The Effect of Fibre-Bundling on the Mechanical Properties of a Short-Fibre Composite

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To Mum and Dad
Abstract

It has been suggested that the use of fibre bundles rather than individual fibres can improve the toughness properties of a short-fibre composite. Previous experimental work on this topic employed materials in which bundles were impregnated prior to manufacture or materials with poorly defined fibre-bundling. This study is the first to consider the mechanical properties of a series of materials where the bundles have been impregnated during manufacture of the material, and the materials tested contained a well-defined proportion of fibres within bundles of a known size.

A novel manufacturing technique has been developed that can be used to produce short carbon fibre reinforced polypropylene materials with a controlled proportion of fibres in bundles. Materials manufactured in this work contained 0 %, 25 %, 50 %, 75 % and 100 % of the fibres in bundles. The fibres had a length of 5 mm or 10 mm and the bundles contained either 1000 or 6000 fibres.

An increase in the proportion of fibres within bundles results in a decrease in the tensile modulus of the short-fibre composites. This decrease was less severe for materials containing bundles with a greater aspect ratio or laminates with a greater thickness. A model for the modulus of the materials has been developed which illustrates some of the effects of fibre-bundling on the structure of a short-fibre composite. For the materials studied, tensile strength of materials containing bundles was one quarter of the tensile strength of the filamentised material.

Only one combination of fibre length and bundle size resulted in a clear increase in toughness, as measured by $J_c$, compared to the filamentised material and this increase appears to be due to areas of unreinforced matrix in the material. Materials containing both filamentised fibres and fibre bundles had relatively low values of $J_c$. The fracture surfaces were imaged and three distinct ways in which a bundle may fail have been identified. Discussion of the fracture mechanisms active in these materials concludes that the use of fibre-bundling to improve toughness is unlikely to be effective due to the mechanism that has been proposed.
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Nomenclature

Material Codes
The materials manufactured in this study are described in the form “x mm / y % z” where x mm is the length of the fibres and y % is the proportion of fibres within bundles containing z fibres. If none of the fibres are within bundles then y = 0 and the quantity z is omitted. For instance, the material described as “5 mm / 50 % 6k” consists of 5 mm long fibres, 50 % of which are within bundles of 6000 fibres.

A series of materials is described in the form “x mm / z” where x mm is the length of the fibres and z is number of fibres per bundle. A series of materials contains the materials with all proportions of fibres within bundles (including 0 %). For instance, the series “5 mm / 6k” describes all materials containing 5 mm long fibres and, if the materials contains bundles, 6000 fibres per bundle.

Symbols
A* area bounded by load-extension curve and secant line
a crack length
af fibre fraction
B thickness of specimen
C compliance of specimen
C̄ non-linear correction factor
d diameter of reinforcing unit
E tensile modulus
Eb tensile modulus of bundle
Ec tensile modulus of composite
Ef tensile modulus of fibre
Em tensile modulus of matrix
G shear modulus
Gb shear modulus of bundle
Gf shear modulus of fibre
\( G_m \)  shear modulus of matrix
\( G \)  strain energy release rate
\( G_c \)  critical strain energy release rate
\( \tilde{G}_c \)  critical non-linear strain energy release rate
\( J \)  J integral
\( J_c \)  critical J integral
\( J_{cc} \)  critical J integral of composite material
\( J_{cm} \)  critical J integral of matrix
\( J_{el} \)  critical J integral due to elastic displacement
\( J_{pl} \)  critical J integral due to plastic displacement
\( k \)  non-linearity parameter
\( K \)  stress intensity factor
\( K_c \)  fracture toughness
\( K_{cc} \)  fracture toughness of composite material
\( K_{cm} \)  fracture toughness of unfilled matrix
\( l \)  fibre length
\( l_c \)  critical length
\( M \)  microstructural efficiency factor
\( m_f \)  mass of fibre
\( m_m \)  mass of matrix
\( n \)  non-linearity parameter
\( P \)  load
\( P_c \)  load at crack extension
\( r_f \)  radius of fibre
\( r_b \)  radius of bundle
\( r_y \)  radius of plastic zone
\( R \)  half of the distance between fibres
\( s \)  length of arc
\( T \)  stress vector
\( u \)  displacement vector
\( U \)  potential energy
\( V_b \)  volume fraction of bundles
\( V_f \)  \hspace{1em} \text{volume fraction of fibres} \\
\( V_{fb} \)  \hspace{1em} \text{volume fraction of fibres within bundles} \\
\( V_{fo} \)  \hspace{1em} \text{volume fraction of fibres outside bundles} \\
\( V_m \)  \hspace{1em} \text{volume fraction of matrix} \\
\( V_{mb} \)  \hspace{1em} \text{volume fraction of matrix within bundles} \\
\( V_{mo} \)  \hspace{1em} \text{volume fraction of matrix outside bundles} \\
\( W \)  \hspace{1em} \text{width or half-width of specimen} \\
\( W_{Charpy} \)  \hspace{1em} \text{energy absorbed in Charpy test} \\
\( W_e \)  \hspace{1em} \text{strain energy density} \\
\( x \)  \hspace{1em} \text{rectangular co-ordinate} \\
\( y \)  \hspace{1em} \text{rectangular co-ordinate} \\
\( Y \)  \hspace{1em} \text{geometric factor} \\
\( \beta_{Cox} \)  \hspace{1em} \text{length efficiency parameter from Cox model} \\
\( \beta_{Nairn} \)  \hspace{1em} \text{length efficiency parameter from Nairn model} \\
\( \gamma_f \)  \hspace{1em} \text{work of fracture} \\
\( \delta \)  \hspace{1em} \text{displacement} \\
\( \delta_{el} \)  \hspace{1em} \text{elastic component of displacement} \\
\( \delta_{pl} \)  \hspace{1em} \text{plastic component of displacement} \\
\( \Gamma \)  \hspace{1em} \text{curve surrounding the crack tip} \\
\( \eta_l \)  \hspace{1em} \text{length efficiency factor for fibres} \\
\( \eta_{lb} \)  \hspace{1em} \text{length efficiency factor for bundles} \\
\( \eta_{lo} \)  \hspace{1em} \text{length efficiency factor for fibres outside bundles} \\
\( \eta_o \)  \hspace{1em} \text{orientation efficiency factor} \\
\( \nu \)  \hspace{1em} \text{Poisson's ratio} \\
\( \theta_f \)  \hspace{1em} \text{fibre orientation relative to loading direction} \\
\( \rho_f \)  \hspace{1em} \text{density of fibre} \\
\( \rho_m \)  \hspace{1em} \text{density of matrix} \\
\( \sigma_{c*} \)  \hspace{1em} \text{fracture stress of composite} \\
\( \sigma_{f*} \)  \hspace{1em} \text{fracture stress of fibre} \\
\( \sigma_y \)  \hspace{1em} \text{yield stress} \\
\( \tau \)  \hspace{1em} \text{interfacial bond strength}
Chapter 1. Introduction
1.1 Short-Fibre Composites

A composite material can be defined as a multi-phase material in which the phase distribution and geometry have been controlled. Although this general definition could be applied to any multi-phase material, the term “composite” is usually applied to systems in which the structure has been deliberately engineered. One phase is usually continuous and is designated the “matrix”; the other phase is distributed and is described as the “reinforcement”. This thesis is concerned with polymer matrix composites, which are defined here as consisting of a continuous polymer matrix containing a reinforcement.

The reinforcing phase of a polymer matrix composite material can be in a wide range of forms. The shape of the reinforcement is often classified in terms of the length to diameter ratio, known as the aspect ratio. At one extreme, the aspect ratio of the reinforcement has a value around one if the reinforcement is particulate. At the other extreme, the aspect ratio of the reinforcement tends to infinity if the reinforcement is a continuous fibre. Between these two extremes is a class of reinforcement known as discontinuous or short-fibre reinforcement. The aspect ratio of short-fibre reinforcement may range from under ten to several thousand.

The best mechanical properties are obtained by the use of continuous fibres, but such composite materials are expensive to produce, particularly in complex shapes. Although short-fibre composites have reduced mechanical properties they offer significant processing advantages and they may be produced in complex shapes using relatively cheap manufacturing techniques. For this reason short-fibre composites are produced in large volumes and are used in commodity products. Since the major advantage of short-fibre composites is based on cost, there is a continued interest in producing a material with the required properties as efficiently as possible.

1.2 Mesostructure in Short-Fibre Composites

The longitudinal Young’s modulus of a continuous aligned composite material can be predicted satisfactorily from the constituent moduli and the fibre volume fraction using
a simple rule-of-mixtures expression. Such an expression may be modified for a short-fibre composite by introducing factors which account for the reduced reinforcing efficiency of short-fibres (e.g. Cox, 1952) and the orientation of the fibres (e.g. Krenchel, 1964). In calculating the reduced reinforcing efficiency, it is usually assumed that individual fibres are the basic unit of reinforcement and that they are straight and regularly spaced.

In real short-fibre composites, such assumptions about the nature of the reinforcement may be invalid because of features such as fibre-bundling and fibre waviness. These structural features, on a scale between that of the individual fibre and that of the material as a whole, have been described as mesostructure (Piggott, 1992). For a short-fibre composite, mesostructure is likely to take the form of additional ordering of fibres within the material which may affect the orientation or packing of the fibres. The development of mesostructure is greatly affected by the processing conditions of the material and Friedrich (1998) has described the development of mesostructure during processing for a number of polymer matrix composite systems. Mesostructures are not usually a feature of models for short-fibre composites and a better understanding of the influence of mesostructure on mechanical properties can assist the more efficient use of these materials.

1.3 Fibre-Bundling in Short-Fibre Composites

It has long been suggested that the toughness of a short-fibre composite could be improved by the use of fibres with a larger diameter, an idea based on the theory of pull-out proposed by Cottrell (1964) which suggests that the energy absorbed by fibre pull-out is proportional to the diameter of the reinforcing fibre. However, the use of a larger diameter fibre has the disadvantage of a reduced reinforcing efficiency and probable reduced fibre strength. An alternative to using a larger diameter fibres is to use fibre bundles since the constituent fibres of the bundles are of the same diameter and quality as an individual fibre, while the pull-out behaviour of the fibre bundle should be similar to that of a large diameter fibre. Hence, the use of fibre bundles offers a possible enhancement of toughness for short-fibre composites and a number of workers have studied the effect of fibre-bundling.
Chapter 1. Introduction

The most frequently used method to manufacture a composite with bundles is to impregnate a fibre tow with a resin, which is then cured, and the resulting rod is subsequently combined with resin to form the laminate. Studies by Fila et al. (1972) and Kim and Mai (1993) recommended this approach for achieving tougher composite materials. However, this approach does not mimic fibre-bundling as it is encountered within mass-manufactured short-fibre composites.

Further complications are associated with the difficulty of quantifying fibre-bundling. It is generally the case that at least some of the fibres within a commercial short-fibre composite are in clusters, or even bundles, reflecting the tow structure from which the fibres were cut originally. Methods for quantifying the fibre-bundling in a random short-fibre composite are currently under development. For example Worrall and Wells (1996) have developed a technique which they have successfully used to analyse the size of bundles in a material. However it is not possible, at present, to find the volume fraction of bundles of a given size using this technique and this information is necessary to assess the effect of fibre-bundling on material properties. Hence, in this study it has been necessary both to produce a fibre mat with a known proportion of fibres within bundles of a known size and to retain this structure during processing in order to be able to relate property changes to fibre-bundling.

1.3 Aims and Outline of Thesis

The present work is aimed at characterising and, where possible, modelling the effect of fibre-bundling on the mechanical properties of a carefully controlled range of short-fibre composite materials in which the proportion of fibres within bundles has been varied in a controlled manner. The mechanical properties to be characterised are tensile strength, tensile modulus and toughness. The objectives are to establish if the use of fibre bundles improves the toughness properties of the short-fibre composites and whether there is a reduction of the modulus and/or strength associated with the use of fibre bundles.
Chapter 1. Introduction

The plan of the thesis is as follows. A review of literature related to the mechanical properties of short-fibre composites and fibre-bundling in composite materials is presented in Chapter 2. Chapter 3 describes the technique developed to manufacture short-fibre composites with a controlled proportion of fibres within bundles and the methods used to characterise the materials. The short-fibre composites used in this work have been manufactured in a way that ensures that the material properties are consistent, apart from effects arising from variation of the proportion of fibres within bundles.

Modulus and strength results are reported and discussed in Chapter 4. A model for the modulus of the materials is described and the predictions from this model are compared with experimental data in Chapter 5. In Chapter 6 toughness results and a discussion of their validity are presented. Chapter 7 describes an investigation of the fracture mechanisms that occur in these materials. Finally, Chapter 8 presents the conclusions and suggestions for further work.
Chapter 2. Literature Review
2.1 Introduction

The addition of short-fibre reinforcement, such as carbon fibre, to thermoplastic materials can considerably enhance their mechanical properties. A large number of factors influence the properties of the resulting composite material and many parameters may need to be specified for the material to be fully described. Even then, mechanical properties, particularly strength and toughness, can be difficult to predict. The present Chapter presents a review of literature relevant to the current study of the effect of fibre-bundling in a short-fibre composite. Sections 2.2 to 2.4 discuss literature related to the stiffness, strength and toughness properties of short-fibre composites. A key parameter is the critical length, which affects both strength and toughness. This may be evaluated experimentally and methods for the evaluation of the critical length are described in Section 2.5. The topic of mesostructure, introduced in Chapter 1, is discussed in more detail in Section 2.6. An example of a mesostructure is fibre-bundling, discussed in Section 2.7. Literature related to experimental and theoretical aspects of fibre-bundling is discussed in Sections 2.8 and 2.9 respectively. Analysis of fibre-bundling in a composite material is discussed in Section 2.10. Finally, conclusions from the present Chapter concerning fibre-bundling in a short-fibre composite are presented in Section 2.11.

2.2 Stiffness Properties

2.2.1 Rule-of-Mixtures

One of the simplest approaches for the prediction of elastic behaviour of a composite material is the "rule-of-mixtures". The equations describe an idealised composite consisting of an array of continuous, straight, aligned fibres, geometrically arranged within a matrix that is perfectly bonded to the fibres. For the case of an applied load parallel to the fibres, both the fibres and matrix experience the same strain. From this iso-strain assumption an expression for the composite modulus $E_c$ can be derived:

$$E_c = E_f V_f + E_m V_m.$$  \hspace{1cm} (2.1)
where $E_f$ and $E_m$ are the modulus of the fibre and matrix, and $V_f$ and $V_m$ are the volume fraction of fibre and matrix. This simple model, known as the Voigt model, provides accurate predictions for the modulus of a uniaxial composite material containing a high volume fraction of continuous fibre. The Voigt model is often described as the upper bound for the Young's modulus.

For the case of a load applied transverse to the fibres, fibres and matrix are assumed to bear the same stress. The resulting expression for the composite modulus, known as the Reuss model, is:

$$E_c = \frac{E_f E_m}{E_f V_m + E_m V_f}.$$  

(2.2)

The Reuss model is less accurate than the Voigt model as strain concentration around the fibres means that the iso-stress assumption is not reasonable. The Reuss model is often described as the lower bound for the Young's modulus.

Both the Voigt and Reuss equations consider a composite material with continuous fibre reinforcement. As fibre length decreases the effect of the fibre ends become increasingly important. To evaluate the reinforcing efficiency of a discontinuous fibre it is necessary to consider the nature of stress transfer to the fibre.

2.2.2 Effect of Fibre Length

Reduced reinforcing efficiency of discontinuous fibres can be accounted for in the Voigt model (Equation 2.1) with the introduction of a length efficiency factor $\eta_l$ thus:

$$E_c = \eta_l E_f V_f + E_m V_m.$$  

(2.3)

By considering a short, stiff fibre of radius $r_f$ and length $l$ in the centre of a lower modulus matrix cylinder of radius $R$, Cox (1952) derived an expression for $\eta_l$: 

8
\[ \eta_l = 1 - \left( \frac{\tanh(\beta_{Cox} l/2)}{\beta_{Cox} l/2} \right) \]  \hspace{2cm} (2.4)

where

\[ \beta_{Cox} = \left( \frac{2G_m}{E_f r_f^2 \ln(R/r_f)} \right)^{1/2} \]  \hspace{2cm} (2.5)

where \( \beta_{Cox} \) is the length efficiency parameter from the Cox model, \( G_m \) is the shear modulus of the matrix and \( R \) is half the inter-fibre spacing.

A number of assumptions are contained in this analysis: it is assumed that no load is transferred by the fibre ends, that the matrix and fibre are both elastic, that the interface between fibre and matrix is perfect and infinitesimally thin and that the matrix at the interface has the same properties as the bulk matrix. Although each of these assumptions may be debatable, the Cox model does provide reasonable, although not exact, predictions for the Young’s modulus of many short-fibre composites (Bader and Hill, 1993).

A deficiency of the Cox analysis is that because the interface between fibre and matrix is assumed to be perfect there is no effect of interfacial strength. An alternative analysis proposed by Kelly and Tyson (1965) is more appropriate for a system in which the matrix or interface has failed resulting in a purely frictional fibre-matrix coupling. In the Kelly-Tyson analysis the strain in the fibre and matrix are equal except over a characteristic length from the fibre end. This characteristic length is half the critical length where the critical length \( l_c \) is given by:

\[ l_c = \frac{\sigma_f^* r_f}{\tau} \]  \hspace{2cm} (2.6)

where \( \sigma_f^* \) is the fracture stress of the fibre and \( \tau \) is the interfacial bond strength. The critical length is the minimum fibre length for which the tensile stress in the fibre can equal the fracture stress of the fibre.
Galiotis et al. (1984) measured the strain at the fibre surface in a polymer matrix composite material using Raman spectroscopy. This work shows that the strain distribution is very similar to that predicted by the Cox model below a certain threshold strain. At this threshold strain interfacial failure occurs, either by shear debonding at the interface or by yielding of the matrix. After this failure has occurred the measured strain distribution was similar to the Kelly-Tyson model. This suggests that where there is a chemical bond between the fibre and matrix Cox-like behaviour will be observed. After the failure of this chemical bond a smaller stress will be transferred across the interface by mechanical or frictional mechanisms, closer to the Kelly-Tyson model.

A number of modifications to the original Cox (1952) analysis have been proposed subsequently. These differ in detail or are appropriate for a particular class of composite materials. For example Fukuda and Chou (1981) proposed a model accounting for stress transferred across the fibre ends, while Clyne (1989) proposed a model for composite materials with a relatively small difference in the moduli of the fibre and matrix.

A more exact shear-lag analysis has been developed by Nairn (1997). This analysis is derived from exact equations of elasticity which eliminate some assumptions of simpler models. The length efficiency factor is found from Equation 2.4:

\[
\eta_l = 1 - \left( \frac{\tanh(\beta_{Nairn} l/2)}{\beta_{Nairn} l/2} \right) \tag{2.7}
\]

where \( \beta_{Nairn} \) is the length efficiency parameter given by:

\[
\beta_{Nairn} = \left[ \frac{2}{r_f^2 E_f E_m} \left( \frac{\frac{E_f V_f}{4 G_f} + \frac{E_m V_m}{4 G_m}}{\frac{V_m}{2} - \frac{1}{V_f} \frac{1 - V_m}{2}} \right) \right]^{1/2} \tag{2.8}
\]
where $G_f$ and $G_m$ are the shear moduli of the fibre and matrix respectively. Nairn (1997) demonstrated that this equation gives better agreement with a finite element analysis approach than the Cox (1952) model.

The methods described so far for prediction of the Young’s modulus of an aligned short-fibre composite have been based on the rule-of-mixtures. Alternative methods have been developed by Halpin (1969) and Eshelby (1957). The method of Halpin (1969) was developed as an extension to the work of Halpin and Tsai (1967). For this method to be used successfully it is necessary to determine quantities empirically from experimental results or from numerical treatments of the material. The equivalent inclusion method of Eshelby (1957) may be used to determine the modulus of a short-fibre composite. This method has been successfully used to predict the modulus of short-fibre composites, however, for polymer matrix materials the large difference between the modulus of the fibre and matrix can reduce the accuracy of predictions from this method.

2.2.3 Effect of Fibre Orientation

The models discussed in Section 2.2.2 have all considered a material in which the fibres are perfectly aligned. Orientation of the fibres may be accounted for by including an orientation efficiency factor $\eta_o$ in Equation 2.3, so that:

$$E_c = \eta_o \eta_l E_f V_f + E_m V_m$$  \hspace{1cm} (2.9)

An expression for $\eta_o$ was proposed by Krenchel (1964). The value of $\eta_o$ can be calculated from a summation of the fibre fraction $\alpha_f$ at an angle $\Theta_f$ to the applied load so:

$$\eta_o = \Sigma \alpha_f \cos^4 \Theta_f .$$  \hspace{1cm} (2.10)
This expression gives a value for $\eta_o$ of 0.375 in the case of a two dimensional random composite and a value for $\eta_o$ of 0.2 for a three dimensional random composite. For a fully aligned composite the value of $\eta_o$ is unity, so Equation 2.9 will reduce to Equation 2.3.

2.3 Strength Properties

2.3.1 Strength of Unidirectional Laminates

Strength of composite materials is considerably more difficult to predict than modulus as it requires both an accurate stress analysis and a failure criterion. A thorough treatment requires that the mode of failure is established by the observation of composite material failures. The simplest strength predictions are, as with stiffness, derived from rule-of-mixtures arguments e.g. Kelly (1973).

For a unidirectional composite material loaded parallel to the fibres, the fibres will fail before the matrix if the fibres have a lower strain to failure. For a composite material with a low volume fraction of fibres, the matrix will carry load after the fibres have failed and therefore strength is controlled by the matrix. While for materials with a higher volume fraction of fibres, the matrix will fail with the fibres as the load transferred to the matrix is too great for the matrix to support. Simple expressions for the strength of the composite material can be developed e.g. Hull (1981).

Tensile strength transverse to the fibres is more difficult to predict than the strength parallel to the fibres because the transverse strength is strongly influenced by many factors. Influences include the properties of the fibre-matrix interface and the presence of voids in addition to the fibre and matrix properties. For a composite material with poor fibre-matrix adhesion the transverse strength is dominated by the matrix. When fibre-matrix adhesion is good the fibre and matrix may remain bonded. In this case there may be strain magnification in the matrix around fibres, particularly between fibres, which results in a reduction in the strength of the composite material.
In reality fibres do not possess a unique failure stress. Fibre strength will vary from fibre to fibre and along the length of an individual fibre. These effects may be described by Weibull statistics. However these effects are less significant in short fibre composite materials so are not discussed further.

2.3.2 Strength of Short-fibre composites

While the stiffness properties of a short-fibre composite can be predicted reasonably well, the prediction of strength values is more challenging. There is an inadequate understanding of fracture processes for these materials, in particular the role of fibre architecture. As with stiffness, the way in which stress is transferred from the matrix to the fibre is critical and may be described by a model such as Cox (1952) or Kelly and Tyson (1965). The critical length has significance for the strength properties as it is the minimum length of fibre for which the stress transferred into the fibre is sufficient for fibre breakage. Fibres of a length less than the critical length cannot be broken during the material failure but may be pulled out.

For an aligned short-fibre composite the rule-of-mixtures may be applied. However, as with stiffness, the properties will be affected by the reduced reinforcing efficiency of short-fibres compared to continuous fibres. In particular, the length of the fibre relative to the critical length is an important factor.

For a random planar short-fibre composite there is no single satisfactory model for predicting strength. It is possible to use the rule-of-mixtures approach for some materials where the properties of the material are dominated by the fibres. An alternative to the rule-of-mixtures approach is the method of Halpin and Kardos (1978) which treats a short-fibre composite as a quasi-isotropic laminate consisting of laminae oriented at 0°, 90° and ±45°. This approach has given good agreement with experimental results but has the disadvantage of requiring accurate values of failure stress and strain for a variety of unidirectional laminates. The prediction of tensile strength for a random planar short-fibre composite is complex and generally requires experimental quantities that can be difficult to obtain. In addition there is a limited amount of experimental data to validate models.
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It should be emphasised that the prediction of strength for short-fibre composites is very different to the prediction of stiffness. Stiffness properties are a result of an averaging process and allow simplifying assumptions to be employed. However strength properties may be affected by microscopic factors and require detailed modelling combined with a statistical approach to the fibre distribution.

2.4 Toughness Properties

2.4.1 Toughness of Short-fibre composites

Toughness may be described as the energy per unit area required to create a new crack surface. Analysis of crack growth in composite materials is more complex than in homogenous materials. The failure of a composite material involves the failure of fibres, matrix and, critically, the interface between fibre and matrix. The energy required to increase the surface area of a crack depends on the failure mechanisms that occur in the composite material. Kim and Mai (1991) have identified a number of mechanisms in the failure of unidirectional composite materials: interfacial debonding, post-debonding friction, stress redistribution, fibre pull-out, creation of fracture surfaces, plastic shear of matrix and plastic shear of fibre. These mechanisms are, with modification, present in short-fibre composites.

For short-fibre composites with a relatively brittle matrix, such as epoxy or polyester resin, the fracture toughness is dominated by fibre pull-out so the presence of fibres increases the toughness of the composite material. In the case of a ductile thermoplastic matrix, such as polypropylene, the presence of fibres may reduce the toughness of the composite material. The reasons for this are that the presence of fibres reduces the ductility of the matrix and leads to a reduction in volume of tough matrix (Kim and Mai, 1991).

There are two widely used models for the prediction of toughness of short-fibre composites; the “microstructural efficiency” approach of Friedrich (1985) and the total fracture toughness theory of Lauke and Pompe (1986). Both approaches have been applied successfully to short-fibre composites.
Friedrich (1985) described the critical stress intensity factor of a short-fibre composite $K_{cc}$ with an equation of the form:

$$K_{cc} = M K_{cm},$$

where $M$ is the microstructural efficiency factor and $K_{cm}$ is the critical stress intensity factor of the unfilled matrix. The factor $M$ is affected by the deformation of the matrix material and the energy absorption associated with the fibres. This approach is semi-empirical and requires measurements at several different fibre volume fractions or fibre orientations.

An alternative approach was described by Lauke and Pompe (1986). The total energy absorbed was determined by a summation of the energy absorbed by each mechanism active in the material. Lauke and Pompe suggested that the relevant important mechanisms are: mode II debonding, sliding within the debonding region, fibre pull-out and matrix fracture. They developed an expression for the energy absorbed due to each mechanism. Predictions from this approach showed good agreement with experimentally evaluated energy release rates.

2.4.2 Linear Elastic Fracture Mechanics

Linear elastic fracture mechanics is a framework for the description of cracking in an elastic body. It is assumed that the crack is sharp and that energy is absorbed very locally around the crack tip. If the energy of the body is considered then a strain energy release rate $G$ may be defined for a given increase in crack length. The critical strain energy release rate $G_c$ is the value of $G$ at crack extension. $G_c$ may be evaluated for a specimen containing a single crack of length $a$ using the expression (Hertzberg, 1996):

$$G_c = \left( \frac{P^2}{2B} \right) \left( \frac{dC}{da} \right)$$

(2.12)
where $P_c$ is the load at crack extension, $B$ is the specimen thickness and $dC/da$ is the change in compliance of the specimen with crack length.

An alternative approach is to consider the stress condition at the crack tip as represented by the stress intensity factor. For a single edge crack in a plate the critical stress intensity factor $K_c$ may be evaluated using the expression:

$$K_c = \frac{YP_c}{BW} \alpha \sqrt{a}$$ \hspace{1cm} (2.13)

where $Y$ is a geometric factor, $P_c$ is the load at crack extension, $W$ is the specimen width and $a$ is the crack length. The value of $Y$ for an anisotropic material may be evaluated experimentally following Barnby and Spencer (1976).

The stress intensity description considers the local stress at the crack tip required for crack extension. This is in contrast to the critical strain energy release rate which considers the energy change in the entire body. For plane strain conditions in an isotropic material the two quantities are related by the expression (Hertzberg, 1996):

$$K = \frac{\sqrt{EG}}{(1-v^2)}$$ \hspace{1cm} (2.14)

where $v$ is the Poisson’s ratio of the material.

For plane stress conditions the quantities are related by:

$$K = \sqrt{EG}$$ \hspace{1cm} (2.15)

For any cracked material a region of plastic deformation will develop around the crack tip where the yield criterion is met. It is in this plastic zone that energy is absorbed. Linear elastic fracture mechanics is applicable to materials with limited plasticity at the crack.
tip. It is possible to make an estimate of the radius of the plane strain plastic zone $r_y$ using the expression (Hertzberg, 1996):

$$
r_y \approx \frac{1}{6\pi} \frac{K_c^2}{\sigma_y^2}
$$

(2.16)

where $K_c$ is the fracture toughness of the material and $\sigma_y$ is the yield stress of the material.

For linear elastic fracture mechanics to be applied to a specific material and geometry it is necessary for the dimensions of the specimen to be fifty times the size of the plastic zone. This condition is expressed in ASTM D 5045 (1996) as:

$$
a, B, (W - a) \geq 2.5 \left( \frac{K_c}{\sigma_y} \right)^2
$$

(2.17)

Additional requirements for the value of $G_c$ to be valid are specified in ASTM D 5045 (1996). If the test does not satisfy all the conditions for linear elastic fracture mechanics then it is necessary to use elastic-plastic fracture mechanics to evaluate toughness.

2.4.3 Elastic-Plastic Fracture Mechanics

An assumption of linear elastic fracture mechanics is that energy is dissipated only very locally at the crack tip. For many materials the plastic zone at the crack tip is too large for this assumption to be reasonable. The most widely used measure of toughness of elastic-plastic materials is the $J$ integral. The $J$ integral was first proposed by Rice (1968) as a path-independent measure of the fracture conditions in a non-linear elastic material. Subsequently, the $J$ integral has been used as a technique for considering fracture in a material experiencing both elastic and plastic deformation. For a crack surrounded by a contour $\Gamma$, the $J$ integral $J$ was defined as:
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\[ J = \int_{\Gamma} \left( W_e \, dy - T \cdot \frac{\partial u}{\partial x} \, ds \right) \]  

(2.18)

where \( x \) and \( y \) are rectangular contours normal to the crack front, \( ds \) is an element of arc length along the contour \( \Gamma \), \( T \) is the stress vector acting on the contour, \( u \) is the displacement vector and \( W_e \) is the strain energy density. From this definition it can be concluded that the J integral is path independent and may be determined from an arbitrary contour away from and surrounding the crack tip. The J integral is a measure of the energy in the body and so is analogous to \( G \). For linear elastic conditions \( J \) and \( G \) are equal. As with \( G \) and \( G_c \) the value of \( J \) at crack extension is termed the critical J integral \( J_c \).

Rice (1968) reported that the J integral may be interpreted physically as the potential energy difference between two identically loaded bodies containing single cracks of length \( a \) and \( a + \Delta a \); this can be stated as:

\[ J = -\frac{\Delta U}{\Delta a} \]  

(2.19)

where \( \Delta U \) is the change in potential energy with crack extension \( \Delta a \). Begley and Landes (1972) showed that the term \(-\Delta U\) could be obtained from the area between the load-displacement curve for a specimen with an initial crack length of \( a \) and the load-displacement curve for a specimen with an initial crack length of \( a + \Delta a \), as illustrated in Figure 2.1. The area corresponding to \(-\Delta U\) is bounded by the displacement at which crack extension occurs, although strictly this analysis is for the case of constant load, the difference for the constant displacement case is negligible (Begley and Landes, 1972). This technique requires a number of samples with different initial crack lengths and forms the basis of multiple specimen techniques for the evaluation of the J integral. Although multiple specimen techniques have been successfully applied to a range of materials, they require significant experimental effort. This is particularly true for materials exhibiting a significant degree of variability in their behaviour.
To reduce the experimental effort required to evaluate the J integral, a single specimen technique was proposed by Rice \textit{et al.} (1973). This technique partitions the displacement of the sample into elastic and plastic components i.e.

$$\delta = \delta_{el} + \delta_{pl}$$  \hspace{1cm} (2.20)

where $\delta$ is the total displacement, $\delta_{el}$ is the elastic component of the displacement and $\delta_{pl}$ is the plastic component of the displacement. From this, the critical J integral can be written as the sum of the elastic and plastic J integrals at the critical point, so:

$$J_c = J_{el} + J_{pl}$$  \hspace{1cm} (2.21)

where $J_{el}$ is the J integral due to plastic displacement at the critical point and $J_{pl}$ is the J integral due to plastic displacement at the critical point.

The following expressions (Equations 2.22 to 2.24) are valid for a double-edge notch specimen of width $2W$ containing two cracks each of length $a$, as used in the experimental work in this thesis. The elastic J integral at the critical point is equivalent to the strain energy release rate and may be evaluated from the equation:

$$J_{el} = G = \left( \frac{P^2}{4B} \right) \left( \frac{dC}{da} \right).$$  \hspace{1cm} (2.22)

The factor four appears here because it is assumed that both cracks grow at the same load. For a deeply notched specimen the plastic critical J integral may be evaluated using the expression:

$$J_{pl} = \frac{1}{2B(W-a)} \left( \frac{\delta_{pl}}{0} \right) \left( \frac{P \delta_{pl}}{0} - P \delta_{pl} \right)$$  \hspace{1cm} (2.23)
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This was interpreted by Atkins and Mai (1988) for a double-edge notch specimen to give the expression:

$$J_{pl} = \frac{A^*}{B(W - a)}$$ \hspace{1cm} (2.24)

where the quantity $A^*$ is the area bounded by the load-displacement curve up to the critical point and a secant line returning to the origin, as shown in Figure 2.2.

The single specimen technique has been used widely for $J$ integral evaluation, particularly for polymers and variable materials such as paper. The technique of Rice \textit{et al.} (1973) has been modified by Yuharu and Kortschot (1993) to overcome an assumption concerning the relation between load and plastic displacement. It is also possible to construct an energy versus crack length plot using a single specimen as with the multiple specimen method. However, this requires a reliable method of monitoring the crack length while the sample is loaded. This is not possible for many types of material. The $J$ integral approach has been used to measure the toughness of short-fibre composites by a number of authors e.g. Agarwal \textit{et al.} (1984), Kim and Kim (1989) and Choi and Takahashi (1996).

The critical $J$ integral $J_c$ is based on elastic-plastic fracture mechanics so the specimen size requirement is less rigorous than that for $G_c$. The current ASTM standard (ASTM E 1737, 1996) for $J_c$ testing requires the specimen dimensions to satisfy the criterion:

$$B, (W - a) \geq 25 \left( \frac{J_c}{\sigma_y} \right)$$ \hspace{1cm} (2.25)

for the thickness of the specimen $B$ and the length of the uncracked ligament $W-a$ where $J_c$ is the critical $J$ integral and $\sigma_y$ is the yield stress of the material. It can be seen that this equation has a similar form to Equation 2.17.
A further parameter proposed by Liebowitz and Eftis (1971), and extended by Eftis et al. (1975), is the non-linear critical strain energy release rate $\tilde{G}_c$ given by:

$$\tilde{G}_c = G_c (1 + \tilde{C})$$  \hspace{1cm} (2.26)

where $G_c$ is the critical strain energy release rate and $\tilde{C}$ is the non-linear correction factor. The factor $\tilde{C}$ is found by fitting the load-displacement curve for the specimen to the expression:

$$\delta = CP + k(CP)^n$$ \hspace{1cm} (2.27)

where $\delta$ is the displacement of the specimen, $C$ is the compliance of the specimen, $P$ is the load on the specimen and $k$ and $n$ are parameters that characterise the non-linearity of the load-displacement curve. Figure 2.3 illustrates the origin of the two terms in Equation 2.27. The factor $\tilde{C}$ is determined from $k$ and $n$.

Westerlund et al. (1991) compared fracture toughness measurements on a paper material using three different techniques: the multiple specimen J integral technique of Begley and Landes (1972), the single specimen J integral technique of Rice et al. (1973) and the non-linear strain energy release rate of Liebowitz and Eftis (1971). There was reasonable agreement between the Liebowitz technique and the multiple specimen technique while the single specimen technique reported higher values.

Jinen (1985) applied this technique to a short-fibre composite and Friedrich et al. (1991) have stated that the Liebowitz and Eftis (1971) technique is suitable for application to short-fibre composites. However, it has not been shown that $\tilde{G}_c$ is equivalent to the J integral. Although there is a similarity in definition, this is not sufficient to justify equating $\tilde{G}_c$ and $J_c$. In addition there is little work to validate the
Liebowitz technique as a measure of toughness for polymers and polymer matrix composite materials.

A major difficulty in applying either a $J_c$ or $\tilde{G}_c$ analysis is assessing the point at which the crack tip advances. This may be avoided by evaluating the J integral at a series of crack extensions and determining the critical value using a blunting line. An alternative approach, proposed by Zhou et al. (1991), can determine the load at crack extension for the single specimen method. This approach involves recording the load-displacement curve up to the point at which the test is stopped and measuring the crack extension at this point. The crack extension may be determined after the test by breaking the sample, at liquid nitrogen temperatures if necessary, and measuring the crack extension from the fracture surface. However Chan and Williams (1983) and Narisawa (1987) found that it was not possible to measure the crack extension in polypropylene when broken at liquid nitrogen temperatures.

### 2.5 Critical Length Determination

A key feature of a short-fibre composite is the critical length of the fibre where the critical length is the minimum fibre length for which the tensile stress can equal the fracture stress of the fibre. The critical length $l_c$ may be calculated using the equation described earlier:

$$l_c = \frac{\sigma_f^* r_f}{\tau}$$  \hspace{1cm} (2.6)

where $\sigma_f^*$ is the failure stress of the fibre, $r_f$ is the fibre radius and $\tau$ is the interfacial strength. The critical length of the fibre can affect the stiffness, strength and toughness properties of a composite material and hence a knowledge of the critical length is a necessary input parameter for modelling some mechanical properties. Techniques available to determine critical length have been critically reviewed by Piggott (1987) and more recently by Herrera-Franco and Drzal (1991). Four types of test to evaluate $l_c$ have been developed and are described here: fragmentation test, single fibre pull-out test, micro-debonding test and micro-indentation test. The
fragmentation test and single fibre pull-out test are the most widely used of these techniques.

The fragmentation test requires a sample of matrix containing a single aligned fibre. The failure strain of the matrix must be greater than the failure strain of the fibre and preferably at least three times the magnitude. The specimen is strained and, as a result, the fibre will fracture due to stress transferred from the matrix. The fibre will continue to fracture until the fragments are too short for stress to build up sufficiently to cause further breaks in the fibre. It is usually taken that the average fragment length will be three quarters of the critical length.

The single fibre pull-out test pioneered by Favre and Perrin (1972) consists of a single fibre partly embedded in a matrix. The free fibre end is then pulled out of the matrix at a constant displacement rate. The debonding load can be determined from a load-time curve and used to calculate the interfacial bond strength. From this the critical length may be calculated using Equation 2.6. A complication with the use of Equation 2.6 is that the strength of the fibre is not single valued but dependent on the length of fibre tested. To determine the critical length using this Equation it is necessary to obtain accurate values for the strength at the critical length.

Both the micro-debonding test and the micro-indentation test may be used to evaluate the interfacial strength and also require a knowledge of the length dependence of the fibre strength to evaluate the critical length. The micro-debonding technique is similar to the single fibre pull-out test; a drop of matrix is placed on a fibre and the force required to pull the droplet off the fibre is measured. This value can be used to calculate the interfacial bond strength and from this the critical length can be calculated. The micro-indentation test is performed on a polished surface of composite material with fibres perpendicular to the surface. The force required to push an individual fibre through the composite material is measured, and this value may be used to calculate the interfacial bond strength and critical length.
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Of the four tests described only the single fibre fragmentation test provides a direct measure of the critical length of fibre. This test also provides a large amount of data from a single specimen. Folkes and Wong (1986) found that the single fibre fragmentation test was suitable for routine assessment of interfacial adhesion in thermoplastic matrix materials. The single fibre fragmentation test may also be used to study other composite material properties such as compressive failure of the fibre. Incardona et al. (1993) reported a “spontaneous fragmentation” of fibre within a thermoplastic matrix caused by the greater thermal contraction of the matrix than the fibre on cooling. This effect was used by Wood et al. (1996) to study the compressive strengths and associated Weibull parameters of carbon fibres loaded in compression.

2.6 Mesostructure

Up to this point in this Chapter it has been shown that the mechanical properties of short-fibre composites are dependent on the individual properties of the fibre, matrix and interface, and also on a variety of structural features such as fibre length and fibre orientation. However, there is a class of structural features that do not usually appear in models for composite materials. Existing models are dominated by (i) micromechanics, i.e. interactions on the scale of the individual fibre, and (ii) macromechanics, i.e. interactions on the scale of the laminate. There is a range of features that are between these two scales, such as fibre-bundling and fibre waviness, which have been described as mesostructures by Piggott (1992). Mesostructure has been defined as structural features in the range 30 μm to 30 mm (Piggott, 1995).

Current knowledge of mesostructure-property relationships has recently been reviewed by Piggott (1996). So far, simple relations have been established for a limited number of mesostructures. A difficulty with this work is the careful processing required to produce material with a controlled mesostructure. Mesostructure of commercial materials is normally introduced inadvertently, sometimes with a deleterious effect on properties. The development of mesostructure during the processing of polymer matrix composite materials has been outlined by Friedrich (1998). The mesostructures that
can develop in a number of commercially manufactured materials were described and the processing conditions that affect the mesostructure were discussed.

There is potential for more efficient commercial materials through the control of mesostructure. For this to be possible it is necessary to relate mesostructure to the final properties of the composite material and to modify the manufacturing process to control the mesostructure of the composite material produced.

### 2.7 Fibre-Bundling

Cottrell (1964) first suggested that the toughness of a fibre composite could be improved by using fibres with a greater diameter due to a longer critical length. As can be seen from Equation 2.6, critical length is proportional to fibre radius:

\[
l_c = \frac{\sigma_f r_f}{\tau}
\]

An increase in critical length will favour fibre pull-out over fibre fracture because a greater length of fibre is required for the stress in the fibre to reach the strength of the fibre. Fibre pull-out absorbs more energy than fibre fracture hence an increase in fibre diameter should improve toughness. This suggestion has been supported theoretically by Piggott (1970) who found that work of fracture was proportional to fibre diameter. A disadvantage of increased fibre diameter is a reduced aspect ratio of the fibre and therefore a lower reinforcing efficiency. It is also probable that larger diameter fibres exhibit reduced fibre strength. An alternative solution is to use fibre-bundles since they provide a larger diameter reinforcing unit, while the constituent fibres of the bundles are of the same diameter and quality as an individual fibre.

It is reasonable to expect that the behaviour of fibres contained in bundles will differ from that of an equivalent number of fibres dispersed more uniformly. The presence of fibre bundles rather than individual fibres will have a number of effects on the structure of a short-fibre composite. There will be a reduced distance between fibres and the possibility of fibres touching within the bundles. The fibres will be locally...
aligned and there will be fibre end synchronisation. For the laminate as a whole, there will be increased variation in the local volume fraction. All these features will have some effect on the mechanical properties of the laminate. However, it is important to establish which effects dominate the mechanical properties.

2.8 Experimental Work on the Effect of Fibre-Bundling

2.8.1 Impregnated Bundle Materials

The most common technique used for the investigation of fibre-bundling is the impregnation of a fibre tow with resin which is then cured. The resulting rod can be incorporated into a laminate in a variety of ways. The earliest work in this area was by Fila et al. (1972) who constructed continuous, aligned laminates of glass and carbon fibre in a polyester resin. The work of fracture \( \gamma_f \) of the materials was measured in notched bending tests. Fila et al. (1972) found good agreement with earlier work by Piggott (1970) who had derived the following equation for \( \gamma_f \):

\[
\gamma_f = \frac{V_f d \sigma_c^*}{12 \tau E_c} \tag{2.28}
\]

where \( V_f \) is the volume fraction of fibre, \( d \) is the diameter of the reinforcing unit, \( \sigma_c^* \) is the failure stress of the composite material, \( \tau \) is the interfacial bond strength and \( E_c \) is the modulus of the composite material. The experimental work of Fila et al. (1972) confirmed the expectation of Piggott (1970) that fibre bundles could be used to produce tough materials because of the relatively large effective diameter of the reinforcing unit.

Hamer and Woodhams (1981) used a similar technique with injection moulded short-fibre composites. Polyethyleneterephthalate (PET) fibres were coated with polyvinylacetate before being combined with polypropylene, the addition of coated bundles resulted in a large increase in impact toughness compared to unreinforced polypropylene. Material manufactured with untreated, and so dispersed, PET fibres
exhibited a smaller increase in impact toughness compared to unreinforced polypropylene.

Kim and Mai (1993) used the impregnated bundle technique to manufacture composite materials with unidirectional continuous fibres and planar random discontinuous fibres. Carbon fibre impregnated with epoxy resin or polycarbonate, within a matrix of epoxy resin, was used. The bundled materials were compared with a composite material of uniform fibre distribution. The impact fracture toughness of the impregnated bundle materials was twice that of the composite materials with a uniform fibre distribution, with no accompanying reduction in strength.

Meraglini and Benzeggagh (1995) manufactured planar random short-fibre composites from bundles of glass fibre coated with epoxy resin. Three types of material were manufactured with 800, 1600 and 2000 fibres per bundle respectively. All the materials were assumed to have the same overall volume fraction of fibre. Materials with more fibres per bundle were found to have a lower stiffness and lower strength.

The impregnated bundle technique has yielded valuable results particularly in showing that an improvement of toughness may be achieved using larger diameter reinforcement. However, there are flaws if the aim is to examine the mechanisms associated with the fibre bundling; the most significant is the extra interface that is created between the polymer used to impregnate the bundle and the polymer matrix. The presence of this interface will certainly modify the mechanical response of the composite material and it is possible that the composite material will fail at this interface. These effects will be more pronounced in cases where the two polymers are different.

2.8.2 Glass Mat Reinforced Thermoplastics
Glass mat reinforced thermoplastic (GMT) materials are commercially produced composite materials consisting of a glass-fibre mat and a thermoplastic matrix, most
commonly polypropylene. GMT materials are supplied as unconsolidated blanks, often cut to the mould size. These blanks are compression moulded in a process known as hot stamping. Berglund and co-workers (Ericson and Berglund, 1992; Ericson and Berglund, 1993; Lindhagen and Berglund, 1997; Lindhagen and Berglund, 1998) have compared the mechanical properties and fracture of GMT materials with various fibre architectures.

Ericson and Berglund (1992) compared two polypropylene GMT materials manufactured by different methods. The Symalit method produces a mat consisting of long fibres, primarily in bundles, looped in the plane of the material. The diameter of the loops is larger than the size of the laminates so fibre loops are not visible within a laminate. The Ahlstrom process produces a mat consisting of 12 mm long dispersed fibres oriented randomly in the plane of the disc. The modulus of the dispersed material is higher, for a comparable volume fraction, than that of the bundled material. Ericson and Berglund (1992) accounted for this difference by assessing the reduced reinforcing efficiency of the bundle. The bundle was treated as a large fibre with the same diameter as the bundle. The modulus of the bundle was determined using the rule-of-mixtures. Predictions from this approach showed good agreement with experimental data. Dispersed fibre material exhibited a slightly higher strength and a higher work of fracture. Increased work of fracture for dispersed materials could be a result of more extensive fibre pull-out and plastic deformation at fibre ends. Fracture surface observation showed that the bundles behaved as a single unit.

Later work by Ericson and Berglund (1993) compared a novel preformed composite material with the Ahlstrom and Symalit materials. The novel preformed composite material was manufactured with 13 mm or 51 mm long fibres which were mainly dispersed but with some bundles present. Fibre length and the use of adhesion promoters had little effect on the mechanical properties of the materials. It was proposed that this was because the properties, in particular strength, are controlled by the inhomogeneity of the materials. Critical inhomogeneities proposed were regions of low fibre content, regions of high fibre content with poor impregnation and
unfavourable fibre orientation. It was concluded that better dispersion of fibres in commercial GMT materials would improve mechanical properties.

Notch sensitivity and damage mechanisms of Symalit and Ahlstrom GMT materials were investigated by Lindhagen and Berglund (1997). The notch sensitivity of the materials was studied by tensile testing coupons with a circular hole of various diameters. It was found that the notch sensitivity of the material containing looped bundles was lower than the notch sensitivity of the dispersed material. This difference in notch sensitivity was attributed to a smaller damage zone in the dispersed material. Mechanisms operating in these two materials were discussed in greater detail in a more recent paper (Lindhagen and Berglund, 1998). For the bundled material it was found that damage initiation occurred at fibre bundles or single fibres oriented transverse to the loading direction while bundles parallel to the loading direction stopped or deviated the crack. For the dispersed fibre material there were no inherent initiation points so initiation occurs at the notch tip. A consequence of this is that a larger material volume is involved in energy dissipation in the bundled material.

The work on GMT materials discussed above is the most detailed study to date of the effect of fibre architecture on the mechanical properties of a composite material. However, there are a number of differences between the GMT materials studied, including variation in type of glass-fibre, surface coating of fibre and type of polypropylene; consequently it is difficult to de-convolute the influences.

2.8.3 Materials with Incidental Fibre-Bundling

This Section considers relevant work in which the fibre-bundling has not been varied in a controlled manner. The difference in fibre-bundling may result from variations in the processing conditions or the surface coating of the fibre. Effects of fibre-bundling are difficult to isolate from other variables in these materials.

Chopped strand mat (CSM), sheet moulding compounds (SMCs) and dough moulding compound (DMC) may all be manufactured using fibre bundles. Surface
coating (size) on the fibre bundle may be selected to control the degree of dispersion (Hull, 1981). If the fibre bundles retain their integrity then the bundle may become the unit of reinforcement rather than the individual fibre.

Mei and Piggott (1996) produced SMC material exposed to different degrees of shear during processing. For materials exposed to greater shear the toughness was reduced. This was probably a result of the breakdown of the fibre bundles and the breakage of individual fibres.

Dispersion of glass fibre in a sheet moulding compound may be strongly affected by the nature of the size on the fibre. An insoluble size will tend to result in fibres being retained in bundles while a soluble size will tend to result in better dispersion of glass fibres. Mechanical properties of these two types of material were compared by Burns (1982). This work reported that material with a soluble size had an almost linear stress-strain response up to the point of fracture. Material with an insoluble size exhibited more ductile behaviour and a lower strength, but a similar initial modulus and a higher impact toughness. Further experiments with materials containing a variety of bundle sizes found that tensile strength decreased and impact toughness increased with increasing bundle size.

In similar work Worrall and Wells (1996) manufactured glass fibre reinforced polyester materials. One type of material contained glass fibre bundles coated with an insoluble size while another type of material had the size removed before processing, resulting in a filamentised material. The material containing bundles exhibited a higher impact toughness then the filamentised material.

2.8.4 Effect of Fibre-Bundling on a Polypropylene Matrix
When the mechanical properties of a short-fibre composite are modelled there is usually an assumption that the matrix is unaffected by the presence of fibres. Such an assumption may be reasonable for thermosetting matrices but there is evidence that this is unreasonable for thermoplastic matrices, such as polypropylene (Seferis, 1986; Ramos and Belmontes, 1991).
A crystallisable thermoplastic, such as polypropylene, exhibits a complex morphology arising from the crystallisation and development of molecular orientation in the matrix; these processes may be modified by the presence of fibres. Polypropylene can crystallise in three forms (Folkes, 1995). The most common is the α-monoclinic form which will develop during non-isothermal crystallisation when there is little or no flow prior to crystallisation. The hexagonal β form is found when the crystallisation occurs under stress, while the γ form is triclinic and unlikely to form during processing.

There are essentially two ways that crystallisation can initiate in a fibre composite material; within the bulk of the polymer or at the fibre surface. Initiation within the bulk of the polymer will occur at nucleation sites, generally believed to be foreign matter in the polymer, although tangling or ordering of the polymer chains could result in a nucleation site. This type of nucleation will result in a spherical crystal, i.e. a spherulite, the size of these spherulites will depend on the density of nucleation sites, fewer sites resulting in larger spherulites. Initiation may also occur at the surface of certain types of fibres. If the density of nucleation sites at the fibre surface is high, then the normal spherical growth will not be possible and instead epitaxial crystal growth from a surface will occur. This will result in a layer of crystallinity around the fibre called transcrystallinity. Transcrystallinity has been observed to occur in polypropylene matrix composite material in the presence of certain types of fibres (Campbell and Qayyum, 1980). Other factors influencing the formation of a transcrystalline phase have been investigated such as the crystallisation temperature, interfacial stress, cooling rate and polymer molecular weight (Thomason and Van Rooyen, 1992; Ye et al., 1995). At present there is no consensus on how transcrystallinity influences the mechanical properties of composite materials.

Ye et al. (1995) manufactured laminates with continuous glass fibre bundles in a polypropylene matrix. These laminates were exposed to various cooling and annealing regimes to modify the crystalline morphology of the matrix. An increased degree of crystallinity was associated with lower $G_c$, but it was concluded that both these effects
arose from the resin rich regions in the composite and variations in fibre-matrix adhesion. It was also found that a larger spherulite size resulted in lower $G_0$.

2.9 Theoretical work on the Effect of Fibre-Bundling

2.9.1 Modelling Materials containing Fibre Bundles

At present the mesostructure of a composite material is not usually considered when modelling mechanical properties. Mesostructure may affect the structure of a composite material in a number of ways. As indicated earlier (in Section 2.7), the presence of fibre bundles will result in reduced distance between fibres, local alignment, end synchronisation, an increase in the variation of local volume fraction and the presence of resin-rich areas. Some of the models described here do not consider fibre-bundling per se but aspects of the presence of fibre bundles.

Models for the mechanical properties of a short-fibre composite were described in Sections 2.2 to 2.4. None of the models discussed consider any effects arising from fibre-bundling. If the fibres are held together in a bundle then the bundle becomes the basic unit of reinforcement rather than the individual fibre. Relevant theoretical approaches for modelling stiffness, strength and toughness properties of a composite material containing fibre bundles must therefore be considered.

2.9.2 Models for Stiffness Properties

Kataoka et al. (1995) proposed a model for the stiffness properties of a short-fibre composite containing individual fibres and fibre clusters. The model is based on the equivalent inclusion method of Eshelby (1957). The equivalent inclusion method evaluates the elastic field around an ellipsoidal inclusion in an infinite elastic medium and thereby enables problems such as the stiffness of a two phase composite material to be solved. Various modifications allow Eshelby’s method to be applied to a material containing two types of inclusions. A cluster, as described here, is different to a bundle in that the fibre ends are not synchronised, as they are in a bundle, and the shape of the cluster is assumed to be ellipsoidal rather than cylindrical. The model of Kataoka et al. (1995) allows the stiffness properties of an aligned short-fibre composite containing
individual fibres and fibre clusters to be predicted. Calculations were made for an aligned short-fibre composite consisting of glass fibre and polyester.

Kataoka et al. (1995) modelled the change in effective Young’s modulus with variation of volume fraction of fibre within clusters, volume fraction of clusters, aspect ratio of the fibres within clusters and aspect ratio of the clusters. The only variable that had a significant effect on effective Young’s modulus was the volume fraction of clusters. Kataoka et al. (1995) also investigated the magnitude of the interfacial stress at the fibre end. This quantity is the force acting to open a crack at the fibre end. The fibre volume fraction in clusters, volume fraction of clusters and cut-off angle of the cluster all had a significant effect on the interfacial stress at the fibre end within a cluster. It was also found that the interfacial stress was greater at the end of fibres outside the cluster than at the end of fibres within the cluster. However, Kataoka et al. (1995) suggested that the interface is weaker for fibres within the cluster than for fibres outside the cluster because of poorer impregnation of matrix within the cluster. So, it was suggested that there is unlikely to be a benefit to composite strength from using clusters.

Carman and Reifsnider (1992) described a micromechanical model for the stiffness of a short-fibre composite. Predictions from this model were compared with experimental results from Kacir et al. (1978) for a composite material of short glass fibre bundles within an epoxy matrix. Carman and Reifsnider (1992) found excellent agreement when the aspect ratio of the bundle was used in the model.

Eduljee et al. (1994) described a model for sheet moulding compound comparing an aggregated and a dispersed microstructure. The aggregated microstructure consisted of domains of similarly aligned fibres which is analogous to a mat of bundled fibres. The dispersed microstructure is analogous to a filamentised composite material. Eduljee et al. (1994) showed that the dispersed microstructure is more efficient for reinforcement. It was also stated that the majority of models for short-fibre composites use an aggregate model since they evaluate properties for an aligned composite material which are then subjected to orientational averaging.
2.9.3 Models for Strength and Toughness Properties

Kacir et al. (1977) suggested that the bundle diameter and not the fibre diameter should be considered in strength calculations, since the short fibres behave as bundles and not as individual fibres. Wells and Beaumont (1985a) described the processes of pull-out and debonding for both fibres and fibre bundles. Models for the debond length and fibre pull-out length distribution were presented. These lengths are functions of the fibre, matrix and interface properties. This approach was developed to consider the energy absorption associated with these processes in terms of debond and pull-out lengths (Wells and Beaumont, 1985b).

A model considering the effect of matrix failure on the overall properties of a short-fibre composite was proposed in the paper by Meraghni and Benzeggagh (1995) discussed earlier. Three types of material were considered containing bundles with 800, 1600 and 2000 fibres per bundle. This model provided good agreement with experimental results for the material with 800 fibres per bundle but overestimated the stiffness of the materials with 1600 and 2000 fibres per bundle. Meraghni and Benzeggagh (1995) suggested this was because the model accounts for matrix failure but not interfacial decohesions which are increasingly important for materials with more fibres per bundle.

Trapeznikov et al. (1992) developed a micromechanical model for the strength of carbon/carbon composite materials. The model is based on the mechanical behaviour of the tows under loading and takes into account sliding of core fibres in tows with respect to peripheral fibres. This has been termed the “stocking effect”. The model proposed by Trapeznikov et al. (1992) shows a dramatic decrease in strength with an increase in the number of fibres per bundle. This effect arises from the reduced stress required to pull out fibres from within a bundle. Karbharti and Wilkins (1991) modelled a mechanism similar to the stocking effect, described as a telescopic type of pull-out. The statistical variation of fibre strength means that individual fibres may fracture and be pulled out individually. This effect could negate the expected increased energy absorption due to the use of reinforcing bundles. Although both mechanisms are
similar, it is unlikely that they are active within all composite materials containing fibre bundles.

Another feature of fibre bundling is an increase in the inhomogeneity of the composite material and this can be described by the variation of local fibre volume fraction. The overall volume fraction represents a mean value and this value is adequate for materials which are relatively homogenous. However, local irregularities are particularly important for fracture properties and Pan (1994) formulated a statistical model which predicts areas of weakness in a composite material using minimum and maximum local volume fractions.

2.10 Analysis of Fibre Bundling

2.10.1 Techniques for the Analysis of Fibre Arrangement
Techniques for the determination of fibre length and orientation are widely used and relatively simple to apply. These techniques have been reviewed by Guild and Summerscales (1993). However, methods for analysis of fibre arrangement are less widely used and are more complex to apply.

The distribution of fibres within a short-fibre composite is routinely described by terms such as planar random and three dimensional random. These terms provide a useful qualitative description of the distribution but are often not strictly accurate. There is a range of statistical tests available for determining whether a distribution is random. These have been compared by Myles et al. (1995) for a number of patterns which exhibit deviations from randomness. The patterns analysed could deviate from randomness in two ways; "clustered" in which particles tend to group together or "inhibited" in which particles must be at least a certain distance apart, which results in regularity. The statistical tests considered by Myles et al. (1995) could test for the randomness of patterns and provide an alternative model if randomness was rejected.

Fibre arrangement in composite materials has been studied by applying a variety of image analysis methods to cross-sections of unidirectional composite materials. Wimolkatitisak et al. (1990) studied fibre arrangement in a carbon fibre-epoxy
composite material and compared the measurements with those of a simulated random composite material. Degree of randomness and degree of contiguity of these images was assessed. Degree of randomness compares the standard deviation of either the fibre spacing or the volume fraction of the real and simulated materials, while degree of contiguity is the proportion of nearest neighbour fibres that are touching a particular fibre. Similar work by Abbé et al. (1990) characterised the morphology of a ceramic matrix composite material in terms of the fibre mean contact number and radial distribution function. Yurgartis and Purandare (1991) proposed the included angle distribution as a measure of fibre arrangement. This measure is independent of volume fraction of fibre and can differentiate between square and hexagonal arrangements of fibre.

These techniques have successfully been applied to composite materials containing continuous, aligned fibres. However, this presents a relatively simple case for analysis and the extension of these analyses to include discontinuous and misoriented fibres is likely to present many additional challenges. In addition, while the information produced is statistically valid, it is not necessarily the information required to model properties of these materials.

2.10.2 Techniques for the Analysis of Fibre-Bundling
None of the techniques described in Section 2.10.1 can be used to describe the bundling or clustering of fibres. Analysis of fibre-bundling presents several further difficulties compared to analysis of fibre arrangement. The problem of defining the clustering of fibres is similar to that of defining spatial distributions in ecology and geography. A method of quantifying these spatial distributions, known as variance analysis, was first described by Greig-Smith (1952). A parameter describing the distribution, such as area fraction, is measured for cells of increasing size. The variance of the parameter with increasing cell size is calculated. If the cell size corresponds to the size of a feature, such as a fibre cluster, then the variance will exhibit a maximum. Therefore peaks in variance will correspond to scales of pattern at that cell size. This technique has been used to describe the distribution of fibres in
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a cross-section of a unidirectional composite of glass fibre and polyester by Guild and Silverman (1978).

Worrall and Wells (1996) developed a fractal-variance characterisation method from variance analysis using elements of fractal geometry. Fractal theory has successfully been applied to physical problems such as the thermal conductivity of a composite material (Pitchumani and Yao, 1991). Fractals can be considered as being defined by non-Euclidian geometry and many objects may be described by a fractal dimension (Mandelbrot, 1982). The key to this approach is that the value of some physical properties depends on the way in which they are measured, known as the Richardson effect. Applying this to a cross section of a composite it is possible to construct a Richardson plot using the log of variance of local volume fraction against the log of cell size used to measure local volume fraction. The slope of this plot is the fractal dimension. A step in the plot will correspond to the size of a change of structure in the sample analysed, such as a fibre bundle. This technique is capable of evaluating the size of bundles from a cross-section. However, at present there is no method of evaluating the volume fraction of bundles of a given size; this information is crucial for relating fibre-bundling to the properties of a composite material.

Yurgartis (1995) stated that the utility of any microstructural measure is only demonstrated when it has successfully been connected to a material property. Until there is experimental evidence to link an analysis of fibre-bundling with the variation of mechanical properties the proposed measures are only suggestions for evaluation. The current situation is that proposed measures are more numerous than experimental studies to evaluate these measures.

2.11 Conclusions

In Section 2.10 the current availability of techniques to quantify fibre-bundling was reviewed. It is clear from this review that there is currently no satisfactory method for quantifying fibre-bundling. To study the effect of fibre-bundling, it is therefore necessary to control fibre-bundling during manufacture of the composite material. To describe
fibre-bundling it is necessary to know the volume fraction of fibres within bundles of a given size within the composite material.

Section 2.8 considered experimental work relevant to the effects of fibre-bundling on the mechanical properties of a short-fibre composite. A variety of techniques have been used to control the degree of fibre-bundling. However, none of these techniques is entirely satisfactory to study the effect of fibre-bundling on the mechanical properties. They either result in a fibre architecture in which the degree of fibre-bundling is unknown, or achieve a known degree of fibre-bundling using a method that invalidates a direct comparison of filamentised and bundled composite materials.

There is clearly a need for a manufacturing method capable of producing a range of composite materials in which the only variable is the degree of fibre-bundling. An experimental study to evaluate the mechanical properties of such a range of composite materials would greatly assist both the development of theoretical models and techniques for the quantification of fibre-bundling. The manufacture of such composite materials, their evaluation and modelling, is the subject of this thesis.
Figure 2.1. Load-displacement curves illustrating the quantity $-\Delta U$ required for the multiple specimen determination of the $J$ integral (after Begley and Landes, 1972).

Figure 2.2. Load-displacement curve illustrating the quantity $A^*$ required for the single specimen determination of the $J$ integral (after Atkins and Mai, 1988).
Figure 2.3. Load-displacement curve illustrating the terms fitted to the load-displacement curve for the analysis of Liebowitz and Eftis (1971).
Chapter 3. Experimental Methods
3.1 Introduction

The present Chapter describes the experimental methods used in this work. Section 3.2 describes the fabrication of fibre mats, with a controlled proportion of the fibres within bundles, and their impregnation with polypropylene. Mechanical testing of the composite materials to determine modulus, strength and toughness is described in Section 3.3. Section 3.4 describes the characterisation of the composite materials in terms of the fibre volume fraction and critical length. Techniques used to obtain images of the fracture surfaces are also described in Section 3.4.

3.2 Material Fabrication

3.2.1 Fabrication of Fibre Mats with Controlled Fibre-Bundling

The major synthetic requirement of this work was the manufacture of laminates in which bundling of the fibres was controlled. Production of materials containing filamentised fibres was relatively straightforward. Retaining fibres within discrete bundles proved difficult to achieve.

A number of methods of retaining fibres within bundles were investigated. These methods generally involved coating the fibre tow to hold it in a bundle and removing this coating after the fibre bundle had been incorporated in a fibre mat. The best material for coating the fibres was found to be paraffin wax with a congealing point of 57 °C to 60 °C. Wax is chemically inert and burns off without residue. It was found that the solid wax was sufficiently strong that the wax-impregnated bundle does not break up during the fabrication of the fibre mat. The melting point of the wax is at a temperature that allows the wax to be applied to the fibre whilst molten. The method used to produce mats with a known proportion of the fibres within bundles is summarised in Figure 3.1 and described in detail below.

The carbon fibre used in this work was Toray T300. Carbon fibre tows with 1000 fibres per tow and 6000 fibres per tow were used. The arrangement used to coat the fibre with wax is illustrated in Figure 3.2. The carbon fibre tow was drawn directly off a bobbin and through a stainless steel bath containing the molten wax. The stainless steel bath rested
on a hot plate which kept the wax molten. The bath contained a set of three rollers immersed in wax which ensured that the fibre tow was coated completely. After exiting the wax, the tow was drawn over a further roller which removed excess wax from the tow. The wax solidified before the fibre tow was drawn into a chopping machine which cut the tow into either 5 mm or 10 mm lengths. This process resulted in bundles of carbon fibres, of a known length and a known number of fibres, impregnated with solid wax.

The required mass of wax-coated bundles was measured out using a balance. To achieve the correct mass of fibre it was necessary to determine the mass of fibre within the coated tow. The mass of ten carbon fibre tows 30 cm in length was measured on a balance to an accuracy of 0.1 mg. Following this the mass of 30 cm lengths of tow coated with wax, as described above, was determined in the same way. The results of these measurements are shown in Table 3.1. These results may be used to determine the ratio of the mass of wax-coated bundles to the mass of carbon fibre within the wax-coated bundles. This ratio was used to determine the mass of wax-coated bundles needed to give the required mass of carbon fibre.

The wax-coated bundles could be incorporated in mats without treatment, resulting in bundles in the mat, or the wax could be burnt off, resulting in filamentised fibres in the mat. By using different proportions of coated and uncoated fibre it was possible to control the proportion of fibres within bundles. The composition of each type of mat is shown in Table 3.2. To remove the wax from fibre, the coated fibres were heated in a furnace for 30 minutes at 300 °C and for 60 minutes at 500 °C.

It was necessary to take into account that the area of the mesh used to produce the mat was greater than the area of the required mat. The masses of fibre quoted in Table 3.2 are for an area of 150 mm by 150 mm which is the area of the frame used to impregnate the mats with polypropylene. The size of the mesh used was 200 mm by 250 mm, so it was necessary to multiply the mass of fibre used by 2.2. This calculation assumes that the fibre was distributed evenly over the mesh.
Chapter 3. Experimental Methods

The mass of both uncoated and coated fibre required for a given mat was added to glycerol solution. The glycerol solution was produced by mixing two parts glycerol with one part water. Before the wax-coated bundles were added to the glycerol solution they were heated in a furnace at 300 °C for 30 minutes. This partially decomposed the wax while still leaving a mechanically intact coating on the bundles. More importantly this increased the density of the bundles so they dispersed uniformly throughout the glycerol solution.

The fibre was dispersed in the glycerol solution using a paddle driven by an electric motor and the fibre and solution were mixed in this way for ten minutes. This mixture was then poured over a stainless steel mesh which retained the fibre whilst allowing the glycerol solution to drain away. A second stainless steel mesh was placed over the mat and the mat was sprayed with warm water for ten minutes to remove any glycerol solution retained on the fibres. The mat was then dried for two hours at 100 °C in an air-circulating oven. Any remaining wax was burnt off by heating the mat for one hour at 500 °C in a furnace. Mats with the following compositions were produced; 0 % of fibres within bundles, 25 % of fibres within bundles, 50 % of fibres within bundles, 75 % of fibres within bundles and 100 % of fibres within bundles

3.2.2 Impregnation of Fibre Mats

Different types of mat contained different masses of fibre so the number of mats required for a laminate varied. The number of mats required to produce laminates with a thickness of 1 mm and 2 mm is shown in Tables 3.3 and 3.4. Although the degree of fibre-bundling varies for each laminate, each 1 mm thick laminate nominally contained 6 g of fibre, while each 2 mm thick laminate nominally contained 12 g of fibre.

Mats were impregnated with the polypropylene matrix using a film stacking technique at the laboratories of Kobe Steel Europe Ltd. The frame that was used to contain the fibre mat and polypropylene during impregnation had internal dimensions of 150 mm by 150 mm by either 1 mm or 2 mm. The fibre mats and polypropylene film, which had a thickness of 50 μm, were cut to fit inside the frame. Layers of carbon fibre were interleaved with the polypropylene film. The number of sheets of polypropylene was
selected to give a nominal volume fraction of fibre within the laminate of 0.15. For a 1 mm thick laminate, 17 sheets of polypropylene were required, while for a 2 mm thick laminate 34 sheets of polypropylene were required. The stack was weighed before pressing and extra sheets were added if the mass of the stack was below the expected mass. In addition, two extra sheets of polypropylene were added to ensure good impregnation.

The stack of fibre mats and polypropylene was placed in a George Moore press and heated to 250 °C with the stack and plates in contact but with no pressure. Contact was maintained for three minutes after the press had reached 250 °C and then maximum pressure (approximately 8 kPa) was applied for one minute. Following this the pressure was briefly released and reapplied three times to "pump" the stack which allowed entrapped air to escape and aided the infiltration of the fibre bundles by the polymer melt. The press was then water-cooled to room temperature while maximum pressure was maintained. Any flash around the laminate was removed using a scalpel. This method of material fabrication produced a laminate 150 mm by 150 mm and either 1 mm or 2 mm thick.

The resulting laminates differed greatly in appearance. A photograph of a laminate with 0 % of the fibres within bundles is shown in Figure 3.3. This material appears to be homogenous with no features apparent at the surface. A laminate with 50 % of the fibres within bundles is shown in Figure 3.4; bundles can be seen at the surface of the laminate against a continuous layer of filamentised fibres. Figure 3.5 shows a laminate with 100 % of the fibres within bundles; individual bundles can be seen throughout the material. The most noticeable feature in Figure 3.5 is the areas of resin containing no reinforcement. In this material, a 1 mm thick laminate of the material with all of the 5 mm long fibres within bundles of 6000 fibres, the areas of unreinforced resin are numerous.

The composite materials manufactured in this work are listed in Tables 3.5 to 3.8. Five materials, 1 mm in thickness, were tested for modulus and strength. These materials are listed in Table 3.5. Table 3.6 lists the five materials, 2 mm in thickness,
tested for modulus and critical strain energy release rate. The widest range of materials was tested for critical J integral and modulus. These ten materials were 2 mm in thickness and are listed in Table 3.7. In addition, four materials were tested for modulus only and then used to develop the testing methods used in this work. These materials are listed in Table 3.8. It can be seen from Tables 3.5 to 3.8 that the overall volume fraction of the laminates showed significant variation. This variation was found to be unavoidable with the manufacturing technique used in this work.

3.2.3 Confirmation of the Integrity of Laminates
Sections from laminates were studied to verify that infiltration of the fibre mats with polypropylene was satisfactory. Samples from laminates were mounted in epoxy resin and sectioned using a Jung microtome with a glass knife. Sections 20 μm thick were viewed using transmitted light on an Axiophot microscope and photographed. Figure 3.6 shows a cross-section of a material with 0% of the fibres within bundles. This cross-section shows that all the fibres are filamentised. Figure 3.7 shows a cross-section of a material with 50% of the fibres within bundles. A single bundle can be clearly seen in this figure while the other fibres are all filamentised. The bundle appears to be elliptical in cross-section which may be because the bundle is not perpendicular to the cross-section or because the pressure applied during processing has deformed the bundle. A cross-section of a material with 100% of the fibres within bundles is shown in Figure 3.8. A single bundle can be seen in Figure 3.8 with a few fibres in the surrounding matrix. These individual fibres may have broken away from the surface of the bundle during processing.

The most important observation from the cross-sections shown in Figures 3.6 to 3.8 is that there is no evidence of porosity in the materials, confirming that the polypropylene satisfactorily impregnated the fibre mats.

3.2.4 Fabrication of Polypropylene Plaques
To manufacture unreinforced polypropylene plaques, the method used to impregnate the fibre mats was applied to a stack of polypropylene sheets. To produce a 2 mm thick plaque of polypropylene 40 sheets of polypropylene were required, although in
practice, 42 sheets were used to ensure good consolidation. Similarly for a 1 mm thick plaque 22 sheets of polypropylene were placed in the press.

3.3 Mechanical Testing

3.3.1 Tensile testing
A water-cooled diamond saw was used to cut laminates into coupons for mechanical testing. It was necessary to back the sample with a laminated sheet to ensure that the coupons had a clean edge. Coupon dimensions were measured using a micrometer.

Mechanical testing was carried out using an Instron 1175 tensile testing machine at a crosshead speed of 0.5 mm/minute. Extension was monitored using an extensometer with a 50 mm gauge-length, attached to the specimen with rubber bands. Coupons were tabbed with 30 mm long friction tabs cut from emery cloth.

3.3.2 Modulus and Strength Evaluation
The Young's modulus and strength of the composite materials were measured using coupons 150 mm long, 20 mm wide and either 1 mm or 2 mm thick. Six specimens were tested for each laminate type. Young's modulus was determined from the slope of a linear regression fit to the stress-strain curve between 0 % and 0.1 % strain. Tensile strength was taken as the maximum stress sustained by the coupon.

3.3.3 Critical Strain Energy Release Rate Evaluation
Critical strain energy release rate \( G_c \) was determined using a method based on a single edge notch (SEN) geometry that had been applied to similar short-fibre composite materials by Hitchen et al. (1994). The geometry of the SEN coupon is illustrated in Figure 3.9. \( G_c \) was calculated from the expression:

\[
G_c = \left( \frac{P_c^2}{2B} \right) \left( \frac{dC}{da} \right)
\]  

(3.1)

where \( P_c \) is the load at crack extension, \( B \) is the specimen thickness and \( dC/da \) is the change in compliance with crack length.
Critical strain energy release rate testing was performed on samples 150 mm long, 20 mm wide and 2 mm thick. The quantity $dC/da$ was found by measuring the compliance of a coupon with a single-edge notch of length 0 mm to 12 mm. Notches were cut using a jewellers saw. Two coupons were measured in this way for each type of material. Coupons were loaded to a strain of 0.1 % and compliance was taken from the slope of a linear regression fit to the load-extension plot. The compliance $C$ was plotted against crack length $a$ for both coupons and a fourth order polynomial was fitted to the average curve. This polynomial was differentiated to give $dC/da$ at a given crack length.

The value of $G_c$ was determined from SEN coupons with crack lengths of 4 mm, 8 mm and 12 mm which corresponds to an initial crack length to width ratio $a/W$ of 0.2, 0.4 and 0.6. Notches were cut using a jewellers saw and the notch was sharpened using a fresh razor blade. Two coupons were tested for each initial $a/W$ and loaded to failure. The value of $P_c$ was taken to be equal to the maximum load sustained by the notched coupon.

3.3.4 Multiple Specimen Critical J Integral Evaluation

The critical J integral $J_c$ was found from the method first described by Begley and Landes (1972). For the double-edge notch (DEN) geometry shown in Figure 3.10, $J_c$ was found from the expression:

$$J_c = \frac{-\Delta U}{2B\Delta a}$$  \hspace{1cm} (3.2)

where $\Delta U$ is the difference in potential energy associated with a crack extension of $\Delta a$ in two cracks and $B$ is the specimen thickness. The term $-\Delta U$ is the area between load-displacement curves for two specimens with a difference between the initial crack lengths of $\Delta a$. 

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Double-edge notch coupons 150 mm long, 20 mm wide and 2 mm thick were used for the $J_c$ determination. The double-edge notched coupon is illustrated in Figure 3.10. Notches were cut in the coupons using a jewellers saw and the notch was sharpened using a fresh razor blade. Coupons with initial crack lengths of 4 mm, 6 mm and 8 mm were tested corresponding to an initial $a/W$ of 0.4, 0.6 and 0.8. A value of $J_c$ at $a/W=0.5$ was obtained by subtracting the area under the load-displacement curve for the coupon with an initial $a/W$ of 0.6 from the area under the load-displacement curve for the coupon with an initial $a/W$ of 0.4. A value of $J_c$ at $a/W=0.7$ was obtained in a similar way by considering coupons with an initial $a/W$ of 0.6 and 0.8. The displacement bounding these areas was taken as the displacement corresponding to the maximum load sustained by the sample with the greater crack length. The areas under the load-displacement curves were found using computer software.

3.3.5 Single Specimen Critical $J$ Integral Evaluation

The critical $J$ integral $J_c$ may also be calculated using the single specimen technique of Rice et al. (1973). This technique was applied to the DEN coupon shown in Figure 3.10. Rice et al. (1973) stated that:

$$J_c = J_{el} + J_{pl}$$  \hspace{1cm} (3.3)

where $J_{el}$ is the $J$ integral due to elastic displacement at the critical point and $J_{pl}$ is the $J$ integral due to plastic displacement at the critical point.

$J_{el}$ was calculated from the expression:

$$J_{el} = \left(\frac{P_c^2}{4B}\right) \left(\frac{dC}{da}\right).$$  \hspace{1cm} (3.4)

where $P_c$ is the load at crack extension, $B$ is the specimen thickness and $dC/da$ is the change in compliance with crack length.
Chapter 3. Experimental Methods

\( J_{pl} \) was calculated from the expression:

\[
J_{pl} = \frac{A^*}{B(W - a)}
\]  

(3.5)

where the quantity \( A^* \) is the area bounded by the load-extension curve up to the critical point and the secant line from that point returning to the origin, \( W \) is the specimen width and \( a \) is the initial crack length.

As with the multiple specimen technique, DEN coupons 150 mm long, 20 mm wide and 2 mm thick were tested to determine \( J_c \). The quantity \( dC/da \) was found by measuring the compliance of a coupon with double-edge notches of increasing length in the range 0 to 8 mm in 1 mm steps. Notches were cut using a jewellers saw. The coupon was loaded to a strain of 0.1 % and the compliance was taken from the slope of a linear regression fit to the load-extension plot. Two coupons were tested in this way for each type of material. The compliance \( C \) was plotted against crack length \( a \) for both coupons and a fourth order polynomial was fitted to the average curve. This polynomial was differentiated to give \( dC/da \) at a given crack length.

For the single specimen technique there is the requirement that the specimen is deeply notched. Previous work using similar materials, but a variety of specimen geometries, has shown that the value of \( J_c \) is independent of crack length for \( a/W \) of 0.4 or greater (Agrawal et al., 1984; Singh and Parihar, 1986). For this reason the smallest value of initial \( a/W \) used was 0.4.

To determine the load at crack extension, coupons with initial crack lengths of 4 mm, 6 mm and 8 mm were tested corresponding to an initial \( a/W \) of 0.4, 0.6 and 0.8. Double-edge notches were cut in the coupons using a jewellers saw and the notch was sharpened using a fresh razor blade. The crack extension load was taken to be equal to the maximum load sustained by the sample. The area corresponding to \( A^* \) was found using computer software.
3.3.6 Impact Testing
Impact testing was performed on a miniature Charpy tester. Specimens were nominally 2 mm thick by 5 mm wide by 60 mm long. The 5 mm dimension was in the direction of impact, as shown in Figure 3.11. The specimens were notched to a depth of 0.5 mm by pressing a fresh scalpel blade into the material. The impact surface energy was calculated from the expression:

\[
\text{Impact surface energy} = \frac{W_{\text{Charpy}}}{B(W - a)}
\]  

(3.6)

where \(W_{\text{Charpy}}\) is the energy absorbed by the specimen in the Charpy test. Six samples were tested for each laminate type and the fracture surface energy was calculated by dividing the energy absorbed by the cross-sectional area of the specimen. The sample dimensions were measured accurately using a micrometer prior to testing.

3.4 Material Characterisation

3.4.1 Volume Fraction
The volume fraction of the laminates was found using a burn-off method. From each laminate, four samples with a mass of approximately 0.5 g were weighed accurately and placed in a crucible. The crucible was heated to 420 °C in a furnace for five hours. After cooling the mass of the residue was determined. The volume fraction of fibres \(V_f\) was calculated from the expression:

\[
V_f = \frac{(m_f/\rho_f)}{(m_m/\rho_m) + (m_f/\rho_f)}
\]  

(3.7)

where \(m_f\) and \(m_m\) are the mass of fibre and matrix, and \(\rho_f\) and \(\rho_m\) are the densities of fibre and matrix respectively. \(m_f\) was taken as the mass of the residue while \(m_m\) was taken as the mass lost during heating. \(\rho_f\) was taken from manufacturer’s data (Lafdi and
Chapter 3. Experimental Methods

Wright, 1998) and was given as 1.76 g/cm³. \( \rho_s \) was taken from manufacturer’s data (Berglund, 1998) and was given as 0.90 g/cm³.

3.4.2 Critical Length

Fragmentation tests were performed to determine the critical length of fibres and bundles. A mould (shown in Figure 3.12) was manufactured from stainless steel. The dimensions of the mould cavity were 150 mm by 20 mm by 1 mm. Strips 150 mm by 15 mm were cut from a 1 mm thick plaque of polypropylene manufactured as described in Section 3.2.4. A single strip of polypropylene was placed onto the base of the mould. Both parts of the mould were heated to 180 °C on a hot plate; this temperature is above the melting point of the polypropylene. The base of the mould was removed from the hot plate and a single fibre or bundle placed carefully on the molten polypropylene. Care was taken that the fibre or bundle was straight and aligned along the length of the mould. A second strip of polypropylene was then gently placed on top of the molten strip and the base of the mould was returned to the hot plate. When the second piece of polypropylene had melted both parts of the mould were removed from the hot plate and the mould was closed under hand pressure and allowed to cool to room temperature. When the mould had cooled the specimen was removed. Any flash on the specimen was removed using a scalpel.

Specimens were tensile tested on an Instron 1175 tensile testing machine at a crosshead speed of 0.5 mm/minute. Coupons were tabbed with 30 mm long friction tabs made from emery cloth. The extension of the specimen was not monitored directly but taken from the crosshead position to avoid straining the specimen while attaching an extensometer. Specimens were extended to a strain of 5%. The failure strain of Toray T300 carbon fibres in tension is 1.5% (Lafdi and Wright, 1998). Since the specimens were strained to over three times the failure strain of the fibres, it is reasonable to assume that fibres were saturated with breaks.

A number of methods were used to measure fragment lengths. An attempt was made to measure the length of fibre fragments using transmitted light microscopy. Unfortunately, it was not possible to identify fibre breaks with any certainty due to
the small depth of focus of the microscope. As an alternative the specimen was
attached to the front of a lightbox so the distance between fibre breaks could be
measured using a travelling microscope. Sometimes a region of plastic deformation
could be observed in the polypropylene matrix which assisted the identification of
fibre breaks. However, even with this technique not all of the fibre breaks could be
identified with confidence.

To ensure that fragment length was measured accurately it was necessary to remove
the polypropylene matrix. This was achieved by enclosing the specimen within the
mould used in its manufacture which was then heated to 420 °C in a furnace for five
hours. The mould was allowed to cool to room temperature and the top of the mould
carefully removed. A piece of adhesive tape 50 mm in length was pressed along the
centre of the mould and carefully removed and attached to a glass microscope slide.
This slide could then be viewed using transmitted light microscopy and the length of
the fragments measured with a graduated eyepiece.

3.4.3 Fracture Surface Images
Photographs of tested coupons and impact specimens were obtained by placing the
specimens on an optical bench illuminated by four lamps. Images were recorded with a
Sanyo digital camera.

The fracture surfaces were imaged on a Hitachi scanning electron microscope. It was
necessary to sputter coat the samples with gold prior to examination to prevent
charging of the matrix. These images were recorded using computer software. It was
found that the best images were obtained with an accelerating voltage of 5 kV and a
working distance of 15 mm.

3.5 Concluding Remarks
A novel manufacturing technique for producing fibre mats with a known proportion of
the fibres within bundles of a known size has been described in this Chapter. The
technique used to impregnate these fibre mats with polypropylene has been outlined.
Photographs and cross-sections of the manufactured materials have been presented. These images confirmed that the impregnation of the fibre mats was satisfactory.

Mechanical testing to determine the mechanical properties of the short-fibre composites has been outlined. Modulus, strength, critical strain energy release rate, critical J integral and impact surface energy were determined. Two methods were used to determine the J integral, a multiple specimen method and a single specimen method.

Methods for the characterisation of the laminates have been presented. The volume fraction of fibre in the laminates and the critical length of fibres and bundles were determined. Photography and scanning electron microscopy of the materials have been described. In the following Chapter the modulus and strength results are presented.
### Table 3.1. Relative masses of uncoated fibre tows and fibre tows coated with wax.

<table>
<thead>
<tr>
<th>Number of fibres in tow</th>
<th>Mass of 30 cm of uncoated fibre tow / mg</th>
<th>Mass of 30 cm of wax-coated fibre tow / mg</th>
<th>Ratio of mass of wax-coated to uncoated fibre tow.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>20.1 ± 0.1</td>
<td>34.3 ± 3.3</td>
<td>1.71</td>
</tr>
<tr>
<td>6000</td>
<td>119.8 ± 0.3</td>
<td>251.0 ± 3.2</td>
<td>2.10</td>
</tr>
</tbody>
</table>

### Table 3.2. Details of fibre mats with a controlled proportion of fibres within bundles.

<table>
<thead>
<tr>
<th>Proportion of fibres in bundles / %</th>
<th>Mass of filamentised fibres in mat / g</th>
<th>Mass of bundled fibres in mat / g</th>
<th>Overall mass of fibre per mat / g</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.5</td>
<td>0</td>
<td>1.5</td>
</tr>
<tr>
<td>25</td>
<td>1.5</td>
<td>0.5</td>
<td>2</td>
</tr>
<tr>
<td>50</td>
<td>1.5</td>
<td>1.5</td>
<td>3</td>
</tr>
<tr>
<td>75</td>
<td>1.5</td>
<td>4.5</td>
<td>6</td>
</tr>
<tr>
<td>100</td>
<td>0</td>
<td>6</td>
<td>6</td>
</tr>
</tbody>
</table>

### Table 3.3. Details of fibre mats required for 1 mm thick laminates.

<table>
<thead>
<tr>
<th>Proportion of fibres in bundles / %</th>
<th>Overall mass of fibre per mat / g</th>
<th>Number of mats required for 1 mm thick laminate</th>
<th>Total mass of fibre within laminate / g</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.5</td>
<td>4</td>
<td>6</td>
</tr>
<tr>
<td>25</td>
<td>2</td>
<td>3</td>
<td>6</td>
</tr>
<tr>
<td>50</td>
<td>3</td>
<td>2</td>
<td>6</td>
</tr>
<tr>
<td>75</td>
<td>6</td>
<td>1</td>
<td>6</td>
</tr>
<tr>
<td>100</td>
<td>6</td>
<td>1</td>
<td>6</td>
</tr>
</tbody>
</table>
Table 3.4 Details of fibre mats required for 2 mm thick laminates.

<table>
<thead>
<tr>
<th>Proportion of fibres in bundles / %</th>
<th>Overall mass of fibre per mat / g</th>
<th>Number of mats required for 2 mm thick laminate</th>
<th>Total mass of fibre within laminate / g</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.5</td>
<td>8</td>
<td>12</td>
</tr>
<tr>
<td>25</td>
<td>2</td>
<td>6</td>
<td>12</td>
</tr>
<tr>
<td>50</td>
<td>3</td>
<td>4</td>
<td>12</td>
</tr>
<tr>
<td>75</td>
<td>6</td>
<td>2</td>
<td>12</td>
</tr>
<tr>
<td>100</td>
<td>6</td>
<td>2</td>
<td>12</td>
</tr>
</tbody>
</table>

Table 3.5. List of composite materials tested for modulus and strength. All laminates are 1 mm thick.

<table>
<thead>
<tr>
<th>Material Code</th>
<th>Fibre Length / mm</th>
<th>Proportion of fibres within bundles / %</th>
<th>Number of fibres within bundle</th>
<th>Overall Volume Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 mm / 0 %</td>
<td>5</td>
<td>0</td>
<td>-</td>
<td>0.122 ± 0.012</td>
</tr>
<tr>
<td>5 mm / 25 % 6k</td>
<td>5</td>
<td>25</td>
<td>6000</td>
<td>0.094 ± 0.017</td>
</tr>
<tr>
<td>5 mm / 50 % 6k</td>
<td>5</td>
<td>50</td>
<td>6000</td>
<td>0.154 ± 0.033</td>
</tr>
<tr>
<td>5 mm / 75 % 6k</td>
<td>5</td>
<td>75</td>
<td>6000</td>
<td>0.163 ± 0.013</td>
</tr>
<tr>
<td>5 mm / 100 % 6k</td>
<td>5</td>
<td>100</td>
<td>6000</td>
<td>0.201 ± 0.014</td>
</tr>
</tbody>
</table>

Table 3.6. List of composite materials tested for modulus and critical strain energy release rate. All laminates are 2 mm thick.

<table>
<thead>
<tr>
<th>Material Code</th>
<th>Fibre Length / mm</th>
<th>Proportion of fibres within bundles / %</th>
<th>Number of fibres within bundle</th>
<th>Overall Volume Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 mm / 0 %</td>
<td>5</td>
<td>0</td>
<td>-</td>
<td>0.114 ± 0.008</td>
</tr>
<tr>
<td>5 mm / 25 % 6k</td>
<td>5</td>
<td>25</td>
<td>6000</td>
<td>0.119 ± 0.017</td>
</tr>
<tr>
<td>5 mm / 50 % 6k</td>
<td>5</td>
<td>50</td>
<td>6000</td>
<td>0.097 ± 0.020</td>
</tr>
<tr>
<td>5 mm / 75 % 6k</td>
<td>5</td>
<td>75</td>
<td>6000</td>
<td>0.145 ± 0.011</td>
</tr>
<tr>
<td>5 mm / 100 % 6k</td>
<td>5</td>
<td>100</td>
<td>6000</td>
<td>0.222 ± 0.023</td>
</tr>
</tbody>
</table>
### Table 3.7. List of composite materials tested for modulus and critical J integral. All laminates are 2 mm thick.

<table>
<thead>
<tr>
<th>Material Code</th>
<th>Fibre Length / mm</th>
<th>Proportion of fibres within bundles / %</th>
<th>Number of fibres within bundle</th>
<th>Overall Volume Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 mm / 0 %</td>
<td>5</td>
<td>0</td>
<td>-</td>
<td>0.115 ± 0.018</td>
</tr>
<tr>
<td>5 mm / 50 % 1k</td>
<td>5</td>
<td>50</td>
<td>1000</td>
<td>0.094 ± 0.009</td>
</tr>
<tr>
<td>5 mm / 50 % 6k</td>
<td>5</td>
<td>50</td>
<td>6000</td>
<td>0.090 ± 0.011</td>
</tr>
<tr>
<td>5 mm / 100 % 1k</td>
<td>5</td>
<td>100</td>
<td>1000</td>
<td>0.106 ± 0.020</td>
</tr>
<tr>
<td>5 mm / 100 % 6k</td>
<td>5</td>
<td>100</td>
<td>6000</td>
<td>0.178 ± 0.015</td>
</tr>
<tr>
<td>10 mm / 0 %</td>
<td>10</td>
<td>0</td>
<td>-</td>
<td>0.112 ± 0.021</td>
</tr>
<tr>
<td>10 mm / 50 % 1k</td>
<td>10</td>
<td>50</td>
<td>1000</td>
<td>0.134 ± 0.021</td>
</tr>
<tr>
<td>10 mm / 50 % 6k</td>
<td>10</td>
<td>50</td>
<td>6000</td>
<td>0.151 ± 0.007</td>
</tr>
<tr>
<td>10 mm / 100 % 1k</td>
<td>10</td>
<td>100</td>
<td>1000</td>
<td>0.196 ± 0.016</td>
</tr>
<tr>
<td>10 mm / 100 % 6k</td>
<td>10</td>
<td>100</td>
<td>6000</td>
<td>0.137 ± 0.025</td>
</tr>
</tbody>
</table>

### Table 3.8. List of composite materials tested for modulus only.

<table>
<thead>
<tr>
<th>Material Code</th>
<th>Fibre Length / mm</th>
<th>Proportion of fibres within bundles / %</th>
<th>Number of fibres within bundle</th>
<th>Overall Volume Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 mm / 0 % (1 mm thickness)</td>
<td>5</td>
<td>0</td>
<td>-</td>
<td>0.135 ± 0.013</td>
</tr>
<tr>
<td>5 mm / 100 % 6k (1 mm thickness)</td>
<td>5</td>
<td>100</td>
<td>6000</td>
<td>0.200 ± 0.034</td>
</tr>
<tr>
<td>5 mm / 0 % (2 mm thickness)</td>
<td>5</td>
<td>0</td>
<td>-</td>
<td>0.131 ± 0.015</td>
</tr>
<tr>
<td>5 mm / 100 % 6k (2 mm thickness)</td>
<td>5</td>
<td>100</td>
<td>6000</td>
<td>0.223 ± 0.014</td>
</tr>
</tbody>
</table>
Figure 3.1. Flowchart detailing the preparation of mats with a known proportion of fibres within bundles.

Figure 3.2. Diagram illustrating arrangement used to coat the carbon fibre with wax.
Figure 3.3. Photograph of a laminate with 0 % of the fibres within bundles. (1 mm thick laminate of 5 mm / 0 % material.)

Figure 3.4. Photograph of a laminate with 50 % of the fibres within bundles. (1 mm thick laminate of 5 mm / 50 % 6k material.)
Figure 3.5. Photograph of a laminate with 100 % of the fibres within bundles. (1 mm thick laminate of 5 mm / 100 % 6k material.)

Figure 3.6. Section of a laminate with 0 % of the fibres within bundles. (1 mm thick laminate of 5 mm / 0 % material.)
Figure 3.7. Section of a laminate with 50 % of the fibres within bundles. (1 mm thick laminate of 5 mm / 50 % 6k material.)

Figure 3.8. Section of a laminate with 100 % of the fibres within bundles. (1 mm thick laminate of 5 mm / 100 % 6k material.)
Figure 3.9. Illustration of the single edge notch geometry used in this work. ($W = 20$ mm, $a = 4$ mm, 8 mm or 12 mm)

Figure 3.10. Illustration of the double-edge notch specimen used in this work. ($W = 10$ mm, $a = 4$ mm, 6 mm or 8 mm)

Figure 3.11. Illustration of the specimen used for impact testing in this work. ($W = 5$ mm, $a = 0.5$ mm)

Figure 3.12. Schematic of the cross section of the mould used to manufacture the fragmentation specimens.
Chapter 4. Modulus and Strength Results
4.1 Introduction

The present Chapter outlines the Young’s modulus results and tensile strength results, for unreinforced polypropylene and for a range of short-fibre composites. Examples of stress-strain curves for unreinforced polypropylene and a selection of short-fibre composites are shown. For the modulus and strength results, comparisons are made between short-fibre composites with different proportions of fibres within bundles. In addition, for the modulus results, a comparison is made between short-fibre composites with different fibre lengths, with different bundle sizes and laminates of different thickness. There is a preliminary discussion of results at this stage. The modulus and strength results will be discussed in further detail alongside modulus and strength modelling in Chapter 5.

4.2 Polypropylene

4.2.1 Stress-Strain Curves

An example of a stress-strain curve for a coupon of unreinforced polypropylene is presented in Figure 4.1. The strain was monitored with an extensometer of 50 mm gauge length. It can be seen that the deformation is linear at very small strains. However, with increasing strain, even below 0.2 % strain, there is some non-linear deformation. At larger strains, the deformation is predominantly non-linear. This stress-strain behaviour is typical of a semi-crystalline thermoplastic.

4.2.2 Modulus Results

A plaque of polypropylene, 2 mm in thickness, was manufactured from film using the same film stacking technique used for the impregnation of the fibre mats. The modulus of polypropylene was taken from the mean of six coupons over a strain range of 0 % to 0.1 %, and was found to be 1.56 ± 0.17 GPa. A typical range for the modulus of polypropylene is 1.1 to 1.6 GPa (Berglund, 1998). The modulus of the polypropylene manufactured in this work is towards the upper end of this range, which suggests that the polypropylene has high crystallinity (Berglund, 1998).
4.2.3 Strength Results

The tensile strength of the polypropylene plaque was determined from six coupons, of 2 mm thickness, as used for the modulus determination. The strength of the polypropylene was found to be 24.4 ± 0.7 MPa. This value of strength is below the typical range for tensile strengths of 30 to 40 MPa for polypropylene (Berglund, 1998). The most likely explanation is that small imperfections in the plaque reduce the strength of the coupon below that expected. These imperfections could be areas of porosity or foreign matter within the polypropylene. Such imperfections would have a far greater effect on the strength of the coupon than on the modulus of the coupon.

4.3 Short-Fibre Composites

4.3.1 Stress-Strain Curves

Typical stress-strain curves for materials with 0 %, 50 % and 100 % of fibres within bundles are shown in Figure 4.2. The curves shown are for 1 mm thick laminates with 5 mm long fibres and bundles containing 6000 fibres per bundle. It can be seen from Figure 4.2 that the proportion of fibres within bundles has a strong effect on the stress-strain curves for the materials and that both modulus and strength decrease with an increasing proportion of fibres within bundles.

The stress-strain curves are increasingly non-linear with an increasing proportion of fibres within bundles. In fact, the stress-strain curve for the material with 100 % of fibres within bundles is very similar in shape to the stress-strain curve for unreinforced polypropylene. This suggests that the bundles have little reinforcing effect on the polypropylene. The mechanical properties of the 100 % of fibres within bundles material appear to be dominated by the mechanical properties of the polypropylene matrix.

The variation of strain to failure with the proportion of fibres within bundles is more complex. It can be seen in Figure 4.2 that the strain to failure of the 50 % of fibres within bundles material is smallest, the strain to failure of the 0 % of fibres within bundles material is larger and the strain to failure of the 100 % of fibres within bundles material is the largest. Possible reasons for this behaviour are discussed in Section 4.4.
4.3.2 Presentation of Modulus Results

The modulus results for the short-fibre composites are shown in Figures 4.3 to 4.12. As mentioned earlier, the modulus is measured over the strain range of 0 % to 0.1 %. Laminates have been grouped with those containing the same fibre length and bundle size, so the only controlled variable in each Figure is the proportion of fibres within bundles. Laminates with up to five different proportions of fibres within bundles are shown on each Figure together with the value for unreinforced polypropylene. Laminates are 2 mm thickness unless stated otherwise.

Figures 4.3, 4.5, 4.7, 4.9 and 4.11 show the variation of tensile modulus with volume fraction. The proportion of fibres within bundles is indicated by different colours and symbols. The error bars show the standard deviation of the modulus and volume fraction values. It is necessary to show results in this way because of the unavoidable variation in the overall volume fraction of fibre within the laminates. The modulus of the unreinforced polypropylene is shown as a composite material with a volume fraction of zero. Typically, the modulus of a composite material will increase approximately linearly with increasing volume fraction. The gradient of a line connecting the unreinforced polypropylene point to that of the short-fibre composite provides a good indication of the effectiveness of the reinforcement; the steeper the gradient the more effective the reinforcement. Hence, in Figure 4.3, the effectiveness of the reinforcement in the material with 100 % of fibres within bundles is much less than the effectiveness of the reinforcement in the material with 0 % of fibres within bundles.

Figures 4.4, 4.6, 4.8, 4.10 and 4.12 show the variation of tensile modulus with proportion of fibres in bundles. As is clear from Figures 4.5 and 4.6, for example, modulus values are not directly comparable due to the variation in volume fraction between laminates. However, notwithstanding the variation in overall fibre volume fraction, it is clear that the effectiveness of reinforcement decreases from the 0 % of fibres within bundles material to the 100 % of fibres within bundles material in each case.
4.3.3 Modulus Results

Modulus results for 5 mm / 1k materials are shown in Figures 4.3 and 4.4. It can be seen from Figure 4.3 that the volume fraction of these laminates lies within a narrow range, less than 0.04. Modulus decreases significantly with an increasing proportion of fibres within bundles, which can be seen more clearly in Figure 4.4, and hence, the values of moduli are comparable because of limited variation in volume fraction. The absolute decrease in modulus is greater between the materials with 0 % and 50 % of fibres within bundles than between the materials with 50 % and 100 % of fibres within bundles, although in both cases the modulus is reduced by approximately half.

The results in Figure 4.5 show a clear trend in the effectiveness of the reinforcement of 5 mm / 6k materials, even though there is a large variation in the volume fraction of these 2 mm thick laminates. In each case the gradient of a line between the unreinforced polypropylene point and a laminate point decreases with an increasing proportion of fibres within bundles. It is therefore clear that an increasing proportion of fibres within bundles reduces the effectiveness of the reinforcement for these materials. Variations in volume fraction reduce the validity of the comparison of modulus values shown in Figure 4.6. However, the trend is clear. In fact, the laminates with 100 % of fibres within bundles have the highest volume fraction and the lowest modulus.

The results for the 1 mm thick laminates of 5 mm / 6k materials are shown in Figures 4.7 and 4.8. These results are comparable to those for 2 mm thick laminates of the same materials (Figures 4.5 and 4.6) and similar conclusions can be drawn. The only significant difference is, that in each case, the modulus of the materials containing bundles (25 %, 50 %, 75 % and 100 % of fibres within bundles) is lower for the 1 mm thick laminates than for the 2 mm thick laminates. Possible reasons for this difference are discussed in Chapter 5.

Results for the 10 mm / 1k materials are shown in Figures 4.9 and 4.10. These laminates are within a narrow range of volume fraction, less than 0.03, so it is possible to compare the modulus values directly. As with other modulus results, the modulus decreases with an increasing proportion of fibres within bundles and the absolute decrease between 0 %
and 50% of fibres within bundles materials is greater than the absolute decrease between 50% and 100% of fibres within bundles materials.

The results for the 10 mm/6k materials are shown in Figures 4.11 and 4.12; these laminates exhibit significant variation in volume fraction. Figure 4.11 reveals that the laminates with a lower proportion of fibres in bundles exhibit the highest modulus, yet have a lower volume fraction. Since laminates with a low volume fraction would be expected to have a low modulus, the results reinforce the earlier observations concerning the effect of fibre bundling (i.e. it is detrimental to modulus). Figure 4.12 shows a decrease in modulus with an increasing proportion of fibres within bundles. A comparison of Figures 4.10 and 4.12 shows that the magnitude of the absolute decrease in modulus for the 10 mm/6k materials is much greater than the absolute decrease in modulus for the 10 mm/1k materials.

There are a number of observations valid across the range of materials that can be made from the results presented here. In every case modulus decreases with an increasing proportion of fibres within bundles. The 10 mm/1k materials (Figures 4.9 and 4.10) show the smallest decrease in modulus, while the largest decrease is for 1 mm thick laminates of the 5 mm/6k material (Figures 4.7 and 4.8). For each type of material a plot of modulus against proportion of fibres within bundles results in a concave curve. However, the degree of curvature varies for the different materials. The results for 10 mm/6k materials are almost linear, while those for 5 mm/6k materials show very pronounced curvature.

Preliminary discussion of the modulus results is presented in Section 4.4. A more detailed discussion is included in Chapter 5 where models for the modulus are also discussed.

4.3.4 Strength Results
The tensile strength of the 5 mm/6k materials, 1 mm thick laminates, was determined and the variation of strength with proportion of fibres within bundles is shown in Figure 4.13. The results show that the strength of the 25% to 100% of
fibres within bundles materials is reasonably constant and about one quarter of the strength of the 0 % of fibres within bundles material. These results suggest that the reduction in strength is due to the presence of bundles.

Strength results are discussed in Section 4.4. Modelling the strength of a material containing bundles is discussed in Chapter 5.

4.4 Discussion of Results

4.4.1 Modulus

Modulus results have been presented for a variety of short-fibre composites. The materials vary in the proportion of fibres within bundles, length of the fibre, number of fibres per bundle and the thickness of the laminate. A complicating factor is the variation of the overall volume fraction of fibre for these materials which is unavoidable. However, it is still possible to deduce the effect of fibre-bundling from these results.

There is no doubt that the reinforcing effectiveness of individual fibres is greater than that of fibre bundles. For a given volume fraction, the modulus decreases with an increasing proportion of fibres within bundles. This is the case irrespective of fibre length, number of fibres per bundle or laminate thickness. The magnitude of the decrease in modulus varies with the details of the material. The decrease in modulus is greater for 1 mm thick laminates than for 2 mm thick laminates.

There is some evidence that a larger bundle aspect ratio results in a reduced decrease in the modulus. The 10 mm / 1k materials show the least decrease in modulus with increasing proportion of fibres within bundles of all the material types. The decrease in modulus for the 5 mm / 1k materials appears to be smaller than for the two material types with 6k bundles which suggests that for these materials, the number of fibres per bundle is more important than the length of the bundle. For the materials containing 6k bundles, the modulus of the 100 % of fibres within bundles materials is comparable to that of the unreinforced polypropylene. This indicates that the 6k bundles provide no reinforcement, which is consistent with the observation in
Section 4.3.1 that the stress-strain curve for the 5 mm / 100 % 6k material is similar to the stress-strain curve for unreinforced polypropylene.

4.4.2 Strength
Tensile strength was determined for a smaller range of short-fibre composites than the tensile modulus. The results show that the strength appears to be dependent on the presence of bundles in the material. It seems reasonable to suggest that the failure is initiated either at bundles oriented transverse to the applied load or at bundle ends when they are aligned parallel to the tensile load. The same sites are likely to be preferred for the propagation of the crack although in this case the transversely oriented bundles are likely to be more significant. This interpretation is supported by the shape of the graph in Figure 4.13, because the presence of bundles is critical rather than the proportion of fibres within bundles.

It is useful to compare the strength of the short-fibre composites with that of the unreinforced polypropylene. The strength of the 0 % of fibres within bundles material is substantially greater than the strength of the unreinforced polypropylene. However, the strength of the 25 %, 50 %, 75 % and 100 % of fibres within bundles materials is slightly lower than the strength of the unreinforced polypropylene. This observation supports the suggestion that the bundles are points of weakness in these short-fibre composites. It is possible that the fibre-matrix interface is weaker within the bundles, and combined with strain magnification around the bundles, this could explain the reduction in strength compared to the unreinforced polypropylene.

4.4.3 Stress-Strain Curves
In the light of the strength and modulus results it is worthwhile re-examining the strain to failure of the stress-strain curves shown in Figure 4.2. The modulus results for these materials show that the moduli of the materials decrease with an increasing proportion of fibres within bundles, as discussed above, but the strength of the materials depend on the presence or absence of fibre bundles. So, taking these two effects together, it is not surprising that for materials containing fibre bundles, that is materials with 25 %, 50 % 75 % and 100 % of fibres within bundles, the strain
to failure increases as the modulus decreases. Although the modulus of the 0 \% of fibres within bundles material is greater than the materials containing fibre bundles, the strength for this material is greater so the strain to failure is greater than would otherwise be expected.

4.5 Conclusions

Stress-strain curves, modulus results and strength results have been presented for unreinforced polypropylene and various short-fibre composites. A single series of materials was studied in terms of stress-strain behaviour and strength properties. A wider range of materials is considered for the modulus results; these materials varied in terms of the length of the fibre, number of fibres per bundle and the thickness of the laminate.

It has been shown that an increase in the proportion of fibres within bundles results in a decrease in the modulus of a material. There is evidence that this decrease is less severe when the material consists of higher aspect ratio bundles or when the thickness of the laminate is greater. For the materials studied, the presence of bundles in a material is associated with a large decrease in strength compared to the fully filamentised material.
Figure 4.1. Typical stress-strain curve for unreinforced polypropylene.

Figure 4.2. Typical stress-strain curves for materials with 0%, 50% and 100% of fibres within bundles.
Figure 4.3. Graph of tensile modulus against volume fraction for 5 mm / 1k materials (2 mm thick laminates).

Figure 4.4. Graph of tensile modulus against proportion of fibres within bundles for 5 mm / 1k materials (2 mm thick laminates).
Figure 4.5. Graph of tensile modulus against volume fraction for 5 mm / 6k materials (2 mm thick laminates).

Figure 4.6. Graph of tensile modulus against proportion of fibres within bundles for 5 mm / 6k materials (2 mm thick laminates).
Figure 4.7. Graph of tensile modulus against volume fraction for 5 mm / 6k materials (1 mm thick laminates).

Figure 4.8. Graph of tensile modulus against proportion of fibres within bundles for 5 mm / 6k materials (1 mm thick laminates).
Figure 4.9. Graph of tensile modulus against volume fraction for 10 mm / 1k materials (2 mm thick laminates).

Figure 4.10. Graph of tensile modulus against proportion of fibres within bundles for 10 mm / 1k materials (2 mm thick laminates).
Figure 4.11. Graph of tensile modulus against volume fraction for 10 mm / 6k materials (2 mm thick laminates).

Figure 4.12. Graph of tensile modulus against proportion of fibres within bundles for 10 mm / 6k materials (2 mm thick laminates).
Figure 4.13. Graph of tensile strength against proportion of fibres within bundles for 5 mm / 6k materials (1 mm thick laminates).
Chapter 5. Modulus and Strength Modelling
5.1 Introduction
The present Chapter describes modelling of the modulus and strength of the short-fibre composites manufactured in this study. A model for the modulus of a material containing bundles has been developed and is described here. Predictions for the modulus of the materials have been calculated using two shear-lag models. These predictions have been compared with the modulus results presented in Chapter 4. Finally, modelling the strength of a material containing bundles is discussed.

5.2 A Model for the Modulus of a Material Containing Bundles

5.2.1 Variables
In Chapter 2, an expression for the modulus of a composite material $E_c$ containing short fibres was introduced:

$$E_c = \eta_o \eta_l E_f V_f + E_m V_m$$  \hspace{1cm} (5.1)

where $\eta_o$ and $\eta_l$ are the orientation and length efficiency factors, $E_f$ and $E_m$ are the modulus of fibre and matrix and $V_f$ and $V_m$ are the volume fractions of fibre and matrix. It is possible to modify this expression for a material containing both individual fibres and fibre bundles. This requires the volume fractions of fibre and matrix to be divided into components outside bundles and within bundles, hence:

$$V_f = V_{fo} + V_{fb}$$  \hspace{1cm} (5.2)

and

$$V_m = V_{mo} + V_{mb}$$  \hspace{1cm} (5.3)

where $V_{fo}$ is the volume fraction of fibres outside bundles, $V_{fb}$ is the volume fraction of fibres within bundles, $V_{mo}$ is the volume fraction of matrix outside bundles and $V_{mb}$ is the volume fraction of matrix within bundles. The definitions of these terms are illustrated in Figure 5.1. Using these terms Equation 5.1 may be modified to include a term for the contribution of bundles:
\[
E_c = \eta_o \eta_{lb} E_f V_{fo} + \eta_o \eta_{lb} E_b (V_{fb} + V_{nb}) + E_m V_{ms}
\]  \hspace{1cm} (5.4)

where $E_b$ is the modulus of the bundle and $\eta_o$ and $\eta_{lb}$ are the length efficiency factors of the fibres outside bundles and of the bundles themselves. The variables used in this model are listed in Table 5.1.

5.2.2 Material Properties

The orientation efficiency factor $\eta_o$ was taken as 0.375 which is the theoretical value for a planar random fibre arrangement (Krenchel, 1964). It is reasonable to assume that individual fibres and fibre bundles have the same orientation distribution, hence the value of $\eta_o$ is the same for individual fibres and fibre bundles.

The radius of Toray T300 fibres have been measured using scanning electron microscopy and the mean radius found was 3.95 $\mu$m (Al-Hassani, 1987). The modulus of Toray T300 fibres $E_f$ has been taken as 230 GPa from manufacturers data (Laﬁdi and Wright, 1998). The shear modulus of the carbon fibre $G_f$ was taken as 18 GPa (Smith, 1996).

Determination of the radius of the bundles was not straightforward but was achieved from a knowledge of the cross-sectional area of the bundle. Microtomed cross-sections of the composite material were viewed with optical microscopy. Observations showed that bundles were not circular, due to the pressure applied during processing, hence the radius could not be measured directly. The cross-sectional area of a bundle perpendicular to the cross-section was used to determine the radius of an equivalent circular bundle. The resulting radii of the bundles are 395 $\mu$m and 161 $\mu$m for the bundles containing 6000 and 1000 fibres respectively.

The area of fibre within the cross-section of a bundle was calculated from the radius of the fibre and the number of fibres per bundle. A comparison of the area of fibre with the area of the bundle revealed that approximately 60% of the area of the bundle was fibre. This translates to a volume fraction of 0.6 within the bundle or in terms of the volume fractions defined earlier:
\[
\frac{V_{fb}}{V_{fb} + V_{mb}} = 0.6.
\] (5.6)

The volume fraction of fibres within the bundle was used to calculate the modulus of the bundles \(E_b\) using the Voigt equation so:

\[
E_b = E_f \left( \frac{V_{fb}}{V_{fb} + V_{mb}} \right) + E_m \left( \frac{V_{mb}}{V_{fb} + V_{mb}} \right). \tag{5.7}
\]

The application of the Voigt equation to an impregnated bundle is justified because the bundle is analogous to a unidirectional continuous composite material. The tensile modulus of the bundle was calculated to be 139 GPa. The shear modulus of the bundles was taken as 5 GPa which is a reasonable value for the shear modulus of a unidirectional carbon fibre-polypropylene material.

The tensile modulus of the polypropylene matrix \(E_m\) was determined experimentally as 1.56 GPa (see Chapter 4). The shear modulus of polypropylene \(G_m\) was calculated from the tensile modulus \(E_m\) using the expression:

\[
G_m = \frac{E_m}{2(1+v)}.
\] (5.8)

assuming a Poisson’s ratio \(v\) of 0.3. The resulting shear modulus value was 0.6 GPa.

The values of the material properties used in the calculation of the predicted moduli are summarised in Table 5.2.

5.2.3 Calculation of Length Efficiency Factors

Length efficiency factors account for the reduced reinforcing efficiency of a discontinuous fibre relative to a continuous fibre and may be calculated from a shear-lag approach. As described in Chapter 2, an early shear-lag approach was developed
by Cox (1952) who showed that the length efficiency factor $\eta_l$ could be evaluated using the expressions:

$$\eta_l = 1 - \left( \frac{\tanh(\beta_{Cox} l/2)}{(\beta_{Cox} l/2)} \right)$$

(5.9)

where

$$\beta_{Cox} = \left( \frac{2G_m}{E_f r_f^2 \ln(R/r_f)} \right)^{1/2}$$

(5.10)

$\beta_{Cox}$ is the length efficiency parameter from the Cox model, $l$ is the fibre length, $G_m$ is the shear modulus of the matrix, $r_f$ is the fibre radius, and $R$ is half the distance between fibres. The term $(R/r_f)$ may be calculated from the expression:

$$V_f = \left( \frac{r_f}{R} \right)^2$$

(5.11)

Recently, a more rigorous shear-lag expression has been developed by Nairn (1997). In this case, the length efficiency factor $\eta_l$ is given by:

$$\eta_l = 1 - \left( \frac{\tanh(\beta_{Nairn} l/2)}{(\beta_{Nairn} l/2)} \right)$$

(5.12)

where

$$\beta_{Nairn} = \left[ \frac{2}{r_f^2 E_f E_m} \left( \frac{E_f V_f + E_m V_m}{\frac{V_m}{4G_f} + \frac{1}{2G_m} \left( \frac{1}{V_m} \ln \frac{1}{V_f} - 1 - \frac{V_m}{2} \right)} \right) \right]^{1/2}$$

(5.13)

where $\beta_{Nairn}$ is the length efficiency parameter from the Nairn model and $G_f$ is the shear modulus of the fibre.
The Nairn expression has been more rigorously derived than the Cox model. Nairn (1997) states that his model starts from the exact equations of elasticity for axisymmetric stress states in transversely isotropic materials and introduces the minimum assumptions required to derive the most commonly used shear-lag equations. Nairn (1997) goes on to state that his shear-lag model gives far closer agreement with finite element analysis results than the Cox model.

Figures 5.2 to 5.4 show a comparison of modulus predictions for a random short-fibre composite material of polypropylene using the Cox and Nairn approaches. Predictions are expressed as the ratio of the predicted composite material modulus to the modulus of the matrix versus the fibre modulus to matrix modulus ratio. Each Figure shows the predictions for a different aspect ratio fibre. Figure 5.2 shows the prediction for a fibre with an aspect ratio of 1265, the aspect ratio of a 10 mm long carbon fibre, the highest aspect ratio for the reinforcement used in this study. There is excellent agreement between the predictions using the two models. Predictions for a fibre of aspect ratio 31.06 are shown in Figure 5.3. This aspect ratio corresponds to that of a 10 mm long bundle containing 1000 fibres, the largest aspect ratio bundle used in this study. In this case it can be seen that there is some difference between the two models at higher values of the fibre modulus to matrix modulus ratio. The ratio of bundle modulus to matrix modulus used in the model described in this Chapter is 89, which is sufficiently large to show a difference between the Cox and Nairn models in Figure 5.3. The difference between the two shear-lag approaches is even greater in Figure 5.4 which shows the predictions for a composite material containing fibres with an aspect ratio of 6.329. This aspect ratio corresponds to that of a 5 mm long bundle containing 6000 fibres, the lowest aspect ratio for the reinforcement used in this study. Figures 5.2 to 5.4 show that the two shear-lag models show excellent agreement when the fibre has relatively high aspect ratio, but as the fibre aspect ratio decreases, the modulus prediction using the Cox shear-lag model becomes substantially lower than the predictions using the Nairn shear-lag model.
Both the Cox and Nairn expressions have been used in the model described here and have been applied to filamentised fibres and fibre bundles within the composite materials. The shear-lag models consider a cylindrical fibre (radius \( r_f \)) at the centre of a cylinder of matrix (radius \( R \)). The radius of the cylinder of matrix, required for the Cox model, was found from the volume fraction of fibre using Equation 5.11.

The application of both versions of the shear-lag analysis to this work requires some care. The 0 % of fibres within bundles material contains only filamentised fibres so the application of the shear-lag models to this material is straightforward. For the 100 % of fibres within bundles materials the bundles were treated as fibres, with the properties described in Section 5.2.2, surrounded by polypropylene only. However, for the 25 %, 50 % and 75 % of fibres within bundles materials the calculation of length efficiency factors is more complex. The length efficiency factors for filamentised fibres were calculated based on a fibre at the centre of matrix of polypropylene, the radius of this cylinder being found from the volume fraction of polypropylene outside bundles. In other words the volume fraction of bundles (both fibre and matrix) was disregarded when calculating the length efficiency factor of the filamentised fibres. The length efficiency factors for fibre bundles were calculated by treating the bundle as a fibre at the centre of a cylinder consisting of filamentised fibres and polypropylene. In this case it was necessary to calculate the modulus of the material surrounding the bundle using Equation 5.1 applied to the fibre and matrix volume fractions outside the bundle.

5.2.4 Combination of Contributions

Equation 5.4 is a modification of Equation 5.1 which is a widely used expression for the modulus of a short-fibre composite. Equation 5.1 is in turn a development of the Voigt model for longitudinal modulus of an idealised unidirectional composite. In the Voigt model it is assumed that the strain is equal in both fibre and matrix, hence the elements are in parallel. Equation 5.4 assumes similarly that the strain is constant in fibres, bundles and matrix. The Reuss model may be used to determine the transverse modulus of an idealised unidirectional composite. In this case it is assumed that the stress is constant in fibres and matrix, hence the elements are in
series. This approach can be applied to the model described here by treating the three elements as being in series. In fact, for the model described here, it is possible to combine the three elements in eight different ways as illustrated in Figure 5.5.

Although there are eight possible combinations, only three of these are applicable to the composite materials manufactured in this study. Combination I has all three elements in parallel which is analogous to the uniform strain case of the Voigt model. A representation of this is shown in Figure 5.6. The example given is that of composite material containing only filamentised fibres. This combination would equally apply to a material where there were few bundles and the bundles are small relative to the thickness of the laminate. These conditions are necessary so that fibres are not excluded from a cross-section of the material by a single, or several, bundles. If these conditions are not met for a material containing both filamentised fibres and fibre bundles then the situation shown in Figure 5.7 will arise. The bundle is large relative to the thickness of the laminate so it will exclude filamentised fibres from around it. In this case it is not reasonable to treat the fibres and bundles as being in parallel so it is appropriate to apply combination IV. This combination has the fibres and bundles in series and the matrix in parallel with both the fibres and matrix. The third relevant description is combination VIII which represents the constant stress case used in the Reuss model. The example for this combination is shown in Figure 5.8 and is an isolated bundle surrounded by matrix. This treatment is applicable to materials with areas of only resin between reinforcement. This may occur when there is a high proportion of fibres within bundles, and it is more relevant when the number of reinforcing units is reduced because each bundle is larger.

It can be seen that a different combination of the elements is appropriate for different situations. In Figures 5.9 to 5.32 all three methods are used to produce predictions of composite modulus. Combination I is described as the parallel model, combination IV is described as the series / parallel model and combination VIII is described as the series model.
5.3 Comparison of Predictions with Experimental Results

5.3.1 Presentation of Predictions

This Section compares the predictions from the models with the experimental results presented in Chapter 4. The data are presented as plots of tensile modulus against volume fraction of fibre which allows a comparison independent of the effect of volume fraction. The error bars indicate the standard deviations of the values. The materials have been divided into three classes and similar materials are discussed together. Section 5.3.2 discusses filamentised materials, materials with 0 % of fibres within bundles. Section 5.3.3 discusses bundled materials, materials with 100 % of fibres within bundles. Mixed materials, materials with 25 %, 50 % and 75 % of fibres within bundles, are discussed in Section 5.3.4.

5.3.2 Filamentised Materials

Two types of material were manufactured containing only filamentised fibres, based on 5 mm and 10 mm long fibres. The predictions and experimental results for 5 mm / 0 % materials are shown in Figures 5.9 and 5.10 and those for 10 mm / 0 % materials are shown in Figures 5.11 and 5.12. The model for these materials consists of only two contributions, fibres and matrix. A result of this is that the series / parallel model reduces to the form of the parallel model so two of the three predictions are identical. A comparison of the predictions using the Cox and Nairn models shows that there is only a slight difference between them. This is reasonable following Figure 5.2, since the reinforcement in these materials has a high aspect ratio.

The experimental results for the 5 mm / 0 % materials show good agreement with the predictions from the parallel model, with no significant difference between the 2 mm and 1 mm thick laminates. Although one of the 1 mm thick laminates has a lower modulus, this is not significantly different from other materials of this type. Similarly the experimental result for the 10 mm / 0 % material shows reasonable agreement with the parallel predictions. The predictions appear to be an underestimate but are within the standard deviations of the laminate properties.
For materials containing only filamentised fibres, Equation 5.4 reduces to Equation 5.1. This expression has been widely used to predict successfully the modulus of short-fibre composites. It is therefore reasonable to expect that predictions for these materials would be accurate and it is reassuring that this is the case.

5.3.3 Bundled Materials

The predictions and experimental results for the materials containing only fibre bundles are shown in Figures 5.13 to 5.20. There are four materials of this type, 5 mm / 100 % 1k, 5 mm / 100 % 6k, 10 mm / 100 % 1k and 10 mm / 100 % 6k. As with the filamentised materials this model contains only two elements, bundles and matrix, so the series / parallel model is identical to the parallel model. For these materials it is appropriate to regard the parallel and series models as upper and lower bounds respectively. For all these materials the experimental results lie between these two bounds.

It can be seen from Figures 5.13 to 5.20 that the predictions from the parallel model using the Nairn model are higher than the predictions using the Cox model. This is reasonable following Section 5.2.3 which showed that the two shear-lag models give different predictions for a short fibre composite containing fibres of a comparable aspect ratio to the bundles. There is little difference between the series model predictions using the two shear-lag models because these predictions are dominated by the matrix. The result of this is that the upper and lower bounds using the Cox model are closer than those using the Nairn model. Since all the experimental results lie within these bounds it appears that the Cox model based predictions are more appropriate for these materials.

Experimental results for the 2 mm thick laminates of 5 mm / 100 % 6k material (Figures 5.15 and 5.16) appear to show a stronger dependence on volume fraction than predicted by the models. This may be due to the reduction in the area of unreinforced resin in the laminate resulting from the increase in the number of reinforcing units. The models presented here account for an effect of an increase in
volume fraction but not an effect of the decrease in area of unreinforced resin. This effect may be accounted for by a change in the combination of the elements; with few bundle, the series model is appropriate (Figure 5.8), but with many bundles the parallel model is appropriate (Figure 5.6). Indeed, in Figure 5.15 the laminate with a lower volume fraction shows good agreement with the series model while, laminates with higher volume fractions show good agreement with the parallel model.

5.3.4 Mixed Materials

The predictions and experimental results for the materials containing both filamentised fibres and fibre bundles are shown in Figures 5.21 to 5.32. For these materials there are three elements to the model so the three combinations each lead to a unique prediction. Many of the observations made earlier about the bundled materials are relevant to the mixed materials. As with the bundled materials all the experimental results for the mixed materials lie between the predictions from the parallel model and the predictions from the series model. The predictions from the series / parallel model will always lie between these two. It is possible, with reasonable confidence, to suggest that the structure of a composite material may be described by the parallel model or by the series model. This is more difficult for the series / parallel model since it represents one specific intermediate case in a range of structures that change gradually from the parallel model to the series model. However the series / parallel model does provide a useful point of reference between these upper and lower bounds.

As with the bundled materials, the predictions using the Cox model produce narrower bounds around the experimental results, so these have been taken as the better predictions and the following observations concern predictions using the Cox model. An overview of the results presented here show that for the 2 mm thick laminates the experimental results for the 25 % of fibres within bundles material (Figure 5.21) lie between the predictions from the parallel model and the predictions from the series / parallel model. Experimental results for the 50 % of fibres within bundles materials (Figures 5.23, 5.25, 5.29 and 5.31) tend to lie around the predictions from the series / parallel model while experimental results for the 75 % of
fibres within bundles material (Figure 5.27) tend to lie between the predictions from the series/parallel model and the predictions from the series model. The experimental results for the 1 mm thick laminates are closer to the series model than the corresponding results of the 2 mm thick laminates. This is consistent with the structures suggested for the models in Section 5.2.4.

5.3.5 Comparison of Predictions using Shear-Lag Models

Predictions in this Chapter have been calculated using the Cox (1952) and Nairn (1997) shear-lag models. Figures 5.2 to 5.4 illustrate that the shear-lag models show good agreement for reinforcement with a high aspect ratio, but differ for low aspect ratio reinforcement. In terms of the materials considered here, the two models predict similar reinforcing efficiencies for filamentised fibres, but predict significantly different reinforcing efficiencies for the fibre bundles. The effect of this can be seen in Figures 5.9 to 5.32. For materials containing only filamentised fibres, predictions using the Cox and Nairn models are virtually identical, whereas, predictions for materials containing bundles are substantially different. The predictions using the Nairn model are consistently higher than the predictions using the Cox model. Hence, the calculated reinforcing efficiencies of the bundles are higher from the Nairn model than the Cox model.

A comparison of predictions with experimental data shows that in no case does the parallel model, using either version of the shear-lag analysis, underestimate the modulus of a material containing bundles. For the series/parallel model the predictions using the Nairn model overestimate the moduli, but predictions using the Cox model give reasonable agreement for some materials. This suggests that the Cox model is more appropriate for these materials.

However, there are factors that are not taken into account in the models used to predict the modulus of the materials which may have an effect. The bundle is considered to be a large fibre and inhomogeneity of the bundles is not taken into account. The transfer of stress to fibres within the bundle could be very inefficient so the effective modulus of the bundle may be less than the value used. This could
possibly make a greater difference to the more sophisticated Nairn model. Alternatively, the modulus of the materials could be reduced by factors unconnected with the fibre or bundle properties. In this Chapter possible effects have been suggested that relate to the distribution of reinforcement and the thickness of the laminates. It is possible that these effects are present to varying degrees in all the materials containing fibre bundles. In which case, a more sophisticated model would be required to predict the modulus of each individual type of material.

5.4 Modelling the Strength of a Material Containing Bundles

The modelling of the strength of a material containing bundles presents a number of additional challenges compared to the modelling of the modulus. The model for the modulus was developed by adapting a satisfactory model for the modulus of random short-fibre composites to a material containing bundles. However, this approach is not possible for modelling the strength because there is no entirely satisfactory model for the strength of a random short-fibre composite. A further complication is that many models for strength require the critical length of the fibre, which it has not been possible to evaluate in this study (as described in Chapter 7). Despite these difficulties it is relevant to discuss aspects of the strength of a material containing bundles.

Modelling the strength of a material requires the identification of the weakest point in the material. For a material containing bundles there are two features that have been identified as likely weaknesses in the material: a bundle oriented perpendicular to the load, and the end of a bundle oriented parallel to the load. A bundle oriented perpendicular to the load can be considered as a unidirectional laminate so, the transverse strength of a unidirectional laminate is a relevant quantity for this failure criterion. As described in Chapter 2, the transverse strength of a unidirectional laminate is influenced by many factors, in particular the properties of the fibre-matrix interface. Additionally, there is the possibility of fibres touching within the bundles and strain magnification within the bundles, both of which would further reduce the strength of the bundle. The failure of short-fibre composites is often initiated at the end of fibres aligned parallel to the applied load. At the end of a bundle there are a
large number of fibre ends in close proximity. It is quite possible that cracks at fibre ends could combine to form a large flaw in the material which could initiate failure.

While both the features mentioned above are weaknesses of the material, it is likely that one will fail at a lower stress than the other. In both cases, the strength of the fibre-matrix interface is critical, either at the end of a fibre or within a bundle. However, it is likely that the transverse bundle would fail at a lower stress because of the possibility of touching fibres or incomplete wetting acting to reduce the strength of the interface.

In addition to identifying the features that initiate failure within a material it is necessary to establish the stress at which failure occurs, for instance by testing a material containing a single bundle. It is likely that it would be necessary to determine this information for a specific material since it may depend on a number of factors including fibre and matrix properties, length of bundle, orientation of bundle relative to load, number of fibres per bundle and shape of bundle.

This discussion has focused on the initiation of failure in the materials. The mechanisms of crack propagation are considered in more detail in Chapter 7.

5.5 Conclusions
A model for the modulus of a short-fibre composite containing bundles has been presented. This model uses a modified rule-of-mixtures to combine the contributions from the fibres, fibre bundles and matrix. Three combinations of the contributions were identified as analogous to the short-fibre composites manufactured in this study.

Predictions were calculated using the Cox (1952) and Nairn (1997) shear-lag models. Comparison of both sets of predictions with experimental results suggest that the Cox model is more appropriate in this case. A comparison of the predictions using the Cox model and the experimental results illustrates the difficulty of defining the effect of fibre-bundling on the structure of a short fibre composite. It is possible to
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suggest extreme examples to which the structure may correspond, and from these produce upper and lower bounds.

The modelling of the strength of a material containing bundles has been discussed. Further characterisation of the materials would be required to develop a successful model for strength. In particular, a knowledge of the effect of a single bundle on the strength of a material.
Table 5.1. Variables used in the model for the modulus.

<table>
<thead>
<tr>
<th>Variable</th>
<th>Symbol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume fraction of fibres outside bundles</td>
<td>$V_{fo}$</td>
</tr>
<tr>
<td>Volume fraction of fibres within bundles</td>
<td>$V_{fb}$</td>
</tr>
<tr>
<td>Volume fraction of matrix outside bundles</td>
<td>$V_{nuo}$</td>
</tr>
<tr>
<td>Volume fraction of matrix within bundles</td>
<td>$V_{nb}$</td>
</tr>
<tr>
<td>Length efficiency factor for fibres</td>
<td>$\eta_{fo}$</td>
</tr>
<tr>
<td>Length efficiency factor for bundles</td>
<td>$\eta_{lb}$</td>
</tr>
</tbody>
</table>

Table 5.2. Material properties used in the model for the modulus.

<table>
<thead>
<tr>
<th>Material Property</th>
<th>Symbol</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Orientation factor</td>
<td>$\eta_{o}$</td>
<td>0.375</td>
</tr>
<tr>
<td>Radius of fibre</td>
<td>$r_f$</td>
<td>3.95 $\mu$m</td>
</tr>
<tr>
<td>Tensile modulus of fibre</td>
<td>$E_f$</td>
<td>230 GPa</td>
</tr>
<tr>
<td>Shear modulus of fibre</td>
<td>$G_f$</td>
<td>18 GPa</td>
</tr>
<tr>
<td>Radius of 1k bundle</td>
<td>$r_b$</td>
<td>161 $\mu$m</td>
</tr>
<tr>
<td>Radius of 6k bundle</td>
<td>$r_b$</td>
<td>395 $\mu$m</td>
</tr>
<tr>
<td>Tensile modulus of bundle</td>
<td>$E_b$</td>
<td>139 GPa</td>
</tr>
<tr>
<td>Shear modulus of bundle</td>
<td>$G_b$</td>
<td>5 GPa</td>
</tr>
<tr>
<td>Tensile modulus of matrix</td>
<td>$E_m$</td>
<td>1.56 GPa</td>
</tr>
<tr>
<td>Shear modulus of matrix</td>
<td>$G_m$</td>
<td>0.60 GPa</td>
</tr>
</tbody>
</table>

Table 5.3. Property values used in Figures 5.2, 5.3 and 5.4.

<table>
<thead>
<tr>
<th>Property</th>
<th>Symbol</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Orientation factor</td>
<td>$\eta_{o}$</td>
<td>0.375</td>
</tr>
<tr>
<td>Radius of fibre</td>
<td>$r_f$</td>
<td>3.95 $\mu$m</td>
</tr>
<tr>
<td>Volume fraction</td>
<td>$V_f$</td>
<td>0.15</td>
</tr>
<tr>
<td>Tensile modulus of matrix</td>
<td>$E_m$</td>
<td>1.56 GPa</td>
</tr>
<tr>
<td>Shear modulus of matrix</td>
<td>$G_m$</td>
<td>0.60 GPa</td>
</tr>
</tbody>
</table>
Figure 5.1. Diagram illustrating the four volume fraction terms used in the model for the modulus.
Figure 5.2. Graph showing predictions from Cox and Nairn models for a short-fibre composite containing fibres with an aspect ratio of 1265.

Figure 5.3. Graph showing predictions from Cox and Nairn models for a short-fibre composite containing fibres with an aspect ratio of 31.06.

Figure 5.4. Graph showing predictions from Cox and Nairn models for a short-fibre composite containing fibres with an aspect ratio of 6.329.
Figure 5.5. Diagram showing the eight possible combinations of three contributions. Combinations I, IV and VIII are used for predictions.
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<table>
<thead>
<tr>
<th>Fibres</th>
<th>Matrix</th>
</tr>
</thead>
</table>

Figure 5.6. Diagram showing an example of a composite material represented by elements in parallel.

<table>
<thead>
<tr>
<th>Fibres</th>
<th>Bundles</th>
<th>Matrix</th>
</tr>
</thead>
</table>

Figure 5.7. Diagram showing an example of a composite material represented by elements in series and parallel.

<table>
<thead>
<tr>
<th>Bundles</th>
<th>Matrix</th>
</tr>
</thead>
</table>

Figure 5.8. Diagram showing an example of a composite material represented by elements in series.
Figure 5.9. Graph showing experimental modulus results and predictions from three models using the Cox analysis for 5 mm / 0% materials.

Figure 5.10. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 5 mm / 0% materials.
Figure 5.11. Graph showing experimental modulus results and predictions from three models using the Cox analysis for 10 mm / 0 % materials.

Figure 5.12. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 10 mm / 0 % materials.
Figure 5.13. Graph showing experimental modulus results and predictions from three models using the Cox analysis for 5 mm / 100 % 1k materials.

Figure 5.14. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 5 mm / 100 % 1k materials.
Figure 5.15. Graph showing experimental modulus results and predictions from three models using the Cox analysis for 5 mm / 100 % 6k materials.

Figure 5.16. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 5 mm / 100 % 6k materials.
Figure 5.17. Graph showing experimental modulus results and predictions from three models using the Cox analysis for 10 mm / 100 % 1k materials.

Figure 5.18. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 10 mm / 100 % 1k materials.
Figure 5.19. Graph showing experimental modulus results and predictions from three models using the Cox analysis for 10 mm / 100 % 6k materials.

Figure 5.20. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 10 mm / 100 % 6k materials.
Figure 5.21. Graph showing experimental modulus results and predictions from three models using the Cox analysis for 5 mm / 25 % 6k materials.

Figure 5.22. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 5 mm / 25 % 6k materials.
Figure 5.23. Graph showing experimental modulus results and predictions from three models using the Cox analysis for 5 mm / 50 % 1k materials.

Figure 5.24. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 5 mm / 50 % 1k materials.
Figure 5.25. Graph showing experimental modulus results and predictions from three models using the Cox analysis for 5 mm / 50 % 6k materials.

Figure 5.26. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 5 mm / 50 % 6k materials.
Figure 5.27. Graph showing experimental modulus results and predictions from three models using the Cox analysis for 5 mm / 75 % 6k materials.

Figure 5.28. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 5 mm / 75 % 6k.
Figure 5.29. Graph showing experimental modulus results and predictions from three models using the Cox analysis for 10 mm / 50 % 1k materials.

Figure 5.30. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 10 mm / 50 % 1k materials.
Figure 5.31 Graph showing experimental modulus results and predictions from three models using the Cox analysis for 10 mm / 50 % 6k materials.

Figure 5.32. Graph showing experimental modulus results and predictions from three models using the Nairn analysis for 10 mm / 50 % 6k materials.
Chapter 6. Toughness: Results and Discussion
6.1 Introduction

In this Chapter, toughness results for the short-fibre composites and unreinforced polypropylene are presented and discussed. Initially, the critical strain energy release rate $G_c$ was determined for a range of short-fibre composites with 0 %, 25 %, 50 %, 75 % and 100 % of fibres within bundles. However, it was found that this approach, which is based on linear elastic fracture mechanics, was not valid for these short-fibre composites. Subsequently, the critical $J$ integral $J_c$, which is based on elastic-plastic fracture mechanics, was used to evaluate the toughness of unreinforced polypropylene and the short-fibre composites.

The $J_c$ approach was applied to unreinforced polypropylene using both a multiple specimen technique and a single specimen technique. $J_c$ results for short-fibre composites with 0 %, 50 %, and 100 % of fibres within bundles from the single specimen technique are reported. Short-fibre composites with two alternative fibre lengths and two alternative bundle diameters were studied. The same short-fibre composites and unreinforced polypropylene were impact tested using a miniature Charpy tester to provide information on the high strain rate toughness. It has not been possible to normalise toughness results for volume fraction of fibre as the effect of volume fraction has not been established.

6.2 Critical Strain Energy Release Rate Testing

6.2.1 Load-Extension Curves

Critical strain energy release rate $G_c$ was determined for 5 mm / 6k materials, with 0 %, 25 %, 50 %, 75 % and 100 % of fibres within bundles, using single-edge notched coupons. Coupons were 20 mm wide and 2 mm thick and the initial crack length to width ratio $a/W$ was 0.2, 0.4 and 0.6. Two coupons were tested for each value of $a/W$. Load-extension curves for the 0 %, 25 % and 100 % of fibres within bundles materials are shown in Figures 6.1 to 6.9. Dashed lines on the plots indicate regions where no points were recorded and consequently the curve has been interpolated.

Load extension curves for coupons of the 5 mm / 0 % material with an initial $a/W$ of 0.2, 0.4 and 0.6 are shown in Figures 6.1 to 6.3. As would be expected, an increase in initial
a/W results in a decrease in the maximum load. It can be seen from Figures 6.1 to 6.3 that the initial a/W also influences the shape of the load-extension curve. The load-extension curves are all initially linear but differences become apparent as the load approaches a maximum. Curves for coupons with an initial a/W of 0.2 are linear until very close to the maximum load. Curves for coupons with an initial a/W of 0.4 exhibit significant non-linearity before the maximum load and the load remains relatively high after the maximum has been passed. Curves for coupons with an initial a/W of 0.6 exhibit an even more pronounced plateau around the maximum load.

Figures 6.4 to 6.6 show load-extension curves for coupons of the 5 mm/25 % 6k material. These curves are very different to those for the material with 0 % of fibres within bundles at a given value of a/W. The maximum load for the 25 % of fibres within bundles material is approximately half of the corresponding value for the 0 % of fibres within bundles material. In addition, the shape of the curves for the 25 % of fibres within bundles material is more rounded than the curves for the 0 % of fibres within bundles material.

Load-extension curves for the 100 % of fibres within bundles material are shown in Figures 6.7 to 6.9. The load-extension curves for the 25 %, 50 %, 75 % and 100 % of fibres within bundles materials at any given a/W are similar. The maximum load values show a slight decrease as the proportion of fibres within bundles increases. Other minor differences are the shape of the curves, which become increasingly rounded, and the extension before failure, which increases with the proportion of fibres within bundles. This may be due to larger areas of unreinforced resin between bundles for the materials with a greater proportion of fibres within bundles.

6.2.2 Critical Strain Energy Release Rate

The method used to determine \( G_c \) is described in Chapter 3. The value of \( G_c \) was calculated from the expression:

\[
G_c = \frac{P^2}{2B} \frac{dC}{da}
\]  

(6.1)
where $P_c$ is the load at crack extension, $B$ is the specimen thickness and $dC/da$ is the change in compliance with crack length.

The method used to determine the compliance of coupons was described in Chapter 3. The compliance of two coupons was determined for each material, for crack lengths of 0 to 12 mm in 1 mm increments. The compliance values for two coupons of the 0 %, 25 % and 100 % of fibres within bundles materials (the materials for which load-extension curves are shown) are shown in Figures 6.10, 6.11 and 6.12. These Figures also show the curve fitted to the average compliance value at each crack length. The value of $dC/da$ was calculated by differentiating the fitted curve and is shown for the 0 %, 25 % and 100 % of fibres within bundles materials in Figure 6.13. It can be seen that the curves for the 25 % and 100 % of fibres within bundles materials are similar. In fact, the curves for all the materials containing bundles (materials with 25 %, 50 %, 75 % and 100 % of fibres within bundles) were similar and significantly higher than the curve for the material with 0 % of fibres within bundles.

The values of $G_c$ for the 5 mm / 6k materials are shown in Figure 6.14. Results for each initial $a/W$ are presented separately. Although there is some variation between $G_c$ values determined at different values of $a/W$ the trends are similar. There appears to be a significant decrease in $G_c$ between the 0 % and 25 % of fibres within bundles materials. Values of $G_c$ for materials with 25 %, 50 %, 75 % and 100 % of fibres within bundles are similar.

The value of $G_c$ is dominated by the maximum load. The values of $P_c$ for the 0 % of fibres within bundles material, at a given crack length, is around twice that for the other materials so the value of $P_c^2$ will be four times as great. The greater values of $dC/da$ for the 25 %, 50 %, 75 % and 100 % are not sufficient to offset the reduced values of the $P_c^2$ compared to the 0 % of fibres within bundles material. The values of $G_c$ are similar for the 25 %, 50 %, 75 % and 100 % of fibres within bundles materials because the values of both $P_c$ and $dC/da$ are both similar for these materials.
The critical strain energy release rate is based on linear elastic fracture mechanics. An assumption of this approach is that energy is only absorbed very locally at the crack tip. For the values of \( G_c \) to be valid this assumption must be reasonable, this is discussed in Section 6.3.

### 6.3 Validity of Toughness Results

#### 6.3.1 Validity of Critical Strain Energy Release Rate

The analysis used to evaluate the critical strain energy release rate \( G_c \) of the short-fibre composites is based on linear elastic fracture mechanics. For linear elastic fracture mechanics to be applied to a specific material and geometry it is necessary for the dimensions of the specimen to satisfy the condition expressed in ASTM D 5045 (1996):

\[
a, B, (W - a) \geq 2.5 \left( \frac{K_c}{\sigma_y} \right)^2
\]  

(6.2)

where \( a \) is the crack length, \( B \) is the thickness of the specimen, \( W \) is the width of the specimen, \( K_c \) is the fracture toughness of the material and \( \sigma_y \) is the yield stress of the material. It is only if this condition is satisfied that valid values of \( G_c \) can be calculated. Although the method employed by ASTM D 5045 (1996) differs from the method used here in a number of ways, the requirement for sample dimensions is based on ensuring plane strain conditions in the specimen. This condition for the specimen dimensions is the same irrespective of the details of the test. A disadvantage of this criterion is that it can only be applied after the value of \( G_c \) has been determined.

It was possible to apply this criterion to the \( G_c \) results presented in Section 6.2, since for plane strain conditions and a homogenous isotropic material:

\[
K_c = \frac{\sqrt{EG_c}}{(1 - v^2)}
\]  

(6.3)
where $K_c$ is the fracture toughness, $E$ is the modulus of the material and $\nu$ is the Poisson’s ratio of the material. It was possible to evaluate the minimum specimen dimensions required for valid $G_c$ determination for the 5 mm / 6k materials with 0 %, 25 %, 50 %, 75 % and 100 % of fibres within bundles. The value of $K_c$ was calculated from an average value of the modulus taken from the results presented in Chapter 4 and values of $G_c$ for each value of initial $a/W$ presented in Section 6.1 of the present Chapter. Strictly a knowledge of the Poisson’s ratio is required. However, in this case, where the calculation was based on an estimate of the plastic zone size, it was sufficient to take the term $(1-\nu^2)$ as unity. ASTM D 5045 (1996) specifies that the yield stress should be taken as the fracture stress of the material, so the strength results presented in Chapter 4 have been used.

The resulting minimum specimen dimensions for valid $G_c$ determination are shown in Table 6.1. The coupons used for $G_c$ determination were 20 mm wide and 2 mm thick with an initial $a/W$ of 0.2, 0.4 and 0.6. It can be seen that the size of specimen required to produce valid $G_c$ values is much greater than the specimens used for the $G_c$ determination. These requirements are greater than could be produced and tested with the techniques employed in this work.

A further condition, which is specified in ASTM D 5045 (1996), is to ensure that the shape of the load-extension curves indicates linear elastic behaviour of the material. This condition is that the maximum load on the load-extension curve is within 10 % of the load at the intercept of the 5 % secant with the load-extension curve. Examination of the load-extension curves for these materials (Figures 6.1 to 6.9) shows that many of these materials are not close to satisfying this condition. In fact this condition was only met by two of the samples of the thirty six tested. These were the two coupons of the 0 % of fibres within bundles material with an initial $a/W$ of 0.2.

Clearly it is not possible to produce valid results with a linear elastic fracture mechanics based analysis for these materials. Therefore an elastic-plastic fracture mechanics based analysis, the critical J integral, was applied.
6.3.2 Validity of Critical J Integral

The critical J integral $J_c$ is based on elastic-plastic fracture mechanics and hence the specimen size requirement is less rigorous than that for $G_c$. The current ASTM standard for $J_c$ testing (ASTM E 1737, 1996) requires the specimen dimensions to satisfy the criterion:

$$B_c(W - a) \geq 25 \left( \frac{J_c}{\sigma_y} \right)$$

(6.4)

for the thickness of the specimen $B$ and the length of the uncracked ligament $W-a$, where $J_c$ is the critical J integral and $\sigma_y$ is the yield stress of the material. It can be seen that Equation 6.4 has a similar form to Equation 6.2.

It was possible to estimate minimum size requirements for the materials studied in Section 6.2, since for elastic conditions:

$$J_c = G_c.$$  \hspace{1cm} (6.5)

Although the behaviour of these materials was clearly not always elastic it was reasonable to use this expression to provide an approximate value of $J_c$ for the purpose of calculating minimum specimen dimensions. The same value of the yield stress was taken as for the calculation of the minimum specimen dimensions for $G_c$ testing.

The minimum specimen dimensions calculated for valid $J_c$ determination are shown in Table 6.2. Although in most cases these requirements are still greater than 2 mm, the specimen thickness, they are only slightly greater and work by Hashemi and Williams (1986) suggests that the ASTM size requirement may be too conservative. While it is not possible to be certain that results for $J_c$ from a 2 mm thick specimen are valid, there is no doubt that $J_c$ testing is preferable to $G_c$ testing for these materials.
6.4 Critical J Integral Testing of Polypropylene

6.4.1 Load-Extension Curves

Coupons with a width of 20 mm were cut from a 2 mm thick plaque of polypropylene. The coupons contained double-edge notches with individual notch lengths of 4 mm, 6 mm or 8 mm, as illustrated in Chapter 3, so that the initial values of $a/W$ were 0.4, 0.6 or 0.8. These values of $a/W$ are greater than those for $G_c$ measurements because it is a requirement of the single specimen J integral analysis that coupons are deeply notched. This requirement is necessary to ensure that plasticity is confined to the region between the two notches. Two coupons were tested with each initial notch length.

Load-extension curves for coupons with initial $a/W$ values of 0.4, 0.6 and 0.8 are shown in Figures 6.15 to 6.17. The same scale has been used for load-extension curves of all double-edge notch coupons of both the unreinforced polypropylene and the short-fibre composites, to allow direct comparison between curves. Dashed lines on the plots indicate regions where no points were recorded and the curve has been interpolated. It can be seen that all the load-extension curves become non-linear at small extensions, indicating a large amount of plastic deformation as would be expected for an unreinforced thermoplastic.

6.4.2 Multiple Specimen Critical J Integral

The multiple specimen analysis for the critical J integral $J_c$ is based directly on the equation of Rice (1968):

$$J = - \frac{\Delta U}{\Delta a} \tag{6.6}$$

where $\Delta U$ is the change in potential energy and $\Delta a$ is the change in crack extension. The J integral may be evaluated from the load-extension curves from two coupons with different initial notch lengths, as described in Chapter 3.

The multiple specimen analysis was applied to pairs of coupons with an initial $a/W$ of 0.4 and 0.6 giving a $J_c$ value at an $a/W$ of 0.5, and to pairs of coupons with an initial
$a/W$ of 0.6 and 0.8 giving a $J_c$ value at an $a/W$ of 0.7. Since there are two curves for each initial $a/W$ this analysis produces four values of $J_c$ for each $a/W$.

The results of the multiple specimen analysis are shown in Figure 6.18. It can be seen that the value of $J_c$ decreases with increasing initial notch length. Since the size of the uncracked ligament $W-a$ is greater with a smaller value of the initial $a/W$ this is more likely to produce valid $J_c$ results. Williams (1984) reports that $J_c$ values of polypropylene are approximately 10 kJ m$^{-2}$ and this value shows good agreement with the value of $J_c$ at an $a/W$ of 0.5.

6.4.3 Single Specimen Critical J Integral

The single specimen J integral analysis of Rice et al. (1973) may be applied to the load-extension curves for polypropylene. The value of $J_c$ is the sum of the critical J integral due to elastic displacement $J_{el}$ and the J integral due to plastic displacement $J_{pl}$, hence:

$$J_c = J_{el} + J_{pl}$$

(6.7)

The method for calculating $J_{el}$ and $J_{pl}$ is presented in Chapter 3. This analysis for $J_c$ gives one value of $J_c$ for each coupon, so there are two values of $J_c$ at each $a/W$.

The variation of compliance with crack length was found for two coupons, the variation of $dC/da$ was determined using the same method used in Section 6.2. The resulting variation of $dC/da$ with crack length, used to calculate $J_{el}$, is shown in Figure 6.19. Results for $J_c$ from the single specimen analysis are shown in Figure 6.18. The value of $J_c$ decreases with increasing $a/W$ as with the values of $J_c$ from the multiple specimen technique. The values at lower $a/W$ from the single specimen also agree with the value of approximately 10 kJ m$^{-2}$ reported by Williams (1984). Minimum specimen dimensions for the valid determination of $J_c$ for unreinforced polypropylene were calculated from the $J_c$ values and are shown in Table 6.3. Clearly, the samples used were too small to produce valid values which accounts for the notch size dependence of the $J_c$ values. The minimum specimen size requirements are the same for the multiple and single specimen techniques. This is supported by the fact that both the multiple
specimen and single specimen analyses give comparable values of $J_c$ for polypropylene at a given value of $a/W$.

6.5 Critical J Integral Testing of Short-Fibre Composites

6.5.1 Testing of Short-Fibre Composites
Due to the inhomogeneity of the short-fibre composites, particularly those containing fibre bundles, a large number of specimens are required to produce a reliable load-extension curve. Hence, it is more appropriate to use the single specimen analysis for the short-fibre composites manufactured in this work. The results presented in Figure 6.18 suggest that both the multiple specimen and single specimen analyses will give similar values of $J_c$ for a polypropylene matrix composite material.

Short-fibre composites with 0%, 50% and 100% of fibres in bundles were tested as coupons with a double-edge notch geometry. These short-fibre composites were manufactured with different fibre lengths (5 mm and 10 mm) and different bundles sizes (1000 fibres per bundle and 6000 fibres per bundle). In total ten types of material were studied, namely: 5 mm/0%, 10 mm/0%, 5 mm/50% 1k, 5 mm/50% 6k, 10 mm/50% 1k, 10 mm/50% 6k, 5 mm/100% 1k, 5 mm/100% 6k, 10 mm/100% 1k and 10 mm/100% 6k. Six coupons of each material were tested, two each with an initial $a/W$ of 0.4, 0.6 and 0.8. Coupons were 20 mm wide and 2 mm thick.

6.5.2 Load-Extension Curves
Load-extension curves for coupons of the short-fibre composites are shown in Figures 6.20 to 6.49. Dashed lines on the plots indicate regions where no points were recorded and the curve has been interpolated.

Two filamentised materials were tested, 5 mm/0% and 10 mm/0%. Load-extension curves for the 5 mm/0% with an initial $a/W$ of 0.4, 0.6 and 0.8 are shown in Figures 6.20 to 6.22. Similarly, load-extension curves for the 10 mm/0% are shown in Figures 6.23 to 6.25. The maximum load for the material with 10 mm fibres is greater than that for the material with 5 mm fibres. Both load-extension curves are similar in shape. The
curves are reasonably linear up to the maximum load and the load falls quickly after the maximum.

Load-extension curves for the bundled materials are shown in Figures 6.26 to 6.37. Four types of bundled material were tested, namely 5 mm / 100 % 1k, 5 mm / 100 % 6k, 10 mm / 100 % 1k and 10 mm / 100 % 6k. The shape of the load-extension curves for the bundled materials are all considerably less linear than those for the filamentised materials. Curves for the bundled materials with 1000 fibres per bundle are more linear than those for the bundled materials with 6000 fibres per bundle. An increase in the non-linearity of the curve indicates more plastic deformation before the maximum load. The maximum load is less for the materials with 6000 fibres per bundle than for the materials with 1000 fibres per bundle and the maximum load for the 10 mm / 100 % 1k is significantly higher than for the other bundled materials. All the maximum loads are lower than those for the filamentised materials. However, the bundled materials support a higher load at larger extensions than the filamentised materials.

Modulus results for the bundled materials with 6000 fibres per bundle (5 mm / 100 % 6k and 10 mm / 100 % 6k) presented in Chapter 4 suggested that the 6000 fibre bundle has no reinforcing effect. A comparison of the load-extension curves for the 5 mm / 100 % 6k (Figures 6.29 to 6.31) and 10 mm / 100 % 6k (Figures 6.35 to 6.37) with those for the polypropylene (Figures 6.15 to 6.17) reveals that the curves are similar in appearance. It appears that the mechanical behaviour of these materials is dominated by the matrix. However, the composite materials show a greater extension before failure than the polypropylene. This suggests that although they are not effective reinforcement they do seem to inhibit crack growth leading to final failure in the short-fibre composites.

Four types of short-fibre composite which contained both individual fibres and fibre bundles were tested. These materials were 5 mm / 50 % 1k, 5 mm / 50 % 6k, 10 mm / 50 % 1k and 10 mm / 50 % 6k. A comparison of the load-extension curves for these materials (Figures 6.38 to 6.49) shows that they are very similar for all the materials, irrespective of fibre length and bundle size. There is some variation in the initial modulus but the maximum load and the shape of the curves are generally the same. In every case
the maximum load is low compared to the filamentised materials and there is limited plastic deformation before the maximum load. This may be because the presence of bundles limits the maximum load while the reinforcing effect of the individual fibres limits plastic deformation.

6.5.3 Elastic J Integral
The critical J integral \( J_e \) calculated here is the sum of the critical J integral due to elastic displacement \( J_{el} \) and the critical J integral due to plastic displacement \( J_{pl} \) (Equation 6.7). Results for the two components are presented separately as it is reasonable to expect that they are influenced by different properties of the material. \( J_{el} \) is equivalent to the elastic strain energy release rate \( G \) and the value of \( dC/da \) is required to calculate \( J_{el} \).

The variation of compliance with crack length was determined for two coupons of each material. The variation of \( dC/da \) was found using the same method used for the \( G_c \) determination in Section 6.2. The variation of \( dC/da \) for each range of materials is shown in Figures 6.50 to 6.53. A noticeable feature is that for each range of materials the curve for the 0 % of fibres within bundles material is the lowest. For the materials containing 5 mm long fibres, the curve for the 50 % of fibres within bundles material is highest, while for the materials containing 10 mm long fibres, the curve for the 100 % of fibres within bundles is highest.

The results of the \( J_{el} \) calculations are shown in Figures 6.54 to 6.57. These results show that the values of \( J_{el} \) for the 10 mm / 0 % material are slightly higher than those for the 5 mm / 0 % material which is a consequence of the higher maximum loads for the material with 10 mm long fibres. The values of \( J_{el} \) for the four 50 % of fibres within bundles materials are all very similar which is reasonable given the similarity of the load-extension curves. The \( J_{el} \) values for the 50 % of fibres within bundles materials are lower than the values for the 0 % of fibres within bundles materials which is a consequence of the lower maximum loads. The lower maximum loads are likely to be a consequence of the presence of fibre bundles.
The values of $J_{el}$ show the greatest variation for the 100 % of fibres within bundles materials. The value of $J_{el}$ is lowest for the 5 mm / 100 % 6k and is slightly higher for both the 5 mm / 100 % 1k and 10 mm / 100 % 6k materials, while the values for the 10 mm / 100 % 1k are significantly higher. The values of $dC/da$ for the 10 mm / 100 % 6k material are higher than for any other material, however, this is not reflected in a higher value of $J_{el}$ because the maximum load values are relatively low. Whereas the higher maximum load for the 10 mm / 100 % 1k material results in the highest values of $J_{el}$ because the square of the maximum load is used to calculate $J_{el}$. It appears that for the bundled materials the value of $J_{el}$ is related to the aspect ratio of the bundle.

6.5.4 Plastic J Integral

The results for the component of $J_e$ due to plastic displacement $J_{pl}$ are shown in Figures 6.58 to 6.61. As with $J_{el}$ the value of $J_{pl}$ is slightly higher for the 10 mm / 0 % material than the 5 mm / 0 % material. For the 50 % of fibres within bundles material there is some variation between materials at individual values of $a/W$, but overall there are no clear trends for the materials with 50 % of fibres within bundles.

As with $J_{el}$, the bundled materials show the greatest variation of $J_{pl}$. It can be seen that the value of $J_{pl}$ is much higher for the 5 mm / 100 % 6k and 10 mm / 100 % 6k materials than the 10 mm / 100 % 1k and 5 mm / 100 % 1k materials. In this case it appears that the diameter of the bundle is the most important factor. This could be a direct effect of the bundle diameter or an effect of the larger areas of unreinforced resin that are present within the 6000 fibres per bundle material. The second suggestion is supported by the similarity between the load-extension curves for these materials and those for the unreinforced polypropylene (as discussed in Section 6.5.2). There is no clear effect with fibre length, the value of $J_{pl}$ for the 5 mm / 100 % 1k material is slightly lower than that for the 10 mm / 100 % 1k material, while the value of $J_{pl}$ for the 5 mm / 100 % 6k material is slightly higher than that for the 10 mm / 100 % 6k material.
6.5.5 Critical J Integral

The values of $J_c$, the sum of $J_{el}$ and $J_{pl}$, are shown in Figures 6.62 to 6.65. For the 5 mm / 6k materials shown in Figure 6.62 the value of $J_c$ tends to decrease with an increasing proportion of fibres within bundles. The trends for the 5 mm / 6k, 10 mm / 1k and 10 mm / 6k materials (Figures 6.63 to 6.65) are all similar and differ from the trend for the 5 mm / 1k materials. For these ranges of material the value of $J_c$ for the 50 % of fibres within bundles material is lower than both the 0 % and 100 % of fibres within bundles materials. Additionally, for the 5 mm / 6k materials the value of $J_c$ for the 100 % of fibres within bundles material is higher than the 0 % of fibres within bundles material, while for the 10 mm / 1k and 10 mm / 6k materials the 0 % and 100 % of fibres within bundles materials have similar values of $J_c$.

For the 5 mm / 6k materials both $G_c$ and $J_c$ have now been determined and these results can be compared by considering Figures 6.14 and 6.63. The values of $G_c$ and $J_c$ are similar for the 0 % and 50 % of fibres within bundles materials. However, for the 100 % of fibres within bundles material the overall value of $J_c$ is higher than the value of $G_c$. This is likely to be a consequence of the more non-linear behaviour of the 100 % of fibres within bundles material indicating more plastic deformation. A linear elastic treatment neglects the energy absorbed through plastic deformation and will consequently underestimate the energy absorbed for a material with significant plastic deformation, whereas this is accounted for in the $J_c$ approach. Clearly, the inclusion of plastic deformation is important for the 100 % of fibres within bundles material.

To assess the validity of the $J_c$ results it is useful to consider the size requirements for the materials studied here. The strength was not determined for the full range of materials so it is only possible to calculate values for three of the short-fibre composites tested for $J_c$. The resulting minimum specimen dimensions are shown in Table 6.3. Although the size requirement is not met for all specimens, the use of $J_c$ is clearly preferable to the use of $G_c$. It can be seen that the dimensions for the 5 mm / 50 % 6k and 5 mm / 100 % 6k materials are greater than the values presented in Table 6.2 because $J_c$ is greater than $G_c$ for these materials. For the 5 mm / 0 %
Chapter 6. Toughness: Results and Discussion

materials the specimen dimensions are greater than those required but even so the results (shown in Figure 6.63) exhibit a notch length dependence. A consequence of this is that the absolute values of $J_c$ should be regarded with caution. However, the trends in $J_c$ values are of most relevance and are enhanced by normalising the values of $J_c$ for the effect of notch length, as discussed in the Section 6.5.6.

6.5.6 Normalised J Integral

The results for $J_{el}$, $J_{pl}$ and $J_c$ all show variation with the value of $a/W$. In this Section a normalisation procedure is proposed to account for the effect of $a/W$, using $J_c$ results from the single specimen analysis for polypropylene (shown in Figure 6.18). The quantity $J_{cc}:J_{em}$ was calculated where $J_{cc}$ is the value of $J_c$ for the composite material at a given value of $a/W$ and $J_{em}$ is the value of $J_c$ for the polypropylene at the same value of $a/W$.

The results of this normalisation are shown in Figures 6.66 to 6.69. It can be seen that this normalisation is successful in reducing, to a significant extent, the dependence on notch length. There is excellent agreement between different values of $a/W$ for the 5 mm / 6k materials (Figure 6.67). The agreement is very good for the 5 mm / 1k and 10 mm / 6k materials (Figures 6.66 and 6.69) and good for the 10 mm / 1k (Figure 6.68). The relative success of the normalisation is likely to reflect the degree to which the properties of the composite material are matrix dominated.

The value of $J_{cc}:J_{em}$ for the 10 mm / 0 % of fibres within bundles material is approximately unity indicating that the toughness of the composite material and the unreinforced polypropylene are similar. $J_{cc}:J_{em}$ is slightly lower for the 5 mm / 0 % material. The values of $J_{cc}:J_{em}$ for all the 50 % of fibres within bundles materials are similar and are significantly lower than unity. This shows that the presence of both fibre and fibre bundles in the composite material, at the relative proportions and overall volume fractions of the present study, significantly reduces the toughness below that of the unreinforced polypropylene.
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For the bundled materials the highest values of $J_{cc}:J_{cm}$ are for the 10 mm / 100 % 1k and 5 mm / 100 % 6k materials, while $J_{cc}:J_{cm}$ for the 10 mm / 100 % 6k materials is slightly lower. It is revealing to compare the contributions of $J_{el}$ and $J_{pl}$ for these materials. The value of $J_{el}$ for the 10 mm / 100 % 1k materials is greater than the other bundled materials, while for the 5 mm / 100 % 6k and 10 mm / 100 % 6k materials the value of $J_{pl}$ is greater than the other bundled materials. It can be seen that higher $J_{cc}:J_{cm}$ values can be achieved by an increase in either $J_{el}$ or $J_{pl}$, suggesting that they originate from different mechanisms. In the case of the 5 mm / 100 % 1k material it appears that neither of these mechanisms is effective since the $J_{cc}:J_{cm}$ value for 5 mm / 100 % 1k is comparable to that for 5 mm / 50 % 1k.

The only range of materials for which the values of $J_{cc}:J_{cm}$ are higher for the 100 % of fibres within bundles material than the 0 % of fibres within bundles material is the 5 mm / 6k materials. However, this is due to an increase in $J_{pl}$ which is likely to originate from the larger areas of unreinforced resin in this material. For the other ranges of material the values of $J_{cc}:J_{cm}$ for the 0 % and 100 % of fibres within bundles materials are comparable.

Failure mechanisms have been investigated in more detail by studying the fracture surfaces. These results are described in Chapter 7.

6.6 Impact Testing

A range of materials were impact tested to investigate fracture behaviour at a higher strain rate. Specimens were impact tested on a miniature Charpy tester which provided an estimate of the impact surface energy. Specimens were 5 mm wide and 2 mm thick. The 5 mm dimension was in the direction of impact and was notched to a depth of 0.5 mm. The short-fibre composites that were impact tested were the same range of materials as those used for the J integral determination. The materials were: 5 mm / 0 %, 10 mm / 0 %, 5 mm / 50 % 1k, 5 mm / 50 % 6k, 10 mm / 50 % 1k, 10 mm / 50 % 6k, 5 mm / 100 % 1k, 5 mm / 100 % 6k, 10 mm / 100 % 1k and 10 mm / 100 % 6k.

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These results are shown in Figures 6.70 to 6.73. For the 5 mm / 1k, 5 mm / 6k and 10 mm / 6k materials the impact surface energy of the 50 % and 100 % of fibres within bundles materials are substantially lower than that for the 0 % of fibres within bundles materials. The exception is the 10 mm / 1k materials, in this case the impact surface energy of the 50 % of fibres within bundles material is slightly lower than 0 % of fibres within bundles material, and the 100 % of fibres within bundles material is slightly higher than the other two materials. The most striking aspect of these results is their similarity to the results for $J_e$, shown in Figures 6.54 to 6.57. This suggests that the impact behaviour of the materials is dominated by the composite elastic response and hence the reinforcement (fibres and/or fibre bundles).

In addition, unreinforced polypropylene was impact tested. The impact surface energy of the unreinforced polypropylene was found to be 9.0 ± 2.0 kJ m$^{-2}$. This is lower than any of the composite materials tested and is a result of the high strain rate used in impact testing. The viscoelastic behaviour of polypropylene means that at high strain rates there is little plastic deformation. Since the plastic deformation requires most energy during the fracture of polypropylene it is reasonable that the impact surface energy of polypropylene is relatively low and that the impact behaviour of the short-fibre composites is dominated by the fibres and/or fibre bundles, rather than the matrix. These results are discussed in relation to the fracture surfaces in Chapter 7.

6.7 Conclusions

Various fracture mechanics parameters have been measured for the materials of the present study. Results have been obtained for both short-fibre composites and unreinforced polypropylene.

$G_c$ measurements were found to be invalid for these materials with the specimen size used. The specimen dimensions required to ensure plane strain conditions at the crack tip were far in excess of the size of the specimen used. However, the specimen
dimensions required for valid determination of $J_e$ were found to be similar to the coupon dimensions.

The value of $J_e$ for the materials with 50% of fibres within bundles was lower than the values for the materials with 0% of fibres within bundles. This may be because the presence of bundles limits the maximum load while the presence of individual fibre inhibits plastic deformation. Some of the 100% of fibres within bundles materials have relatively high values of $J_e$, resulting from increased contributions of either $J_{el}$ or $J_{pl}$ in different cases. This suggests that increases in $J_e$ can arise from different mechanisms. $J_{el}$ appears to be greater for materials containing bundles with a greater aspect ratio which are more effective reinforcement. On the other hand, $J_{pl}$ appears to be greater for materials containing bundles with a greater volume, meaning fewer bundles overall, which results in larger areas of unreinforced polypropylene between bundles. The $J_e$ results showed a notch dependence but it was possible to normalise for this using the results for unreinforced polypropylene. The results of the normalisation showed that only one range of materials showed a clear improvement in toughness with the use of fibre bundles, and this increase is likely to be due to the larger unreinforced areas of matrix in these material.

Impact testing results were similar in appearance to $J_{el}$ results. This is reasonable as plastic deformation is reduced at higher strain rates due to the viscoelasticity of polypropylene.
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#### Table 6.1. Minimum specimen dimensions for valid $G_c$ determination.

<table>
<thead>
<tr>
<th>Material</th>
<th>Minimum size of $a$, $B$ and $(W-a)$ in mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$a/W = 0.2$</td>
</tr>
<tr>
<td>5 mm / 0 %</td>
<td>13.2</td>
</tr>
<tr>
<td>5 mm / 25 % 6k</td>
<td>40.9</td>
</tr>
<tr>
<td>5 mm / 50 % 6k</td>
<td>25.3</td>
</tr>
<tr>
<td>5 mm / 75 % 6k</td>
<td>39.8</td>
</tr>
<tr>
<td>5 mm / 100 % 6k</td>
<td>15.2</td>
</tr>
</tbody>
</table>

#### Table 6.2. Minimum specimen dimensions for valid $J_c$ determination calculated from $G_c$.

<table>
<thead>
<tr>
<th>Material</th>
<th>Minimum size of $B$ and $(W-a)$ in mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$a/W = 0.2$</td>
</tr>
<tr>
<td>5 mm / 0 %</td>
<td>1.05</td>
</tr>
<tr>
<td>5 mm / 25 % 6k</td>
<td>2.43</td>
</tr>
<tr>
<td>5 mm / 50 % 6k</td>
<td>1.35</td>
</tr>
<tr>
<td>5 mm / 75 % 6k</td>
<td>2.30</td>
</tr>
<tr>
<td>5 mm / 100 % 6k</td>
<td>2.55</td>
</tr>
</tbody>
</table>

#### Table 6.3. Minimum specimen dimensions for valid $J_c$ determination calculated from $J_c$.

<table>
<thead>
<tr>
<th>Material</th>
<th>Minimum size of $B$ and $(W-a)$ in mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$a/W = 0.4$</td>
</tr>
<tr>
<td>5 mm / 0 %</td>
<td>1.69</td>
</tr>
<tr>
<td>5 mm / 50 % 6k</td>
<td>4.62</td>
</tr>
<tr>
<td>5 mm / 100 % 6k</td>
<td>10.97</td>
</tr>
<tr>
<td>Unreinforced polypropylene</td>
<td>11.78</td>
</tr>
</tbody>
</table>
Figure 6.1. Load extension curves for two single-edge notched coupons of the 5 mm / 0 % material with an initial $a/W$ of 0.2.

Figure 6.2. Load extension curves for two single-edge notched coupons of the 5 mm / 0 % material with an initial $a/W$ of 0.4.

Figure 6.3. Load extension curves for two single-edge notched coupons of the 5 mm / 0 % material with an initial $a/W$ of 0.6.
Figure 6.4. Load extension curves for two single-edge notched coupons of the 5 mm / 25 % 6k material with an initial $a/W$ of 0.2.

Figure 6.5. Load extension curves for two single-edge notched coupons of the 5 mm / 25 % 6k material with an initial $a/W$ of 0.4.

Figure 6.6. Load extension curves for two single-edge notched coupons of the 5 mm / 25 % 6k material with an initial $a/W$ of 0.6.
Figure 6.7. Load extension curves for two single-edge notched coupons of the 5 mm / 100 % 6k material with an initial $a/W$ of 0.2.

Figure 6.8. Load extension curves for two single-edge notched coupons of the 5 mm / 100 % 6k material with an initial $a/W$ of 0.4.

Figure 6.9. Load extension curves for two single-edge notched coupons of the 5 mm / 100 % 6k material with an initial $a/W$ of 0.6.
Figure 6.10. Graph showing variation of compliance with crack length and the fitted curve for the 0 % of fibres within bundles material tested for $G_c$.

Figure 6.11. Graph showing variation of compliance with crack length and the fitted curve for the 25 % of fibres within bundles material tested for $G_c$. 

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Figure 6.12. Graph showing variation of compliance with crack length and the fitted curve for the 100 % of fibres within bundles material tested for \( G_c \).

Figure 6.13. Graph showing variation of \( dC/da \) with crack length for the 0 %, 25 % and 100 % of fibres within bundles materials tested for \( G_c \).
Figure 6.14. Graph of $G_c$ against proportion of fibres within bundles for 5 mm / 6k materials.
Figure 6.15. Load extension curves for two double-edge notched coupons of polypropylene with an initial $a/W$ of 0.4.

Figure 6.16. Load extension curves for two double-edge notched coupons of polypropylene with an initial $a/W$ of 0.6.

Figure 6.17. Load extension curves for two double-edge notched coupons of polypropylene with an initial $a/W$ of 0.8.
Figure 6.18. Graph of $J_c$ against $a/W$ for unreinforced polypropylene. The values of $J_c$ have been determined by two techniques.

Figure 6.19. Graph showing variation of $dC/da$ with crack length used in the single specimen determination of $J_c$ for unreinforced polypropylene.
Figure 6.20. Load extension curves for two double-edge notched coupons of the 5 mm / 0 % material with an initial $a/W$ of 0.4.

Figure 6.21. Load extension curves for two double-edge notched coupons of the 5 mm / 0 % material with an initial $a/W$ of 0.6.

Figure 6.22. Load extension curves for two double-edge notched coupons of the 5 mm / 0 % material with an initial $a/W$ of 0.8.
Figure 6.23. Load extension curves for two double-edge notched coupons of the 10 mm / 0 % material with an initial $a/W$ of 0.4.

Figure 6.24. Load extension curves for two double-edge notched coupons of the 10 mm / 0 % material with an initial $a/W$ of 0.6.

Figure 6.25. Load extension curves for two double-edge notched coupons of the 10 mm / 0 % material with an initial $a/W$ of 0.8.
Figure 6.26. Load extension curves for two double-edge notched coupons of the 5 mm / 100 % 1k material with an initial $a/W$ of 0.4.

Figure 6.27. Load extension curves for two double-edge notched coupons of the 5 mm / 100 % 1k material with an initial $a/W$ of 0.6.

Figure 6.28. Load extension curves for two double-edge notched coupons of the 5 mm / 100 % 1k material with an initial $a/W$ of 0.8.
Figure 6.29. Load extension curves for two double-edge notched coupons of the 5 mm / 100 % 6k material with an initial $a/W$ of 0.4.

Figure 6.30. Load extension curves for two double-edge notched coupons of the 5 mm / 100 % 6k material with an initial $a/W$ of 0.6.

Figure 6.31. Load extension curves for two double-edge notched coupons of the 5 mm / 100 % 6k material with an initial $a/W$ of 0.8.
Figure 6.32. Load extension curves for two double-edge notched coupons of the 10 mm / 100 % 1k material with an initial $a/W$ of 0.4.

Figure 6.33. Load extension curves for two double-edge notched coupons of the 10 mm / 100 % 1k material with an initial $a/W$ of 0.6.

Figure 6.34. Load extension curves for two double-edge notched coupons of the 10 mm / 100 % 1k material with an initial $a/W$ of 0.8.
Figure 6.35. Load extension curves for two double-edge notched coupons of the 10 mm / 100 % 6k material with an initial $a/W$ of 0.4.

Figure 6.36. Load extension curves for two double-edge notched coupons of the 10 mm / 100 % 6k material with an initial $a/W$ of 0.6.

Figure 6.37. Load extension curves for two double-edge notched coupons of the 10 mm / 100 % 6k material with an initial $a/W$ of 0.8.
Figure 6.38. Load extension curves for two double-edge notched coupons of the 5 mm / 50 % 1k material with an initial $a/W$ of 0.4.

Figure 6.39. Load extension curves for two double-edge notched coupons of the 5 mm / 50 % 1k material with an initial $a/W$ of 0.6.

Figure 6.40. Load extension curves for two double-edge notched coupons of the 5 mm / 50 % 1k material with an initial $a/W$ of 0.8.
Figure 6.41. Load extension curves for two double-edge notched coupons of the 5 mm / 50 % 6k material with an initial $a/W$ of 0.4.

Figure 6.42. Load extension curves for two double-edge notched coupons of the 5 mm / 50 % 6k material with an initial $a/W$ of 0.6.

Figure 6.43. Load extension curves for two double-edge notched coupons of the 5 mm / 50 % 6k material with an initial $a/W$ of 0.8.
Figure 6.44. Load extension curves for two double-edge notched coupons of the 10 mm / 50 % 1k material with an initial \( a/W \) of 0.4.

Figure 6.45. Load extension curves for two double-edge notched coupons of the 10 mm / 50 % 1k material with an initial \( a/W \) of 0.6.

Figure 6.46. Load extension curves for two double-edge notched coupons of the 10 mm / 50 % 1k material with an initial \( a/W \) of 0.8.
Figure 6.47. Load extension curves for two double-edge notched coupons of the 10 mm / 50 % 6k material with an initial $a/W$ of 0.4.

Figure 6.48. Load extension curves for two double-edge notched coupons of the 10 mm / 50 % 6k material with an initial $a/W$ of 0.6.

Figure 6.49. Load extension curves for two double-edge notched coupons of the 10 mm / 50 % 6k material with an initial $a/W$ of 0.8.
Figure 6.50. Graph showing variation of $dC/da$ with crack length for the 5 mm / 1k materials tested for $J_c$.

Figure 6.51. Graph showing variation of $dC/da$ with crack length for the 5 mm / 6k materials tested for $J_c$. 

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Figure 6.52. Graph showing variation of $\frac{dC}{da}$ with crack length for the 10 mm / 1k materials tested for $J_c$.

Figure 6.53. Graph showing variation of $\frac{dC}{da}$ with crack length for the 10 mm / 6k materials tested for $J_c$. 

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Figure 6.54. Graph of $J_{el}$ against proportion of fibres within bundles for 5 mm / 1k materials.

Figure 6.55. Graph of $J_{el}$ against proportion of fibres within bundles for 5 mm / 6k materials.
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Figure 6.56. Graph of $J_{el}$ against proportion of fibres within bundles for 10 mm / 1k materials.

Figure 6.57. Graph of $J_{el}$ against proportion of fibres within bundles for 10 mm / 6k materials.
Figure 6.58. Graph of $J_{pl}$ against proportion of fibres within bundles for 5 mm / 1k materials.

Figure 6.59. Graph of $J_{pl}$ against proportion of fibres within bundles for 5 mm / 6k materials.
Figure 6.60. Graph of $J_{pl}$ against proportion of fibres within bundles for 10 mm / 1k materials.

Figure 6.61. Graph of $J_{pl}$ against proportion of fibres within bundles for 10 mm / 6k materials.
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Figure 6.62. Graph of $J_c$ against proportion of fibres within bundles for 5 mm / 1k materials.

Figure 6.63. Graph of $J_c$ against proportion of fibres within bundles for 5 mm / 6k materials.
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Figure 6.64. Graph of $J_c$ against proportion of fibres within bundles for 10 mm / 1k materials.

Figure 6.65. Graph of $J_c$ against proportion of fibres within bundles for 10 mm / 6k materials.
Figure 6.66. Graph of ratio of $J_c$ of 5 mm / 1k materials to $J_c$ of polypropylene against proportion of fibres within bundles.

Figure 6.67. Graph of ratio of $J_c$ of 5 mm / 6k materials to $J_c$ of polypropylene against proportion of fibres within bundles.
Figure 6.68. Graph of ratio of $J_c$ of 10 mm / 1k materials to $J_c$ of polypropylene against proportion of fibres within bundles.

Figure 6.69. Graph of ratio of $J_c$ of 10 mm / 6k materials to $J_c$ of polypropylene against proportion of fibres within bundles.
Figure 6.70. Graph of impact surface energy against proportion of fibres within bundles for 5 mm / 1k materials.

Figure 6.71. Graph of impact surface energy against proportion of fibres within bundles for 5 mm / 6k materials.
Figure 6.72. Graph of impact surface energy against proportion of fibres within bundles for 10 mm / 1k materials.

Figure 6.73. Graph of impact surface energy against proportion of fibres within bundles for 10 mm / 6k materials.
Chapter 7. Fracture Mechanisms
7.1 Introduction

The present Chapter reports the results of a study of the fracture mechanisms operating in the short-fibre composites. Photographs of tested materials and scanning electron micrographs of fracture surfaces obtained from quasi-static and impact testing are shown. Interpretation of the appearance of the fracture surfaces is assisted by a knowledge of the critical length of the reinforcement. The critical length of the fibres and bundles was investigated using specimens containing a single fibre or bundle. The possible mechanisms active in these materials are discussed in the context of the appearance of the fracture surfaces and the critical length results. The relationship between the fracture mechanisms and the toughness of the short-fibre composites is also discussed.

7.2 Images of Fracture Surfaces

7.2.1 Tensile Tested Coupons

Photographs of typical tensile tested notched coupons of materials containing 5 mm long fibres and bundles are shown in Figures 7.1 and 7.2. Figure 7.1 shows coupons of the 5 mm / 0 %, 5 mm / 50 % 1k and 5 mm / 100 % 1k materials. Figure 7.2 shows coupons of the 5 mm / 0 %, 5 mm / 50 % 6k and 5 mm / 100 % 6k materials. Figures 7.3 and 7.4 show typical tensile tested notched coupons of the materials containing 10 mm long fibres. Figure 7.3 shows coupons of the 10 mm / 0 %, 10 mm / 50 % 1k and 10 mm / 100 % 1k materials. Figure 7.4 shows coupons of the 10 mm / 0 %, 10 mm / 50 % 6k and 10 mm / 100 % 6k materials.

All the tensile tested coupons exhibit a region of white matrix at the edge of the fracture surface. This is believed to be a result of stress-whitening of the polypropylene matrix as it undergoes plastic deformation. The white appearance of the polypropylene is due to crazes and microcracks in the polypropylene (Karger-Kocsis, 1995). There is some correlation between the amount of whitened material and the amount of plastic deformation in a material. For example, the 10 mm / 0 % material exhibits least whitened material and the load-extension curves shown in
Chapter 6 indicate that this material experiences the least plastic deformation of the materials studied.

As indicated previously in this Section, the tensile tested coupons shown in Figures 7.1 to 7.4 are typical examples from the six coupons that were tested for each material. The appearance of pulled out fibres and bundles varied in detail but coupons of the same composite material had a similar overall appearance. The pulled out fibres are clearly visible at the fracture surface of the 5 mm / 0 % and 10 mm / 0 % materials. Closer examination of the fracture surfaces revealed that the average length of the pulled out fibres is less for the 10 mm / 0 % material than for the 5 mm / 0 % material and that the 50 % of fibres within bundles materials show a fibre pull-out length shorter than in either of the 0 % fibres within bundles materials.

There appear to be a greater number of pulled out bundles in the materials containing 1k bundles (Figures 7.1 and 7.3) than in the materials containing 6k bundles (Figures 7.2 and 7.4). However, the length of pulled out bundles is greater in the materials with 10 mm long bundles than in the materials with 5 mm long bundles. These observations are discussed further in Section 7.4.3 of the present Chapter.

7.2.2 Impact Tested Specimens
Photographs of notched impact tested specimens of materials containing 5 mm long fibres and bundles are shown in Figures 7.5 and 7.6. Figure 7.5 shows specimens of the 5 mm / 0 %, 5 mm / 50 % 1k and 5 mm / 100 % 1k materials. Figure 7.6 shows specimens of the 5 mm / 0 %, 5 mm / 50 % 6k and 5 mm / 100 % 6k materials. Figures 7.7 and 7.8 show notched impact tested specimens of the materials containing 10 mm long fibres. Figure 7.7 shows specimens of the 10 mm / 0 %, 10 mm / 50 % 1k and 10 mm / 100 % 1k materials. Figure 7.8 shows specimens of the 10 mm / 0 %, 10 mm / 50 % 6k and 10 mm / 100 % 6k materials. In Figures 7.5 to 7.8 the specimens have been arranged so that the notch in the specimen is on the left of the specimen.
In contrast to the tensile tested coupons, none of the impact tested specimens shown in Figures 7.5 to 7.8 exhibit stress-whitened material around the fracture surface. This is entirely reasonable and is a result of the higher rate of deformation in the impact tests. Polypropylene exhibits viscoelastic behaviour which results in very little plastic deformation of the matrix at high deformation rates. This is consistent with the suggestion in Chapter 6 that the impact behaviour of these materials is dominated by toughening mechanisms associated with the fibres and bundles in the materials since the toughness of polypropylene is largely due to plastic deformation which is greatly reduced at these higher strain rates.

7.3 Critical Length Results

7.3.1 Specimens for Critical Length Determination

The manufacture of specimens for critical length determination was described in Chapter 3. The resulting specimens had dimensions of 150 mm by 20 mm by 2 mm and consisted of polypropylene containing either a single carbon fibre, a single bundle of 1000 carbon fibres or a single bundle of 6000 carbon fibres. The single fibre or fibre bundle was aligned with the length of the specimen. Great care was taken to preserve the integrity of the fibres and bundles during the manufacture of these specimens. A photograph of each type of specimen, illuminated using a lightbox in contact with the specimens, is shown in Figure 7.9.

The specimens containing either a 1k or 6k fibre bundle exhibited a number of small ridges at the surface of the specimen across the width of the fibre bundle. The distance between these ridges varied between 2 mm and 22 mm. These ridges were investigated by burning off the matrix as described in Chapter 3, but before the specimen had been mechanically strained. It was found that the fibre bundle was fractured in a number of places corresponding to the ridges at the fracture surface. A similar effect was observed in the single fibre specimens, after burning off the matrix it was found that the fibre was broken into fragments. It is reasonable to attribute these observations to the spontaneous fragmentation reported by Incardona et al. (1993) and studied further by Wood et al. (1996). The spontaneous fragmentation of
fibre occurs under the compressive thermal stress generated in the fibre bundle as a result of the greater thermal contraction of the matrix than the fibre on cooling.

Fragmentation of fibres and bundles during manufacture means that it is not possible to estimate the critical length of the fibres and bundles from the distribution of fragment lengths using the usual method, as described by Folkes and Wong (1987). However it is still possible to obtain some information concerning the critical length of the fibres and bundles based on the further fragmentation of the single fibre (or fibre bundle) when the specimen is strained.

7.3.2 Critical Length of Single Fibres
Specimens containing a single fibre were extended to strains of 5% and 10% and the fibre fragment length distribution was determined as described in Chapter 3. The fibre fragment length distribution for the mechanically strained specimens and the as-manufactured specimens are shown in Figure 7.10. It can be seen from Figure 7.10 that for fragment lengths below 3 mm the distribution is very similar for all specimens. However, for fragment lengths greater than 3 mm the percentage of fragments decreases with increasing strain on the sample until, when the sample has been strained to 10%, there are no fragments with a length greater than 3 mm.

A single fibre fragmentation test will fracture a fibre fragment that has a length equal to or greater than the critical length. Hence, from the results presented here it may be concluded that a fragment with a length greater than 3 mm is longer than the critical length. Hence the critical length of the fibres studied here is less than 3 mm.

7.3.3 Critical Length of Fibre Bundles
Samples containing single 1k bundles or single 6k bundles were strained in the same way as the single fibre. No further breaks were observed in either type of fibre bundle. There are two possible explanations for this, firstly, that the critical length of the bundle is sufficiently large that it was not possible to fracture the bundle fragment further. If this is the case, since the largest unbroken fragments of bundle were 22 mm in length, it may be deduced the critical length of the bundles is greater than
Chapter 7. Fracture Mechanisms

22 mm. The second possible explanation is that the matrix is not strong enough to support sufficient stress to fragment the bundle further. If this explanation is correct then it is not possible to draw any deductions concerning the critical length from these tests.

7.4 Discussion of Fracture Mechanisms

7.4.1 Failure of Individual Fibres
The critical length is a useful parameter when considering failure mechanisms in short-fibre composites. For fibres aligned with the loading direction, a fibre with a length less than the critical length will be pulled out rather than fractured. If the fibre length is greater than the critical length, then the fibres will be fractured and the resulting fragments, shorter than the critical length, may be pulled out.

Although it has not been possible to determine the critical length of the fibre in these materials precisely, there is evidence that the critical length is less than 3 mm. Hence, both the 5 mm and 10 mm fibres are longer than the critical length. For fibres longer than the critical length, the average length of fibre pull-out decreases with increasing fibre length because the proportion of fibres that is pulled out decreases (Kim and Mai, 1991). This is consistent with the observations of the fracture surfaces of tensile tested coupons of the 5 mm / 0 % and 10 mm / 0 % materials.

A scanning electron micrograph of the fracture surface of a tensile coupon of the 5 mm / 0 % material is shown in Figure 7.11. A similar image of the fracture surface of an impact specimen of the same material is shown in Figure 7.12. It can be seen that the length of pulled out fibre is significantly reduced in the impact tested specimen.

7.4.2 Failure of Fibre Bundles
The results of the critical length determination together with observations of the fracture surfaces of the materials suggest a fundamental difference in the pull-out behaviour of fibres and bundles. The pull-out length of individual fibres is controlled by the critical length which is a function of the fibre strength, fibre diameter and
interface properties whereas the pull-out length of bundles appears only to be limited by the length of the bundles. In other words, the pull-out length of bundles does not appear to be controlled by the critical length. Although it was not possible to determine the critical length from the samples manufactured in this work, there is evidence that the critical length is greater than 22 mm. Hence, it is reasonable to conclude that any bundles oriented with the loading direction will be completely pulled out and not fractured.

Observation of the fracture surfaces of the tensile tested coupons and the impact tested specimens suggest that there are three ways in which the bundles can fail during fracture. These three mechanisms are described in turn below.

The first case is where the bundle is approximately aligned with the loading direction, that is perpendicular to the fracture surface. In this case the bundle is pulled out as a single unit. This process involves no breakage of the bundle or the fibres constituting the bundle. A bundle that has failed in this manner is shown in Figure 7.13 and the resulting socket is shown in Figure 7.14.

The second case is where the bundle is oriented at an angle between parallel and perpendicular to the applied load. In this case the crack propagates through part or all of the bundle, resulting in some or all of the fibres within the bundle being broken. The corresponding fracture surface containing parts of a bundle which has failed in this manner are shown in Figures 7.15 and 7.16.

The third case occurs when the bundle is oriented approximately perpendicular to the loading direction. In this case the bundle will break along its length, this type of fracture involves no breakage of constituent fibres. An example of a bundle in a 100 % of fibres within bundles material which has failed in this way is shown in Figure 7.17. It is also possible to observe the two parts of a bundle in a 50 % of fibres within bundles material that has failed in this way in the lower left of Figure 7.13 and the upper left of Figure 7.14.
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The three ways in which bundles may fail in these materials have been differentiated in terms of the orientation of the bundle relative to the loading direction. In reality there are a range of possible orientations that will result in a similar type of failure of the bundle. In particular, the first type of failure associated with bundles oriented parallel to the loading direction may be observed for bundles at relatively large angles to the loading direction. In some cases this type of failure is observed in bundles oriented up to approximately 45° to the loading direction. However, there does not appear to be a clear transition from one type of failure to another, so similarly oriented bundles may be observed to fail in different ways in different specimens.

It is useful to speculate on the relative toughening effect of these three types of bundle failure. For maximum toughening effect the area of fibre pulled out should be maximised which requires that fibres do not fracture. The first mode of fracture with the bundle being pulled out in a similar way to an individual fibre is the toughening mode envisaged by Cottrell (1964). The critical length results and observation of the fracture surfaces indicates that the pulled out length of bundles is greater than individual fibres as would be expected from Cottrell (1964). The second mode would be expected to have relatively less toughening effect since the area of pull-out is reduced by the fracture of some of the fibres within the bundles. The toughening effect of a bundle in the third case is likely to be minimal since the fracture takes place within the polypropylene so the presence of a bundle at the fracture surface would have no beneficial effect. It is reasonable to assume that only bundles failing in the first way will make a significant contribution to the toughness of the materials.

7.4.3 Comparison of Materials Containing Fibre Bundles

Many of the observations made of the fracture surfaces in Section 7.2 can be explained by the different number of bundles within the materials. It is possible to calculate the number of bundles within any specified volume of each material. This calculation is based on a knowledge of the volume fraction of the laminates and the mass of fibre per unit length, as presented in Chapter 3. The only assumption, which seems a reasonable one, is that the proportion of fibres within bundles is accurate.
The resulting estimates of the number of bundles within a laminate (150 mm by 150 mm by 2 mm) of each material are shown in Table 7.1. It can be seen that the number of bundles within a laminate changes significantly depending on the proportion of fibres within bundles, the bundle length, the number of fibres within the bundle and the overall volume fraction of fibre within the laminate. Table 7.1 also shows an estimate of the number of bundles within a 10 mm square of the laminate. This quantity provides a measure of the distribution of bundles that is more relevant to observations of the fracture surfaces.

The specimens and coupons shown in Figures 7.1 to 7.8 are “typical” but cannot represent the range of fracture surfaces observed. When observations from all the coupons and specimens for a material are considered, the number of bundles at the fracture surface is greater for the 100% of fibres within bundles materials than the corresponding 50% of fibres within bundles materials. Similarly there are more bundles present at the fracture surfaces of materials containing 1k bundles compared to materials with materials containing 6k bundles. Both these observations can be explained with reference to Table 7.1 which shows that they are consistent with the number of bundles per unit area of the laminate.

The effect of bundle length is less straightforward since Table 7.1 shows, as expected, materials with 10 mm bundles contain half the number of bundles per unit area of a corresponding material with 5 mm long bundles with the same volume fraction. However, observation of the fracture surfaces shows that materials with 10 mm long bundles appear to exhibit more bundles at the fracture surface. This may be because the length of bundle pull-out is longer in the materials with 10 mm long bundles, which makes the bundles more visible although there is a smaller number of bundles present. Alternatively, the longer bundles may be involved in fracture at a greater distance from the crack. So although the number of bundles per unit area is less in the materials with 10 mm long bundles, the area involved with the fracture is greater. Hence, overall, the number of bundles involved with fracture could be the same or greater even though the number of bundles per unit area is less.
7.4.4 Comparison of Individual Fibres and Fibre Bundles

In this study it is possible to compare the toughness of materials containing fibres and/or bundles. It is generally accepted that the most important toughening mechanism in short-fibre composites is fibre pull-out (Kim and Mai, 1991). It was suggested in Section 7.4.2 that the length of pull-out for bundles is limited only by the bundle length. This results in a maximum pull-out length of 2.5 mm and 5 mm for a 5 mm and a 10 mm long bundle respectively. For individual fibres the maximum pull-out length is half of the critical length i.e. less than 1.5 mm. So, for both fibre and bundle lengths the use of bundles increases the length of pull-out.

These observations confirm that the use of bundles increases the pull-out length of the fibre which would be expected to increase the toughness of the material. However, there is a disadvantage to the use of bundles which is that bundles contain a large number of fibres. The materials studied here contain either 1000 or 6000 fibres. Calculations of the surface area of a fibre and the two types of bundle are presented in Tables 7.2 and 7.3. These calculations are based on the radii presented in Chapter 5 and do not include the surface area of the fibre and bundle ends. The surface area of the bundles is calculated as though the fibre was a solid cylinder; while this is not the case it is believed to be a reasonable first approximation. It can be seen that the surface area of a bundle is equivalent to the surface area of only 41 or 100 individual fibres for a bundle containing 1000 or 6000 fibres respectively. Hence, the surface area available to pull-out is reduced by a factor of either 24 or 60 by using 1000 or 6000 fibre bundles respectively compared to using the individual fibres constituting the bundle.

The use of bundles compared to individual fibres will increase the pull-out length at the expense of reducing the surface area available for pull-out. In the case of the materials studied here the pull-out length has been slightly increased but the surface area of fibre available to pull-out has been reduced by a factor of 24 and 60. With the fibre length and bundle diameters used in this study it appears impossible that the use of bundles could increase the toughness of the composite materials based on pull-out considerations.
For fibre-bundling to increase the pull-out area it would be necessary for the increase in pull-out length to outweigh the reduction in area caused by the inclusion of fibres within bundles. For this to occur would require longer fibres than those considered here. For bundles containing 1000 and 6000 fibres the pull-out length of the bundle would have to be 24 and 60 times respectively the pull-out length of an individual fibre to achieve the same area of pull-out, although this ratio would decrease for smaller numbers of fibres per bundle. This indicates that the use of fibre-bundling is unlikely to be beneficial for short-fibre composites. However, there is the possibility that the use of fibre-bundling may be beneficial for long-fibre or continuous fibre composites.

The present Chapter and Chapter 6 have considered the effects of fibre-bundling on the toughness of polypropylene reinforced with carbon fibres. The polypropylene matrix of the materials studied here is a relatively tough matrix material. For a lower toughness matrix material, such as an epoxy, it might be expected that the use of fibre-bundling would have a more deleterious effect on the toughness properties because the regions of unreinforced resin present in bundled materials would not have a beneficial effect on the toughness.

7.5 Conclusions
Fracture surfaces of tensile tested coupons and impact tested specimens have been examined. The tensile tested coupons showed stress-whitened regions, indicating plastic deformation. No such regions were exhibited by the impact tested specimens. The fracture surfaces showed variation in the pull-out length of the individual fibres and also the number and pull-out length of fibre bundles.

The results of an investigation of the critical length of the single fibres and bundles containing 1000 and 6000 fibres are presented. It was not possible to determine a precise value for the critical length of the fibres or bundles because of fragmentation in the as-manufactured specimens as a result of thermal stresses. However it was
possible to establish that the critical length of the fibres was less than 3 mm and to deduce that the critical length of the bundles was greater than 22 mm.

The fracture mechanisms active in these materials have been discussed with reference to photographs and scanning electron micrographs of the fracture surfaces, and critical length results. Three distinct ways in which a fibre bundle can fail have been identified. The effect of the fracture mechanisms on the toughness of these materials has been discussed.

Finally, the relative toughening effect of individual fibres and fibre bundles is discussed. The use of bundles slightly increases pull-out length but drastically reduces the area of fibre available to pull-out. A material would have to be substantially different from the materials studied here for the use of fibre bundles to increase the toughness of a short-fibre composite.
Chapter 7. Fracture Mechanisms

Table 7.1. Estimated number of bundles in materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>Estimated number of bundles in laminate</th>
<th>Estimated number of bundles per 100 mm²</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 mm / 50% 1k</td>
<td>11270</td>
<td>50.1</td>
</tr>
<tr>
<td>5 mm / 100% 1k</td>
<td>32100</td>
<td>142.6</td>
</tr>
<tr>
<td>5 mm / 50% 6k</td>
<td>1810</td>
<td>8.0</td>
</tr>
<tr>
<td>5 mm / 100% 6k</td>
<td>6030</td>
<td>26.8</td>
</tr>
<tr>
<td>10 mm / 50% 1k</td>
<td>6300</td>
<td>28.0</td>
</tr>
<tr>
<td>10 mm / 100% 1k</td>
<td>16390</td>
<td>72.9</td>
</tr>
<tr>
<td>10 mm / 50% 6k</td>
<td>1780</td>
<td>7.9</td>
</tr>
<tr>
<td>10 mm / 100% 6k</td>
<td>3930</td>
<td>17.5</td>
</tr>
</tbody>
</table>

Table 7.2. Surface area of 5 mm long reinforcement.

<table>
<thead>
<tr>
<th>Reinforcing unit</th>
<th>Radius in m</th>
<th>Surface area in m²</th>
<th>Number of fibres equivalent to bundle</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 mm long fibre</td>
<td>3.95x10⁻⁶</td>
<td>1.24x10⁻⁷</td>
<td>-</td>
</tr>
<tr>
<td>5 mm long 1k bundle</td>
<td>1.61x10⁻⁴</td>
<td>5.06x10⁻⁶</td>
<td>41</td>
</tr>
<tr>
<td>5 mm long 6k bundle</td>
<td>3.95x10⁻⁴</td>
<td>1.24x10⁻⁵</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 7.3. Surface area of 10 mm long reinforcement.

<table>
<thead>
<tr>
<th>Reinforcing unit</th>
<th>Radius in m</th>
<th>Surface area in m²</th>
<th>Number of fibres equivalent to bundle</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 mm long fibre</td>
<td>3.95x10⁻⁶</td>
<td>2.48x10⁻⁷</td>
<td>-</td>
</tr>
<tr>
<td>10 mm long 1k bundle</td>
<td>1.61x10⁻⁴</td>
<td>1.01x10⁻³</td>
<td>41</td>
</tr>
<tr>
<td>10 mm long 6k bundle</td>
<td>3.95 x10⁻⁴</td>
<td>2.48 x10⁻⁵</td>
<td>100</td>
</tr>
</tbody>
</table>
Figure 7.1. Photograph of the fracture surfaces of tensile tested coupons of 5 mm / 1k materials. Labels show proportion of fibres within bundles.

Figure 7.2. Photograph of the fracture surfaces of tensile tested coupons of 5 mm / 6k materials. Labels show proportion of fibres within bundles.
Figure 7.3. Photograph of the fracture surfaces of tensile tested coupons of 10 mm / 1k materials. Labels show proportion of fibres within bundles.

Figure 7.4. Photograph of the fracture surfaces of tensile tested coupons of 10 mm / 6k materials. Labels show proportion of fibres within bundles.
Figure 7.5. Photograph of the fracture surfaces of impact tested specimens of 5 mm / 1k materials. Labels show proportion of fibres within bundles.

Figure 7.6. Photograph of the fracture surfaces of impact tested specimens of 5 mm / 6k materials. Labels show proportion of fibres within bundles.
Figure 7.7. Photograph of the fracture surfaces of impact tested specimens of 10 mm / 1k materials. Labels show proportion of fibres within bundles.

Figure 7.8. Photograph of the fracture surfaces of impact tested specimens of 10 mm / 6k materials. Labels show proportion of fibres within bundles.
Figure 7.9. Photograph of typical specimens manufactured for critical length determination. (From left to right; single fibre, single 1k bundle and single 6k bundle.)

Figure 7.10. Graph showing the distribution of fragment lengths from the single fibre specimens.
Figure 7.11. Scanning electron micrograph of a fracture surface of a tensile tested coupon of the 5 mm / 0 % material.

Figure 7.12. Scanning electron micrograph of a fracture surface of an impact tested specimen of the 5 mm / 0 % material.
Figure 7.13. Scanning electron micrograph of a fracture surface of a 50% of fibres within bundles material showing a bundle with no broken fibres.

Figure 7.14. Scanning electron micrograph of a fracture surface of a 50% of fibres within bundles material showing a socket with no broken fibres.
Figure 7.15. Scanning electron micrograph of a fracture surface of a 50% of fibres within bundles material showing a bundle with broken fibres.

Figure 7.16. Scanning electron micrograph of a fracture surface of a 50% of fibres within bundles material showing a socket with broken fibres.
Figure 7.17. Scanning electron micrograph of a fracture surface of a 100 % of fibres within bundles material showing a bundle broken along its length.
Chapter 8. Conclusions and Further Work
8.1 Conclusions

The aim of the present work was to investigate the effect of fibre-bundling on the mechanical properties of a short-fibre composite. From a review of literature it was found that, at present, it is not possible to quantify fibre-bundling within a short-fibre composite. For this reason a study of the effect of fibre-bundling must consider materials with well-defined fibre-bundling. To produce materials for the present study, a novel manufacturing technique was developed enabling carbon fibre mats with a known proportion of fibres within bundles of a known size to be produced. This technique was used to produce fibre mats with 0 %, 25 %, 50 %, 75 % and 100 % of fibres within bundles. Mats were produced with either 5 mm or 10 mm long fibres and with bundles containing either 1000 or 6000 fibres. These mats were impregnated with polypropylene by a film stacking technique so their structure was retained in the short-fibre composites.

It was found that an increase in the proportion of fibres within bundles resulted in a decrease in the tensile modulus of the short-fibre composites. The decrease in modulus was less severe for materials with longer fibres, materials with a smaller number of fibres per bundle and laminates with a greater thickness. For the materials studied, tensile strength of materials containing bundles was one quarter of the tensile strength of the filamentised material.

A model has been developed for the modulus of a short-fibre composite containing fibre bundles. Three variants of this model, based on structural variations within the materials, were used to predict the modulus of the materials. Experimental data for the materials fell within the range of the predictions and it was found that different variants of the model were more appropriate for different materials.

The critical J integral $J_c$ was found to be the most useful toughness measure for these materials. The use of fibre bundles only resulted in a clear increase in $J_c$, compared to the filamentised material, for one combination of fibre length and bundle size and this increase is likely to be a result of areas of unreinforced matrix in the material. $J_c$ appeared to be increased by either more effective reinforcement or larger areas of
unreinforced resin. Materials containing both filamentised fibres and fibre bundles generally exhibited a lower $J_c$ than materials containing only filamentised fibres or only fibre bundles. The impact surface energy results for the same range of materials show that, as with $J_c$, the use of bundles does not significantly improve the impact surface energy compared to the filamentised material for either fibre length or bundle size studied.

An investigation of the critical length of the fibres and bundles suggested that the critical length of both types of bundles is substantially longer than that of individual fibres. The effect of this can be seen in the fracture surfaces where bundles parallel to the loading direction are not fractured but are completely pulled out. Three distinct ways in which bundles may fail have been identified. It has been proposed that fibre-bundling slightly increases pull-out length but drastically reduces the area of fibre available for pull-out. Hence, for the materials studied here, the use of fibre bundles does not increase the energy of pull-out and so does not improve the toughness.

Following the results of this study it is difficult to envisage a short-fibre composite which would exhibit improved toughness properties from the use of fibre-bundling. Certainly, for a typical mass-manufactured short-fibre composite the use of fibre bundles is undesirable since an improvement in toughness is unlikely and a reduction in modulus and strength is expected. A corollary to these conclusions is that the mechanical properties of a short-fibre composite are optimised by ensuring that fibres are filamentised and distributed evenly throughout the material.

8.2 Further Work

The results of this work have shown that fibre-bundles are not desirable in the short-fibre composites studied here. However, there is a possibility that a short-fibre composite consisting of small diameter bundles and/or fibres with a very short critical length would show an improvement in toughness with the use of fibre bundles. Short-fibre composites that could show an improvement in toughness from the use of bundles could be identified from a calculation of pull-out areas.
Chapter 8. Conclusions and Further Work

The short-fibre composites investigated in this study were manufactured with a single size of bundle. Mass-manufactured materials may contain bundles with a variety of sizes. A study of these materials would require a technique for the assessment of more complex fibre-bundling. A study of fibre-bundling in mass-manufactured materials may be concerned with ensuring fibres are fully dispersed, since it seems that the mechanical properties are maximised for such materials.

Although fibre bundles appear to be undesirable in short-fibre composites, they may be beneficial in long-fibre or continuous fibre composite materials. The greater pull-out length of bundles would be more effective in these materials because the pull-out length of fibres in these materials is generally lower. In addition, the use of bundles may inhibit crack growth in these materials.

More fundamental work should consider the behaviour of individual fibres compared to fibre bundles. An attempt was made in this work to compare the critical length of single fibres and fibre bundles. With modification, this method may be successfully applied to both single fibres and fibre bundles. Other factors that merit study are the effect of the number of fibres per bundle, the orientation of bundles and the shape of bundles. Further insight into the behaviour of bundles may be obtained by a comparison of the behaviour of fibres at the centre of a bundle compared to those at the edge of the bundle.
References
ASTM D 5045,
Plane strain fracture toughness and strain energy release rate of plastic materials.

ASTM E 1737,

Abbé, F, Chermant, L, Coster, M, Gomina, M, and Chermant, J L,
Morphological characterisation of ceramic-ceramic composites by image analysis.

Agarwal, B D, Patro, B S, and Kumar, P,
J integral as fracture criterion for short fibre composites: an experimental approach.

Al-Hassani, A, Mills, P J, and Ogin, S L,
Fatigue of Short-Fibre Reinforced Composites.

Atkins, A G, and Mai, Y W,
Elastic and Plastic Fracture,

Bader, M G, and Hill, A R,
Short Fibre Composites.

Barnby, J T, and Spencer, B,
Crack propagation and compliance calibration in fibre reinforced polymers.

Begley, J A, and Landes, J D,
The J integral as a fracture criterion.

Berglund, L A,
Thermoplastic Resins.

Burns, R,
Polyester Moulding compounds,
References

Campbell, D, and Qayyum, M M,
Melt crystallisation of polypropylene: effect of contact with fibre substrates.

Carman, G P, and Reifsnider, K L,
Micromechanics of short-fibre composites.
Composites Science and Technology, 43, 137-146, 1992.

Chan, M K V, and Williams, J G,
J integral studies of crack initiation of a tough high density polyethylene.

Choi, N S, and Takahashi, K,
Toughness and microscopic fracture mechanisms of unfilled and short glass fibre filled poly(cyano arylether).

Clyne, T W,
A simple development of the shear lag theory appropriate for composites with a relatively small modulus mismatch.

Cottrell, A H,
Strong solids.

Cox, H L,
The elasticity and strength of paper and other fibrous materials.

Edujjee, R F, McCullough, R L and Gillespie, J W,
The influence of aggregated and dispersed textures on the elastic properties of discontinuous-fibre composites.

Eftis, J, Jones, D L, and Liebowitz, H,
On fracture toughness in the nonlinear range.

Ericson, M L, and Berglund, L A,
Deformation and fracture of glass mat reinforced polypropylene.

Ericson, M L, and Berglund, L A,
The effect of microstructure on the elastic modulus and strength of preformed and commercial GMTs.
Polymer Composites, 14, 35-41, 1993.

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References

Eshelby, J D,
The determination of the elastic field of an ellipsoidal inclusion and related problems.

Favre, J P, and Perrin, J,
Carbon fibre adhesion to organic matrices.

Fila, M, Bredin, C and Piggott, M R,
Work of fracture of fibre-reinforced polymers.

Folkes, M J, and Wong, W K,
Determination of interfacial shear strength in fibre reinforced thermoplastic composites.

Folkes, M J,
Interfacial crystallisation of polypropylene in composites

Friedrich, K,
Microstructural efficiency and fracture toughness of short fibre / thermoplastic matrix composites.

Friedrich, K, Carlsson, L A, Gillespie, J W, and Karger-Kocsis, J,
Fracture of thermoplastic composites.

Friedrich, K,
Mesoscopic aspects of polymer composites: processing, structure and properties.

Fukuda, H, and Chou, T W,
An advanced shear-lag model applicable to discontinuous fibre composites.

Galiotis, C, Young, R J, Yeung, P H J, and Batchelor, D N,
The study of model diacetylene/epoxy composites: part 1 - the axial strain in the fibre.

Greig-Smith, P,
The use of random and contiguous quadrats in the study of the structure of plant communities.
Guild, F J and Silverman, B W,
The microstructure glass fibre reinforced polyester resin composites.

Guild, F J and Summerscales, J,
Microstructural image analysis applied to fibre composite materials: a review.
*Composites*, 24, 383-393, 1993

Halpin, J C, and Tsai, S W,
*Environmental Factors in Composite Materials Design.*

Halpin, J C,
Stiffness and expansion estimates for orientated short fibre composites.

Halpin, J C, and Kardos, J L,

Hamer, J W and Woodhams, R T,
The fracture toughness of polypropylene containing poly(ethylene terephthalate) fibres.
*Polymer Engineering and Science*, 21, 603-611, 1981.

Hashemi, S, and Williams, J G,
The effects of specimen configuration and notch tip radius on the fracture toughness of polymers using $J_c$.

Herrera-Franco, P J, and Drzal, L T,
Comparison of methods for the measurement of fibre/matrix adhesion in composites.

Hertzberg, R W,

Hitchen, S A, Ogin, S L, Smith P A and Soutis, C,
The effect of fibre length on fracture toughness of short carbon fibre/epoxy composites.

Hull, D,
*An Introduction to Composite Materials.*
Incardona, S, Migliaresi, C, Wagner, H D, Gilbert, A H, and Marom, G,
The mechanical role of the fibre/matrix transcrystalline interphase in carbon fibre
reinforced J-polymer microcomposites.
**Composites Science and Technology, 47, 43-50, 1993.**

Jinen, E,
The determination of $\tilde{G}_c$ fracture toughness values for short carbon fibre reinforced
nylon 66 (FRTIP) by acoustic emission.
**Engineering Fracture Mechanics, 21, 157-171, 1985.**

Kacir, L, Narkis, M and Ishai, O,
Oriented short glass fibre composites. III. Structure and mechanical properties of
moulded sheets.
**Polymer Engineering and Science, 17, 234-241, 1977.**

Kacir, L, Ishai, O and Narkis, M,
Oriented short glass fibre composites. IV. Dependence of mechanical properties on
the distribution of fibre orientations.
**Polymer Engineering and Science, 18, 45-52, 1978.**

Karbhari, V M, and Wilkins, D J,
Constituent scale and property effects on fibre-matrix debonding and pull-out.
**Journal of Materials Science, 26, 5888-5898, 1991.**

Karger-Kocsis, J,
Microstructural aspects of fracture in polypropylene and in its filled, chopped fibre and
fibre mat reinforced composites.

Kataoka, Y, Taya, M and Saito, M,
Effect of fibre clustering on the stiffness and strength of a short fibre composite.
**Proceedings of the Japan-CCM, 7, 505-514, Kyoto, June 19-22, 1995.**

Kelly, A and Tyson, W R,
Tensile properties of fibre-reinforced metals: Copper/Tungsten and
Copper/Molybdenum.

Kelly, A,
**Strong Solids**

Kim, B H, and Kim, H S,
Fracture characterization of short glass fibre reinforced thermoplastic polyester by the
J-integral.
**Journal of Materials Science, 24, 921-925, 1989.**
Kim, J K, and Mai, Y W,
High strength, high fracture toughness fibre composites with interface control - a review.

Kim, J K, and Mai, Y W,
Fracture of CFRP containing impregnated fibre bundles.

Krenchel, H,
**Fibre Reinforcement**.

Lafdi, K, and Wright, M A,
Carbon Fibres.

Lauke, B, and Pompe, W,
Fracture toughness of short-fibre reinforced thermoplastics.

Liebowitz, H, and Eftis, J,
On nonlinear effects in fracture mechanics.

Lindhagen, J, and Berglund, L A,
Notch sensitivity and damage mechanisms of glass mat reinforced polypropylene.

Lindhagen, J, and Berglund, L A,
Microscopical damage mechanisms in glass fibre reinforced polypropylene.

Mandelbrot, B B,
**The Fractal Geometry of Nature**.

Mei, T and Piggott, M R,
Mesoscopic structure development during moulding of sheet moulding compounds.
**Polymer Composites**, 17, 548-555, 1996.

Meraghni, F and Benegagh, M L,
Micromechanical modelling of matrix degradation in randomly oriented discontinuous fibre composites.
References

Myles, J P, Flenley, E C, Fieller, N R J, Atkinson, H V, and Jones, N,
Statistical tests for clustering of second phases in composite materials.
Philosophical Magazine A, 72, 515-528, 1995.

Nairn, J A,
On the use of shear-lag methods for analysis of stress transfer in unidirectional
composites.

Narisawa, I,
Fracture and toughness of crystalline polymer solids.

Pan, N,
Analytical characterisation of the anisotropy and local heterogeneity of short-fibre
composites: fibre fraction as a variable.

Piggott, M R,
Theoretical estimation of fracture toughness of fibrous composites.

Piggott, M R,
Debonding and friction at fibre-polymer interfaces: I. criteria for failure and sliding.

Piggott, M R,
Mesosstructures: an overview.
Proceedings of the 37th International SAMPE Symposium, 37, 738-746,
Anaheim, March 9-12, 1992.

Piggott, M R,
Preface: realistic models for fibre composites.

Piggott, M R,
Mesosstructures and their mechanics in fibre composites.
Advanced Composite Materials, 6, 75-81, 1996.

Pitchumani, R, and Yao, S C,
Correlation of thermal conductivities of unidirectional fibrous composites using local
fractal techniques.

Ramos, M A, and Belmome, F A,
Polypropylene/Low density Polyethylene blend matrices and short glass fibre based
composites: 3. morphology and fibre orientation.
Polymer Composites, 12, 7-12, 1991.
References

Rice, J R,
A path independent integral and the approximate analysis of strain concentration by notches and cracks.

Rice, J R, Paris, P C and Merkle, J G,
Some further results of J integral analysis and estimates.

Seferis, J C,
Polyetherketone (PEEK): processing-structure and properties studies for a matrix in high performance composites.

Singh, R K, and Parihar, K S,
The J integral as a fracture criterion for polycarbonate thermoplastic

Smith, P A,
Private communication, 1996.

Thomason, J L, and Van Rooyen, A A,
Transcrystallised interphase in thermoplastic composites: part 2 influence of interfacial stress, cooling rate, fibre properties and polymer molecular weight.

Trapeznikov, D A, Toropov, A I, and Loskutov, O D,
Modelling approach to optimisation of mechanical properties of discontinuous fibre-reinforced C/C composites.

Wells, J K and Beaumont, P W R,
Debonding and pull-out processes in fibrous composites.

Wells, J K and Beaumont, P W R,
Crack-tip energy absorption processes in fibre composites.

Westerlind, B S, Carlsson, L A, And Andersson, Y M,
Fracture toughness of liner board evaluated by the J integral.

Williams, J G,
*Fracture Mechanics of Polymers*.
Chichester, Ellis Horwood, 1984.

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Wimolkiatisak, A S, Bell, J P, Scola, D A and Chang, J,
Assessment of fibre arrangement and contiguity in composite materials by image

Wood, J R, Wagner, H D, and Marom, G,
The compressive fragmentation phenomenon: using microcomposites to evaluate
thermal stresses, single fibre compressive strengths, Weibull parameters and the

Worrall, C M, and Wells, G M,
Fibre distribution in discontinuous fibre reinforced plastics: characterisation and effect
on material performance. *Proceedings of the Seventh European Conference on Composite Materials*, 1, 247-

Ye, L, Beechag, A and Friedrich, K,
Mesostructural aspects of interlaminar fracture in thermoplastic composites: is

Yuharu, T, and Kortschot, M T,

Yurgartis, S W, and Purandare, M N,

Yurgartis, S W,

Zhou, J, Yin, J, Liu, W, and Li, B,