>
<th>S.d (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>34</td>
<td>0.30</td>
<td>0.02</td>
</tr>
<tr>
<td>12</td>
<td>34</td>
<td>0.29</td>
<td>0.02</td>
</tr>
<tr>
<td>30</td>
<td>34</td>
<td>0.30</td>
<td>0.03</td>
</tr>
<tr>
<td>75</td>
<td>34</td>
<td>0.30</td>
<td>0.03</td>
</tr>
</tbody>
</table>

Table 6.3 Regression estimates of the Weibull parameters for the impregnated bundles.

<table>
<thead>
<tr>
<th>Gauge length/mm</th>
<th>n</th>
<th>W</th>
<th>σo(L)</th>
<th>α</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>34</td>
<td>19.98</td>
<td>4.85</td>
<td>5.26</td>
</tr>
<tr>
<td>12</td>
<td>34</td>
<td>19.46</td>
<td>4.61</td>
<td>5.24</td>
</tr>
<tr>
<td>30</td>
<td>34</td>
<td>19.78</td>
<td>4.42</td>
<td>5.25</td>
</tr>
<tr>
<td>75</td>
<td>34</td>
<td>20.36</td>
<td>4.15</td>
<td>5.13</td>
</tr>
</tbody>
</table>
Table 6.4 Adjusted characteristic strength values using the corrected bundle mean diameter of the fibres only.

<table>
<thead>
<tr>
<th>Gauge length / (mm)</th>
<th>Adjusted $\sigma_0(L) ;/ \text{(GPa)}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>5.57</td>
</tr>
<tr>
<td>12</td>
<td>5.29</td>
</tr>
<tr>
<td>30</td>
<td>5.08</td>
</tr>
<tr>
<td>75</td>
<td>4.77</td>
</tr>
</tbody>
</table>
Figure 6.1 A global Weibull probability plot using failure load.
Figure 6.2 A plot of characteristic strength per gauge length against their respective (a) mid-value of a diameter range for the unsupported fibres and; (b) measured mean diameter for the supported fibres (in circle).
Figure 6.3 Probability of failure versus breaking stress for the impregnated bundles.

Figure 6.4 Weibull probability plot for the impregnated bundles.
Figure 6.5 $\ln$ (strength) versus $\ln$ (length) plot for the impregnated bundles.
CHAPTER SEVEN

7.0 Conclusions

The work involved examining and comparing the strength distribution of individual fibres in two different environments: in air (unsupported fibres) and embedded in epoxy-resin (supported fibres). It also involved measuring the strength of impregnated bundles of fibres for comparison purpose. The main objective is to improve the understanding of the fracture of fibres in the context of the weak-link theory. In order to examine them thoroughly, it was essential to test a large bank of specimens under similar experimental conditions, where a long length of a single fibre, or a bundle of fibres, was cut into four specimens of gauge lengths 5, 12, 30 and 75mm respectively. In all cases the carbon fibre used was taken from the same spool (Celion fibre), thus ensuring a consistent statistical base for the comparison. However, for the unsupported single fibre another PAN-based fibre (XAS fibre) was used to provide a direct comparison to the data of the Celion fibre under similar experimental conditions. All the data produced were characterised using the Weibull model and other statistical tests and the following conclusions were drawn.

7.1 Unsupported single fibres

1. The single unsupported Celion fibres were found to deviate strongly from the Weibull model and weak-link behaviour, this was thought to be due to diameter variation. The XAS fibre, on the other hand, was shown to comply with both the Weibull model and the weak-link behaviour, due to its diameter uniformity.
2. Diameter variation found in the Celion fibre raised serious doubt to the usual assumption made by previous authors that a nominal diameter value could be used in calculating stresses.

3. There was an increasingly strong dependence of strength on the fibre diameter as the gauge lengths decreases, this was due to diameter which produced different effects on the different lengths. In fact longer lengths (75 and 30mm) were shown to be length dependent and the shorter lengths (12 and 5mm) were dependent on both diameter and length.

4. The variation of diameters about the mean values across the set of gauge lengths were shown to vary in behaviour for both carbon fibres. This could be due to their respective manufacturing processes involved during their productions. The diameter variability about the mean value increased with gauge length increase for the Celion fibre. This was explained in terms of the likelihood that the reduced laser beam used to measure fibre diameter has a higher chance to measure diameter at the fibre's weak-link for the longer lengths than the shorter lengths. This was probably due to the weak-links being more uniformly randomised as the length increases. The X4s fibre, on the other hand, showed uniform diameter values irrespective of gauge length. This behaviour could be due to non-randomised weak-link distribution.

5. The 12mm length was shown to be the critical length, where diameter effect becomes important, since it displayed a pronounced bi-modal behaviour of mixed effect. Its lower stresses behaved like the 30mm and the higher stresses like the 5mm length.

6. The overall behaviour of the Celion fibre data was found to be complex and
this was shown to stem from two effects: firstly the presence of two classes of failure which may be represented by surface and volume flaws, and secondly within the volume flaw mode itself, there was a strong dependence on diameter.

7.2 Supported single fibres

1. There was a marked increase in strength for the 75, 30 and 12 mm length comparing to strength values obtained for the unsupported fibres. However, the 5 mm gauge length actually reduced in strength value comparing to its equivalent unsupported length.

2. The Weibull model conformed well to the data over the four gauge lengths tested and the weak-link property was restored, due to the apparent uniformity of the measured diameters for all the lengths.

3. The apparent uniformity of diameter values obtained for the supported fibres were due to the large averaging effect produced by the technique employed (W.I.S.E.) for measuring diameter. This technique was different to the one used for the unsupported fibres (laser), where there was little averaging effect.

4. The effect of length was found to be the only parameter affecting the failure of the fibre, since diameter was shown to be uniformly randomised.

5. The effect of the resin has reduced the role of surface flaws in all the gauge lengths.

6. Volume flaws, therefore, were the only dominant class of failure present.
7.3 Impregnated bundles

1. The impregnated bundles, tested under similar experimental conditions to the unsupported and supported single fibres, were found to be much stronger than even the supported fibres.

2. Their mean diameter was found to be similar irrespective of gauge length.

3. The strength values for the four gauge lengths were found to be relatively uniform suggesting that impregnated bundles do not fail as a result of an isolated first fibre failure but rather due to the interaction of a group of fibres.

4. The data were shown to adequately satisfy the strength/length relationship implicit in the Weibull model and the weak-link property.
APPENDIX 1

Assuming circular section for impregnated bundle, diameter I.

Therefore, area of impregnated bundle \( = \frac{\pi}{4} I^2 \)

But area available of fibre assuming a closed packed \( = 0.76 \cdot \frac{\pi}{4} I^2 \)

structure

Now we know:

Diameter of a single filament, \( d = 7.75 \mu m \)

One bundle \( = 1000 \) filaments

Area of impregnated bundle \( = 1000 \cdot \frac{\pi}{4} \cdot d^2 \)

Therefore, \( 0.76 \cdot \frac{\pi}{4} I^2 = 1000 \cdot \frac{\pi}{4} \cdot d^2 \)

\( 0.76 \cdot I^2 = 1000 \cdot d^2 \)

\( I^2 = 36.27 \cdot d \)

As an example for working out, using the 5mm g.l impregnated bundle.

Measured diameter of impregnated bundle (fibres + resin) = 0.30mm

corrected diameter of impregnated bundle (no resin) = 0.28mm

Now if we use 0.28mm as the diameter of the bundle ignoring the resin,

the strength values of the 5mm g.l will increase to 5.57GPa from 4.85GPa.

The percentage increase in strength is \((5.57 - 4.85)/4.85 \%\)

i.e. \(14.85\%\)

Similarly, applying 14.85% to other gauge lengths gives the strength values presented in Table 6.3.
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SUPPLEMENTARY APPENDIX

The following supplementary materials are included to amplify some of the points raised in the thesis.

Supplementary Tables 1 and 2 are representative data sets for three diameter readings for each gauge length specimen for the unsupported and the supported fibres respectively. Table 1 shows the range of the three diameter readings for the unsupported fibres about their mean value using the laser diffraction method. The Table shows the diameter range increases with increasing gauge length. However, this behaviour across the fibre lengths is never so significant as to seriously affect the breaking stress values derived from using their mean diameter. This behaviour suggests the likelihood that the average mean diameters coincide at or near the breaking points for the 5mm lengths is higher than for the 75mm specimens. Table 2, on the other hand, shows the diameter values for the supported fibres (measured by the Watson shearing eye-piece) to have a much smaller range about their respective mean value and is randomised across the four lengths. The other data sets tested for both the unsupported and the supported fibres also behaved in the same manner.

Typical load-extension curves are shown in Supplementary Figure 1. This Figure shows, as an example, the first eight observations comprising four gauge length specimens pertaining to data set '88 (their respective diameter measurements are shown in Supplementary Table 1). Other data sets tested behaved in similar fashion to this one. There is a noticeable inconsistency in the initial portion of each curve up to approximately 0.1% strain. This can be attributed to a slack in the specimen being removed during tensile testing. This portion is not included in the calculations of the Young's modulus. Each curve, although almost straight, displays an upward curvature indicating an increase in Modulus as strain increases. The load-extension curves show an increasing slope for strains beyond approximately 0.3 and 0.4%. The fibre can therefore be characterised in terms of an initial elastic modulus, which behaves in a hookian fashion, and a non-hookian final breaking modulus (beyond 0.3 and 0.4% strains). This non-linear stress-strain relationship in the latter part of each curve can be related to stress-induced stiffening effect in the fibre preferred orientation, where an initial alignment of the C=C bonds results in a consequent rise in stiffness. The existence of this feature in the curve means that the Young's modulus values cannot be calculated in the conventional manner. Therefore, two Young's modulus values were determined, one between approximately 0.1 and 0.4% strain and a second value was calculated by obtaining the fibre breaking elongation from the abscissa of an extrapolated straight line and the final breaking load. Supplementary Table 3 gives these two respective values for each specimen shown in Figure 1. The Table indicates that the difference between the two moduli values increases with increasing fibre length for each set of observation. This behaviour may be due to the diameter range about the mean value which increases with the fibre length.
(Supplementary Table 1). The Table also shows that large and small diameter values have different effects on the modulus of the 5mm length fibres only. This observation is very important since the use of different moduli value for small and large diameters may be relevant for calculating the strength of the 5mm supported fibres, since this length showed its strength to be diameter dependent.

Supplementary Figures 2 and 3 show atomic force microscopy (AFM) images for the Celion and the XAS fibres respectively. Supplementary Figure 2 shows several AFM images of the Celion fibre. The important feature is that this fibre indicates a complex corrugated structure. The Figure also reveals the presence of a defect. The XAS fibre, on the other hand, was also examined for comparison purposes and its images are shown in Supplementary Figure 3. The Figure shows this fibre having a structure which is mainly typical of most modern carbon fibres.

Overall conclusions

There were many conclusions drawn from this work, however the overriding ones are listed below:

1. In the fibre material two classes of flaws can be recognised from the statistical data.

2. An effect of diameter can be recognised when the fibre length is shorter than 12mm in length.

3. Embedding the single fibres in epoxy-resin has the effect of eliminating one of the two classes of flaws.
Supplementary Table 1  Representative data set (Data set '88) of three diameter (d) readings for each unsupported fibre specimen showing their diameter variation about their respective mean value.

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* o.m.d. is the overall mean diameter for the data set.
Supplementary Table 2 Representative data set (Data set two) of three diameter (d) readings for each supported fibre specimen showing their diameter variation about their respective mean value

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**Supplementary Table 3** Two calculated Young's modulus values for each gauge length (g.l.) shown in Figure 1

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<td>Young's modulus from breaking strain</td>
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*Manufacturer's Published value for the Young's modulus is 228GPa*
Supplementary Figure 1  Representative load-extension curves for the first five observations (n) for data set '88 comprising the four gauge lengths. (Full-load scale is 20g and magnification ratio of chart speed to cross head speed is 300:1) N.B. (a), (b), (c) and (d) are 5, 12, 30 and 75mm gauge lengths respectively.
Supplementary Figure 2  AFM images of desized Celion fibre.
Supplementary Figure 3  AFM images of desized X6 fibre.