THE MECHANICAL PROPERTIES OF
GLASS FIBRE REINFORCED
AND RUBBER TOUGHENED
POLYPROPYLENE

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A Thesis Submitted for the Degree of Doctor of Philosophy
at the University of Surrey
I would like to dedicate this wonderful piece of scientific script to my mother and father, as a small token of thanks for their unquestioning support and boundless love over twenty-six maturing years.

This work would not have been possible without the poignant guidance of my supervisors, Mr Mike Bader and Dr Paul Smith. Without their help this work would have lacked the interest, originality and scientific background which made it such an enjoyable project with which to be associated. A substantial proportion of this work was carried out at the I.C.I Advanced Materials Centre where Dr Roy Moore, Dr Martin Rayner and Dr Robin Chivers allowed me seemingly limitless use of their equipment, time and knowledge. For which, I am whole heartily indebted and grateful.

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The Swiss Cheese Plant - from a single leaf a jungle appeared.
SUMMARY

The mechanical properties and fracture mechanics of a series of short glass fibre reinforced and rubber toughened polypropylene composite grades has been studied. The microstructural characteristics of composite grades were examined and, through appropriate models, related to the observed mechanical properties.

The moulded material was modelled as being composed of fibre reinforced plies of varying average fibre orientation. The rubber was distributed uniformly throughout the specimens. The main effect of the rubber was to reduce the stiffness of the matrix and hence also the efficiency of the load transfer at the fibre/matrix interface while at the same time improving the fracture toughness and critical strain energy release rate of the matrix.

Automated image analysis has been used to characterise the rubber particles' size, shape and distribution, and glass fibres' length and orientation distributions. The fibre/matrix interface has been studied using a novel single fibre fragmentation technique. Iterative computer simulations have been developed to accurately predict the stress-strain response of the various grades.

The fracture mechanics properties of this series of materials are highly strain rate sensitive. At low strain rates the addition of glass fibres reduces the toughness of the material because the fibres act as discontinuities within the matrix, aiding initiation and propagation of a crack. At higher strain rates the fibres toughen the material by increasing the energy dissipation associated with fibre pull-out. These effects result in changes in the fracture surface morphology. Fibres
pulled-out at low strain rates had clean surfaces. At higher strain rates the surfaces of pulled-out fibres were coated in an adherent sheath of matrix material. These effects are considered to be a consequence of the viscoelastic nature of the matrix. At low strain rates the matrix deforms plastically. At impact speeds the matrix responds in a predominantly brittle manner.
NOMENCLATURE

a  Crack Length
AH1_ Material Grade Code - Campaign 1
AH2_ Material Grade Code - Campaign 2
an  Charpy Impact Strength
An  Charpy Impact Energy Loss
ao  Initial Crack Length
b  Crack Length Increment
BN  Specimen Thickness (Notched)
Bmin  Fracture Mechanics Minimum Specimen Dimension Criteria
C  Plastic Constraint Factor
Ci  Total Count of Measurable Fibres
CT  Compact Tension
d  Fibre Diameter
DWd b  Fracture Mechanics Specimen Dimensions
da  Crack Extension/Growth
da_max  Crack Growth Exclusion Criteria
DMTA  Dynamic Mechanical Thermal Analyser
E  Modulus
EGF  European Group on Fracture
EPFM  Elastic/Plastic Fracture Mechanics
Exp  Experimentally Determined Results
\bar{\sigma}_f  Average Stress in Fibre
F  Force
FC  IFWIT Peak Load
FLD  Fibre Length Distribution
FOD  Fibre Orientation Distribution
Fr  Volume Fraction of Rubber in Matrix
FRP  Fibre Reinforced Polymer
g  Glass
G  Strain Energy Release Rate
G_{IC} G_C  Critical Strain Energy Release Rate
G_m  Matrix Shear Modulus
IFWIT  Instrumented Falling Weight Impact Test
J  Elastic/Plastic Crack Driving Energy
$J_{0.2}$  \( J_C \) Evaluated using 0.2mm Vertical Line

$J_{0.2/BL}$  \( J_C \) Evaluated using 0.2mm Blunting Line

$J_C$ Critical $J$ integral

$J_0$ Fracture Resistance not allowing for Crack Growth

$k$ Constant of Proportionality

$K$ Stress Intensity Factor

$K_{IC}$ $K_C$ Critical Stress Intensity Factor

$l$ Fibre Length

$l_C$ Critical Fibre Length

LEFM Linear Elastic Fracture Mechanics

$L_i$ Fibres of Length $i$

$l_{i,j}$ Sub/Super Critical Fibres of Length $i,j$ Respectively

$m$ Matrix

MFD Principal Melt Fill/Flow Direction

$M_i$ Total Number of Mismeasured Fibres

$\text{Mod}$ Model Prediction

$n$ Fracture Mechanics Specimen Geometrical Factor

$N_C$ Count of Correctly Measured Fibres

$n_i$ True Count of Fibres of Length $L_i$

$N_i$ Fibre Count of Length $L_i$

$N_m$ Count of Incorrectly Measured Fibres

$P$ Load

$P_i$ Probability of Mismeasuring a Fibre of Length $L_i$

$\text{PP}$ Polypropylene

$r$ Fibre Radius

$R$ Cox Inter-Fibre Spacing

$r_y$ Crack Tip Plastic Zone Radius

SEM Scanning Electron Microscopy

$\text{SENB}$ Single Edge Notched Bend

SFRTF Short Fibre Reinforced Thermoplastic

$T_g$ Glass Transition Temperature

Theo Theoretical Prediction

$U$ Work Done

$U_c$ Elastic Energy of IFWIT Specimen at Peak Load

VDU Visual Display Unit

$V_f$ Volume Fraction

$W_e$ Energy to Bend Charpy Specimen to Crack Initiation
\( W_f \) Weight Fraction
\( W_{\text{kin}} \) Kinetic Energy of Charpy Sample
\( X \) Fibre End Coordinates
\( Y \) IFWIT Geometrical Factor
\( f \) CT Geometrical Factor
\( \alpha, \beta \) Angular Coordinates
\( \beta \) Cox Fibre Stress Factor
\( \gamma_{e,p} \) Surface Energy (elastic, plastic)
\( \delta \) Loss Modulus
\( \Delta a \) Change in Crack Length
\( \Delta U \) Change in Work Done
\( \varepsilon \) Strain
\( \eta_l \) Length Correction Factor
\( \eta_o \) Orientation Correction Factor
\( \Theta \) Fibre Orientation Relative to MFD
\( \xi \) Halpin-Tsai Geometrical Factor
\( \rho \) Density
\( \sigma \) Stress
\( \sigma_c^* \) Fracture Strength of Composite
\( \sigma_f^* \) Fracture Strength of Fibre
\( \sigma_m' \) Stress in Matrix at Fibre Failure Stress
\( \sigma_t \) Transverse Stress
\( \sigma_{uc} \) Ultimate Composite Strength
\( \sigma_{uf, fu} \) Ultimate Fibre Strength
\( \sigma_{um} \) Ultimate Matrix Strength
\( \sigma_y \) Yield Stress
\( \tau \) Shear Stress
\( \tau_i \) Interfacial Shear Stress (Strength)
\( \tau_c \) Composite Shear Strength
\( \tau_{\text{max}} \) Measured Maximum Fibre/Matrix Interfacial \( \tau \)
\( \tau_{\text{Mmax}} \) Predicted Maximum Fibre/Matrix Interfacial \( \tau \)
\( \tau_{\text{PPmax}} \) Maximum Fibre/Polypropylene Interfacial Shear Stress
\( \phi \) Specimen Geometrical Factors
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1. INTRODUCTION

Short glass fibre reinforced thermoplastics (SFRTF) are utilised in a wide range of low-cost high-volume applications. With lower specific mechanical properties than continuous-fibre reinforced composites, SFRTF's are attractive on account of their low-cost enhanced properties in relation to unreinforced thermoplastics, and their ability to be injection moulded into complex shapes at high production rates.

The general effect of short glass-fibre additions to the polymer is to raise the elastic modulus and strength whilst reducing ductility. Toughness is also influenced, but it is more difficult to characterise because it is affected by stress concentrations, test piece geometry, microstructural morphology, loading strain rate and environmental conditions.

The present project is concerned with a polypropylene base polymer, to which glass fibre, a polyolefin rubber and a coupling agent have been added. Rubber introduced to unreinforced thermoplastics resides as a separate phase within the base polymer. These polymer "alloys" have enhanced toughness but with an associated reduction of the mechanical strength and stiffness. Rubber additions to short fibre reinforced thermoplastics produce similar general effects but the behaviour is more complex due to the different toughening mechanisms and effects in the region of the fibre/matrix interface. The coupling agent is designed to promote adhesion of the polymer matrix to the glass fibre surface.
It is well known that the flow of a fibre filled melt into the mould leads to a complex fibre orientation distribution (FOD); the FOD often changing in layers through the thickness and this pattern itself changing in location in the moulded part. This leads to anisotropy and inhomogeneity of mechanical properties. It must also be noted that the fibre length distribution affects mechanical properties and can itself be influenced by the injection moulding process. Since the behaviour and properties of SFRTP are strongly dependent on microstructure a major aim of the present work was to develop techniques for characterising microstructure accurately. To this end, automated image analysis has been used to characterise the composite microstructure. The fibre/matrix interface has been studied using a novel single fibre fragmentation technique. Iterative computer simulations have been developed to accurately predict the stress-strain response of the grades.

It is important to provide designers and engineers with measurements of toughness. Within the work, recently developed test methods (Linear Elastic and Elastic Plastic fracture mechanics) were applied. Testing was performed at high strain rates to investigate impact resistance and at slower strain rates to produce controlled crack propagation. Polymeric materials are viscoelastic solids. Their propensity to anelastic and plastic deformation is reduced when they are tested at high strain rates and/or at low temperatures. Fracture surface morphology was studied to identify the toughening mechanisms which operate in these materials within different loading regimes.
A project which is intended to cover the broad spectrum of the mechanical properties and characteristics of a short fibre filled thermoplastic material, by its very nature will include many varied topics of study. The most influential parameters for any particular material will be the levels and properties of the component materials. It is a description of these base materials which will commence this chapter. Compounding and fabrication of the composite material designates the spacial and physical nature of the individual components within the final moulding. Understanding the fabrication route and its effect upon the microstructure of the final component is essential for a later explanation of the mechanical properties.

Arguably the most important chemical/physical micro-characteristic of any composite is the interface between the fibre and the matrix. It is the interface which transfers the load from the matrix into the fibre, and hence determines how the fibre will be stressed. The manner by which a fibre reinforces a composite is the basis for any models of composite stiffness and strength, and these are also discussed.

The most unusual feature of the family of material grades under study within this work, is the inclusion of a rubber phase. As will become apparent later, this addition is made to enhance the impact properties of the material. The final section of this chapter is concerned with discussing the merits of different fracture toughness tests for measuring an absolute value to quantify this property.
2.1. FABRICATION

Fabrication of short fibre reinforced thermoplastic materials requires the execution of a number of discrete steps. Firstly the constituent elements need to be weighed and roughly dry mixed. Thorough dispersion, fibre attrition and interfacial bond development between the composite elements occurs during extrusion compounding. Finally the chopped extrudate is injection moulded into the component shape.

2.1.1. Extrusion Compounding

The first stage of thermoplastic commodity fabrication is production of a quasi-homogeneous mixture of the component materials, i.e. polypropylene, rubber, glass, stabilisers and coupling agents. It is essential that effective blending of the component materials occurs during extrusion, as later processing has only a limited mixing effect.

Melt-blend extrusion consists essentially of a rotating screw conveyor carrying cold plastic pellets or powder forward and compacting it in the compression section with heat from external heaters and from the friction of viscous flow. Glass fibre is added to the base polymer as loose chopped fibre or continuous roving. It is drawn into the barrel by the rotating screws and broken up by shear of the molten material around them. The pressure is highest at the point where the plastic enters the die that shapes the extrudate. The screen pack or breaker plate between the screw and the die filters out dirt and unfused
polymer lumps. When thermoplastics are extruded, it is necessary to cool the extrudate lace below the crystallisation temperature, $T_c$, in order to gain dimensional stability. This is done by running the lace through a tank of water, or simply air-cooling, depending on the heat capacity of the polymer blend. The continuous lace of extrudate can be chopped before cooling at the die face, or after cooling at the end of the cooling baths. The granules are dried thoroughly prior to being fed to an injection moulding machine.

2.1.1a. Fibre Attrition and Dispersion

Extrusion compounding has been shown to give good wetting and distribution of fibres; hence it is used widely in the large scale production of fibre reinforced injection moulding granules. The major disadvantage of this type of production route is the high level of fibre attrition associated with the large shearing forces within the melt. However, a reduction in the level of shear would result in a poor dispersion of fibres.

The balance between fibre dispersion and fibre attrition has been the object of much study. Krenchel\textsuperscript{1} showed that relatively small compressive stresses applied to non-woven glass fabric gave rise to fibre breakage. Bader and Bowyer\textsuperscript{2} have shown that the level of shear and fibre attrition going on around the screw is controlled by the processing variables. Lunt and Shortall\textsuperscript{3} likewise reported that fibre degradation is dependant on screw speed and diameter. Their work concluded that the majority of fibre breakage occurs in a small region along the extruder barrel.
and that, in steady state operation, it is the region of the melt pool which is associated with this effect.

Johnson and Lunt\(^4\) postulate three mechanisms by which fibre length can be reduced during the compounding operation. These are:

i) by monofilaments bending around the high curvature surfaces of other filaments;

ii) by the turbulent motion of the polymer melt producing sufficient amounts of stress in a filament to cause tensile failure;

iii) by the film of sizing material between adjacent filaments breaking down under conditions of high shear - a phenomenon that results in a contraction of the high modulus glass surfaces and consequently leads to crack propagation through the filaments.

Similarly the results of Gupta et al\(^5\) indicate that during extrusion of glass fibre-filled polypropylene in single-screw plasticising extruders, the breakage of fibres occurs predominantly in the melting zone at the solid-melt interface. The fibre attrition is visualised as a two stage phenomenon. First the fibres, which are exposed by the surface of the granules melting close to the barrel wall of the extruder, interact with the flowing melt and experience a bending moment which can result in fibre breakage. The broken pieces flow with the melt and can experience further breakage due to post-buckling deformation. A model was developed to predict this attrition\(^6\).
Essentially it determines the bending moment experienced by a single fibre, anchored at one end, being subjected to drag forces produced by the flow of the polymer past it. For fibres which are free to move in the molten polymer, buckling introduced by the shearing motion of the molten polymer produces attrition as is predicted by the Forgas and Mason model

Fibre can be added to the extruder as a dry-blend into the hopper, or feeding the fibres into the melt at some distance along the barrel. Franzen et al observed that for both systems the bulk of the fibre degradation process occurred within a relatively short period after introducing the blend into the compounding extruder. Additional fibre damage was noted to occur in the exit orifice of the compounders as well as in the injection moulding step.

The fibres in the extrudate have a maximum length designated by the largest dimension of the extruded granules. However, fibres of that length are very seldom observed in uniform dispersions, which generally contain a range of fibres from the maximum possible length down to fracture fragments whose lengths are of the order of a few fibre diameters.

2.1.2. Injection Moulding

The process of producing complex shaped components by injection moulding was first devised and used for unfilled thermoplastics. The process is now successfully used with fibre filled materials, albeit with a few alterations due to the different physical nature of the filled materials.
During injection moulding the compounded extruded granules fall from a hopper into the channels of a single rotating screw which carries the granules forward into a heating zone where they melt and become compressed. The molten material is stored in a section of the barrel in front of the screw and behind the mould. The screw is forced forward in a plunger like manner, forcing the molten material through a narrow sprue into the mould cavity. The screw may remain forward during freezing of the moulding. It then returns and threads its way back to the rear of the barrel. It is in this part of the cycle that the screw is invaluable because it increases heat transfer at the walls and also causes considerable heat by the conversion of mechanical energy into heat in an adiabatic system. When the finished part is ejected from the mould, the entire cycle is repeated.

To produce optimal conditions for moulding, rheology, heat conductivity and fibre attrition need to be considered. Typical processing conditions are set out below:

i) Low injection speed:
   - prevents fibre breakage through the sprue;
   - controls fibre orientation distribution.

ii) Low screw speed and back pressure:
   - minimises fibre breakage.

iii) Melt temperature maintained at the high end of a range recommended for unfilled thermoplastics:
   - keeps melt viscosity low;
   - assists in reducing premature solidification.

iv) Long mould cooling times:
   - ensures moulding dimensions are stabilised;
- avoids voids.

v) Screw torque:
- as a result of the reinforcement, the density and viscosity of the material are increased and thus a greater torque is required by the screw, as compared to that for unreinforced material. However, reducing the volume fraction of the matrix material results in a reduction in the energy required to ensure plasticisation of the matrix. The two process act against each other.

vi) Screw size and injection pressure:
- due to the higher viscosity of the fibre filled material, the injection pressures must be raised. Modern injection moulding machines are designed for a selection of different size screw diameters for each injection unit. For processing short fibre reinforced materials, the smaller screws with higher injection pressures are preferred.

vii) Clamping pressure:
- due to the higher viscosities the internal pressures are higher than in non-reinforced materials. Clamping units for processing reinforced materials must be designed to take high internal pressures with very little bending.

The resultant wear on the screw from the short fibre reinforcement is influenced by:
- type of fibre;
- amount of fibre reinforcement;
-type of polymer matrix;
-processing conditions.

It has been found by Eckardt and Munschek\textsuperscript{9} that the most abrasive effect on the screws is in the compression zone. This is due to the fact that in the high pressure compression zone, the viscosity of the material remains high. The fibres can therefore attack the screw and the barrel. To reduce abrasion the screw and barrel can be ion-nitrided and the flights of the screw worst affected can be armoured.

2.1.2a. Molecular Orientation

During moulding the molten polymer undergoes large shear and extensional flow which, if frozen in, may lead to molecular orientation in the final product. The crystallisation of the polymer may also be affected by the melt flow and by the presence of fibres: for example, epitaxial crystallisation of polymer molecules has been observed around carbon fibres. It is therefore not always correct to assume that the matrix is isotropic and homogeneous. Kantz et al\textsuperscript{10} observed a skin-core crystallite morphology in unreinforced polymer injection mouldings. A correlation was found between the skin thickness and the mechanical properties of the test-bar mouldings.

Schmidt\textsuperscript{11} went further and by use of a colour tracer technique, showed the skin-core structure to be a direct consequence of the unsteady flow conditions going on within the mould cavity. More precisely, the splitting melt front arises
as the melt enters the mould through a constricted gate. It then diverges and decelerates, thus inducing these effects. Schmidt noted that the surface layer was highly orientated and non-spherulitic for crystalline polymers.

2.1.2b. Fibre Orientation

The effects of matrix crystallite orientation are usually insignificant when compared with those of fibre orientation. Melt flow during mould filling results in orientation of the fibres. This will vary from one part of the material to another depending on the way in which mould filling occurs. Thus, the orientation distribution depends upon the position of the gate and on the process conditions, since these affect the flow properties of the fibre-filled melt. Fully three-dimensional random distributions of fibres are not expected, and because of the orientation distribution variation within the material it is impossible to obtain experimental data which is entirely characteristic of random fibre material.

Layer structures are often observed in the through thickness sections of mouldings$^{12,13,14}$. The extent of this layering can be influenced by mould design and processing parameters. Bright et al$^{15}$ moulded short fibre reinforced polypropylene (SFRPP) into a two cavity mould to show that fast injection speed, slow constant injection speed and stepping the injection stroke gave rise to differences in fibre orientation. Slow injection speed produced a core region (which was transverse to that observed at fast injection speeds) in which the fibres were
aligned in the melt flow direction and were thicker than for fast injection strokes. These mouldings also contained a distinct fibre free region surrounding the core which was absent in faster injected shots. Consequently the skin region was thicker in fast injected mouldings. Malzahn and Schultz\textsuperscript{16} proposed that the frequent observation that fibres in the core of a plaque lie predominantly in the plane of the plaque and perpendicular to the mould fill direction (MFD) whilst at the edges they lie in the MFD, is due to a squeeze flow effect. This is caused by the prolonged ram pressure used to suppress shrinkage on freezing. Similar observations were made by Singh and Kamal\textsuperscript{17}. Surprisingly, a layer with random fibre orientation was always observed in the vicinity of the wall. Also a layer was observed below the surface with a low concentration of fibres.

In a study by Bright and Darlington\textsuperscript{18} it was shown that mould geometry plays a far more significant role in fibre orientation distribution (FOD) than process variables. A more complex mould filled with short glass fibre reinforced polypropylene was used in this series of experiments. The experiments emphasised that real commercial components inevitably contain flow peculiarities which are not observed in simple moulds, and that the flow singularities often give rise to the weakest regions. The most common of these is the 'weld' or 'knit' line, which is produced where two approaching melt fronts, caused by multiple gating or projections within the mould, meet. There is insufficient shear where the melt fronts collide to allow the fibres to straddle the two solidifying fronts and consequently a plane of weakness is produced. Compositions
reinforced with flakes and fibres show the greatest loss in strength. This is due to flow induced filler orientation in the weld line zone\textsuperscript{19}. Hogg\textsuperscript{20} investigated the impact and indentation properties of short fibre reinforced polypropylene containing a central weld line. Slow indentation testing could not distinguish between discs with or without a central weld line whereas instrumented falling-weight impact tests produced different failure modes and significant changes in the amount of energy absorbed between specimen subsets.

It should also be noted that different base polymers (e.g. polypropylene and nylon 66) may give different patterns of fibre orientation in the same mould geometry\textsuperscript{21}.

The fibre orientation distribution in a moulded part cannot be predicted with any certainty at the design stage, except in the simplest of geometries. Even if it were possible to predict the exact FOD throughout the moulding, the complexity of any stress analysis required to handle the resulting anisotropy and inhomogeneity would in most cases be impracticable. Hence, when components are designed to comply with strength and stiffness criteria, the use of lower bound tensile strength data with a "maximum stress" failure criterion may in many instances lead to over-design.

2.1.2c. Shrinkage, Warping and Voiding

General defects found to be present in injection moulded components include shrinkage, warpage and voiding:
i) Shrinkage is common in unreinforced mouldings and is a result of the contraction of the polymer upon freezing and cooling. This problem can be reduced by retaining a residual pressure on the polymer during cooling; the pressure being dependent upon the compressibility of the polymer. In reinforced polymers the ability to apply a back pressure during cooling is very limited. The fibres increase the heat conductivity, and the reduced volume fraction of polymer reduces the latent heat of the system resulting in the sprue freezing off soon after the mould is filled. Cloud and Wolverton\textsuperscript{22} studied shrinkage and warpage of glass fibre and glass bead filled thermoplastic matrices. They observed that as the level of reinforcement was increased the degree of warpage increased, but the degree of shrinkage decreased.

ii) Wetherhold, Dick and Pipes\textsuperscript{12} observed an asymmetrical difference in the skin thickness of mouldings. They proposed that this is caused by different temperature gradients in opposite walls of the mould. This resulted in the observed asymmetric warpage, which is particularly pronounced in thicker cross-sections. Mascia and Speck\textsuperscript{23} observed that warpage became more pronounced as mould temperature increased. They suggest the elimination of warpage by encouraging a differential mould wall temperature. The warp itself, they attribute to differing molecular orientation and degree of crystallinity in each moulding surface. It is thought that there is a close relationship between fibre and molecular orientation, and that annealing treatments moderate the warping by relaxing molecular orientations and promoting crystallisation.
iii) Voiding is a problem particularly associated with short fibre reinforced thermoplastics. After the surfaces of the moulding freeze, the central core cools and contracts but is restricted by the stiffer surface layers. The central core then voids as it is unable to sink as would an unfilled moulding. Voiding weakens the moulding, and is particularly undesirable as it is clearly observable in finished components moulded in translucent matrix materials such as polypropylene ornylons. This type of defect can be overcome by raising the mould wall temperature and prolonging the injection stroke. This results in the surface of the moulding staying molten and pliable for longer thus allowing prolonged feeding of the core and controlled sinking.
2.2. MICROSTRUCTURE

2.2.1. Polypropylene

Polypropylene is a polyolefin which can be produced in isotactic, syndiotactic, or atactic form. The crystallisability of isotactic polypropylene makes it the predominant form to hold properties of commercial interest.

Isotactic polypropylene is essentially a linear, semi-crystalline polymer, with a melting point of 165°C. Polypropylene crystallizes with a helical configuration in which alternate chain bonds take trans and gauche positions. The helix has exactly three units per turn. The crystals nucleate and grow as the temperature drops in the mould: thus direction of heat flow is likely to influence the crystal morphology. The molecular weight of the polypropylene will strongly influence the final extent of crystallinity, and, in the presence of unmelted crystallites or impurities acting as nucleating sites, will alter the crystal size. Furthermore, the mould wall itself can provide additional nucleation sites for crystal growth, as, in a composite, do fibre surfaces.

The poor thermal conductivity of unreinforced polymers influences the cooling rates and gradients within the moulding, particularly in the core. This produces the skin-core molecular orientation observed by Schmidt, and discussed in the previous section.

Investigations conducted by Bowman et al. into the relationship between processing conditions, microstructure and
mechanical properties of injection moulded semi-crystalline thermoplastics showed the existence of a strong correlation between microstructure, crystalline texture and processing conditions.

In the process of melting during fabrication there will inevitably be some level of degradation occurring to the molten polymer, due to oxidation with the atmosphere. This is likely to affect the morphology of the material as the molecules become shortened and different structurally.

2.2.2. Fibre Reinforced Polypropylene

Of the commonly used polyolefins, polypropylene has a significantly high crystalline melting point. Compared with other polymers, this property, combined with its low density, chemical and environmental stability, commercial availability, relative cost, and wide processing range make it the only polyolefin bulk polymer with any high temperature pretensions in common usage.

The introduction of fibres into the system affects the matrix microstructure, both by their presence and their influence on the surrounding matrix. To this are also added the microstructural changes resultant from the different processing conditions necessary for injecting fibre reinforced polymers. As previously discussed in section 2.1.2 the addition of glass fibre to the molten polymer will effect the melt viscosity and hence alter its rheological behaviour.
The thermal conductivity of glass fibres is far greater than that of the surrounding matrix. Each individual fibre will act as a heat sink for its encompassing matrix and thus provide nucleation sites and a thermal gradient encouraging crystallite growth. Bless et al.\textsuperscript{25} prepared nylon reinforced with carbon fibres using an in-situ polymerisation process. An epitaxial crystal growth of nylon on the surface of the fibres was reported. This effect, sometimes referred to as transcrystallinity, has been investigated further by Burton and Folkes\textsuperscript{26}. Using a hot stage microscope, they studied the solidification behaviour of a range of nylon compounds. Transcrystallinity was induced in fibre composites for which there was an epitaxial relationship between the fibre surfaces and nylon crystallites. Using polarised optical microscopy, it was found by Tan, Kitano and Hatakeyama\textsuperscript{27}, that the nucleation of polypropylene started at the crossing point of two or more carbon fibres. Although carbon and Kevlar composites demonstrated transcrystallinity, the phenomenon has not been observed in the glass filled composites, even when additional nucleating agents are applied to the fibres. It is believed that transcrystallinity is caused by a mismatch in the thermal coefficient of expansion between the matrix and the fibre. For systems with a high level of mismatch, i.e., carbon and Kevlar fibres in nylon, as the blend cools there is a thermal contraction mismatch at the fibre-matrix interface which initiates transcrystalline nucleation. For systems which do not possess this mismatch, for example glass fibres in polypropylene or nylon, initiation does not occur. Thomason\textsuperscript{28} has
demonstrated this effect by placing a glass fibre in molten polypropylene on a hot stage microscope. Nucleation will not initiate until the fibre is physically drawn a small distance through the melt. The friction at the interface induces transcry stalline growth instantaneously.

Observing transcry stallinity on a hot stage microscope will not represent the true crystallographic nucleation and growth processes going on within a mould because the massive flow found therein is not represented. This may affect the level of transcry stallinity in two ways: firstly, by affecting nucleation at the fibre surface; and secondly, by influencing nucleation in the matrix. Maxwell et al\textsuperscript{29} showed that flow actually increases the rate of nucleation in a polymer. This would impede transcry stalline growth due to the competing spherulite growth in the matrix.

Investigating the effect of processing variables on the microstructure of injection moulded short fibre reinforced polypropylene, Singh and Kamal\textsuperscript{17} observed that the crystalline structure of the injection moulded parts change from the skin to the core. This is due to the thermo-mechanical history experienced in each region. Crystalline orientation is low, with a maximum near the skin and random or slightly transverse orientation in the core.

2.2.3. Rubber Particle Dispersion

Another type of composite is the polymer "alloy". Two polymers of marginal compatibility and widely differing moduli
will act in a similar manner to glass and polymer, ie having a rigid, load-bearing element and at the same time a softer, energy-absorbing element.

Rubber is dispersed in the polymer during extrusion compounding. In long time-scale tests such as ordinary tensile testing, the rigidity and strength of pure base polymer are only reduced by a proportion exemplified by the simple rule of mixtures approximation. However, in a rapid test, such as the Izod impact test, the rubbery portion will deform, absorbing much more energy than the more rigid polypropylene matrix.

The ability of the rubber to toughen the polypropylene effectively is governed by a number of factors as described by Hobbs et al\textsuperscript{30}:

i) Rubber concentration;

ii) Size and dispersion of rubber particles;

iii) Level of interfacial adhesion;

iv) Inherent matrix ductility;

v) Shear modulus of the rubber;

vi) Glass transition of the rubber;

vii) Craze initiation stress and shear yield stress of the matrix.

In a theoretical evaluation, the stress distribution inside a spherical inclusion embedded in an otherwise homogeneous matrix was discussed by Liu and Nauman\textsuperscript{31}. They concluded that the need for strong interfacial bonding depends on the stiffness ratio of the constituents. Stress calculations inside a soft inclusion, (simulating rubber) show that Van der Waals adhesion
can be sufficient to relieve the high localized stresses in the matrix. Therefore the soft inclusion acts as yield initiator as well as crack arrestor.

Coppola et al\textsuperscript{32} used an etching technique to remove the rubber from the matrix. By measuring the remnant holes they were able to deduce the size of the original rubber particles. They concluded that the rubber particles were spherical in shape and had a narrow distribution of diameters, depending on the type of polypropylene matrix used. For polypropylene with melt viscosity close to that of rubber, the rubber spheres had diameters of 1 micron, but for polypropylene with lower melt viscosity the diameters increased to around 4 microns.

Differential Interference Contrast Microscopy can reveal the size of a second phase and was used by Hodgkinson\textsuperscript{33} to confirm that the rubber particles within the polypropylene reside as spheres of diameter between 1 and 2 microns. He stated that as the level of rubber content is increased, the frequency of the rubber inclusions also increases, although their size does not appear to do so.

2.2.4. Fibre Length Distribution

Incorporation of fibres into a thermoplastic matrix such as polypropylene is achieved by glass and polymer being drawn into the screws of an extruder. The process involves large shear fields which result in fibre breakage due to tensile and bending stresses. Subsequent processing such as injection moulding leads to further attrition and degrees of alignment. Since the
properties of composite materials are dependent on the distribution of fibre lengths and their orientation, it is important to monitor the fibre length and orientation distributions at all stages of processing and fabrication.

If glass is added to the base polymer as chopped fibres during extrusion, very little fibre breakage occurs prior to the polymer melting. If the fibre is added as a continuous roving, a large proportion of fibre attrition occurs in the early flights of the screw before the polymer becomes molten. Barring early attrition of continuous fibres, the majority of fibre attrition occurs in the compression zone of the extruder where the molten polymer is subject to highest shear (Lunt and Shortall\textsuperscript{3}). Further fibre length degradation is incurred during the injection stroke of the moulding cycle. The extent of fibre breakage during flow through constricted gates and runners will depend upon the forces associated with the injection stroke, the matrix material rheology and the filler loading. This effect was studied by Schweitzer\textsuperscript{34} using polypropylene and polyester matrices. He concluded that the level of fibre attrition was highly dependant upon the shear strength of the polymer.

A number of methods have been used to determine individual fibre lengths and the length distributions within a composite. They can be classified broadly into indirect and direct methods. The indirect methods involve the measurement of some physical property of the composite, which is dependent upon the fibre length, such as strength or modulus. This is an imprecise and unsatisfactory approach, although it maintains some value in quality control. In direct methods, the lengths of all the
fibres in a given volume are measured. The very wide distribution of lengths and the large number of measurements which have to be made to obtain a statistically meaningful result make this a difficult task. Firstly the fibres need to be separated from the matrix by burning off or dissolving the resin. There are two main approaches to the measurement stage: dispersion of the fibres in a low viscosity liquid followed by the separation of the different length fractions on a series of sieves (Lunt and Shortall\textsuperscript{3}); or, direct measurement of the length of each fibre by using optical microscope and photographic techniques. The latter method offers the most accurate and satisfactory approach, but it may be tedious and time consuming.

Sawyer\textsuperscript{35} considered the use of automatic image analysis techniques and the possibility of a more rapid evaluation of results. A problem that may arise with this technique is if the image analyser is unable to distinguish between touching and adjacent fibres; also the large range of fibre lengths associated with SFRTP may cause difficulties due to the limited size of the video image. However advanced computer processing allows touching and adjacent fibres to be digitally separated and then measured accurately, Jenkinson\textsuperscript{36}.

2.2.5. Fibre Orientation Distribution

The orientation distribution of short fibres in injection moulded products requires a three-dimensional description and is consequently complex and difficult to determine. The orientation
of fibres crossing a thin section of matrix material can be described in two ways:

i) by the subtended angles of the fibres to predefined cartesian coordinates, figure 2.1;

ii) by the shape (major and minor axes) and the orientation of the ellipse produced by the intersection of the fibre with the plane-sided surface of the section, figure 2.2. It is assumed that the fibre has a circular cross-section.

For method (i) Thomas and Meyer, and Darlington and McGinley determined the angles experimentally by viewing a thin section in transmission using X-ray microradiography; whereas Folkes used optical microscopy. The fibres, which are oriented in three-dimensions in the thin section, appear as a two dimensional array. The first angle can be measured directly from the photograph and the second angle is obtained from the projected length of the fibre and the thickness of the thin section.

For method (ii) the angle between the major axis of the ellipse and the reference axis $y$ is $\alpha$. Angle $\beta$ can be determined from the geometry of the ellipse since:

$$\beta = \sin^{-1}(b/a) \quad (2.1)$$

where $2b$ is the diameter of the fibre ($2r$) and $2a$ is the length of the major axis of the ellipse. The values $\alpha$, $a$ and $b$ can be determined experimentally from the measurements on optical
micrographs of polished sections cut from the composite material. This area of work has been extensively studied by Thomas and Meyer\textsuperscript{37}. This technique does not provide an accurate description for fibres with a small aspect ratio.

A novel technique for measuring FOD has been developed by McGee and McCullough\textsuperscript{39}. A negative film is prepared from a reflected light micrograph of the polished thin section of the specimen. A laser beam is then passed through the negative and the far-field (Fraunhofer) diffraction pattern is recorded. This has brightness proportional to the intensity of the sum of the individual fibres and can thus be related to the proportion of fibres at any particular angle.

Neither of these methods characterises the orientation of the fibres fully because there are two possible positions for a fibre having angles $\alpha$ and $\beta$. Graphical representation of the three-dimensional distribution corresponding to the values of alpha and beta is required. Barrett and Massalski\textsuperscript{40} developed one of the more straightforward methods, the stereographic projection. The values $\alpha$ and $\beta$ are plotted on a planar net with each point on the net representing a single fibre orientation. The population of points can then be mapped out as regions of different orientation densities in a way analogous to a pole figure diagram for grain orientation in polycrystalline metals, figure 2.3. The main problem with this and other similar techniques, is the large number of fibres which need to be sampled so as to be an accurate representation of the whole.

Digital processing of images has been improved by the use of mathematical transforms to detect image features (lines in
this case). The Fourier transform method has been applied to
determine fibre orientation distributions. This method
is extremely accurate as an estimator of FOD, but contains a
great deal of additional information which is not required for
the prediction of elastic properties. The digital two-
dimensional Hough transform offers less detailed
information about the fibre ensemble, but is formulated
principally to detect straight line segments. Thus, the Hough
transform offers a faster and more direct technique.

2.2.6. The Fibre-Matrix Interface

The structure and properties of the fibre-matrix interface
play a major role in the mechanical and physical properties of
composite materials. In particular, the large differences in
elastic properties have to be accommodated through the interface
and thus the load is transferred from the matrix to the fibre.
The ability of the interface to perform this task effectively is
paramount to successful reinforcement. For a satisfactory
transfer of stress between the polymer matrix and the fibre
reinforcement, it is necessary that sufficient adhesive strength
is developed along the fibre-polymer interface. A poorly bonded
region at the interface will cause rupture of the interface at
a very low shear stress. This debonded region will then spread
along the interface until the whole fibre is separated from the
matrix. Around the resulting cavity, high stress concentration
can develop and this will tend to promote overall failure of the
sample.
Surface treatments to the fibre, and chemical modification of the matrix/interface (e.g. by addition of a coupling agent) can promote this adhesive bond. Size is applied to the surface of the glass fibres immediately after they have been drawn. The size fulfils many functions:

i) It acts as a lubricant. The fibres themselves are highly abrasive, and when formed into a bundle or tow they could easily damage the surface of each other. Such surface abrasions reduce the strength of the fibres drastically;

ii) It acts as a binder. It is much easier to handle the fibres in the form of a bundle;

iii) It allows for an electrostatic correction;

iv) It enhances the interfacial bond.

For glass fibre composite systems the chemical bonding mechanisms that involve silane coupling agents or other bifunctional molecules apply generally to thermosetting polymer matrices. This is because the organo-functional group is locked chemically into the cross-linked structure of the resin during the chemical curing reactions which change the resin from a liquid to a rigid solid. This type of chemical bonding will not occur so readily in the case of glass fibres introduced into thermoplastic matrices, because the molecules are already polymerised fully. However, the virgin fibre will still be susceptible to water pick up and to abrasive damage during processing, making it necessary to apply a protective size. It is also important that the surface is wetted out fully by the plastic and that interfacial bonding occurs. The fibres are

27
given a size treatment which includes a silane coupling agent and
a film forming resin. This ensures protection from water
degradation and damage during the injection moulding processes.
For other thermoplastic matrices it is possible to obtain some
chemical bonding if there are reactive side groups on the polymer
molecule.

Reactivity can be enhanced by "tailoring" an unreactive
polymer molecule so that it contains special functional groups
which can bond with the coupling agent. Another approach is to
include chemicals in the size which induce local chain scission
of the molecules near the fibre and allow chemical reactions to
ensure coupling occurs directly into the molecule.

The addition of acrylic acid to polypropylene improves the
adhesion between the matrix and the fibres. The properties of
the modified polypropylene were compared with a plain
polypropylene matrix by Avella\textsuperscript{45}. It was found that for
similar glass-fibre contents a better value of the elongation at
break and superior creep behaviour are observed in the reinforced
blends having the acrylic acid modified polypropylene matrix than
those with the pure polypropylene.

The effect of the silane coupling agent on the morphology
and fracture performance of particulate glass-filled
polypropylene was investigated by Denault and Vu-Khanh\textsuperscript{46}.
Optical microscopy revealed that even at low concentrations the
silane alters the matrix morphology by inducing nucleation at the
filler-matrix interface. This was found to influence the
fracture performance. Subsequent increases in silane
concentration had no further effect. Furthermore, the effect of
the coupling agent depended on concentration of filler. Silane had a detrimental effect at low concentrations of filler. However, at high concentrations of filler, an improvement in the filler-matrix adhesion had a beneficial effect on the fracture performance.

2.2.7. Quantifying the Fibre-Matrix Interface

It is useful to be able to determine quantitatively the interfacial bond strength, \( r_i \). Ostensibly the most direct technique, pioneered by Favre\(^47\), is the single fibre pull-out test. In this test a fibre is embedded in a very thin resin button, and the force required to initiate pull-out is measured. Control of the compliance of the experimental set-up makes it possible to use the technique to determine the frictional parameters of the pull-out process after debonding, as well as the debonding force.

In the single fibre 'composite' test, a single fibre is embedded in a bar of resin having a breaking strain somewhat greater than that of the fibre. When the resin is stressed in tension, the fibre breaks into fragments. When this process is taken to completion, the fibre has broken into average lengths of about \( \frac{2}{3} l_c \), where \( l_c \) is the critical length\(^{(1)}\). For any particular composite system the critical fibre length \( l_c \) is the maximum length a uniform fibre can be but still sustain the maximum composite strain without itself breaking. If \( r_i \) is

\[ \text{(1) The concept of a critical length fibre is discussed in detail in section 2.3.1 with respect to the models of Cox, and of Kelly and Tyson.} \]
assumed to be constant along the fibre surface, simple analysis leads to:

\[ l_C = \frac{\sigma_{fu} d}{2\tau_i} \]  

(2.2)

and hence for a fibre of strength \( \sigma_{fu} \) and diameter \( d \), the interfacial shear stress \( \tau_i \) can be estimated. The value of \( \sigma_{fu} \) appropriate to this test is much higher than the strength measured on a substantial length of fibre. The appropriate value is usually established by using weak link scaling to determine the strength of the fibres of the critical length.

Two other methods have been developed for measuring \( \tau_i \). The microdebond technique uses a small drop of polymer which is placed on a fibre, and the force required to initiate slip is measured. The micro compression technique uses a small indenter to push on the end of a fibre in a polished surface of the composite.

Piggott\(^4^8\) has critically reviewed the principles underlying these tests and has shown that the different tests are measuring different properties:

- The single fibre "composite" test gives mainly frictional data, but does not permit the separate estimation of the coefficient of friction and the interface pressure arising from cure and/or cooling from the moulding temperature. Since weak interfaces tend to give low coefficients of friction the test is effective in giving comparative results;
- The microdebond test has also proved itself useful in providing comparative data;
- The microcompression test also gives useful comparative data;
- The single fibre pull-out test is capable of providing the most comprehensive data since it can, under suitable conditions, give debonding energies, together with frictional coefficients and shrinkage pressures. However, it is the most difficult test to carry out successfully, and if debonding energy is affected by matrix pressure, as some experiments suggest, the energy values obtained in simple tests with no external pressure applied may not always be valid.

Bowyer and Bader, Ramsteiner and Theysohn and Mittal and Gupta have applied the aforementioned techniques to composite systems. The first two methods are based on the fibre pull-out model produced by Kelly and Tyson. Bowyer and Bader's method allows the determination of \( r \) from studies on a composite sample containing discontinuous fibres. It is based on the assumption that \( r \) is constant at all composite strains. The method suggested by Ramsteiner and Theyson is based on a linear relationship between the strength of the composite and the fibre volume fraction. It requires the measurement of the strengths of samples having well-aligned discontinuous fibres with different volume fractions and is therefore necessarily time consuming. In Mittal and Gupta's work they use the experimentally determined stress-strain relation of a single
sample of the composite with fibres aligned along the load axis in order to determine $\tau$ at failure. The method takes into account the variation of the interfacial shear stress during the deformation process and consequently yields very consistent results at all values of the composite strain. In a later piece of research by the same workers\textsuperscript{53} two methods were developed to determine the effect of orientation on the interfacial shear stress in well aligned short glass fibre reinforced polypropylene. The study confirmed that the interfacial shear stress varies linearly with the tensile stress on the specimen. This assumption is more consistent than that of Bowyer and Bader\textsuperscript{2}, namely that the shear stress is constant for all strains. It was also demonstrated that the load transfer from matrix to the fibres in short-fibre reinforced thermoplastics is characterised by a single parameter which varies little with fibre inclination to the load axis. The interfacial shear stress varied between 2MPa (45°) and 9MPa (0°) at 0.01$\varepsilon$, and 3MPa (45°) and 14MPa (0°) at 0.02$\varepsilon$.

The models of Bowyer and Bader, and Cox\textsuperscript{54}, have been further developed by Gupta et al\textsuperscript{55} to predict this interfacial shear stress behaviour. Up to a composite strain of 0.8\%, $\tau$ increases linearly with composite strain for both models. At higher strains, however, the rate of increase is smaller for the Bowyer and Bader model. This is attributed to the nonlinear behaviour of the matrix. At small strains, when both the fibre and the matrix may be assumed to be elastic, the deformation in the matrix is relatively large and thus causes the shear stress to develop at the polymer-fibre interface. With increasing
composite strain within the elastic limit of the matrix, this deformation difference goes on increasing linearly and thus the interfacial shear stress is also linear. At larger strains, the matrix can begin to flow and consequently loses its capability to transfer the stress to the fibre. Therefore the actual value of the interfacial shear stress at large strains is less than that predicted by the model of Cox which assumes the matrix to be elastic and the bond perfect.

Traditional fibre debonding theories upon which the fibre pull-out test is based may only be applicable to certain systems. Leung and Li\textsuperscript{56} consider that classical debonding from the loading end of the fibre only (figure 2.4) is applicable to composites with low fibre volume fraction, low fibre/matrix moduli ratio and high interfacial strength/interfacial friction ratio. For other cases; a two-way debonding theory is proposed where the fibre not only debonds from the loaded end, but also the unloaded end. In a strained coupon containing an embedded discontinuous fibre, high interfacial shear at the fibre end, where the stress is zero, transferring load back into the fibre may lead to debonding. In such a case the fibre will debond from both ends. Under such conditions previous debonding theories are no longer valid.
2.3. TENSILE MECHANICAL PROPERTIES

2.3.1. Reinforcement Using Short Fibre

Calculating the elastic response of a short fibre filled polymer is relatively complex, due to the large number of structural variables present and the difficulty in determining them accurately.

Two of the simplest models for elastic response for continuous fibre lamina lead to the Voigt and Reuss equations, figure 2.5. The Voigt equation represents a well-bonded lamina where there is a continuity of strain at the interfaces and is treated as a parallel arrangement of springs. The Reuss equation has a continuity of stress and is treated as a series arrangement of springs.

Resulting expressions for the lamina moduli are:

\[ E_C = E_m V_m + E_f V_f \quad (\text{Voigt}) \]  \hspace{1cm} (2.3)

\[ 1/E_C = V_f/E_f + V_m/E_m \quad (\text{Reuss}) \]  \hspace{1cm} (2.4)

where;

\[ V_m + V_f = 1 \]  \hspace{1cm} (2.5)

\( V_m \) and \( V_f \) are the matrix and fibre volume fractions respectively.

In an aligned short fibre filled composite with the load transmitted parallel to the fibres, the modulus will lie between the bounds of the Voigt (upper) and Reuss (lower) elements. The equations may be modified further by the inclusion of a length
correction factor, $\eta_l$, to account for the lower efficiency of short fibre reinforcement. The Voigt model becomes:

$$E_C = \eta_l E_f V_f + E_m(1-V_f)$$  \hspace{1cm} (2.6)

Cox\textsuperscript{54} derived the following expression for $\eta_l$:

$$\eta_l = 1 - \left( \tanh \frac{\beta l}{2} \right) / (\beta l/2)$$  \hspace{1cm} (2.7)

where:

$$\beta = \left[ \frac{2G_m}{E_f r^2 \ln(R/r)} \right]^{1/2}$$  \hspace{1cm} (2.8)

$\beta$ expresses the rate of stress build from the fibre ends. $G_m$ is the shear modulus of the matrix, $r$ is the radius of the fibre, and $R$ approximates the inter-fibre spacing. Cox derived this by applying the so-called "shear-lag analysis" to a fibre of length $l$ embedded in a matrix of lower modulus, figure 2.6, with the assumptions that:

i) the matrix and the fibre behave as elastic materials;

ii) the bond between the fibre and the matrix is perfect, which implies that there is no strain discontinuity across the interface;

iii) the interface is infinitesimally thin; and

iv) the matrix close to the fibre has the same properties as the matrix in the bulk form.

If the fibre is well bonded to the matrix, an applied stress on the composite will be transferred to the fibre via the fibre\textbackslash matrix interface, as represented in figure 2.7. The fibre will carry the maximum load at the centre of its length and zero
load at the fibre ends where the shear stress is at a maximum. This shear-lag analysis, as proposed by Cox, showed that for an applied stress on the resin parallel to the fibre the tensile stress along the fibre is given by:

$$
\sigma = \frac{E_f \varepsilon_m \{1 - \cosh \beta (1/2 - x)\}}{\cosh \beta l/2}
$$  \hspace{1cm} (2.9)

Regions at the ends of the fibres carry little load and so the reinforcing efficiency decreases as the average fibre length decreases because a greater proportion of the total fibre length is not fully loaded.

Other similar theories of how the load is shed from the matrix into the fibre have been proposed. Outwater\textsuperscript{57} suggested that the stress in the fibre was dependent upon the coefficient of friction between the fibre and the matrix in a linear manner. Kelly and Tyson\textsuperscript{52} proposed a shear-lag analysis, based upon tungsten fibres in a metal matrix, relying upon a constant interfacial shear stress, where the stress in the fibre builds up linearly from the end of the fibre to a maximum value at its centre, figure 2.8. In their system, the metal matrix would eventually yield around the fibre. This can be likened to frictional slippage in a polymer matrix. The strain in the fibre and the matrix are equal, except over a characteristic length known as the semi-critical transfer length $l_c/2$. This term may be described by an expression which includes the interfacial bond strength, $r$, and the fibre strength. Since the stress is transferred from a plastically deforming matrix to the constraining elastic fibres, it is assumed that for a fibre to
break, the stress transferred across the interface must have exceeded the fibre tensile strength.

If:

\[ x = \frac{l_c}{2} \quad (2.15) \]

then:

\[ l_c = \frac{q_{uf-L_f}}{\tau} \quad (2.16) \]

Kelly described the critical length as the minimum length of fibre required to attain the ultimate fibre strain.

It has recently been shown by Galiotis et al\textsuperscript{58} that the strain at the surface of the fibre can be measured by use of Raman spectroscopy. This technique has demonstrated that the Cox model gives the more accurate prediction of the stress carried by the fibre in a short fibre composite. This latest work has also shown that the fibre ends carry load because the end faces of the fibres are bonded to the matrix. Fukuda and Chou\textsuperscript{59} have developed an analysis based on the shear-lag theory taking the load transfer at the fibre ends into consideration, which they conclude is more important in the early stages of loading.

The maximum possible value of the strain in the fibre is the strain applied to the composite material as a whole so that the maximum stress in the fibre is \( \varepsilon E_f \). According to the Cox model this is never achieved, but very closely approached in long fibres. To approximate this maximum stress the fibre length must be greater than a critical value \( l_c \). Kelly and Tyson\textsuperscript{52} proposed that for fibres longer than \( l_c \) the regions at the ends of the fibre which are not fully loaded have a length \( \frac{1}{2} l_c \). The stress
in the fibre of the Cox model never actually achieves the maximum composite stress, however for fibres longer than the critical length it is very closely approached. Using the Kelly model, the angular fibre loading curve exhibits a clear point signifying the transfer length of the fibre. The smoother loading curve of the Cox model has no such clearly defined point. For such cases the critical transfer length can arbitrarily be taken as the length at which 90% of the maximum load is shed into the fibre, figure 2.9.

An alternative to equation (2.6), which was developed by Halpin\textsuperscript{60} as an extension of the Halpin-Tsai equations\textsuperscript{61}, expresses the longitudinal Young's modulus as:

\[
E_c = \frac{E_m(1+\eta V_f)}{(1-\eta V_f)}
\]  
(2.10)

where;

\[
\eta = \frac{(E_f/E_m)-1}{(E_f/E_m)+\xi}
\]  
(2.11)

and;

\[
\xi = f(l/r)
\]  
(2.12)

\(\xi\) depends on various characteristics of the reinforcing phase such as the shape and aspect ratio of the fibres, packing geometry and regularity, and also on loading conditions i.e, the strain rate. It is necessary to determine \(\xi\) empirically by fitting the stress/strain curves to experimental results.

The properties of an aligned fibre composite depend to a large extent upon the orientation of the fibres relative to the applied stress. As the angle of loading is shifted away from the fibre orientation, the interfacial and matrix shear stresses
become more important because they are required to transfer higher stresses between the fibres.

A number of theories have been used to predict the variation in composite modulus with fibre angle. Tsai\textsuperscript{62} used elasticity theory to solve equations for changes in modulus with fibre angle, figure 2.10. Good experimental agreement was found by Dimmock and Abrahams\textsuperscript{63}, who compared measured moduli with the Tsai predictions using continuous carbon and glass fibre reinforced epoxy resin composites.

Fibre orientations in short fibre reinforced composites are extremely complex. The fabrication route gives rise to fibre dispersions which are themselves difficult to measure or predict. For this reason, predictive mechanical property relationships are generally inadequate because the assumptions upon which they are based are oversimplified.

Where there is a distribution of fibre orientations, the reinforcing efficiency of the fibres is further reduced due to misaligned fibres. Krenchel\textsuperscript{1} included an additional term in the rule of mixtures equation (2.3) to take this into account, thus:

\[ E = \eta_0 \eta_1 E_f V_f + E_m (1-V_f) \]  

(2.13)

where \( \eta_0 \) is the Krenchel orientation factor. The term \( \eta_0 \) is calculated from a summation of the fibre fraction, \( a_k \), oriented in angular increments away from the applied load, \( \theta_k \):

\[ \eta_0 = \Sigma a_k \cos^4 \theta_k \]  

(2.14)
Krenchel\textsuperscript{1} has calculated $\eta_0$ for various fibre orientation distributions. For unidirectional laminae $\eta_0=1$ and 0 when tested parallel and perpendicular to the fibres respectively. $\eta_0=3/8$ for in-plane random fibre distributions and $\eta_0=1/5$ for three dimensional random distributions. The initial modulus may be predicted accurately by this type of relationship. However, this expression overlooks fibre/fibre interactions and stress concentrations which induce interfacial debonding. It is therefore clearly unsatisfactory at higher strains and for composites containing higher fibre contents. Furthermore, in SFRTPs a unique fibre length is not observed. The range of fibre lengths observed in actual SFRTP's gives rise to fibres of different lengths sustaining different loads. To account for this, an iterative summation needs to be performed to account for the contribution of the individual fibre length fractions at the respective composite strains.

2.3.2. Composite Strength Predictions

Strength properties are difficult to model because failure modes are important, although many authors have used a simple rule of mixtures to predict composite strengths\textsuperscript{64,65,66}. In man-made continuous fibre composites the fibres usually fail first. The load is then transferred back into the weaker matrix. The matrix is unable to sustain the whole of the applied load if there is a reasonably high fibre volume fraction, $V_f$. Rapid failure of the composite ensues. In continuous reinforced
composites, this situation leads to a unique failure stress of the composite:

\[ \sigma_{uc} = \sigma_{uf} \cdot V_f + \sigma_m' \cdot V_m \]  \hspace{1cm} (2.17)

where \( \sigma_m' \) is the stress in the matrix at fibre failure stress, \( \sigma_{uf} \). In its simplest form this assumes a unique fibre failure stress. However all fibres contain weaker sections, and it is these weak regions that are statistically more likely to fail first. Weibull statistics may be used to describe the distribution of these regions and hence the strength of fibres thus modifying equation 2.17.

At low fibre fractions, the matrix material is weakened by the presence of the fibres. In this instance, the matrix may be strained sufficiently to cause fibre breakage, but the matrix still strong enough to support the additional load, so that the strength is governed by the matrix, \( \sigma_{um} \):

\[ \sigma_{uc} = \sigma_{um} (1 - V_f) \] \hspace{1cm} (2.18)

The two regimes (2.17 & 2.18) give rise to minimum composite strength when these expressions equate:

\[ V_{min} = \frac{\sigma_{um} - \sigma_m'}{\sigma_{uf} + (\sigma_{um} - \sigma_m')} \] \hspace{1cm} (2.19)

Below \( V_{min} \) fibre failure occurs below the matrix ultimate strength. The work-hardening capacity of the matrix then
determines composite strength. There is good experimental evidence for justification of these concepts$^{64}$.

The variations of composite strength with fibre angle have been the object of much study$^{62,63,64}$. If the strengths of a composite parallel with the fibres, $\sigma_c$, perpendicular to the fibres, $\sigma_t$, and in shear parallel with the fibres, $\tau_c$, are all known, there are several ways of predicting the strength as a function of orientation. The simplest is the maximum stress theory of Stowell and Lui$^{67}$. This model defines three modes of failure for continuous fibre reinforced materials. The maximum stress permitted within each criterion, $\sigma_\theta$, is characterised by the three equations:

$$\sigma_\theta = \sigma_c \sec^2\theta$$  \hfill (2.20)

for failure controlled by tensile failure of the fibres;

$$\sigma_\theta = 2\tau_c \csc\theta$$  \hfill (2.21)

for failure controlled by shear parallel with fibres;

$$\sigma_\theta = \sigma_t \sec^2\theta$$  \hfill (2.22)

for tensile failure normal to the fibres.

The lowest of these failure mode relationships is likely to dominate the composite fracture behaviour. Good experimental agreement has been found for a wide range of materials$^{62}$. 

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Tsai\textsuperscript{62} has shown that in some GRP materials a so called maximum distortion energy theory loosely based on von Mises' yield criterion fits the data more closely, the variation of $\sigma_\theta$ with orientation being:

$$1/\sigma_\theta^2 = \cos^4\theta/\sigma_C^2 + \sin^4\theta/\sigma_C^2 + \cos^2\theta\sin^2\theta \left(1/\tau_C^2 - 1/\sigma_C^2\right) \quad (2.23)$$

This is frequently referred to as the Tsai-Hill failure criterion, and its advantage over the maximum stress criterion is that it is a continuous function rather than the lower bounds of equations (2.20) to (2.22), figure 2.11.

Short fibres in a composite cannot be directly loaded at their ends alone. The stress is transmitted into them by shear forces at the fibre/matrix interface in a manner described by one of the shear-lag theories\textsuperscript{52,54}. The load carried by the fibre builds up from the fibre ends in a manner determined by the shear modulus and shear strength of the interfacial bond. The interfacial shear modulus determines the rate at which load can be transferred into the fibre elastically. If the interfacial shear modulus is low then the rate at which load can be shed into the fibre will be low. The transfer and critical lengths will be long, and the average load carried by the fibre will be lower than for a system with a higher interfacial shear modulus. The interfacial shear strength determines the maximum load that can be transmitted into the fibre. If this is low then an adhesive failure of the interface will result before the ultimate tensile
strength of the fibre is achieved, again reducing the efficiency of the composite.

The mean load carried by the short fibres will be lower than that carried by a continuous fibre in a composite strained to the same level because of the less efficient fibre ends. As the average fibre length becomes shorter the contribution of the fibre ends will become more dominant and the average load carried by each fibre less.

If the fibres are shorter than the critical length, \( l_c \), so that the length available for transfer is less than \( l_c/2 \), the level of stress that can be transferred into the fibre will never reach the fibre failure stress and such short fibres can never be broken by loading the composite. Only when fibres have a transfer length of at least half the critical length can the maximum potential of the reinforcement be approached. If a Kelly-Tyson shear-lag interface is assumed then a fibre of length \( l_c \) will carry an average stress of only \( \sigma_{uf}/2 \). The maximum stress that can be achieved at the midpoint of the fibre length is the fibre breaking stress. The average stress in a fibre of length \( 2l_c \) will be \( 0.75\sigma_f \), and a general expression for the average stress in a fibre of length \( l \), \( \langle \sigma \rangle_f \), is thus:

\[
\langle \sigma \rangle_f = \sigma_f (1 - l_c/2l) \tag{2.24}
\]

The composite strength, calculated from the rule of mixtures, based on the average tensile stress carried by the short fibres all of length \( l \), is therefore:
\[ \sigma_c = \sigma_f V_f (1-l_c/2l) + \sigma_m (1-V_f) \quad (2.25) \]

for \( l > l_c \), ignoring statistical variations in the fibre strength.

When the fibres are shorter than the critical length they cannot be broken by direct tensile stress transfer from the matrix. The average tensile stress in a fibre is determined from a force equilibrium balance of an embedded fibre being pulled-out of a matrix, illustrated in figure 2.12. The maximum fibre tensile force, \((\sigma)_f \cdot \pi d^2/4\), acting across the centre plane is balanced by an interfacial frictional force which must exceed the fibre/resin interfacial bond strength, \( \tau_i \), or the matrix yield stress, \( \tau_{my} \), if a failure other than fibre fracture is to occur. Letting the limiting shear stress simply be \( \tau_i \) (i.e. invoking the more common mode of failure), the force balance at failure will be:

\[ \tau_i \pi d l/2 = (\sigma)_f \pi d^2/4 \quad (2.26) \]

for \((\sigma)_f < \sigma_f \), and the average fibre tensile stress, \((\bar{\sigma})_f \), is therefore:

\[ (\bar{\sigma})_f = (\sigma)_f/2 = \tau_i l/d \quad (2.27) \]

The composite tensile strength, from the mixture rule again, is then for \( l < l_c \):

\[ \sigma_c = (\tau_i l/d)V_f + \sigma_m (1-V_f) \quad (2.28) \]
where $\sigma_{tu}$ is the matrix ultimate tensile strength. Equation (2.28) makes clear the importance of good fibre/matrix interfacial bonding if reasonable strength levels are to be obtained in short fibre composites.

For injection moulded components it is very difficult to predict strengths on the basis of the above equations. Regular arrays of fibres have been assumed in most of the previously discussed micro-models. However, regular arrays of fibres are seldom observed in actual composite micrographs. There is also an uncertainty about the parameters $r_f$ and $l_c$ (which are difficult to measure for conditions appropriate to the real composite), and a lack of knowledge of the effects of processing on the strength of the reinforcement.

Wetherhold\textsuperscript{68} accounted for two further fibre probabilistic aspects: fibre crossover reinforcement, and crack gap bridging by fibres before fracture. The fibre crossover density is proposed as a measure of mutual fibre strengthening, due to providing local reinforcement for stressed fibres. It is possible to have different fibre orientations with the same elastic properties but different crossover densities, hence different strengths. The density of fibre crossovers will depend on fibre orientation, fibre length and number of fibres. Wetherhold shows that the crossover density is a random variable whose average increases roughly as a function of the square of the fibre length, and whose coefficient of variation decreases with increasing fibre length. The fracture mechanism of fibre bridging microcracks is described by probabilistic expressions for fibres whose length exceeds the critical length appreciably.
These additional factors give a more accurate representation of the strengthening effect of fibres in a composite at high strains.

Fukuda and Chou's critical damage zone model considers the distribution function of both the fibre orientation and the fibre length as it varies along the composite. The weakest cross-section of the composite is considered to be a zone of finite thickness where the fibres either bridge the zone or have both ends in that zone. The fibre length and orientation distributions are then used to find the probability of any fibre being a bridging fibre. The stress that the bridging fibres sustain can be predicted by considering the fibre effectiveness for a particular orientation, length, and critical length. Fukuda and Chou consider this value to be representative of injection moulded compound strength.

2.3.3. **Stress-Strain Response**

Bader and Bowyer have attempted to account for the effect of damage to fibres during injection moulding of reinforced thermoplastics by studying the fibre length distributions in mouldings. They used a mathematical model in which contributions to composite strength from fibres of subcritical and super-critical lengths were summed separately over the effective range of lengths. Bowyer and Bader suggested the final expression as:

\[ \sigma_c = \eta(X+Y) + Z \]  

(2.29)
where;

\[ X = \varepsilon_{\text{subcritical}} \tau l_i V_i / 2 \tau_f \]  

(2.30)

\[ Y = \varepsilon_{\text{supercritical}} E_f \varepsilon_c (1 - \{ E_f \varepsilon_c r_f / 2 l_j \}) V_j \]  

(2.31)

and;

\[ Z = E_m \varepsilon_c (1 - V_f) \]  

(2.32)

where \( \eta \) is an orientation factor; \( V_i \) and \( V_j \) are the volume subfractions of fibres with lengths \( l_i \) and \( l_j \), respectively, \( E_m \) is the matrix modulus and \( V_f \) the total fibre volume fraction of fibre. In equation (2.30), the summation extends over all fibre-length intervals below the critical length and in equation (2.31) over all intervals above the critical length.

While applying the equations given above, Bowyer and Bader assumed that the value of the shear stress, \( \tau \), is the same at all values of the composite strain, \( \varepsilon_c \). The second assumption made by them was that the Kelly and Tyson model can be applied at all fibre orientations to the load axis by incorporating an orientation factor, \( \eta \), and thus an imperfectly oriented sample can be analysed by their procedure and a representative value of \( \eta \) obtained. It was later shown by Mittal and Gupta\(^{51}\) that the constancy of \( \tau \), particularly at low values of \( \varepsilon_c \), leads to widely varying values of \( \eta \) for the same sample.

In contrast to Bowyer and Bader's procedure, Ramsteiner and Theysohn\(^{50}\) used well characterised and aligned composite samples with fibres oriented along the load axis. However, they assumed that the volume fraction of supercritical fibres was negligible for their samples and hence \( Y=0 \) in the above equations. The
value of $\tau$ was obtained from the slope of curve between the composite stress at failure and the fibre volume fraction. From macromechanical analysis, Ramsteiner and Theyson were able to correlate the stiffness and strength of aligned fibre samples with different orientations to the load axis, and showed good agreement with the Tsai-Hill criteria$^{71}$.

Mittal and Gupta$^{51}$ used a similar method but developed a linear relationship between the interfacial shear stress and the composite stress, i.e. $\tau=K\sigma_c$. The principal contribution of the equation $\tau=K\sigma_c$ is the proper evaluation of the critical length during the deformation process. The Kelly and Tyson definition of the critical length can now be written as:

$$l_c = \frac{(E_f \tau_f \varepsilon_c)}{K\sigma_c} = \frac{(E_2 \tau_f / K) \cdot (\varepsilon_c / \sigma_c)}{ } (2.33)$$

Hence the critical length is inversely proportional to the secant modulus of the stress-strain curve of well-aligned short glass fibre-reinforced polypropylene sample loaded in tension in the direction of the fibres. On the other hand, according to Bowyer and Bader's procedure, $l_c$ is proportional to $\varepsilon_c$ since $\tau$ is constant.
2.4. TOUGHNESS

Typical engineering reinforced plastics consist of brittle fibres in a weak brittle matrix. Despite the brittle constituents, the resultant composite is surprisingly tough. An understanding of how a material will respond to a defect within its structure is essential to successful service life. Fracture mechanics is concerned with the propagation of defects and the ability of materials to absorb energy during the fracture process.

2.4.1. Linear Elastic Fracture Mechanics

The subject of fracture mechanics has been developed from the basic concept of Griffith. He considered an infinite plate of unit thickness containing a through thickness crack, subjected to uniform tensile stress (applied at infinity). For the case where no work is done by external forces, (the so-called fixed grip condition), the introduction of the crack decreases the elastic strain energy of the plate. When the elastic energy release due to a potential increment of crack growth outweighs the demand for surface energy for the same crack growth, the crack will propagate unstably. Griffith's energy balance for fracture gives the failure stress as:

\[ \sigma = \left( \frac{2Ey}{\pi a} \right)^{1/2} \]  

(2.34)
2\gamma is the increase in fracture surface energy owing to infinitesimal crack growth. The strain energy release rate, G, is closely related to 2\gamma and represents the elastic energy per unit crack surface area that is available for infinitesimal crack extension. Propagation of a crack occurs when G reaches a critical value G_c. In 1948 Irwin\textsuperscript{73} suggested that the Griffith theory for ideally brittle materials could be modified and applied to both brittle materials and metals that exhibit plastic deformation. A similar modification was proposed by Orowan\textsuperscript{74}. The modification recognised that a material's resistance to crack extension is equal to the sum of the elastic surface energy and the plastic strain work accompanying crack extension. Consequently, equation (3.34) was modified to:

\[
\pi \cdot \frac{a^2}{a} = 2(\gamma_e + \gamma_p) = G_c \quad (3.35)
\]

For relatively ductile materials \( \gamma_p > \gamma_e \), i.e. the surface energy can be neglected. Although Irwin's modification includes a plastic energy term, the energy balance approach to crack extension is still limited to defining the conditions required for instability of an ideally sharp crack.

Irwin later went on to develop the so called "stress intensity" approach to fracture. Using elastic stress analysis he described the stresses in the vicinity of a crack tip in a cylindrical polar coordinate system in terms of a parameter, K, which governs the magnitude of the elastic stress field. It is called the stress intensity factor.
The general form of the stress intensity factor is given by:

\[ K = \sigma(\pi.a)^{1/2}.f(a/w) \]  \hspace{1cm} (2.36)

where \( f(a/w) \) is a dimensional parameter that depends on the geometry of the cracked specimen.

Irwin then demonstrated that if a crack is extended by an amount \( da \), the work done by the stress field ahead of the crack when moving through the displacements corresponding to a crack of length \( (a+da) \) is formally equivalent to the change in strain energy \( Gda \). Thus the achievement of a critical stress intensity factor, \( K_c \), is exactly equivalent to the Griffith-Irwin energy balance approach, which requires the achievement of a stored elastic strain equal to \( G_c \).

The parameter governing fracture may therefore be stated as a critical stress intensity, \( K_c \), instead of a critical energy value \( G_c \).

Irwin's elastic stress distribution in the vicinity of the crack tip shows that as the crack tip radius tends to zero the stresses become infinite. Since structural materials tend to deform plastically at high stress, there will be a plastic zone surrounding the crack tip. A plastic zone at the crack tip toughens the material in three ways:

1) It dissipates the high local stresses to some degree, thereby reducing the severity of the excitation;
ii) It absorbs more energy than would be required for growth of a brittle crack of the same length;

iii) It dissipates energy in the sense that the energy absorbed in it is not recoverable, as it would be from an elastic deformation, and therefore is not available to support crack growth.

Irwin argued that the occurrence of plasticity makes the crack behave as if it were longer than its physical size. He showed that the crack may be viewed as having a notional tip at distance \( r_y \) ahead of the real tip. The increased effective length of the crack may be interpreted alternatively as the so-called "crack tip opening displacement". An alternative analysis, attributed to Dugdale\(^7^5\), regards the plastic zone as a wedge-shaped extension to the crack, figure 2.13.

The profile of the crack tip plastic zone changes through the thickness, reflecting the transition from plane stress at the surface to plane strain in the central plane of a thick specimen. When \( r_y \) is large compared to the plate thickness, yielding will take place freely in the thickness direction but when it is small such yielding is constrained by the surrounding material. In the former case, the stress state is plane stress and \( r_y \) has to be much smaller than the thickness if a state of plane strain is to exist through the greater part of the thickness. It seems to have been established that a plastic zone radius less than of the order of 0.025 times the thickness does not suppress plane strain fracture in metals, but the situation is not well documented for
plastics. Plastic zones several millimetres across are common and test geometry thicknesses of 10cm are therefore necessary for some materials if plane strain is to dominate in tests at room temperature. This condition is very difficult to satisfy and one practical expedient is the use of low temperatures, which generally reduce $K_c$ and raise $\sigma_y$ thereby reducing the size of the plastic zone. However, a measure of fracture toughness at a particular temperature and straining rate may say very little about the property under another set of conditions, since the failure processes which govern may not be the same.

In order to obtain reliable stress intensity factor and strain energy release rate ($K_{IC}$ and $G_{IC}$) data of polymeric materials workers have developed experimental methods working at high strain rates at room temperature. This has the effect of minimising viscoelastic losses and plastic deformation in a similar way to lowering the temperature, but is also difficult to extrapolate to other conditions. Instrumented pendular or falling weight impactors are used to break notched specimens in three point bending. Casiraghi et al $^7$ found that for their conditions the LEFM criteria were satisfied for polyvinylchloride, but not by polypropylene. Using a Charpy instrumented pendulum Greco and Ragosta $^7$ found that $K_{IC}$ and $G_{IC}$ increase markedly with increasing molecular weight. The opposite trend was observed with increasing crystallising temperature.
2.4.2. *Application of Fracture Mechanics to Composites*

The use of any classical fracture mechanics approach to the toughness of composite materials is limited by the heterogeneity and complex nature of the energy absorbing processes occurring at and away from the crack front.

Composites are heterogeneous materials which contain uneven stress distributions under load. As a crack propagates through the material its stress field will change depending upon the proximity of fibre and matrix. Cooper and Kelly\textsuperscript{78} observed this effect as a crack tip zone size which alternated between small and large dimensions as the crack propagated through brittle fibres and ductile metal matrix.

Interpretation of data from fracture toughness tests can lead to ambiguity. Phillips and Tetelman\textsuperscript{79}, and Beaumont and Phillips\textsuperscript{80} investigated the correlation between LEFM and work of fracture type tests. The discrepancies in results between the two tests occur primarily because the fracture mechanics data are calculated from the crack initiation; while the work of fracture results includes crack propagation contributions.

Under some circumstances, linear elastic fracture mechanics can be used directly. Typically this applies to brittle composites with high interfacial shear strengths and brittle matrices. In such materials the crack face is planar and the damage zone size small (e.g. <1mm). The effects of composite heterogeneity are therefore not apparent. When these conditions are deviated from, the assumptions of LEFM no longer hold, and can no longer be applied. Tough composites damage in a complex
manner and single crack failure may not occur. The damage zone may also be large, and for most testing geometries it will be outside the range of the plane strain testing criteria.

2.4.3. Elastic-Plastic Fracture Mechanics

Linear Elastic Fracture Mechanics (LEFM) was originally developed to describe crack growth and fracture under essentially elastic conditions, as the name implies. However, such conditions are met only for plane strain fracture of high strength metallic materials and for fracture of intrinsically brittle materials like glass, ceramics, rock and ice.

Later it was shown that LEFM concepts could be slightly altered in order to cope with limited plasticity in the crack tip region. In this category fall the treatment of fracture problems in plane stress, eg the R-curve concept. Nevertheless, there are many important classes of materials that are too ductile to permit description of their behaviour by LEFM: the crack tip plastic zone is simply too large. For these cases other methods must be found.

Elastic-Plastic Fracture Mechanics (EPFM) methods significantly extend the description of fracture behaviour beyond the elastic regime, but they too are limited. Thus EPFM cannot treat the occurrence of general yield leading to so-called plastic collapse.

The J integral concept is based on an energy balance approach. It was first introduced by Rice\textsuperscript{81}. 

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The Griffith energy balance of an elastic, remotely loaded cracked plate can be stated as:

\[ U = U_o + U_a + U_\gamma - F \]  \hspace{1cm} (2.37)

where;

- \( U \) = total energy content of cracked plate,
- \( U_o \) = elastic energy content of the loaded uncracked plate (a constant),
- \( U_a \) = change in the elastic strain energy caused by the introducing the crack in the plate,
- \( U_\gamma \) = change in elastic surface energy caused by the formation of the cracked surfaces,
- \( F \) = work performed by external forces.

Previously this has only been considered for linear elastic behaviour. However, equation (2.37) will remain valid as long as the behaviour remains elastic. It need not be linear. Thus, even for a material exhibiting a nonlinear load displacement response the equation is valid.

An important consequence of this extended validity of equation (2.37) is that under certain restrictions this nonlinear elastic behaviour can be used to model plastic behaviour of a material. This is known as the deformation theory of plasticity. The main restriction is that no unloading may occur in any part of a body since for actual plastic behaviour the plastic part of the deformation is irreversible.

As long as equation (2.37) remains valid it is possible to derive an instability condition leading to:
\[
\frac{d}{da} (F - U_a) \rightarrow \frac{dU_Y}{da} 
\]

Furthermore, a nonlinear elastic equivalent of \( G, J \) can be defined:

\[
J = \frac{d}{da} (F - U_a) \quad (2.39)
\]

For elastic behaviour \( J = G \) by definition.

Returning again to equation (2.37) the potential energy \( U_p \) can be defined as:

\[
U_p = U_0 + U_a - F
\]

i.e.
\[
U = U_p + U_Y \quad (2.40)
\]

Thus \( U_p \) contains all the energy terms that may contribute to nonlinear elastic behaviour, while \( U_Y \) (the change in elastic surface energy owing to crack extension) is generally irreversible. Since \( U_0 \) is a constant, differentiation of \( U_p \) gives:

\[
\frac{dU_p}{da} = \frac{d}{da} (U_a - F) = - \frac{d}{da} (F - U_a) \quad (2.41)
\]

From equation (2.39) it is seen that by definition:

\[
J = - \frac{dU_p}{da} \quad (2.42)
\]

\( dF/da \) represents the energy provided by the external force \( F \) per increment of crack extension. \( dU_a/da \) is the increase of elastic energy owing to the external work \( dF/da \). The quantity \( dU_p/da \) therefore represents the change in stored energy. A decrease in stored energy, \(-dU_p/da\), means a release of crack driving energy.
In order to provide the energy $dU_y/da$, for an increase in crack surface by $da$.

During crack growth the newly formed crack flanks are completely unloaded from stresses as high as $\sigma_{ys}$ (or considerably higher, $C\sigma_{ys}^{(2)}$, in the case of plane strain). Therefore in principle $J$ should be expected to apply only up to the beginning of crack growth. Despite this, some success in using $J$ to characterize crack growth has been claimed.

Huang and Williams\textsuperscript{82} followed a multiple specimen technique similar to ASTM E813 to measure $J$ for three toughenednylons. The notch tip radius was fairly large (5-12$\mu$m), and was not sharpened with a scalpel, for example. Their results were self consistent but may be an over estimate due to notch blunting at the crack tip. Values between 15 and 30 kJm$^{-2}$ were obtained. This compared well with values obtained for polypropylene by Hashemi and Williams\textsuperscript{83} of 15.5 kJm$^{-2}$.

2.4.4. Observations of Fibre Toughening Mechanisms

Mandell et al\textsuperscript{84} attempted to measured stress intensity, $K$, values for a range of thermoplastic matrix materials reinforced by carbon and glass fibres. It was shown that the fibre length distribution influenced $K$ values to a greater extent than the actual matrix type. The fibre orientation distribution gave rise to variations in $K$ of a factor of two. They also observed that the fracture took a "fibre avoidance" propagation route. As this

\textsuperscript{(2)} In situations of high constraint, e.g. cracks in thick sections, the effective yield strength will increase to $C\sigma_{ys}$, where $C$ is the plastic constraint factor.
must be the most energetically favourable route, it suggests that a larger proportion of long fibres should lead to an increase in the toughness as it decreases the likelihood of the fibre avoidance path. This contrasts with the Cottrell and Kelly models which predict that fibres of the critical length are most desirable for fibre pull-out toughening. A balance of fibres of and above the critical length in SFRTP's may therefore give optimum toughness.

Sato et al conducted microstructural investigations into the initiation and propagation of cracks in unnotched SFRTP's. They made three observations:

i) Initially, cracks form at the fibre ends;

ii) These cracks propagate along the fibre-matrix interface;

iii) Finally, cracks growing into the matrix link up and give rise to the catastrophic failure of the composite.

This type of damage accumulation process has been confirmed in many studies. It is generally believed that the fibres within the matrix can act as stress raisers and/or internal notches due to the differing elastic response of the two materials and their interfacial bonding. These mechanisms occur in the equivalent of a plastic zone. However, as the material does not undergo entirely plastic deformation it is more appropriate to refer to this damage region as the "process zone". The "process zone" contains very localised regions of intense plastic deformation at and around the fibre ends, as well as matrix rich regions which undergo little or no plastic deformation.
Contradictory to Mandell et al\textsuperscript{84}, Lhymn and Schultz\textsuperscript{13} observed that fibre fracture, and microcracks from fibre ends, are induced by a "far-field" effect, and not by an immediate field near the crack tip.

It is generally held that crack initiation from fibre ends holds in most cases, but the propagation of the crack is highly influenced by the matrix material.

There are many observed possible energy absorbing processes which occur prior to final crack surface formation and thereby enhance crack growth resistance. Outwater and Murphy\textsuperscript{57} observed that during the fracture of glass-fibre resin composites, the adhesive bond between fibres and matrix was destroyed for some distance on either side of the fracture path. They concluded that this is a result of local Poisson's contraction which, exacerbated by the lateral contraction of tensile stress ahead of the crack tip, initiates fibre/resin debonding. The interfacial shear stress resulting from fibre/matrix modulus mismatch will then cause extension of the debond along the fibre in both directions away from the crack plane. In some circumstances debonding can lead to large scale deviation of the crack tip parallel with the fibres, resulting in effective blunting of the crack tip.

After debonding, the fibre and the matrix move relative to each other as the crack opening continues. Work must be done against frictional resistance during the process.

Fibre pull-out is another energy absorbing process associated with the interfacial shear stress. Cottrell\textsuperscript{88} and Kelly\textsuperscript{64} proposed that the energy contribution arises when the
fibres are restrained by the interfacial shear stresses between themselves and the matrix. The energy required to pull the fibres free of the matrix is termed the "pull-out energy". In an aligned short fibre reinforced composite the work of pull-out will be a maximum when the reinforcing fibres are of exactly the critical length (Cooper\textsuperscript{89}). Helfet and Harris\textsuperscript{90} observed that if the fibres are at an angle to the crack face, brittle ones will fail prematurely without pull-out, but plastically deformable ones will undergo extra work of shearing as they pull-out, contributing to extra toughness. Wells and Beaumont\textsuperscript{91} presented a statistical method whereby various potential contributions to the toughness of a polymer containing short brittle fibres are quantified. The energetics of fibre debonding, fibre fracture and fibre pull-out are predicted from theory. This data are then applied to a model which predicts the cumulative probability distribution of fibre pull-out lengths. The method reveals that toughness increases to a maximum value with increasing fibre length, and good experimental agreement is shown.

Composite toughness can be increased by raising the fibre volume fraction, increasing the fibre diameter, or using stronger fibres. Improving the fibre/matrix bond may not increase the toughness because it inhibits and therefore reduces pull-out, as postulated by Beaumont and Harris\textsuperscript{92}. However, if the matrix is relatively ductile, the fibres are well bonded to the matrix and the stress intensity factor at the crack tip is not sufficient to propagate the crack through the fibre, the crack may be forced to run around the fibre. When pull-out occurs the crack will
have to propagate in a cohesive failure through the matrix around the surfaces of the fibre. This may be more energy intensive than if the failure were an adhesive failure at the fibre surface, and hence it may increase the toughness of the material.

2.4.5. Rubber Dispersion Toughening

Energy absorbing processes occurring during loading of ductile polymers at temperatures at and above that of the $T_g$ of the matrix components are shear flow and craze formation. The crazes contain between 20% and 90% voids, the rest consisting of fibrils oriented in the direction of the applied stress. The shear bands, in contrast, have very little void volume. Unlike actual cracks, both crazes and shear bands are capable of supporting stresses because of the oriented polymer involved. This helps to account for the increased toughness of these materials.

The deformation behaviour in tension of notched specimens of blends of polypropylene with ethylene-propylene rubber was studied as a function of temperature and composition by Chou et al. Stable crack growth was not obtained up to 99% of the maximum stress. However, a hierarchy of failure events was observed. First, there was a gradual occurrence of a fan-shaped damage zone at the crack tip. Then an intense damage zone initiated. Finally, a slow tearing mode of crack growth occurred at the maximum stress and the sample failed. Different mechanisms were active in the growth of the damage zones. In rubber-modified blends microvoids were dominant in the fan zone,
with some crazes close to the intense zone. In the case of unmodified polypropylene, only crazes were observed. In both cases, the crazes coalesced to form a network of deformation bands in the intense zone. Using identical material, Chou et al. investigated the irreversible deformation mechanisms in the region of the ductile-to-brittle transition. At -40°C, the controlling micro deformation process in polypropylene was crazing. In the blends, two kinds of damage zones were observed: a diffuse zone due to voiding at rubber particles and an intense damage zone due to craze-like damage and deformation bands. In general, the size and density of the damage zones increased in a gradual manner through the ductile-to-brittle transition whether examined as a function of temperature, strain rate or blend composition.

2.4.6. Impact Behaviour

Polymeric materials are viscoelastic solids. Their propensity to anelastic and plastic deformation is reduced when they are tested at high strain rates and/or at low temperatures. The reduced deformability causes a formerly tough or highly elastic polymer to respond in a brittle manner. Evidence of this is shown by the impact fracture of natural rubber at liquid nitrogen temperature.

Impact resistance depends strongly on the presence and shape of stress concentrators, on the sample geometry, and on the testing conditions. This makes it very difficult to define and measure a unique material property "impact resistance". Since
the impact behaviour under one condition (eg impact bending) cannot very well be predicted from the behaviour under different conditions (eg falling weight) quite a number of impact tests have been devised. The four best known are the three point bending (Charpy notched and unnotched), two point bending (Izod), tensile-impact, and falling weight tests which have been standardized (DIN 53453, 53448, 53373, 5344E; ASTM D 256, 1822, 2444, 3029).

In the Charpy three point bending impact test a measurement is taken of the energy loss $A_n$ which a pendulum incurs in striking and breaking a prismatic specimen (of thickness $D$ and width $B$), which may or may not be notched. The observed energy loss basically comprises of 4 different terms:

- the energy $W_e$ to bend the sample up to the point of crack initiation;
- the energy $G_{cBD}$ to propagate the crack through the specimen;
- the kinetic energy $W_{kin}$ of the broken sample;
- vibrational or otherwise dissipated energy.

The ratio of the fracture energy $A_n$ to the sample cross section $B.D$ is termed impact strength, $a_n$. Such a notation creates the illusion that $a_n$ is a fracture surface specific material property. It has been pointed out frequently that this is not the case$^{95,96}$. Neither $W_e$ nor $W_{kin}$ is proportional to the sample cross section. A comparison of $a_n$ values should only be made, therefore, if all values are obtained from one type of
test and preferably from specimens of identical geometry. Impact strength values $a_n$ of Charpy unnotched specimens (DIN 53453) at 20° C range from 3.5-12 kJ/m$^2$ for filled phenolic melamine and urea resins, 4-22 kJ/m$^2$ for various filled epoxy and polyester resins, 12-20 kJ/m$^2$ for PMMA, PS and SAN, and 50-90 kJ/m$^2$ for ethyl cellulose, CA, styrene-butadiene copolymers, and POM. For many thermoplastics (ABS, CAB, PE, PP, PTFE, PC, PVC, PA) no fracture is observed under these test conditions$^{97}$.

The above impact strength values designate brittle behaviour if $a_n<40$ kJ/m$^2$. The materials with $a_n$ between 50 to 90 kJ/m$^2$ will generally be brittle if bluntly notched. Of those polymers not breaking in the Charpy unnotched test, some will respond in a brittle manner if they are sharply notched, whereas others remain tough. Vincent$^{98}$ and Bucknall et al.$^{96}$ propose the following qualitative impact resistance rating: brittle ($a_n<40$kJ/m$^2$), brittle if bluntly notched, brittle if sharply notched, tough but crack propagating , and very tough and crack arresting.

Impact behaviour is a very important property of SFRTP's as high impact rate damage tolerance is a required feature of most modern fabricated components. Notching a test specimen in an impact test will produce a stress concentration at the notch and will result in a lower initiation energy for crack production. Hence, it will have a lower impact energy than for an unnotched ligament of similar length. Theberge and Hull$^{99}$ have indicated discrepancies between notched and unnotched specimens for glass
fibre filled thermoplastics over a range of thermoplastics. They observed opposite trends for toughness versus fibre fraction in the notched and unnotched specimens. Davis\textsuperscript{100} confirmed similar observations. The impact resistance of the unnotched samples decreased with increasing volume fraction of fibres because each additional fibre is a new potential discontinuity. The notched specimens demonstrated an increase in impact resistance with increasing volume fraction of fibres because the biggest discontinuity was already built in, in the form of the notch. The fibres in the notched samples acting mainly to impede the crack running out of the notch.

Thomas and Meyer\textsuperscript{37} have investigated the effect of fibre dispersion upon the toughness. They showed that optimal toughness is achieved when the fibres are orientated perpendicularly to the plane of the crack. This configuration produces the maximum contribution from fibre pull-out, and makes fibre avoidance difficult.

In the case of low fibre fractions, there may be a reduction in impact resistance over the unreinforced material. Ramsteiner and Theysohn\textsuperscript{50} explained this by suggesting that adding brittle fibres to a ductile matrix will reduce the elongation to failure of the composite, yet, at the same time, the contribution due to fibre pull-out will be too small to influence toughness. At higher fibre fractions, when the ductility is further reduced the pull-out contribution is large enough to compensate for this matrix embrittlement. Increasing the fibre fraction further results in even higher toughness up to a maximum value, after
which it declines due to fibre/fibre interactions as the average distance between adjacent fibres gets smaller.

Bailey and Bader\textsuperscript{101} performed fracture toughness tests on a series of glass-fibre filled polyamide compounds, containing rubber dispersions. They demonstrated that the improvement of toughness was far greater at higher strain rates. From a study of fracture morphology they postulated that fibre-matrix debonding is the controlling factor at low strain rates, but that the matrix has more influence at higher strain rates.

The influence of the adhesive forces at the fibre-matrix interface on the impact behaviour and fracture morphology of short fibre reinforced elastomer modified polypropylene, was investigated by Stamhuis\textsuperscript{102}. Poor adhesion results in interfacial fracture solely by fibre-matrix debonding, as evidenced by scanning electron microscopy on the fracture surfaces. Pulled-out fibres exhibited pristine fibre surfaces. This is accompanied by relatively low impact strengths. By contrast, increased adhesion leads to fracture not only by fibre-matrix debonding, but also by crack propagation through the elastomeric phase at the fibre surface. This mechanism is thought to be responsible for the remarkable increase of the impact strength. After this process, pulled-out fibres are left with an adherent sheath of matrix on their surface.
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Figure 2.1  Schematic representation of projected length method of determining fibre orientation in a three dimensional random composite material.

Figure 2.2  Determination of fibre orientation in a thin section, (a) orientation defined by angles alpha and beta, and (b) orientation defined by the shape and orientation of fibre cross-section.
Figure 2.3  Stereographic projection representing orientation of fibres in a thin section. Injection moulding direction is shown and the plane of the diagram is parallel to the plane of the section. Fibres are oriented preferentially in the mould fill direction.

Figure 2.4  Leung & Li consider four different cases: (a) elastic stage; (b) debonding from the loaded fibre end; (c) debonding from the embedded fibre end; and (d) debonding from both fibre ends.
Figure 2.5  The micromechanical models of Voigt and Reuss.
Figure 2.6 Cox model fibres embedded in a polymer matrix.

Figure 2.7 Load transfer model as proposed by Cox. As the composite strain is increased, the length at the fibre ends over which the load is shed, up to the maximum load, is increased.
Figure 2.8 Variation of the fibre tensile stress, and the interface stress, along the length of a short fibre embedded in a matrix under tensile load:
(a) Cox model;
(b) Kelly-Tyson model.
The transfer length for a Cox model fibre can be taken at the intersection of the fibre stress curve where 90% of the maximum composite load is carried in the fibre.
Figure 2.10  
Tsai elastic theory of the modulus response with change of fibre angle to the applied load.

Figure 2.11  
Tensile strength of aligned continuous fibre material as a function of angle
Figure 2.12  Force equilibrium when an embedded fibre is pulled out of a matrix.

Figure 2.13  Dugdale argued that the effective crack length is larger than its physical length, by a distance $\Delta a_n$
3. COMPOSITE FABRICATION AND MECHANICAL TESTING

3.1. MATERIALS PRODUCTION

3.1.1. Base Materials

The base polymer used in this study is a polypropylene homopolymer powder to which a premixed addition of powdered stabilisers, antacid phenolics and antioxidants is made, at a level of 0.9% by weight of the base polymer. Polyolefin rubber granules are introduced at levels between zero and 40% by weight of the base polymer. A commercially available E-glass is added to produce nominal fibre fractions of 0, 15, 30 and 40% by weight of the final compound. A coupling agent may also be incorporated to promote greater adhesion between the polymer and the fibre. In grades where the coupling agent is used it is added at a standard percentage by weight of the base polymer, (the exact level is not disclosed for reasons of confidentiality). Grades containing levels of the coupling agent higher and lower than the standard have also been produced.

By varying the composition, a number of grades were produced, each of which is given a simple reference code. The code consists of four characters. The first two letters, AH, signify that the material was produced by myself. The next character signifies when the material was originally compounded. Material coded with a 1 was compounded in November 1987, material coded with a 2 was compounded in May 1988. Unless otherwise stated, the coupons and plaques were injected at the same time.
that the material was compounded. The final character is a letter which denotes a particular grade, A-N. Table 3.1, details all the grades produced.

All grades were produced from nominally identical stock, and processed under conditions as similar as was practicably possible.

3.1.2. Extrusion Compounding

It is essential to mix fully the constituent components of the material grade which are to be fed to the injection moulding machine. The degree of homogenisation is controlled by the level of shear. The shear within the injection moulder is not sufficient to compound the "ingredients" fully, and wet out the fibres to the necessary extent to produce a homogeneous matrix and fully bonded composite. This task is performed by extrusion compounding.

Polypropylene base polymer, stabilisers, coupling agent powders and rubber granules are weighed into quantities appropriate to the required grade. These components, with the exception of the glass fibre, are mixed roughly in an Henschel\(^3\) mixer. The mix is fed to the extruder and the fibre is added. Flights on the screws are angled in such a manner that they increase the shear in the melt, mix the components and break up the fibres. A vacuum is applied to the

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\(^{3}\) The Henschel mixer consists of a large stainless steel drum into which powder and granules are loaded. Counter-rotating paddles within the drum produces turbulent motion and mix the constituent elements.
vented extruder barrel at the decompression zone, next to the nozzle, to facilitate the removal of unwanted volatiles such as any remaining water or decomposition products from the molten polymer. The amalgam of components melts and becomes mixed within the barrel, before being squeezed out of the die. A continuous lace of material is hauled from the die and cooled in air or water. This lace is chopped into granules approximately 3mm in length and 2mm in diameter, and then dried in ovens prior to injection moulding.

3.1.3. Injection Moulding

The single screw injection moulding machine remelts the granules prior to the hydraulic ram injecting the molten polymer blend into a mould. The cast component is then cooled and ejected from the mould.

As far as practicable, the moulding conditions were kept constant in order to minimize possible microstructural differences which may occur during this procedure. Moulding was carried out at the maximum possible injection pressure, without inducing "flashing"\(^\text{(4)}\), in order to obtain good dimensional stability, avert voiding and reduce sinking. A minimum level of back pressure was applied consistent with successful mould filling and fibre dispersion. The degree of preplasticisation

\[^\text{(4)}\] "flashing" is exhibited as excess polymer being squeezed out of the mould cavity into the space between the mould halves. It is generally caused by the injection pressure exceeding the mould clamping pressure.
was kept to a minimum in order to avoid significant fibre attrition.

Each system grade had an initial production run of thirty 'dog bone' tensile test specimens, sixty unnotched charpy type specimens, thirty heat distortion bars, and thirty 150mm x 150mm coat hanger gated plaques, all nominally 3mm in thickness, figure 3.0. The 'dog bone' tensile specimens, heat distortion bars, and Charpy test pieces were all side gated. Side gating reduces the probability of jetting\(^\text{(5)}\) and produces a more uniform mould fill. Coat hanger gating the plaques produces a uniform flow of material into the mould from the whole of one edge. This results in greater alignment and orientation of fibres in the final product.

Plaques of 450mm x 450mm x 10mm from grades AH2A, B, C, and D were injection moulded in February 1989. These thicker plaques were produced primarily to be machined into fracture mechanics test pieces. The mouldings were gated so as to produce consistent fibre alignment either side of a central weld line.

\(^{(5)}\) "jetting" results in poor mould filling, and is caused by irregular squirting of the polymer melt into the mould cavity.
3.2. MECHANICAL TESTING

3.2.1. Test Piece Annealing

All test pieces were placed between glass plates, to ensure that they remained flat, and annealed for 2 hours at 130°C. They were then left to furnace cool prior to testing. This is done to alleviate any internal strains or ageing effects resident within the test coupon.

3.2.2. Tensile Mechanical Testing

Tensile testing was performed at room temperature using an Instron 1196 testing machine at a cross head speed of 50mm/min. All tensile mechanical property tests were carried out on the injection moulded dog-bone tensile test pieces.

Extensions of up to 12% of the 80mm gauge length were measured using an extensometer, with a 50mm gauge length. Extensions above 12% are taken directly from the Instron chart recording of load against cross head travel. A minimum of ten specimens from each grade were tested. Each datum presented on the graphs of tensile properties represents the average of all the test results. The error bars represent the full spread of results within a grade.
3.2.3. **Modulus**

For the base polymer and its formulations the tensile stress strain response is non-linear from its origin. A \( \frac{1}{2} \)% secant regression was considered the most suitable method of evaluating the modulus, figure 3.1.

A Polymer Laboratories Dynamic Mechanical Thermal Analyser (DMTA) was used to evaluate the flexural modulus of the preliminary grades \((AH1A-J)\), under three-point bending at room temperature. Heat distortion bars were tested at a maximum displacement of 62.5 microns and at a frequency of 1 Hz, with an unclamped free length of 16mm.

Further flexural tests were performed on plaque material from a limited number of grades. The purpose of the tests was to compare the flexural properties of nominally identical grades which were produced in different campaigns. The tests were performed using an Instron on 3mm thick plaques at 1mm/min at room temperature. Material grades investigated in this manner were the 30% rubber filled base polymer \( AH1D \) and \( AH2B \), and the 30% glass filled base polymer \( AH1F \) and \( AH2C \).

3.2.4. **Strength**

The tensile yield strength is taken at the upper yield point, and is determined from the stress/strain curves of the individual grades.
Uniaxial compression tests were carried out on 3mm thick plaque material from grades AH1D, 1F, 2B, 2C. In this test the stress is applied to the plaques by parallel dies. The technique allows a yield stress to be determined for plane strain conditions. This yield value differs from that measured by tensile testing, or indeed any measurement that can be taken from the tensile stress strain response. The reason for this distinction lies in the fact that the plane strain compression test produces a yield value which is more of a true materials property and is not subject to the fracture nucleation processes associated with tensile fracture. In this manner it is possible to measure accurately a representative yield strength for a material which exhibits fracture mechanisms at or near yield in tension.

3.2.5. Strain-to-Yield/Failure

As indicated above, extensions below 12% are measured using an extensometer. Above that, the extensions are taken from the plot of cross-head travel. The strain-to-failure is taken as the strain, both elastic and plastic, at the point of fracture. The strain-to-yield is taken at the tensile yield point. Strain results were only taken as being "significant" if the coupon failed between the extensometer grips. However, those which failed outside or at the extensometer grips, showed good agreement with the valid results.
3.3. MECHANICAL PROPERTY RESULTS

3.3.1. Data Presentation of Mechanical Properties

The results of all the mechanical testing carried out in this project are depicted in this section both in tabulated form (table 3.2) and as graphical plots (figures 3.3-3.14). The large number of variables incorporated in this work makes the graphical representation of any one property very confusing where all the systems are presented on a single plot. It is therefore necessary to plot each mechanical property three times, each time against a different composite component (i.e. glass content, rubber content, coupling level). This makes property trends easier to observe. Individual points on the plots are labelled with the last two digits of their reference code i.e. AH2D is shown as 2D, and AH1J as 1J. The purpose of this is to enable easier reference and to show in a clearer manner the difference between materials produced in the two campaigns.

3.3.2. Tensile Tests

The stress/strain response of the individual grades differed quite considerably. There follows a brief description of the basic tensile stress/strain trends for the general grades, figure 3.2.

Composite grades which do not contain any glass undergo "drawing" when tested in tension. This is exhibited as a further increase in the load carried by the specimen after initial yield
load drop and necking of the specimen. This effect is caused by alignment of the polymer molecules within the necked region of the specimen when loaded. The energy required to initiate fracture in the necked region is greater than the energy required to draw the regions outside the neck. The neck will thus grow down the length of the specimen. Necking is promoted by increasing temperature and decreasing strain rate.

The uncoupled fibre filled grades, and rubber modified fibre filled grades, continue to support a reduced load following the initial yield point. The coupled fibre filled grades fail at initial maximum load.

The yield stress results, figures 3.3, 3.4, 3.5, show the trends expected from rule-of-mixture approximations. Increasing glass and rubber contents leads to increase and decrease respectively in the yield stress in a linear manner. The level of coupling appears to increase the yield stress linearly up to the standard concentration. Beyond this, some increase is observed but not at the same rate.

The effect of adding any glass to the polymer blend is to reduce its strain-to-yield significantly, figure 3.6. This is a consequence of two mechanisms which bring about embrittlement and control the tensile failure mechanisms. The first of these, reported by Sato et al\(^1\), proposes that debonding is initiated at the fibre ends of oriented fibres, whilst misaligned fibres were observed to be more prone to debond along their interfaces. The second embrittlement effect was observed by Collins\(^2\), who showed that matrix cracking results from the fibre end debonding, giving
rise to penny shaped cracks from which failure of the matrix initiates subsequently. These effects combine to reduce the tensile strain-to-yield.

Increasing rubber content in general tends to increase the strain-to-yield by increasing the ductility of the matrix, figure 3.7. This will delay debonding of the fibres and thus the ability for penny shaped cracks to initiate at fibre ends. In the non-coupled fibre filled systems the trend is reversed and the strain to yield falls slightly with increasing rubber content. In the non coupled grades it is probable that the matrix strength dominates the failure processes. As rubber is added to the polypropylene the matrix strength is reduced and hence the strain-to-yield is also diminished.

Increasing the level of coupling increases the strain-to-yield linearly up to the maximum level used in this series of systems, figure 3.8. As the level of coupling is increased the interfacial shear strength may also be increased. This would manifest itself as an increase in the load required to initiate matrix cracking and debonding at fibre ends, and thus an increase in the load and strain to yield. The level of coupling has a more pronounced effect than the level of glass on the strain to yield.

The addition of glass to any of the systems reduces the strain to failure, figure 3.9. The glass particles due to their modulus mismatch and weak interfacial bond, at high loads act as internal defects and initiate failure processes.

The strain to fracture increases with rubber content as the ductility and compliance of the matrix is increased, figure 3.10.
The effect of the rubber on the fracture mode is to enhance the energy absorbing processes occurring at and around the crack tip. Microcavitation and crazing, has been observed in rubber toughened materials by Wu\(^3\). The compliance of the rubber serves to make the particles act as holes in the polypropylene. This reduces the stiffness and initiates voiding and crazing. In this way, extra energy is absorbed during fracture by increasing the volume of material which participates around the crack.

The 0.5% secant moduli increase and decrease linearly with increasing glass and rubber content respectively, figures 3.11, 3.12. These trends are predicted by the rules of mixtures approximations when adding second and third phases materials with substantially higher and lower moduli. The effect of adding rubber to the polypropylene matrix is to reduce the modulus of the composite to an extent far greater than that predicted by particulate rule of mixtures approximations. This will be discussed in greater detail in chapter 6.

Increasing the level of coupling to any particular system improves the modulus at low additions, but has little effect at higher levels, figure 3.13. The addition of the coupling agent to an uncoupled system produces a visco-elastic bond thus increasing both the interfacial shear strength and interfacial shear modulus. The additional load now able to be transferred into the fibres will account for the composite modulus increase at low levels of the coupling agent. Once the coupled visco-elastic bond is created any further addition of coupling agent will not significantly improve the interfacial shear modulus. Hence, the composite modulus does not increase with further
additions of coupling agent. However, this is not to be confused with the shear strength of the interface. The shear strength of the interface determines the maximum load which the interface can support before debonding occurs. The interfacial shear strength may be increased by the further additions of coupling agent and would thus affect processes controlled by fibre debonding, such as yield strength and toughness. However, such a mechanism by which this would occur appears unlikely.

The mechanical property results show a clear difference between batches produced in the two campaigns. Within each batch AH1 and AH2 the property trends are consistent, but between the batches there is an inconsistency. The specimens from the two campaigns were tested at different times. To ensure that the observed differences were features of the different production campaigns, and not of the testing technique, a limited number of retests was performed. Specimens from nominally identical grades from the two batches were annealed and tested together. The results are given in table 3.3. From these retests it appears that there is a clear difference in mechanical properties between nominally identical grades from the different production campaigns.

The base polymer exhibits identical properties for both batches. However, the addition of fibre or rubber produces differing results. The moduli of nominally identical grades are the same. Comparative specimens from different batches, which have nominally identical grades, follow identical load/extension relationships when strained. However, specimens from the second batch yield at a far lower strain than specimens of the same
grade from the first batch. This results in a lower yield stress and strain to yield/fracture. This is probably caused by a difference in the microfracture/yield modes occurring in the region of the fibre/matrix interface. The reason for this difference is not clear since materials and process conditions were kept constant as far as possible.

The plane strain modulus results are in agreement with the tensile test results and show a slightly higher modulus for the coupled glass from the first batch of materials, and a higher modulus for the rubber toughened grade of the second batch. The results of the yield strength measured in uniaxial compression show an 18% difference between the glass filled material. There was no difference in the rubber toughened grades, as was similarly observed in the tensile tests.

3.3.3. Dynamic Mechanical Modulus Analysis

The modulus values obtained from the DMTA are far below those obtained from tensile tests, figure 3.14. The modulus in three point bending should be higher than that obtained from a tensile test because there is a greater contribution from the fibres oriented in the stressed direction at the surface. Also, as the DMTA uses a lower strain, the effective gradient of the load/extension graph at that point is higher and should thus produce a higher modulus. The machine produces a strain rate of approximately one sixth of that of a tensile test, \(0.0018 \text{s}^{-1}\) DMTA, \(0.01 \text{s}^{-1}\) tensile). The strains involved in the two tests vary, 444 microstrain maximum for the DMTA and 5000 microstrain
for the tensile test. The results have been confirmed by the
equipment manufacturers on similar analysers, table 3.4.

Even though it is not easy to explain the generally low
modulus values obtained by this testing technique, some
interesting observations and hypotheses can be made. The moduli
of the coupled grades are consistently lower than those from the
uncoupled grades. This trend completely contradicts the results
from the tensile tests. A possible explanation for this lies in
the different nature of the fibre/matrix interface in the
different grades.

The primary difference between the coupled and uncoupled
grades is the interfacial bond between the fibre and the matrix.
In the uncoupled grades the interfacial bond will be of a
frictional nature, which is developed from a difference in
thermal contraction between the fibre and matrix upon freezing.
Fibres in the coupled grades are physically bonded to the matrix
due to the addition of the coupling agent. The resultant bond
will have a visco-elastic response.

In the uncoupled grades as the composite is strained, load
is transferred from the matrix through the frictional interface
into the fibres. At low loads the frictional interface will be
too weak to transfer the load, and the fibre will slip within the
matrix. In the coupled grades the applied load is transferred
into the fibres through the visco-elastic interface in a similar
manner. However, the interfacial shear strength is higher than
that of the frictional bond, and can thus transfer greater
maximum loads. This accounts for the increased stiffness of the
coupled grades in the tensile test. The DMTA test applies much
lower loads and strains to the specimens than does the tensile test. It is possible that at these low loads the frictional interfacial shear strength of the uncoupled grades is not exceeded. If the shear modulus of the frictional interface of the uncoupled grades was greater than that of the visco-elastic interface of the coupled grades, then at low loads the observed modulus trends could be accounted for.

References

1. Sato N., Kurauchi T., Sato S. and Kamigaito O.
   Mechanism of Fracture of Short Glass Fibre Reinforced Polyamide Thermoplastic.

2. Collins J.F.
   The Strength and Behaviour of Short Glass Fibre Reinforced Polyamide 6.

3. Wu S.J.
   Impact Fracture Mechanisms in Polymer Blends.
TABLE 3.1

NOMINAL COMPOSITE GRADES

All compositions are in weight percent, with the exception of the level of coupling which is given as a fraction of the standard loading.

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<th>Code</th>
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### TABLE 3.2

**MECHANICAL PROPERTY RESULTS**

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<th>Strain-to-Failure (%)</th>
<th>Secant Modulus (GPa)</th>
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<tr>
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<td>AH2D</td>
<td>5.60</td>
<td>5.37</td>
<td>4.23</td>
<td></td>
</tr>
</tbody>
</table>
**TABLE 3.4**

**DYNAMIC MECHANICAL MODULUS**

Machine: Polymer Laboratories D.M.T.A.

Clamping: Dual Cantilever
16mm free length

Calibration: $A = 9.49$
$B = 0.160$
$C = 5.019$
$D = 2.417$

Frequency: 3Hz

Strain: $\times 4$

<table>
<thead>
<tr>
<th>Code</th>
<th>Log $E'$ Polymer Laboratories</th>
<th>Log $E'$ University of Surrey</th>
<th>$\tan \delta$ Polymer Laboratories</th>
<th>$\tan \delta$ University of Surrey</th>
</tr>
</thead>
<tbody>
<tr>
<td>AH1D</td>
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</tr>
<tr>
<td>AH1E</td>
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<td>0.026</td>
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</tr>
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<td>AH1J</td>
<td>9.410</td>
<td>9.251</td>
<td>0.031</td>
<td>0.033</td>
</tr>
</tbody>
</table>

The Polymer Laboratories data are in closer agreement with that obtained on the tensile tester at Surrey. Measured modulus can be corrected to allow for the movement of the sample under the clamp bar. By a careful study of the effect of sample free length on the measured modulus and correct sample clamping, then the data which correlate well with other techniques may be obtained.
Figure 3.1 Typical stress/strain response for a rubber toughened polypropylene. The mechanical properties extracted from such graphs are shown.

Figure 3.2 Typical stress/strain response for the reinforced and toughened grades.
Tensile Yield Strength
The Effect of Glass Content

Tensile Yield Strength
Effect of Rubber Content
**Tensile Yield Strength**

Effect of Coupling Content

- PP & 15% Glass
- PP & 30% Glass
- PP & 40% Glass

All grades contain 30% Rubber by weight of base polymer.

**Figure 3.5**

---

**Strain to Yield**

The Effect of Glass Content

- Polypropylene
- PP & Coupling
- PP & 30% Rubber
- PP & R & Coupling

**Figure 3.6**
Strain to Yield
Effect of Rubber Content

Strain to Yield
Effect of Coupling Content

All grades contain 30% Rubber by weight of base polymer.

Polypropylene
+ PP & 30% Glass
* PP & G & Coupling

PP & 15% Glass
+ PP & 30% Glass
* PP & 40% Glass
Strain to Failure
The Effect of Glass Content

- Polypropylene
- PP & Coupling
- PP & 30% Rubber
- PP & R & Coupling

Figure 3.9

Strain to Failure
Effect of Rubber Content

- Polypropylene
- PP & 30% Glass
- PP & G & Coupling

Figure 3.10
0.5% Secant Modulus
The Effect of Glass Content

![Figure 3.11](image1)

0.5% Secant Modulus
Effect of Rubber Content

![Figure 3.12](image2)
0.5% Secant Modulus
Effect of Coupling Content

Youngs Modulus Evaluation
From Dynamic Mechanical 3-Pt Bending

Figure 3.13

Figure 3.14
The qualitative interpretation and modelling of the mechanical properties of short fibre composites, requires an accurate knowledge of their microstructure. In short fibre reinforced thermoplastics the fibres generally have a range of lengths and lie in a three dimensional distribution within the matrix. The quantification of the distribution of fibre lengths and their respective orientation is necessary for the development and application of mechanical models. The size and distribution of the rubber particles aid an understanding of possible toughening mechanisms. In the present chapter the range of techniques used and developed for microstructural characterisation are described (sections 4.1 to 4.4) and subsequently (section 4.5) the detailed results are presented.

4.1. FIBRE VOLUME FRACTION

Constituent mixing and extrusion compounding as described in section 3.1.2 produces nominal fibre fractions of 0 to 40 percent (table 3.1). It is necessary to confirm the actual fibre fractions of the final mouldings. There may also be differences in the fibre loading through the thickness and breadth of mouldings. This is due to flow characteristics within the mould. The fibre fraction is evaluated by taking the representative section of moulding, burning off the matrix, and calculating the resultant mass of fibres as a fraction of the original moulding.
4.1.1. Fibre Fraction Evaluation

Sample sections under investigation are the extruded granules, bulk moulded test specimens and plaques, and also sections through their thickness and breadth. Firstly, ceramic crucibles containing the desired moulding section are weighed. The matrix material is then burnt-off for six hours in an oven heated to 400°C. The melting and oxidising of polypropylene can be quite vigorous, but the loss of fibres due to convection and escaping gases from the melting polypropylene can be restricted by using a crucible with a loose fitting lid. The severity of the burning of the matrix is reduced when the specimen is heated up from room temperature, rather than by placing the specimen inside the oven at temperature.

After burning-off, the crucibles are reweighed. The volume fraction of fibres is calculated using the following equation:

\[
V_f = \frac{W_f/\rho_f}{(W_f/\rho_f) + (W_m/\rho_m)}
\]

where;

\[
W = \text{weight fraction}
\]

\[
\rho = \text{density}
\]
4.2. FIBRE LENGTH DISTRIBUTION

Fibre length distributions are produced from representative batches of fibres from burn-off tests. The fibres are observed under an optical microscope and the image fed to an image analyser. The analyser edits crossed and touching fibres digitally before they are measured and counted. A large number of fibres from many different fields needs to be counted to produce an accurate representation of the length distribution. A total count of about 7000 fibres appears to produce a representative distribution of fibre lengths.

Errors are incurred by the image analyser due to long fibres crossing between separate fields. These errors can be corrected by use of a computer simulation and correction program. Fibre length distributions are displayed as histograms. They can be shown either as fibre count versus length, or more informatively as fibre volume versus length. Fibre volume can be envisaged more simply as the total length of fibres within a particular length fraction. The following sections 4.2.1 to 4.2.10 give a detailed account of the image analysis, simulation and correction techniques developed to produce accurate fibre length distributions.

4.2.1. Matrix Burn-Off

To produce fibre fractions separate from the matrix, ceramic crucibles containing representative mouldings are prepared. The
matrix material is burnt-off in an oven at 400°C for six hours in a manner identical to that described in section 4.1.1.

### 4.2.2. Optical Observation of the Fibres

A small quantity of fibres from the burn-off is placed in the centre of a microscope slide. The slide is agitated to enable the fibres to become dispersed more uniformly and separated. The slide and fibres are observed in optical transmission microscopy at a relatively low magnification, typically x40. The optimum magnification is a compromise between several factors:

i) it must be possible to observe and measure the longest actual fibre from the burn-off within a single frame of the image analyser. It will become apparent in section 4.2.8. that this parameter sets up limitations within the simulation and correction analysis. This limits the highest magnification;

ii) each fibre feature within the display requires eleven spacial parameters to be measured and recorded to enable a description of its position, angle, etc. These parameters are used to separate crossed and touching fibres. If too many fibre features exist within one field, their associated parameters will cause an overflow within the analyser's memory. This determines the lowest magnification;
iii) low magnifications incur errors in resolution when measuring short fibre lengths of the order of a few pixels.

4.2.3. Process Screen of the Analyser

A monochrome multishade image from the microscope is fed to the monochrome screen of a Cambridge Instruments Quantimet 920 image analyser. The visual display unit (VDU) of the analyser is effectively made up of two overlapping areas. The first, larger image, is the visual screen which encompasses the whole of the observable field of the display. The visual screen has dimensions 880 x 688 pixels. The second, smaller image, is the live frame and is positioned centrally within the visual screen. The dimensions of the live frame can be altered, but for the present analysis are set as 800 x 625 pixels, figure 4.1.

The purpose of having two image frames is to help remove ambiguities associated with features overlapping visual fields and being measured twice. When features are finally measured, it is only those features for which the lowest point lies within the live frame which are measured. In this way, any feature whose lowest point lies outside the present live frame but within the present visual screen will only be measured in a contiguous live frame, figure 4.2. Hence the stage of the microscope must be incrementally moved one live frame at a time. In this way every feature is measured just once.
4.2.4. Image Processing

Features lying wholly or partly within the visual screen and which are darker than a preset shade, are treated as a positive feature. Lighter features are treated as negative features. As the fibres are considerably darker than the background, the analyser has little trouble in recognising them as discernable features. A black and white digital image is thus achieved, figure 4.3. A shading correction can also be applied at this point to remove any point or shading aberrations caused by the microscope or video camera, e.g. dust on the lenses, or uneven light distribution.

The total fibre length distribution is regarded as two separate length distributions. The first of these encompasses fibre fragments, i.e. features less than 60 microns in length. Fibres satisfying this criterion which lie within the live frame are counted and measured at this stage. This is necessary as the subsequent routine, which separates crossed fibres, effectively removes very small fibres. Fortunately fibre fragments do not touch or cross other fibres very often. The probability of fibres touching increases with fibre length, therefore errors incurred by touching fibres are more significant at longer fibre lengths.

Many longer fibres are found to be crossing or touching. If such fibre features were not separated the analyser would assume that any group of touching fibres were a single fibre, and hence produce an incorrect measurement, figure 4.4. Fibres
within the visual screen are uncrossed and separated by a series of image processing steps:

i) before the automated digital manipulation proceeds it is possible to edit the visual screen manually to remove any ambiguities, such as dust or erroneous particles on the microscope slide;

ii) positive features are skeletised. This has the effect of reducing the thickness of any feature down to a single pixel. A bare skeleton of the original digital image is thus produced, figure 4.5. The skeletising process has the effect of reducing the length of the fibres by half of their original thickness at each end. This results in a reduction in fibre length of about 13 microns. More significantly, fibre fragments can be completely obliterated, or considerably shortened, by this process. It is for these reasons that the fibre fragments are measured prior to skeletisation, as detailed above;

iii) fibre crossing and touching points are detected;

iv) crossing and touching fibres are separated by the insertion of a space at their intersection. This has the effect of cutting any crossing fibres into two pieces, and reducing the length of any touching fibres, figure 4.6;

v) the computer program detects all the skeletised fibre ends. These may be naturally occurring fibre ends, or those artificially produced by separating crossed fibres;

vi) at each fibre end the computer calculates the angle at which the fibre is lying. The analyser detects any other
fibre ends present within a preset vicinity. If another fibre end is present, the analyser calculates the angle at which the second fibre is lying. If the second fibre is lying within a preset angular range of the first fibre the computer assumes that the second fibre is an extension of the first fibre, figure 4.7. The two fibre features are then remeasured as a single fibre, and the two previous features replaced with the single new one. The difficulty with this process lies in the choice of values for the box size within which the computer scans for adjacent fibres ends, and the angular range within which the fibres can be assumed to be part of the same fibre. These values are dependant upon fibre diameter, level of magnification, and the degree to which fibres are separated by the uncrossing process. The values for these parameters are best evaluated from trial-and-error simulations of known field distributions;

vii) the digitally adjusted fibres are remeasured. Fibres whose lowest point lies within the live frame are counted and their length recorded, see section 4.2.3;

viii) the long fibre distribution is adjusted to account for the length lost during skelletization (add 13 microns). The short and long fibre distributions are combined;

ix) The fibre length distributions are displayed as histograms of fibre count, or summed fibre length (volume), against fibre length.
4.2.5. Errors Incurred by Image Analysis

The technique described in section 4.2.4. results in errors across nearly the whole distribution of fibre lengths. These are caused by the manner in which the analyser measures fibres whose lengths intersect the perimeter of the visual screen. As stated in section 4.2.3, the purpose of setting up the live frame within the visual screen is to eliminate the possibility of measuring fibres twice in adjacent image frames. However, with a range of fibre lengths from 20 to 2600 microns the live frame would need a minimum safety surround of 2600 microns between itself and the perimeter of the visual screen, figure 4.8. This is be impracticable as it would:

- require exceptionally low magnifications;
- exceed the memory capacity due to the larger number of fibres within the visual screen caused by the low magnification;
- result in a loss of resolution and accuracy as many fibre features would be so small.

The circumstances under which the analyser will count and measure fibres needs to be reiterated in more depth. The analyser maps the points of the visual screen using $X,Y$ coordinates. Position (0,0) is placed at the top left corner of the screen. $X$ increases across the screen, and $Y$ increases down the screen. The analyser will only count and measure a fibre if its lowest point on the screen, $Y_{min}$, is within the live frame. If both $Y_{max}$ and $Y_{min}$ are within the live frame then the fibre is
measured correctly (figure 4.9, fibre A). If $Y_{\text{max}}$ is within the live frame and $Y_{\text{min}}$ is within the visual screen but outside the live frame, the fibre is also measured. In these circumstances, if the fibre does not intersect the perimeter of the visual screen it is measured correctly (fig 4.9, fibre B). A fibre whose $Y_{\text{max}}$ is within the live frame, but whose length breaks the perimeter of the visual screen, is measured. However, the length is measured as that from $Y_{\text{max}}$ to where the fibre exits the perimeter of the visual screen, and is thus measured incorrectly (fig 4.9, fibre C).

Measuring a fibre which intersects the perimeter of the visual screen causes:
- an increase in the fibre count of a smaller length range;
- the loss of a count to the fibres' actual length range.

The other end of the fibre will not be measured again in an adjacent frame because its lowest point will be outside the live frame. Over a total distribution, this incorrect measuring of fibres will produce an artificial distribution with an increased count of shorter fibres and a reduced count of longer fibres than are actually present within the sample.

These problems have been realised in a computer simulation which copies the process by which the image analyser measures fibres. The results of the simulation are applied through a probabilistic model to the actual results from the image analyser resulting in accurate fibre length distributions. A full description of the manner in which these correcting processes is applied and calculated are complex and lengthy. For these
reasons a full description of the simulation and correction processes are given in an appendix at the end of this chapter.

4.2.6. Log-Normal Fibre Length Distributions

The corrected fibre length distributions from the image analyser approximate log-normal distributions, i.e. if the FLD data is plotted out as fibre volume versus log fibre length, then a normal distribution is observed, figure 4.10.

The log-normal curve is obtained from the data in following manner. The fibre volume data are logged and square rooted. This produces a pointed distribution. A regression is applied to the points on both sides of the peak and lines of best fit calculated. The gradient of the lines is related to the standard deviation of a normal distribution. The standard deviation is inserted into the equation representing a normal distribution, and the area below the curve equated to that below the original curve.

Hence the corrected FLD can be represented graphically either in the form of bar charts, or in the form of log-normal distributions as smooth curves.
4.3. FIBRE ORIENTATION DISTRIBUTION

Fibre orientation distributions are measured quantitatively in a similar manner to the fibre length distributions. However, for fibre orientation distributions the fibres are observed whilst still embedded in the matrix, figure 4.11.

4.3.1. Section Choice

A cross-sectional plane of moulded material is selected for analysis. Central cross-sections from the three major axes (figure 4.12) give good overall descriptions of the three-dimensional orientation structure of the moulded component, but are not easily applied to mechanical models. The sections which need to be examined depend upon the model description in use. For example, if the moulded component is being likened to a cross-ply laminate, then representative samples from three parallel planes need to be taken, figure 4.13.

4.3.2. Thin Section Preparation

The thin section can be prepared in one of two ways:

i) the section is microtomed to a thickness of about 30 microns at room temperature. The microtomed slice is immersed in microscope oil on a slide under a cover slip. This technique works fairly well at low fibre fractions. At higher fibre fractions and when rubber is present, the
surface of the slice becomes heavily disfigured by pulled-out, broken fibres and fibre ends;

ii) a far more successful technique is to grind, rather than slice, the section down to the required thickness. The specimen is bonded to a microscope slide and coarsely ground down near to the thickness required for analysis. This surface is then ground down carefully on successively finer grades of paper. The final paper used is a 4000 grit. This produces an almost polished surface. Actual polishing is not recommended as the polishing fluids tend to become embedded and deface the surface of the sample. The sample is removed from the microscope slide by dissolving away the adhesive. The specimen is then bonded with the freshly prepared surface face down on a fresh slide. The adhesive needs to bond as a thin transparent layer as this slide will become the cover slip for the sample. An acrylonitrile glue was found to be very successful in this application. The opposite face of the sample is ground down in a similar manner until the overall thickness is about 25 microns, depending upon the fibre loading of the sample. Another slide is bonded to the fresh surface of the sample enclosing it. This preparation technique produces little surface degradation and fibre damage.
4.3.3. **Image Analysis of FOD**

The image analyser can be used to record the fibre orientation distribution in a similar manner to the evaluation of the fibre length distributions. The image analyser is set up as for evaluation of the fibre length distribution with a few changes;

i) the image frame is set to the same size as the visual screen. As we are interested in the total length of fibres at a particular angle it is not essential that a single fibre be measured on a single screen. A fibre can overlap between separate fields, but its total length will be measured at a single angle (assuming the fibre is not curved). This alleviates the need for the complex simulation and correction procedure developed for the FLD as all the fibres are measured correctly;

ii) the magnification may need to be increased as the embedded fibres are harder to distinguish due to the surrounding matrix and proximity of other fibres;

iii) the final output is recorded as total fibre length versus fibre angle. The fibre angle is measured with respect to the sample edge (the principal melt flow direction).

The analyser is set up to observe its first field at the surface of the moulding. The fibres within this field are separated digitally and measured. The field of view is moved to the next adjacent area of unmeasured sample moving down the melt flow direction (Z direction). This procedure is repeated until
sufficient frames have been measured to obtain a representative distribution. At the end of one traverse the data are logged. The field of view is moved one frame distance away from the surface, i.e. one unit into the moulding. The whole process is repeated for all the sections through the samples thickness or breadth. The result for each sample is a set of values representing the orientation distributions across the width or breadth of the sample, taken down its length.

4.3.4. Computer Generated FOD

The FOD and FLD data were combined in a computer model to produce artificial orientation distributions\(^{(6)}\). The model takes into account the probability that a fibre at any position within the two-dimensional plane has a particular orientation. This fibre with its 'weighted' orientation is then given a length similarly 'weighted' by the FLD statistics. The result is a 2-dimensional image analogous to that of a thin section in which all fibres lie within the plane of the section. A visual comparison can be made between the observed sections, figure 4.11, and the computer model, figure 4.14, based upon the image analysis.

---

\(^{(6)}\) A listing of the Turbo Basic programme developed to perform this operation is given in appendix B.
4.4. RUBBER PARTICLE DISPERSION

The position of the rubber phase can be determined experimentally in many ways. The rubber particles can be tagged using a dye or heavy element for optical or electron probe microscopy. It is also possible to determine the position of the rubber particles within thin sections using phase contrast transmission electron microscopy. The position of any particles which intersect a surface can be found by simply dissolving the rubber out of the matrix and observing the resultant holes.

4.4.1. Section Preparation

With the above techniques a thin specimen section or specimen cross section is required. The ductility of rubber and polypropylene are very different. In order to slice the material it is necessary to take both phases below their respective glass transition temperatures. This ensures that the rubber will not smear when the surface or slice is prepared. The $T_g$ of polypropylene is around room temperature, and the $T_g$ of rubber is $-90^\circ$C. Hence specimens must be sliced under cryogenic conditions. Small specimens were sliced in a cryogenic microtome at $-100^\circ$C. This produces a thin sample which can be used in transmission electron microscopy, and also a flat face on the donor sample from which the rubber can be dissolved and the resultant holes observed using scanning electron microscopy. The rubber is dissolved from the freshly prepared surface by suspending the specimen above boiling heptane for 60 seconds.
This method dissolves the rubber but does not affect the polypropylene. The specimens are then sputter coated with gold and observed by SEM.

The above preparation technique proved to be very successful with the rubber toughened polypropylene grades, where no glass was included. In the glass filled grades, the fibres are sheared out of the matrix during the microtoming. The broken fibre fragments destroy the prepared surface. Using this technique it is not possible to observe the possibility of preferential segregation of the rubber to the fibre surface and an alternative method had to be found.

Notched charpy type specimens were machined from the injected fibre reinforced grades. These were placed in liquid nitrogen for five minutes and then broken open in the charpy testing machine. From each specimen two fracture surfaces were created. One surface had the rubber dissolved from it using boiling heptane, the other did not. The fracture surfaces were observed under the scanning electron microscope and compared to see if the rubber could be distinguished clearly from the polypropylene.
4.5. MICROSTRUCTURAL CHARACTERISATION RESULTS

4.5.1. Fibre Fraction Confirmation

Many burn-off tests were performed during this project, specifically for glass fibre fraction confirmation, but also as a necessary stage for evaluation of fibre length distributions. The fraction of fibres was calculated for all of these burn-offs and the average tabulated below. The standard deviations of all the fibre fraction evaluations were less than 1%.

<table>
<thead>
<tr>
<th>Nominal Weight Fraction (%)</th>
<th>Actual Weight Fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>15.00</td>
<td>14.58</td>
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<tr>
<td>30.00</td>
<td>29.42</td>
</tr>
<tr>
<td>40.00</td>
<td>38.56</td>
</tr>
</tbody>
</table>

Table 4.1 Weight fraction of fibres from 3mm moulded specimens

Sections through the thickness of the 10mm thick plaques were taken to see whether there is any difference in fibre loading between the skin and the core of these specimens, listed in table 4.2. Grades were produced at a nominal 30% weight fraction of fibre.

The fibre fractions actually achieved during production were consistently slightly less than those which were aimed for.

The fibre loading in the skin and the core to all intents and purposes are identical. As will become apparent in the following section, the average fibre length is substantially less than the dimensions of the specimens. This allows the fibres to distribute themselves uniformly throughout the moulding (with
Table 4.2 Fibre fractions at different thicknesses through the 10mm plaques.

<table>
<thead>
<tr>
<th>Section</th>
<th>PP &amp; Glass (%)</th>
<th>PP &amp; Glass &amp; Rubber (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>skin</td>
<td>29.54</td>
<td>29.43</td>
</tr>
<tr>
<td>core</td>
<td>29.00</td>
<td>29.12</td>
</tr>
<tr>
<td>skin</td>
<td>28.99</td>
<td>28.87</td>
</tr>
<tr>
<td>average</td>
<td>29.17</td>
<td>29.14</td>
</tr>
</tbody>
</table>

respect to volume fraction, orientation will be dealt with later). When the fibre length distributions have lengths approximating the minimum dimensions of the specimen, the fibre loading of the core can become far greater than that in the skin.¹

4.5.2. Fibre Length Distributions

Figure 4.15 demonstrates how the computer simulation and correction programs adjust the output from the image analyser. The peak of the corrected curve is moved down and across towards the longer fibre range. The correction program decreases the count of the shorter fibres and increases the count of longer fibres.

Many fibre length distributions have been produced in the course of this project. The FLD curves which have been produced have shown great consistency within mouldings, grades, and also between batches and campaigns. Figures 4.16, 17, 18 show typical fibre length distributions from samples taken from mouldings containing 30% rubber with 15%, 30%, 40% glass by weight respectively.

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The corrected FLD bar charts of the three volume fractions exhibit the same features. The fibres are distributed in a log-normal manner. The majority of the fibre volume is made up from fibres whose length is greater than the fibre length at the peak of the curve. A secondary feature of all the curves is the much smaller initial peak produced by the very short fibres. This initial peak is produced by fibres whose maximum dimension is approximately the same as the fibre diameter. It was found that these very small fibre fragments are produced when long fibres undergo high curvature bending and break during extrusion. The attrition of long fibres produces two shorter fibres, and numerous small thumb nail shaped shards of glass fibres. As the volume fraction of fibres is increased the average fibre length is reduced. This must be a consequence of greater attrition, and thus more fibres being broken. The volume fraction of the fibre shards increases with fibre volume fraction confirming this point.

Figure 4.19 shows the log-normal distributions for the three fibre filled grades. As the volume fraction of fibres is increased the level of attrition during extrusion increases due to a greater probability of fibre-fibre interactions. This manifests itself as a general reduction in the average fibre length. The maximum fibre length is reduced as is the total fraction of long fibres. The proportion of short fibres is increased and the peak of the curve moves towards the shorter fibres.

The majority of fibre attrition occurs in the compression zone of the twin screw extruder where the molten polymer is
subject to highest shear, Lunt and Shortall\textsuperscript{2}. Further fibre length degradation is incurred during the injection stroke of the moulding cycle. The extent of fibre breakage during flow through constricted gates and runners will depend upon the forces associated with the injection stroke, the matrix material rheology and the filler loading. This effect was studied by Schweitzer\textsuperscript{3} using polypropylene and polyester matrices. Fibre attrition occurring in the injection moulding cycle has been studied by comparing the fibre length distributions from both the extruded granules, figure 4.20, and the moulded specimens, figure 4.21. The results confirm that the majority of fibre attrition occurs during extrusion compounding. The injection moulding stage reduces further the maximum and average fibre length. Although the level of attrition is far less during the injection moulding stage, it is still significant, particularly in relation to the mechanical properties of the moulded product. Decreasing the length of a short fibre, for example a fibre whose total length is less than $2l_c$, reduces its load bearing capacity to a far greater extent than does a similar reduction in length of a long fibre. A small reduction in the average fibre length in a SFRTP in which the large proportion of the fibres have lengths around the critical length, will result in a considerable reduction in the strength and stiffness of the composite.

4.5.3. Fibre Orientation Distribution

Thin sections were taken perpendicular to the three major axis of tensile test bars, figure 4.12, and examined. It was
found that sections taken down the length of the specimen, through the $x$ and $y$ planes, showed good fibre definition and quantitative data. The specimen taken through the thickness and breadth of the sample, through the $z$ plane, only revealed fibre ends. The fibres are naturally oriented with their length in the mould fill direction. This section was of little quantitative use. For this reason the rest of the results and discussion will deal with sections taken from the $x$ and $y$ planes. As these two planes incorporate the $y,z$ and $x,z$ directions, all three dimensions are accounted for.

The results from the fibre orientation distributions are shown in this chapter as area graphs. Each shaded area on the graph represents the volume fraction of fibres, at that distance across the section, which lie within a specific orientation range. Each range covers $30^\circ$. $0^\circ$ lies parallel to the melt fill direction, i.e. down the length of the sample. $90^\circ$-$180^\circ$ is directly equivalent to $0^\circ$-$(−90)^\circ$. The actual image analysis results were measured in angular ranges of $10^\circ$. For calculation of orientation factors the narrower distribution were used.

Five sections were taken from each of the $x$ and $y$ planes, figure 4.22. The results were found to be symmetrical about the core. Figures 4.23 to 4.25, show the $y$ plane results taken through the thickness of a 15% glass 30% rubber specimen and observed across its breadth. The sample taken at the surface, figure 4.23, shows a large degree of misoriented fibres across the whole breadth of the sample. They are not specifically restricted to the centre. These results correspond with the work of Singh and Kamal$^4$ who also observed a layer of randomly
orientated fibres in the vicinity of the wall. The wall of the mould acts as a heat sink. Consequently it produces a higher frictional drag than the viscous effect of the melt. The flow of molten material past the surface will cause fibres in contact with the wall to be dragged across its surface and hence off orientation.

The section taken just below the surface of the specimen, figure 4.24, shows a much greater degree of alignment. The fibres in the proximity of the moulding edge are consistently oriented in the melt fill direction. Flow of the melt in this region is constrained by the mould surfaces, producing good alignment of the fibres. In the centre of the specimen the melt flow is not constrained to the same extent and fibres begin to align themselves normal to the fill direction. The distribution is not symmetrical about the centre. This is a result of the side gating of the specimen and the non-symmetric flow of melt down the mould. This becomes even more apparent in the central section, figure 4.25. This section is taken through the core of the sample. The core exhibits the greatest degree of misoriented fibres. Again, the misoriented fibres lie predominantly in the centre of the section. The reason for the increase in misoriented fibres is the greater distance to the top and bottom surfaces of the mould. The central section has the least constraints from any surface.

Figures 4.26 to 4.28 show the x plane results taken through the breadth of an identical sample and observed across its thickness. The degree of misorientation for all three samples is far less than that observed in the y plane samples. The
breadth of the sample is four times greater than the thickness. Therefore, even in the centre of the thickness of the sample there is still a large constraint influence. This is due to the close proximity of the top and bottom surfaces. In the $y$ plane samples, the well oriented edges extended two millimetres from each edge into the sample. The total thickness of the sample is only three millimetres. It is therefore not surprising that the fibres through the thickness of the sample are highly constrained. This produces the finding that most of the fibres are oriented within just one plane, namely the $y$ plane. This is of greater consequence later when trying to evaluate a orientation factor for the composite as a whole. The other features of the $x$ plane samples are the same as for the $y$ plane samples.

Figures 4.29, 4.30 show fibre orientation distributions for 30% glass and 40% glass grades, both containing 30% rubber. The distributions are nominally identical. The addition of glass to a grade would be expected to increase the viscosity of the melt. This change in viscosity does not appear to alter greatly the orientation distributions. Moulding conditions and specimen geometry will have the greatest effect upon fibre orientation distributions.

4.5.4. Krenchel Orientation Factor

The fibre orientation distributions given above are of little quantitative use in that form. Where there is a distribution of fibre orientations, the reinforcing efficiency
of the fibres is reduced due to misaligned fibres. Krenchel\textsuperscript{5} included an additional term in the rule of mixtures equation (2.3) to take this into account: $\eta_o$, the Krenchel orientation factor. The term $\eta_o$ is calculated from a summation of the fibre fraction, $a_k$, oriented in angular increments away from the applied load, $\theta_k$:

$$\eta_o = \sum a_k \cos^4 \theta_k$$  \hspace{1cm} (2.14)

Krenchel\textsuperscript{5} has calculated $\eta_o$ for various fibre orientation distributions. For unidirectional laminae $\eta_o=1$ and 0 when tested parallel and perpendicular to the fibres respectively.

Calculating the Krenchel factor relative to the $z$ direction for the 15\% glass 30\% rubber grade distributions, figures 4.23 to 4.28, gives:

<table>
<thead>
<tr>
<th></th>
<th>$x$ plane</th>
<th>$y$ plane</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Surface</strong></td>
<td>0.867</td>
<td>0.649</td>
</tr>
<tr>
<td><strong>Near Surface</strong></td>
<td>0.930</td>
<td>0.848</td>
</tr>
<tr>
<td><strong>Centre</strong></td>
<td>0.907</td>
<td>0.716</td>
</tr>
</tbody>
</table>

Table 4.3 Krenchel orientation factors calculated at different distances through the thickness and breadth

The contribution from the different sections to the composite as a whole is not evenly distributed. The layer at the surface of the moulding which contains a high degree of misoriented fibres is only about 100 microns thick. The contribution of these layers will be negligible to the overall composite and can be disregarded. The two remaining sections can be broadly described as the skin (near surface) and core (centre). It is fortunate that the thickness of these sections...
is distributed with the approximate ratio of 2:1. With regard to the x plane, there is little difference between the skin and core. The weighted average of the Krenchel factors is 0.923 (twice the skin thickness to the core thickness). Applying Krenchel factors from two 2-dimensional planes to a 3-dimensional composite is very complex. However, as has been mentioned in the previous section, the majority of fibres appear to be oriented solely in one plane, the y plane. This is confirmed by the x plane Krenchel factor of 0.923 (average fibre misorientation of 11.4° from the melt fill direction). Such a low degree of misorientation within the x plane enables, for the sake of simplicity, the composite to be treated as a three ply laminate with all the misorientation within the y plane. This approximation results in an overestimation of the Krenchel factor for the composite.

Treating the composite as a three ply laminate, the Krenchel factor for the 15% glass reinforced composite is calculated as 0.804.

Using the same analysis for the 30% and 40% glass filled grades, the following results are produced:

<table>
<thead>
<tr>
<th></th>
<th>Skin</th>
<th>Core</th>
<th>Composite</th>
</tr>
</thead>
<tbody>
<tr>
<td>15% Glass</td>
<td>0.848</td>
<td>0.716</td>
<td>0.804</td>
</tr>
<tr>
<td>30% Glass</td>
<td>0.834</td>
<td>0.675</td>
<td>0.781</td>
</tr>
<tr>
<td>40% Glass</td>
<td>0.839</td>
<td>0.616</td>
<td>0.765</td>
</tr>
</tbody>
</table>

Table 4.4 Krenchel orientation factors for 30% and 40% glass filled grades

Increasing the volume fraction of glass increases the degree of misorientation within the composite. Adjusting the volume fraction of glass in the melt will alter its flow, viscosity and
shear properties and hence affects the final orientation distribution.

4.5.5. Rubber Particle Distribution

An accurate analysis of the distribution and size of the rubber particle was not performed. Although such a characterisation would have been of microstructural interest, it is not essential to the mechanical modelling. The following discussion is based upon broad conclusions obtained from careful observations of rubber particle distribution in scanning electron microscopy.

In grades not containing any glass the rubber particles were distributed uniformly throughout the matrix, figure 4.31. The rubber particle size was approximately 1 to 3 microns in diameter. Increasing the volume fraction of rubber did not appear to alter the average particle size significantly. These results confirm similar observations by Hodgkinson\(^6\) and Coppola\(^7\). The shape of the rubber particles was not truly spherical, figure 4.32. Their actual shape is best described as a sphere elongated in the direction of the melt flow. This effect is caused by the elastic constraints of the mould surfaces upon freezing and the relative expansion coefficients and moduli of the constituent phases.

The fibre reinforced Charpy type specimens were broken open under cryogenic conditions. The purpose of these test pieces was to determine the rubber particle distribution around the fibre surfaces. Figure 4.33 shows that there is no segregation of
rubber to the fibre surfaces. Figure 4.35 shows the fracture surface of a charpy specimen without the rubber having been dissolved from its surface. Nodular protrusions can be seen emanating from the fracture surface of the polypropylene. Figure 4.34 is the opposite fracture face of the same specimen where the rubber is dissolved. The nodular protrusions are no longer present. It can be assumed therefore that the nodules are pulled-out rubber particles. At the low temperatures under which these fracture faces were produced, the polypropylene/rubber interfacial shear strength is lower than the rubber shear strength, hence causing this effect. Confirming that these nodules are rubber particles will be of importance in explaining the fracture surfaces of fracture mechanics specimens in chapter 8.

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APPENDIX A

4.6. COMPUTER SIMULATION AND CORRECTION PROCESS

The following appendix details the manner by which the fibre measuring process of the image analyser is simulated and errors resulting from this process recorded. These results are used in a probabilistic manner to correct the fibre length distributions from the image analyser. A full listing of the computer programme is given at the end of appendix A.

4.6.1. Simulating the Fibre Count

The fibre count, $N_i$, for a particular fibre length, $L_i$, from the analyser is made up of two distinct portions. The first portion is made up of those fibres which are counted and measured correctly, $N_c$. The second portion is made up of counting those fibres which are actually longer than $L_i$, but which have been mismeasured and consequently counted into that band, $N_m$, figure 4.36:

$$N_i = N_c + N_m \quad (4.2)$$

If it were possible to take the count from the analyser, $N_i$, and subtract the miscounted fibres, $N_m$, the remainder would be the number of fibres which had been measured correctly, $N_c$. This
would not be the true total count, $n_i$, as it does not take into account those fibres of length $L_i$ which have been mismeasured as fibres of a shorter length. If it were possible to calculate the probability, $P_i$, for a fibre of length $L_i$ to be mismeasured as a shorter fibre, it would also be possible to adjust the count to allow for these missing fibres. This would be done by taking the number of correctly measured fibres, $N_c$, of length $L_i$, and applying a probability factor, $P_i$, to adjust the count to allow for mismeasured fibres of length $L_i$. This would give a probabilistic approximation to the true count, $n_i$, based on the actual count from the analyser, figure 4.37:

$$n_i = \frac{N_c}{1-P_i} \quad (4.3)$$

4.6.2. The Computer Simulation: An Overview

A computer simulation has been developed to model the manner in which the analyser counts and measures fibres. The program sets up an imaginary visual screen and live frame within its memory. Imaginary fibres of known length are randomly distributed into the simulated visual screen. Upon distributing a statistically significant number of fibres, the simulation evaluates probabilistic parameters simulating the manner in which fibres are measured by the analyser:

i) The probability, $P_i$, of a fibre of length $L_i$ being mismeasured is calculated;
ii) if a fibre of length $L_i$ is measured incorrectly, the fibre length range into which it would be counted, $L_{i-n}$ is found;

iii) for each number count from the analyser, $N_i$, the incorrectly measured fibres, $N_m$, are removed from the count to leave only those fibres which have been measured correctly, $N_c$. To this count a correction factor $(1/(1-P_i))$, is applied to account for the fibres of that length which were mismeasured into fibre counts of smaller lengths.

iv) fibre counts are converted into volume fractions allowing for fragment shape as well as fibre length.

The steps in this simulation are described in detail in the sections which follow.

4.6.3. Simulation Input Parameters

The computer simulation necessitates the input of specific parameters to simulate and adjust the analyzers fibre length distributions correctly:

i) Maximum Fibre Length. The simulation is set up within bounds imposed by the total fibre length distribution. The maximum fibre length within the sample imposes the upper bound, and the 'fibre step range' (note iii) the lower bound. Longer fibres contribute to the miscount contribution, $N_m$, of shorter fibres. Thus, the number of miscounts within a short fibre count will alter depending upon the maximum fibre length. The longest fibres are
very rare within a distribution as they are statistically few in number. However, the probability of them being mismeasured is high, as will be shown later. It is therefore necessary for careful image analysis of a very large number of fibres to ensure that the longest possible fibre is measured. The observed maximum fibre length starts at about 1600 microns for a 40% glass grade and increases with decreasing fibre volume fraction.

ii) *Pixel Size.* This refers to the magnification of the microscope and hence the size that one pixel of the display represents. The visual screen and live frame are defined in pixels, but the fibres are measured in microns. The program mainly works in pixels, 2.74 microns per pixel is typical for the magnifications used in this series of tests.

iii) *Fibre Step Range.* Individual fibres are measured to the resolution of a pixel. However the analyser groups the data into fibre length ranges to simplify later analysis. This input refers to the step size of these bands. 32 microns is fairly typical. This value also determines the lower bound and the step size for the fibre length loop within the program.

iv) *The Number of Fibres per Length Range.* The simulation randomly distributes fibres over the visual screen. It does this for each length range. The number of fibres randomly distributed per length range is defined by this input. The larger the number, the more accurate the final simulation. Values of 5,000 and 10,000 fibres
appear satisfactory. There may be approximately 50 fibre length range loops:

\[
\frac{\text{max' fibre length}}{\text{fibre step range}} = \frac{1600\mu m}{32\mu m} = 50
\]

and 10,000 fibres within each loop. Consequently this process can take a substantial length of time to run.

4.6.4. Simulating the Distribution

The computer sets up two 2-dimensional data arrays akin to the pixel dimensions of the visual screen and live frames respectively. A series of nested loops is set up within the program, the first of which determines the fibre length, \( L_i \). The initial fibre length is the value of the fibre step range (section 4.2.8.iii), and with each successive loop it increases by the fibre step value, up to the maximum fibre length (section 4.2.8.i). A second loop is set up from 0 to the number of fibres per length range (section 4.2.8.iv). The program now simulates the random distribution of fibres over the visual screen. \( X \) and \( Y \) coordinates within the visual screen array are randomly selected by the computer for each fibre. This represents the random position of one end of a fibre. A random fibre angle, \( \theta \), is also chosen. The length of the fibre is known for each step range, \( L_i \), and hence the coordinate position of the opposite end of the fibre, \( X' \) and \( Y' \) can be calculated by use of simple trigonometry:
\[ X' = X + L_i \sin \Theta \]  
\[ Y' = Y + L_i \cos \Theta \]  

(4.4)  
(4.5)

If \( Y \) is greater than \( Y' \) then the coordinates \( X \) and \( X' \), and, \( Y \) and \( Y' \) are exchanged. This is done to simplify the program and ensure that the lowest point of the fibre is at the \( X', Y' \) coordinate.

The program takes the \( X', Y' \) fibre end coordinate and determines whether it lies within the live frame array. If it does, the fibre will be measured and counted:

\[ C_i = C_i + 1 \]  

(4.6)

where:

\( C_i \) is the total number of measurable fibres of actual length \( L_i \), whether measured correctly or incorrectly.

Any fibre whose \( X', Y' \) coordinate lies outside the live frame is not measured at all. For any measured fibre, if the \( X, Y \) fibre end coordinate lies within the live frame or visual screen the fibre will be measured correctly. If however the \( X, Y \) coordinate lies outside the visual screen, the fibre will be incorrectly measured and will therefore be a miscount:

\[ M_i = M_i + 1 \]  

(4.7)

where:

\( M_i \) is the total number of mismeasured fibres of actual length \( L_i \).
The total number of mismeasured fibres divided by the total number of measured fibres within the length range will give the proportion of miscounts, \( P_i \), for fibres of length \( L_i \), figure 4.38:

\[
P_i = \frac{M_i}{C_i}
\]

(4.8)

For any mismeasured fibre the distance between the \( X', Y' \) fibre end coordinate and the position where the fibre intercepts the perimeter visual screen is calculated. This is the fibres' mismeasured length. It is determined into which fibre length range this effectively shorter fibre would be recorded. A large array is set up to log the number of mismeasured fibres of length \( L_i \), and into which shorter length range they are counted into. The procedure is repeated, stepping a fibre at a time, up to a maximum determined by the value of the fibres per length range (about 10,000). The procedure is then repeated for the next length range in the loop, and so on up to the maximum fibre length. Finally the number of mismeasured fibres attributed to shorter fibre length ranges is converted into fractions of the total miscount for the shorter length range.

The final result is a triangular array of data which describes the way in which a randomly distributed group of fibres of known lengths are measured, mismeasured, and into which length ranges they are attributed in a simulated analyser.
4.6.5. Correcting the Distribution

Imagine the count for a single fibre step range from the analyser of length $L_i$. The count is made up of the correctly measured fibres, $N_c$, plus all of those fibres which are actually longer fibres, but which have been mismeasured, $N_m$. This mismeasured portion is made up from many longer fibre ranges, figure 4.39. If a very large number of fibres is measured, then the contribution to $N_m$ for a fibre length range of $L_i$ will be made up from all ranges longer than $L_i$ up to the maximum fibre length, $L_{\text{max}}$. $N_m$ can thus be broken down into a series of smaller contributions from the individual fibre ranges:

$$N_m = N_{m_i+1} + N_{m_i+2} + N_{m_i+3} + \ldots + N_{m_{\text{max}}} \quad (4.9)$$

The mismeasured contributions from the larger fibre ranges, $N_{m_i+x}$, can be broken down further. $N_{m_i+x}$ represents the fraction of fibres of length $L_i+x$ that are measured as having length $L_i$, multiplied by the true fibre count for fibres of length $L_i+x$ i.e:

$$N_{m_i+x} = f(L_i+x).n_{i+x} \quad (4.10)$$

where;

$f(L_i+x)$ is the fraction of fibres of length $L_i+x$ that are measured as having length $L_i$;

$n_{i+x}$ is the true fibre count of fibres of length $L_i+x$.

The fraction of fibres of length $L_i+x$ that are measured as having length $L_i$, $f(L_i+x)$ in the above equation, can be equated
further. It is made up of the fraction of incorrectly counted fibres of length $L_{i+x}$ which are measured as length $L_i$, multiplied by the fraction of the total number of fibres of length $L_{i+x}$ which are incorrectly measured:

$$f(L_{i+x}) = g_{i,i+x} \cdot f_{i+x}$$

(4.11)

$$N_{mi+x} = g_{i,i+x} \cdot f_{i+x} \cdot n_{i+x}$$

(4.12)

where:

- $g_{i,i+x}$ is the fraction of incorrectly measured fibres of length $L_{i+x}$ which are counted as length $L_i$;
- $f_{i+x}$ is the fraction of fibres of actual length $L_{i+x}$ which are incorrectly measured;
- $n_{i+x}$ is the true number of fibres of length $L_{i+x}$.

Combining equations (4.9) and (4.12) gives:

$$N_m = g_{i,i+1} \cdot f_{i+1} \cdot n_{i+1} + g_{i,i+2} \cdot f_{i+2} \cdot n_{i+2} + \ldots + g_{i,max} \cdot f_{max} \cdot n_{max}$$

(4.13)

Adding equation (4.2) to (4.13) describes the analyser's count:

$$N_i = N_c + g_{i,i+1} \cdot f_{i+1} \cdot n_{i+1} + \ldots + g_{i,max} \cdot f_{max} \cdot n_{max}$$

(4.14)

Substituting from equation (4.3) gives a relation between the image analyser count of length $L_i$ ($N_i$) and true counts of all fibre lengths greater than or equal to $n_i$: 135
Equation (4.15) is the fundamental relation basis for correcting the distribution.

Evaluating the parameters of equation (4.15):

\( P_i, g_{i,i+x}, \text{ and } f_{i+x} \) are calculated for the individual fibre length ranges in the computer simulation. \( N_i \) is obtained from the analyzers output.

The unknown quantities are the true counts of fibres of length, \( n_i \) and \( n_{i+x} \) to \( n_{\text{max}} \). To solve the system of equations represented by (4.15) we note that as \( L_i \) gets larger the number of \( N_{mi+x} \) contributions becomes less. For \( L_{\text{max}} \) there is no \( N_m \) contribution at all, as there are no fibres longer than \( L_{\text{max}} \) which can be mismeasured as having length \( L_{\text{max}} \). Hence:

\[
N_{\text{max}} = n_{\text{max}}(1-P_{\text{max}}) \tag{4.16}
\]

Rearranging gives:

\[
n_{\text{max}} = N_{\text{max}} / (1-P_{\text{max}}) \tag{4.17}
\]

\( N_{\text{max}} \) and \( P_{\text{max}} \) are known, hence \( n_{\text{max}} \) is found. Equating the solution for fibres one fibre step range shorter, \( L_{\text{max}-1} \). Equation (4.15) can be expressed as:

\[
N_{\text{max}-1} = n_{\text{max}-1}(1-P_{\text{max}-1}) + g_{\text{max}-1,max}f_{\text{max}}n_{\text{max}} \tag{4.18}
\]
\( n_{\text{max}} \) is known from equation (4.17). The only unknown is \( n_{\text{max}-1} \), and is thus solvable. This process can be carried on for subsequent values of \( L_i \), eventually reaching \( L_{\text{min}} \), and thus adjusting the whole distribution.

This iteration is performed by a computer program interfaced to the computer simulation. The program takes the triangular array from the simulation (section 4.2.7). The array contains the values of \( P_i, g_i,i+x, \) and \( f_{i+x} \) for all fibre lengths \( L_i \). The analyser's fibre count for the maximum fibre length, \( N_{\text{max}} \), is input manually. The program solves equation (4.15/17). \( N_{\text{max}-1} \) is input manually and equation (4.15) is solved, and so on until \( n_i \) is solved for all \( L_i \).

The program finally converts the adjusted number count to a length count. Longer fibres are assumed to be cylindrical. Very short fibres (<30 microns) are fibre fragments produced by high curvature bending fracture. The ends of such fibres are triangular in profile, figure 4.40. For short fibres the fibre ends are a large proportion of the total fibre length, and thus the fibres can not be assumed to be cylindrical. A correction factor of 0.35 is applied to fibres below 60 microns in length to account for this. The final print-out gives volume percent versus length.
4.6.6. Correction Programme Listing

Listing of Turbo Basic programme to correct the output from the image analyser.

REM THIS PROGRAM ADJUSTS FIBRE LENGTH DISTRIBUTIONS FROM THE IMAGE ANALYSER

GOSUB initialise
GOSUB setuparrays

REM mainloop
FOR lq=0 TO (mfl/fsr)
  l=lq*fsr/mpp
  count=0;fail=0;tot=0
  FOR n=0 TO fplr
    y=INT(vssy*RND)
    x=INT(vssx*RND)
    th=RND*2*4*ATN(1)
    xo=x+l*SIN(th)
    yo=y+l*COS(th)
    IF yo>vssy THEN GOSUB switch
    IF y>(vssy-lfsy)/2 AND y<=(vssy-lfsy)/2 AND x>(vssx-lfsx)/2 AND x<=(vssx-lfsx) THEN LET count=count+1
    IF y<(vssy-lfsy)/2 OR y>=(vssy-lfsy)/2 OR x<(vssx-lfsx)/2 OR x>=(vssx-lfsx) THEN GOSUB nocount
    IF yo<vssy OR xo<0 OR xo>vssx THEN GOSUB miscount
  NEXT n
GOSUB missort
NEXT lq
GOSUB matrixsort
restart:
GOSUB fibdistribution
GOSUB volumecount
GOSUB printout
ERASE np
DIM np(2,mfl/fsr)
GOTO np
END

initialise:
PRINT "TURN YOUR PRINTER ON"
INPUT "Reference",ref$
LPREFIX "Reference",ref$
INPUT "Maximum Fibre Length (microns)", mfl
LPREFIX "Max Fibre Length (microns)",mfl
INPUT "Microns Per Pixel",mpp
LPREFIX "Microns Per Pixel",mpp
INPUT "Fibre Step Range (microns)",fsr
LPRINT "Fibre Step Range (microns)",fsr
INPUT "How many fibres per length range",fplr
LPRINT "Fibres per length range",fplr
INPUT "Visual Screen Size (x,y)",vssx,vssy
LPRINT "Visual Screen Size (x,y)",vssx,vssy
INPUT "Live Frame Size (x,y)",lfsx,lfsy
LPRINT "Live Frame Size (x,y)",lfsx,lfsy
RETURN

setuparrays:
DIM pf(mfl/fsr)
DIM mat(mfl/fsr,mfl/fsr)
DIM vf((mfl/fsr),2):ntot=0
DIM ifd(mfl/fsr,mfl/fsr)
DIM np(2,mfl/fsr)
RETURN

switch:
SWAP x,xo
SWAP y,yo
RETURN

nccount:
xo=vssx/2
yo=vssy/2
RETURN

mccount:
fail=fail+1
IF xo>0 AND xo<vssx AND yo>vssy THEN GOSUB topl
IF xo<0 AND yo<vssy THEN GOSUB sidell
IF xo>vssx AND yo<vssy THEN GOSUB sideln
IF xo<0 AND yo>vssy THEN GOSUB corl
IF xo>vssx AND yo>vssy THEN GOSUB corr
lo=INT(lo*mpp/fsr)
ifd(lq,lo)=ifd(lq,lo)+1
RETURN

topl:
lo=l*((vssy-y)/(y-y))
RETURN
	sidell:
lo=l*(x/(x-xo))
RETURN
	sideln:
lo=l*((vssx-x)/(x-x))
RETURN

corl:
lot=l*(x/(x-xo))
lol=l*((vssy-y)/(y-y))
IF lot<lol THEN LET lo=lot
IF lol<lot THEN LET lo=lol
RETURN
corr:
  \[ \text{lot} = l \times \left( \frac{(vssx-x)}{(xo-x)} \right) \]
  \[ \text{lor} = l \times \left( \frac{(vssy-y)}{(yo-y)} \right) \]
  IF \( \text{lot} < \text{lor} \) THEN LET \( \text{lo} = \text{lot} \)
  IF \( \text{lor} < \text{lot} \) THEN LET \( \text{lo} = \text{lor} \)
RETURN

missort:
  \[ \text{prob} = \text{fail} \times 100 / \text{count} \]
  \[ \text{pf}(lq) = \text{prob} / 100 \]
  LPRINT "length (microns)"; \( lq \times \text{fsr} ", " \text{probability }"; \text{prob} \]
  FOR \( z = 0 \) TO \( lq \)
      \( \text{tot} = \text{tot} + \text{id}(lq, z) \)
  NEXT \( z \)
  IF \( \text{tot} = 0 \) THEN \( \text{tot} = 1 \)
  REM data logging
  FOR \( z = 0 \) TO \( lq \)
      \( \text{id}(lq, z) = \text{id}(lq, z) / \text{tot} \)
  NEXT \( z \)
RETURN

matrixsort:
  FOR \( y = 0 \) TO \( mfl / \text{fsr} \)
      FOR \( z = y \) TO \( mfl / \text{fsr} \)
          \( \text{mat}(y, z) = \text{id}(z, y) \times \text{pf}(z) \)
      NEXT \( z \)
  NEXT \( y \)
  FOR \( x = 0 \) TO \( mfl / \text{fsr} \)
      \( \text{mat}(x, x) = 1 - \text{pf}(x) \)
  NEXT \( x \)
RETURN

fibdistribution:
  FOR \( y = (mfl / \text{fsr}) \) TO \( 0 \) STEP \(-1\)
      PRINT "fibre count for length "; \( y \times \text{fsr} ": \)\n      \( \text{INPUT n} \)
      \( \text{np}(1, y) = n \)
      FOR \( z = (mfl / \text{fsr}) \) TO \( y \) STEP \(-1\)
          \( n = n - \text{mat}(y, z) \times \text{np}(2, z) \)
      NEXT \( z \)
      \( \text{np}(2, y) = n / \text{mat}(y, y) \)
  NEXT \( y \)
RETURN

printout:
  LPRINT "fibre length", "number", "revised", "volume", "revised"
  FOR \( z = 0 \) TO \( mfl / \text{fsr} \)
      LPRINT USING "#####.###";
      \( z \times \text{fsr} ", \text{np}(1, z) ", \text{np}(2, z) ", \text{vf}(z, 1) ", \text{vf}(z, 2) \)
  NEXT \( z \)
RETURN

volumecount:
  \( \text{ntot} = 0 : \text{nrtot} = 0 \)
  FOR \( z = 0 \) TO \( (mfl / \text{fsr}) \)
      IF \( z \times \text{fsr} \leq 60 \) THEN LET \( \text{ntot} = \text{ntot} + 0.35 \times \text{np}(2, z) \times (z \times \text{fsr} - (\text{fsr} / 2)) \)
      IF \( z \times \text{fsr} > 60 \) THEN LET \( \text{ntot} = \text{ntot} + \text{np}(2, z) \times (z \times \text{fsr} - (\text{fsr} / 2)) \)
140
nrtot=nrtot+np(1,z)*(z*fsr-(fsr/2))
NEXT z
FOR z=0 TO (mfl/fsr)
   IF z*fsr<60 THEN
      vf(z,2)=.35*np(2,z)*(z*fsr-(fsr/2))*100/ntot
   IF z*fsr>60 THEN vf(z,2)=np(2,z)*(z*fsr-(fsr/2))*100/ntot
   vf(z,1)=np(1,z)*(z*fsr-(fsr/2))*100/nrtot
NEXT z
RETURN
Listing of Turbo Basic programme to produce artificial fibre orientation plots.

REM *****PROGRAMME TO PRODUCE FIBRE ORIENTATION PLOTS*****

$STACK 10000
RANDOMIZE TIMER
INPUT "COLOUR(1), B/W(2) HIGH RES'(3)'",col%
IF col%=1 THEN INPUT "File Name"; fname$
IF col%=1 THEN OPEN fname$ FOR OUTPUT AS #1
IF col%=1 THEN GOSUB colour
IF col%=2 THEN GOSUB bandw
IF col%=3 THEN GOSUB highres
IF col%=3 OR col%=1 THEN mag=1.75
IF col%=2 THEN mag=1
LINE(0,2)-(640,2),1
LINE(0,2+189*mag)-(640,2+189*mag),1
GOSUB main
CLOSE #1
IF col%=2 THEN GOSUB savpic
END
colour:
  SCREEN 9
  PALETTE 0,7
  PALETTE 1,1
  PALETTE 2,2
  PALETTE 3,3
  PALETTE 4,4
  PALETTE 5,5
  PALETTE 6,6
RETURN

bandw:
  SCREEN 2,0
  PALETTE 0,15
  PALETTE 1,0
RETURN

highres:
  SCREEN 9,0
  PALETTE 0,15
  PALETTE 1,0
RETURN

savpic:
  DEF SEG=&H800
  BSAVE "FPIC",0,16000
  PRINT "HELLO"
  CLS
DEF SEG = &HB800
LOAD "FPIC"
RETURN

main:
  FOR th% = 2 TO 182*mag STEP 9*mag
  FOR an% = 0 TO 150 STEP 30
    tl=0
    READ od
    IF od>0 THEN GOSUB orient
    NEXT an%
    NEXT th%
  RETURN

orient:
  DO UNTIL tl*10/(mag*1.5)>od
    GOSUB fibselect
    IF ok%=1 THEN GOSUB plot
  LOOP
  RETURN

fibselect:
  l%=1+INT(3000*RND)
  p=(0.4*RND)
  li=((1/(2*3.14159)^0.5)*EXP(-0.5*((2.492912-((LOG(l%))^-0.5))/0.084606)^2))
  ok %=0
  IF p<li THEN GOSUB fibreok
  RETURN

fibreok:
  x%=INT(640*RND)
  y%=INT(9*mag*RND)
  a%=INT(30*RND)
  ye%=y%+th%
  a%=a%+an%
  xe%=x%+INT((l%*0.016*mag)*(2.26)*COS(0.01745*a%))
  ye%=y%+INT((l%*0.016*mag)*SIN(0.01745*a%)*mag)
  IF xe%<0 THEN xe%=0
  IF xe%>640 THEN xe%=640
  IF ye%>191*mag THEN ye%=191*mag
  IF ye%<0 THEN GOTO fibreok
  IF x%<0 OR x%>640 OR y%<0 OR y%>191*mag THEN GOTO fibreok
  ok%=1
  tl=tl+l%/1000
  RETURN

plot:
  c%=1+INT(l%/500)
  IF col%=2 OR col%=3 THEN c%=1
  LINE(x%,y%)-(xe%,ye%),c%
  IF col%=1 THEN GOSUB fyle
  RETURN

fyle:
  xe%=x%+INT((l%*0.041)*COS(0.01745*a%))
  143
\( y_{e\%} = y_{\%} + \text{INT}\left((1\% \times 0.028) \times \text{SIN}\left(0.01745 \times a_{\%}\right)\right) \)

IF \( c_{\%} = 1 \) THEN WRITE \#1, \( x_{\%}, y_{\%} \)
IF \( c_{\%} = 1 \) THEN WRITE \#1, \( x_{e\%}, y_{e\%} \)
IF \( c_{\%} = 2 \) THEN WRITE \#1, \( x_{\%}, "x", y_{\%} \)
IF \( c_{\%} = 2 \) THEN WRITE \#1, \( x_{e\%}, "x", y_{e\%} \)
IF \( c_{\%} = 3 \) THEN WRITE \#1, \( x_{\%}, "x", "x", y_{\%} \)
IF \( c_{\%} = 3 \) THEN WRITE \#1, \( x_{e\%}, "x", "x", y_{e\%} \)
IF \( c_{\%} = 4 \) THEN WRITE \#1, \( x_{\%}, "x", "x", "x", y_{\%} \)
IF \( c_{\%} = 4 \) THEN WRITE \#1, \( x_{e\%}, "x", "x", "x", y_{e\%} \)
IF \( c_{\%} = 5 \) THEN WRITE \#1, \( x_{\%}, "x", "x", "x", "x", y_{\%} \)
IF \( c_{\%} = 5 \) THEN WRITE \#1, \( x_{e\%}, "x", "x", "x", "x", y_{e\%} \)
IF \( c_{\%} > 5 \) THEN WRITE \#1, \( x_{\%}, "x", "x", "x", "x", "x", y_{\%} \)
IF \( c_{\%} > 5 \) THEN WRITE \#1, \( x_{e\%}, "x", "x", "x", "x", "x", y_{e\%} \)

WRITE \#1,
RETURN

\[ \text{DATA} \]
\[ 49.50, 00.00, 00.12, 00.00, 00.18, 50.18 \]
\[ 49.83, 00.00, 00.00, 00.00, 00.00, 50.16 \]
\[ 49.62, 00.00, 00.00, 00.00, 00.00, 50.37 \]
\[ 47.62, 00.00, 00.00, 00.00, 00.24, 52.13 \]
\[ 26.99, 00.94, 00.00, 00.16, 00.49, 71.41 \]
\[ 09.42, 01.13, 00.58, 00.49, 13.57, 74.80 \]
\[ 11.60, 01.40, 00.72, 00.29, 07.71, 78.28 \]
\[ 06.02, 02.69, 00.86, 04.52, 20.45, 65.45 \]
\[ 03.05, 00.50, 00.77, 03.31, 36.50, 55.87 \]
\[ 00.91, 07.87, 08.31, 28.36, 43.78, 10.75 \]
\[ 01.07, 28.92, 16.04, 26.44, 24.72, 02.79 \]
\[ 13.51, 43.36, 19.32, 16.95, 04.03, 02.82 \]
\[ 48.60, 34.76, 03.61, 03.42, 03.57, 06.03 \]
\[ 61.44, 17.17, 01.17, 01.37, 00.92, 17.92 \]
\[ 65.56, 05.42, 00.32, 00.00, 01.57, 27.13 \]
\[ 56.98, 00.15, 00.16, 00.18, 00.00, 42.53 \]
\[ 52.06, 00.25, 00.25, 00.37, 01.23, 45.82 \]
\[ 48.17, 01.48, 00.22, 00.00, 00.00, 50.12 \]
\[ 48.62, 00.28, 00.00, 00.00, 00.00, 51.00 \]
\[ 47.90, 00.00, 00.00, 00.00, 00.00, 52.10 \]
\[ 48.33, 00.14, 00.00, 00.00, 00.00, 51.52 \]
RETURN
Figure 4.1  The size and shape of the Visual Screen and Live Frame of the image analyser. The visual screen around the live frame is the region designed to act as a safety net for overlapping fibres.

Figure 4.2  Fibres are only measured in the live frame in which their lowest point lies. In this way each fibre is only measured once. The Visual screens must overlap if the live frames are to measure every fibre.
Figure 4.3 The refraction of light at the surfaces of the fibres produces a clear black and white digital image.

Figure 4.4 Problems associated with measuring the fibre length are caused by crossing fibres (a) and touching fibres (b) and (c).
Figure 4.5  The digital fibre images are reduced in thickness until they are just one pixel thick. This process is called skeletisation and is essential for the unambiguous use of later digital manipulation.

Figure 4.6  Fibre crossing and touching points are detected. A digital blank spot is inserted at the fibre joins. This has the effect of separating the fibres.
Figure 4.7 Fibres are rejoined and measured. Only fibres within a predetermined distance and angular range of each other will be joined. The length of fibre lost due to the skeletising process can be also be readded at this point.

Figure 4.8 In order for no long fibre to be mismeasured the border surrounding the image frame would need to be at least as large as the longest possible fibre, e.g. 2.6mm. This would impose huge memory and resolution restrictions upon the image analyser.
Figure 4.9 Fibres whose lowest point lies within the live frame are measured. Fibre (A) and Fibre (B) are measured correctly. Fibre (C) is measured, but only to the edge of the visual screen. The measured lengths are represented by the arrows.

LOG-NORMAL F.L.D
15% Glass Fibres

Figure 4.10
Figure 4.11 Fibre Orientation Distributions observed in optical transmission microscopy through thin sections taken through the specimens mid-plane. Section through the thickness shown above. Section through the breadth shown below.
Figure 4.12  Sections taken perpendicular to the three major axes produces a good description of the overall fibre orientation distribution of the composite. However, the quantitative use of this data is highly complex.
Figure 4.13  The composite can be treated as a three ply laminate. The Krenchel orientation factor can be calculated for the skin and core and then for the composite as a whole.

FIBRE ORIENTATION DISTRIBUTION
CENTRAL PLANE OF SPECIMEN

Figure 4.14  Computer generated FOD from the data obtained from the image analyser.
Fibre Length Distribution
Original and Adjusted Curves

Quantimet Curve
Corrected Curve

2000 iterations per point.

LOG-NORMAL F.L.D
15% Glass Fibres
LOG-NORMAL F.L.D
30% Glass Fibres

Figure 4.17

LOG-NORMAL F.L.D
40% Glass Fibres

Figure 4.18
LOG-NORMAL F.L.D
Comparing Fibre Loading

Fibre Volume (%) vs Fibre Length (microns)

Fibre Length Distribution
30% Glass Extruded Granule

Figure 4.19

Figure 4.20
Fibre Length Distribution
30% Glass Moulded Sample

Figure 4.22 Thin slices were taken from five positions through the thickness (y plane) and breadth (x plane). Image analysis techniques were employed to determine the fibre orientation distributions of these sections.
Fibre Orientation Distribution
15% Glass & 30% Rubber

Section taken at surface

Figure 4.23

Fibre Orientation Distribution
15% Glass & 30% Rubber

0.5 mm from surface

Figure 4.24
Fibre Orientation Distribution
15% Glass & 30% Rubber

Section taken near surface

Section taken at centre
Fibre Orientation Distribution
30% Glass & 30% Rubber

Fibre Orientation Distribution
40% Glass & 30% Rubber
Figure 4.31  Holes left after the rubber has been dissolved from the PP show an even distribution and size.

Figure 4.32  The remnant holes are not spherical in shape. They exhibit an ellipsoid with the primary axis in the melt fill direction.
Figure 4.33 The remnant rubber holes, in and around the channel left by a fibre which was pulled-out under cryogenic conditions, show that there is no segregation of rubber to the fibre surfaces.

Figure 4.34 Rubber particles dissolved from the fracture surface of a Charpy specimen broken under cryogenic conditions leave holes showing their original position.
Figure 4.35 The opposite fracture face to that shown in figure 4.34, in this case without the rubber having been dissolved. The rubber can be seen protruding from the fracture surface.

Fibre Length

Figure 4.36 The fibre length counts from the image analyser, $N_i$, are made up from counts of correctly measured fibres, $N_c$, and counts from incorrectly measured fibres $N_m$. 

Fibre Volume

- Proportion of fibres mismeasured, $N_m$
- Proportion of fibres correctly measured, $N_c$
The counts from the image analyser can be corrected by discounting the mismeasured fibres and then applying a correction factor based on the probability that a fibre of length $L_i$ would be mismeasured.

\[ n_i = \frac{N_c}{1 - P} \]

**The Probability of Mismeasuring Fibres**

Visual Screen 880x688, Live Frame 800x625, Pixel Size 2.74 microns
\[ g_{i,i+x} \] is the fraction of mismeasured fibres of length \( L_{i+x} \) which are measured as length \( L_i \).

\[ f_{i+x} \] is the fraction of fibres of length \( L_{i+x} \) which are incorrectly measured.

\[ n_{i+x} \] is the true number of fibres of length \( L_{i+x} \).

**Figure 4.39** The total contribution from mismeasured fibres is made up from fibres of all the longer fibre ranges.

**Figure 4.40** Fibres are broken during extrusion by the action of high curvature bending. The resultant fibre ends and fibre fragments have a triangular profile.
5. FIBRE/MATRIX INTERFACE

When an external stress is applied to a SFRTF specimen, the load is transmitted from the matrix to the fibre through the fibre/matrix interface. Two models describe the stress profile developed in the fibre, namely the Cox and the Kelly and Tyson models (for detailed descriptions see section 2.3.1). In both models the tensile stress in the fibre increases from approximately zero at the fibre ends to a maximum value at its centre. The maximum stress is limited by one of two competing processes. If the load transferred into the fibre reaches the failure strength of the fibre the fibre will break into two pieces. A small increase in the external load perpetuates the fibre breakage process until all fibre fragments are less than or equal to a minimum fibre length at which the maximum tensile stress can reach the fibre tensile strength. This length is termed the critical fibre length, $l_c$. At this point the tensile stress in the fibre cannot reach the fracture stress of the fibre and no further fibre fragmentation will occur.

As discussed in chapter 2.3.1 Kelly and Tyson\(^1\) have related the critical length to the interfacial shear strength, $\tau$, through a force balance around an embedded fibre (figure 2.12) and obtained the following equation for the critical length:

$$l_c = (\sigma d)/(2\tau) \quad (5.1)$$

where $\sigma$ is the ultimate tensile strength of the fibre, and $d$ is the diameter of the fibre.
Thus, if the critical length for a fibre embedded in a matrix can be determined experimentally, the fibre/matrix interfacial shear strength between the fibre and the matrix can be evaluated for a fibre of known strength and diameter. Attempts have been made previously by many other workers to measure directly the critical length of a fibre embedded in a thermoset resin. Most of these studies are based on the measurement of fibre pull-out lengths, fibre pull-out forces, or the fibre fragment lengths of an embedded fibre subjected to a tensile force.

Work on fibres embedded in thermoplastic matrices is limited and more complex to carry out due to the difficulties associated with specimen preparation. Thermosetting resins can be handled and prepared with ease under standard laboratory conditions resulting in a representative interface. Thermoplastic matrices need to be heated to temperatures in excess of their melting point in inert conditions, as they are subject to oxidation, and commonly under the influence of shear forces. The equipment necessary to achieve these conditions does not lend itself to the preparation of specimens containing a single fibre. The fibre/matrix interface in the specimen needs to be as similar as possible to that produced during extrusion and injection moulding. Representative fibres need to be dispersed well, so as not to influence each other, and aligned to the loading direction. It was with these problems in mind that a series of specimen preparation techniques was developed.
5.1. SINGLE FIBRE SPECIMEN PREPARATION

Ideally one would like to produce well dispersed aligned glass fibres through an injection moulding technique, but this was not possible with the equipment available. It was thus decided to attempt to mould polymer around single long fibres through a combination of pressure and temperature. A fibre fragmentation technique could be employed to evaluate $l_c$ from the resultant specimen. Early techniques utilised powder and granules melting around individual fibres. Problems associated with these techniques were entrapped gases and degradation of the polymer. Degradation was overcome to a large extent by enclosing the system in an inert gas. The entrapped bubbles were reduced through moulding thinner sections. However the problems were still encountered to an unacceptable extent and the material was not of an acceptable standard.

The failures of the earlier techniques led to a number of criteria that any subsequent technique would need to fulfil in order to work successfully:

- The specimen needs to be consolidated under pressure and temperature to reduce entrapped gases in the specimen;
- The preparation technique is required to be as brief as possible and in the absence of oxygen in order to minimise polymer degradation;
- The final specimen needs to be as thin as possible to enable the fibre to be observed in situ and hence the fibre fragment lengths to be measured directly.
5.1.1. **Compression Moulding**

A completely new lamination procedure was developed. Strips of polypropylene, less than 1mm in thickness, were produced. Two such strips were laminated together with aligned fibres positioned between their opposing faces. This resulted in a thin strip of polypropylene with fibres bonded in its centre.

A new mould was designed to produce the thin strips. A drawing of the mould is shown in figure 5.1. The compression mould consisted of a male tool that slots exactly into a rectangular female slot where the polymer was positioned. At the base of the female slot was a sheet of PTFE supported on a glass fibre mesh. This acted as a release film, and also appeared to help the flow of the molten material.

Both parts of the mould were placed on a hot plate, and heated to 250°C. The mould parts were taken from the hot plate and spacers placed on the female part to determine the thickness of the final strip. A previously moulded unnotched Izod specimen was placed into the centre of the slot of the female mould. The male part was introduced to the female slot, aligned and inserted. The mould was now at a considerably lower temperature, circa 210°C, than when it left the hot plate. The mould was placed in a press, which was at room temperature, and placed under considerable pressure. This forced the polymer to flow down the channel left between the mould parts by the spacers. Excess material was forced out of the ends of the mould. The platens of the press were cooled with water, and acted as heat sinks for
the mould. The mould was cooled directly by compressed air jets aimed at its exposed flanks.

When the mould was cool, after 20 minutes, the mould was opened and the new thin tape removed. These tapes were usually defect free. The tape was then cut into two, half way along its length. One face of each half was ground down to produce a pristine surface uncontaminated by reaction with either the mould or PTFE surfaces. These two faces would become the bonding surfaces for the fibres in the centre of the final coupon. A dozen or so glass fibres were laid parallel down the length of one tape and a drop of superglue applied to hold them in position prior to final consolidation. The other tape was positioned face down on the fibres and bonded to the other tape by the superglue at its ends.

The mould parts were reheated to a temperature of about 225°C, i.e. slightly less than the that of the first cycle. The consolidation process was repeated as in the first stage, but with the laminated tapes and fibres replacing the Izod specimen. The spacers were set to 1.5 times the thickness used in the tape preparation. This was done to promote limited polymer flow down and around the fibre length to ensure good bonding.

Defect free specimens were possible, but usually small bubbles of air became entrapped between the two tapes and as a result there were small bubbles in the final specimen. These defects were cut out, or positioned so that they lay in the grip face of the Instron during testing.

Prior to testing the coupons faces were prepared for optical microscopy. Any surface defects, such as the texture imprinted
from the glass fibre matt of the PTFE, were ground away, using carborundum papers, to a 4000 grit finish. This produced an almost polished surface appearance. The surfaces were not actually polished as this tended to disfigure the surface.

Ideally the coupons should have been tested in situ under an optical microscope on a straining stage. However this was not possible during the period of this work due to an updating of microscope facilities at the University. Instead the coupons were strained to 10% in an external testing machine. At this extension the matrix was extended well beyond the breaking strain of the fibre, ensuring that fibre fragmentation was complete. The single fibre tensile test specimens were not loaded to complete failure in order to avoid the failure of the loaded specimen sending shock waves through the coupon and possibly producing further damage in the fibre. The coupon was slowly unloaded and removed for further analysis.

The coupons'surfaces'were given a further grind on the 4000 grit, and then observed using transmission optical microscopy. Representative samples are shown in figures 5.2 and 5.3 for the rubber toughened grade specimens, polarised light showed regions of residual stress after unloading.

In the untoughened grades, subsequent to loading, transmission microscopy revealed fibre breaks and debonds as dark regions in and along the fibres length respectively, figure 5.4. The graduated microscope stage travel and the cross-hairs in the optical turret made it possible to measure the distance between the fibre breaks.
A histogram can be constructed from the measured fragment lengths. Following Kelly and Tyson theory a normal distribution of fragment lengths should be obtained varying from $\frac{1}{4}l_c$ to $l_c$, with the peak of the histogram at $3/4 l_c$. Normally the fragment length distribution is broader than the 2:1 ratio predicted. This is due to the existence of flaws in the fibre, thereby causing its strength to depend on length. However the peak of the broadened histogram should still correspond to $3/4 l_c$. On a cumulative frequency curve this corresponds to the 50% count.

5.1.2. Matrix Selection for Single Fibre Coupons

Initial single fibre specimens were limited to the unreinforced grades which had already been moulded into Izod bars. This allowed samples to be produced with polypropylene, PP and 10% rubber, PP and 30% rubber, and PP and coupling matrices. Problems were encountered with the rubber toughened grades. These grades stress-whitened as they were loaded, obscuring the fragmented fibre. It was possible to obtain results from the 10% rubber grades, but not from grades with higher rubber contents. This limited the testing to three grades. To see the influence of the rubber and the coupling combined it was necessary to produce a new grade, (PP, 10% rubber and coupling). It would have been very arduous to carry out the full compounding, extrusion and injection moulding. Fortunately, small quantities of extruded granules and constituent elements from all of the previously prepared grades had been kept. Granules of polypropylene with coupling, AH1B, were mixed with
rubber granules to produce a 10% rubber coupled grade. This was then injection moulded into Izod bars using the small injection moulder at the University of Surrey. Conditions were kept as similar as possible to those used at ICI.

Single fibre specimens were thus produced in polypropylene with combinations of 10% rubber and/or coupling.

5.2. FIBRE/MATRIX INTERFACE RESULTS

It has been shown by other workers\textsuperscript{2} that the fragmentation technique gives results which are not absolute interfacial properties, but which are highly dependent on the initial stress state of the fibre. However the technique does give good comparative results. An important consideration is to try to ensure that the specimen preparation technique mimics the creation of the fibre/matrix interface normally produced in extrusion compounding and injection moulding. Determining the effectiveness of this procedure is virtually impossible without more complex analytical techniques. The above considerations mean that the results detailed in this chapter must at best be approximations of the interfacial shear properties.
5.2.1. Interfacial Shear Strength

The fragment lengths are measured and the count of each length is plotted against fibre length either on a bar chart or as a cumulative curve. Figures 5.6 and 5.7 show such curves for the combined number of measured fibre fragments from several coupons containing many fibres. All curves show a reasonable approximation to a normal distribution of fibre lengths. The curves with the largest critical length have the greatest range, as would be expected by weak link fibre theory.

The critical lengths for the four matrices are determined as:

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Critical Length</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polypropylene Homopolymer</td>
<td>2.20 mm</td>
</tr>
<tr>
<td>Polypropylene and Coupling</td>
<td>1.23 mm</td>
</tr>
<tr>
<td>Polypropylene and 10% Rubber</td>
<td>4.67 mm</td>
</tr>
<tr>
<td>PP, 10% Rubber and Coupling</td>
<td>2.45 mm</td>
</tr>
</tbody>
</table>

Table 5.1 Fibre/Matrix critical length

The critical length would be expected to decrease with the addition of coupling and increase with the addition of rubber and this is observed experimentally. However, the effect of the 10% rubber addition on the measured critical length is greater than might be expected from simple particulate theory.

The Kelly and Tyson interfacial shear strength, \( \tau \), is calculated from:

\[
l_c = \frac{\sigma_{uf} - r_f}{\tau}
\]

where:

\[
\begin{align*}
    r_f &= 7.0 \times 10^{-6} \text{ m} \\
    \sigma_{uf} &= 3.4 \times 10^9 \text{ Pa}
\end{align*}
\]

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The bulk strength data of the fibre were obtained courtesy of the fibres manufacturer. The interfacial shear strength is calculated as:

<table>
<thead>
<tr>
<th>Material</th>
<th>Shear Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polypropylene homopolymer</td>
<td>10.80</td>
</tr>
<tr>
<td>Polypropylene &amp; coupling</td>
<td>19.30</td>
</tr>
<tr>
<td>Polypropylene &amp; 10% rubber</td>
<td>5.10</td>
</tr>
<tr>
<td>PP &amp; 10% rubber &amp; coupling</td>
<td>9.70</td>
</tr>
</tbody>
</table>

Table 5.2 Measured interfacial shear strength

The work of Folkes et al.\textsuperscript{3} measured that for glass fibres in polypropylene the critical length is about 700 microns and the interfacial shear strength 30 MPa. The results obtained in this work suggest a substantially weaker interfacial bond. This is probably a result of the reduced wetting of the fibre by the molten polypropylene. Possible reasons for these discrepancies are most likely to be associated with the specimen preparation. Poor interfacial bonding may be due to:

- very limited flow of polymer around the fibre;
- entrapped air around the fibre;
- degradation of the polymer due to four melting cycles.

Despite the quantitative results of these series of experiments falling short of the expected results good comparative behaviour can be seen. The addition of coupling and rubber increases and decreases respectively the interfacial shear strength and modulus.

The results of these tests are applied, with the results of the microstructural characterisation and base material properties to theoretical mechanical models in chapter six.
References

1. Kelly A. and Tyson W.R.
   Tensile Properties of Fibre-Reinforced metals: Copper/Tungsten and Copper/Molybdenum.

2. Piggott M.R.
   Debonding and Friction at Fibre-Polymer Interfaces. I: Criteria for Failure and Sliding.

3. Folkes M.J., Hornsby P.R., Shipton P.D. and Wong W.K.
   Evaluation of Interfacial Shear Strength in Glass, Carbon and Kevlar Fibre Reinforced Thermoplastics
Fig 5.1  The single fibre compression mould. Izod bars are compressed in the central slot to produce thin strips. Laminated strips and continuous fibres are compression bonded to produce final thin specimens.
Figure 5.2  Rubber toughened grades exhibit residual stress which can be observed as different colours under polarised light. Scale bar = 10 microns.

Figure 5.3  A Fibre end shows the redistribution of stress into the matrix. Scale bar = 50 microns.
Figure 5.4 Grades not containing rubber were observed in plane transmission microscopy. Fibre breaks and debonds can be observed as dark regions down the fibres length. Scale bar = 50 microns.

Figure 5.5 Many fibre fragment lengths need to be measured in order to evaluate the fibre/matrix critical length. Scale bar = 100 microns.
SINGLE FIBRE FRAGMENTATION
Effectiveness Of Coupling

Figure 5.6

SINGLE FIBRE FRAGMENTATION
Effect Of Coupling & Rubber

Figure 5.7
6. MECHANICAL MODELLING

Many workers have attempted to model the mechanical properties of short fibre reinforced polymers, with differing levels of success. The fundamental problem in modelling such materials is the quantification of the large number of compositional variables, such as $\tau$, $l_c$, and the fibre length/orientation distributions. In the course of this project all these parameters have been measured. The aim of the present chapter is to use these data in appropriate models to predict the stress/strain response and strength of any rubber toughened and glass reinforced polypropylene composite accurately.

6.1. ASSUMPTIONS WITHIN THE MODEL

To aid in modelling this particular system, certain assumptions have been made:

i) The duplex matrix phase of rubber spheres in continuous polypropylene is treated as a single homogeneous phase using modified particulate theory;

ii) Fibres carry load in a Kelly and Tyson model type profile. This allows the results of the single fibre fragmentation test to be used directly;

iii) The interfacial shear stress is a function of the composite strain (discussed in section 2.3.3).

The models are based on a modified version of the Bader and Bowyer\textsuperscript{1} concept of a strain dependent critical length. At any
particular strain the response of the composite can be separated into two distinct contributions - that of the matrix, (the mixture of rubber and polypropylene) and that of the fibres, (those of sub and super critical fibres). Firstly we will consider the behaviour of the matrix. Only then can the fibre contribution be evaluated since the fibre/matrix interface is affected by the matrix properties.

6.2. THE MATRIX

Modelling of the stress-strain response of a fibre filled polymer requires initially a knowledge of the loading response of the unreinforced matrix. The stress/strain response of the polypropylene was measured and recorded in the tensile tests described in chapter 3. Using particulate theory the effect of the rubber can then be modelled in a simple way as outlined below.

The rule-of-mixtures approximation we use treats the filler, (rubber in this case) as a cube at the centre of a unit cube of matrix, figure 6.1. The cube of rubber has sides of length $F_r^{1/3}$ so that the volume fraction is $F_r$.

The horizontal slab containing the reinforcement has the Reuss modulus (section 2.3.1), $E_B$:

$$E_B = E_r V_r + E_p V_p$$  \hspace{1cm} (6.1)

where;

$E_p$ is the Youngs modulus of polypropylene,
$E_r$ is the Youngs modulus of rubber,

$E_m$ is the Youngs modulus of composite matrix.

The volume fraction of rubber, $V_r$, in slab B is $F_r^{2/3}$.

Hence:

$$E_B = E_r F_r^{2/3} + E_p (1 - F_r^{2/3}) \quad (6.2)$$

Considering the cube as a whole, slab B is in parallel with slab A and C, and hence the Voigt model (section 2.3.1) is applicable, thus:

$$E_m = \frac{E_B E_p}{E_B (1 - V_B) + E_p V_B} \quad (6.3)$$

where $V_B$ is the volume fraction of slab B in the unit cube, given by:

$$V_B = F_r^{1/3} \quad (6.4)$$

Hence:

$$E_m = \frac{(E_r F_r^{2/3} + E_p (1 - F_r^{2/3})) \cdot E_p}{(E_r F_r^{2/3} + E_p (1 - F_r^{2/3})) (1 - F_r^{1/3}) + E_p F_r^{1/3}} \quad (6.5)$$

Or more elegantly:

$$E_m = \frac{(E_p + (E_r - E_p) \cdot F_r^{2/3}) \cdot E_p}{E_p + (E_r - E_p) \cdot F_r^{2/3} \cdot (1 - F_r^{1/3})} \quad (6.6)$$

The results from the mechanical testing discussed in section 3 give the modulus of the composite matrix at different rubber levels. The modulus of the pure rubber is not known but can be
estimated from a knowledge of the mechanical properties of various rubber toughened polypropylene grades using equation (6.6). This gives the modulus of the rubber as being negative, and varying significantly between different rubber levels. Obviously this rule-of-mixtures model can not be a true representation of the influence of the rubber upon the matrix. The magnitude of the modulus of a typical polyolefin rubber is of the order of a few megapascals, whilst the modulus of the polypropylene is of the order of 2 gigapascals, three orders of magnitude difference. A small addition of rubber will reduce the modulus of the matrix substantially as the difference in compliance is so great. If it is assumed that the rubber contributes effectively zero stiffness, then the rubber particles can be envisaged as small voids within the matrix.

Making this assumption, equation (6.6) reduces to:

\[ E_m \approx (1-F_r)E_p \quad (6.7) \]

Even using the above simplification, the mechanical response of the rubber toughened polypropylene grades is only approached loosely. The problem arises in that the reduction of matrix stiffness does not equate in a linear manner to the addition of rubber. Equation (6.7) underestimates the reduction in modulus as a result of small additions of rubber. Further additions of rubber have a further but less significant detrimental effect. This type of behaviour can be represented by a power law response of the form:
where $x$ is a constant describing the rate at which the rubber softens the matrix. Equation (6.8) can be rearranged and $x$ evaluated from the best fit to the experimental data for the rubber toughened polypropylene grades. The constant is found to be 0.69 for the AH2 grade materials and 0.56 for the AH1 grade materials. This difference in the exponent is consistent with the differences in mechanical properties of identical grades produced in different campaigns.

Substituting stress and strain for the moduli, equation (6.8) can be rewritten:

$$\frac{\sigma_m}{\varepsilon_m} = \frac{\sigma_p}{\varepsilon_p} \cdot (1-F_r)^x$$

Equating the strain in the matrix, $\varepsilon_m$, to the strain in the polypropylene, $\varepsilon_p$, allows the stress in the matrix, $\sigma_m$, to be calculated from the stress in the polypropylene, $\sigma_p$, at that same strain. Figure 6.2 shows the curve fits for the 10% rubber (AH1C) and 30% rubber (AH1D & AH2B) modified grades.

It will be important to incorporate the deviation from the rule-of-mixtures when modelling the fibre/matrix interface behaviour in subsequent sections.
6.3. THE COMPOSITE

The stress/strain response of the composite is to be modelled using the analysis developed by Bader and Bowyer\(^1\) as described in section 2.3.3. In this manner the fibre length distributions evaluated in chapter 4 can be used to calculate the fibre contribution through an iterative process in a computer program. In an advancement to the basic model, the interfacial shear stress is modelled as changing with the composite stress, and the maximum interfacial shear stress is dependant on the matrix composition.

The yield strength of the composite grades can also be approximated from a modified rule-of-mixtures type prediction. This is a very simplistic model and does not account for any of the microstructural weakening factors such as fibre cross-over density and fibre end fracture initiation.

To reiterate, in a material containing short fibres it is necessary to relate the stress in the fibres to the applied stress. The Kelly-Tyson\(^2\) relationship assumes a linear build-up of stress from the fibre ends. The critical length of fibre is defined as the minimum fibre length required for the stress in the fibre to build up to the fracture strength of the fibre, \(\sigma_f^*\),

\[
 l_c = \frac{r \sigma_f^*}{\tau} \tag{6.10}
\]

where \(2r\) is the diameter of the fibre and \(\tau\) is the interfacial shear stress. This is related to the shear strength of the
matrix or the strength of the fibre-matrix interface. Throughout this chapter a Kelly and Tyson fibre stress profile will be assumed.

### 6.3.1. Stress/Strain Response

The non-linear response of a short fibre composite is the result of two processes. Firstly, the non-linear base polymer response, discussed in the previous section, and secondly the reinforcing contribution of the fibres. As there is a range of fibre lengths the individual fibres do not contribute equally. As the strain in the composite is increased the shorter fibres reach the maximum load beyond which they can carry no greater load. At any composite strain there will be a discrete fibre length at which that fibre is supporting its maximum load. This length is termed the 'critical length'. This redefinition of the critical length allows the critical length to be related to the strain. Increasing the strain in the composite will allow eventually the stress in a sufficiently long fibre to reach the tensile strength of the fibre. At this point the fibre will fracture - this length being the previously defined 'composite critical length'. A slight modification to the Kelly and Tyson\(^3\) critical length gives the strain dependant critical length, \(l_c\):

\[
l_c = r\varepsilon_c E_f/\tau
\]

where:

- \(r\) is the fibre radius;
- \(\varepsilon_c\) is the strain in the composite;
$E_f$ is the fibre modulus;
$	au$ is the interfacial shear strength.

Bader and Bowyer\textsuperscript{1} developed a summation process to evaluate the contribution of individual components to the composite stress. For any particular strain $X$, $Y$ and $Z$ (equations 6.12 to 6.15) are the individual contributions from the subcritical fibres, supercritical fibres and matrix respectively to the composite stress.

\begin{equation}
E_C \varepsilon_C = \sigma_C = \eta_0 (X + Y) + Z 
\tag{6.12}
\end{equation}

where;

\begin{equation}
X = \Sigma_{\text{subcritical}} \tau l_i V_i / 2r_f \tag{6.13}
\end{equation}

\begin{equation}
Y = \Sigma_{\text{supercritical}} E_f \varepsilon_C(1-(E_f \varepsilon_C r_f/2l_i \tau)) \tag{6.14}
\end{equation}

and;

\begin{equation}
Z = E_m \varepsilon_C (1-V_f) \tag{6.15}
\end{equation}

where $V_i$ and $V_j$ are the volume subfractions of fibres with lengths $l_i$ and $l_j$, respectively, $E_m$ is the matrix modulus and $V_f$ the volume fraction of all fibres. The $\eta_0$ term has been added to account for the non-alignment of the fibres.

Equations 6.12 to 6.15 are calculated within a computer model which takes as input parameters the fibre length distributions and the base polypropylene response. The computer programme steps at small increments of strain from zero to an input specified strain at yield (this value is very difficult to calculate theoretically as will become apparent later in this chapter). At each step it calculates the critical length at that strain and then calculates the sum of the contributions from the
sub and supercritical fibres using an interfacial shear strength assigned as discussed in the next section. The stress carried by the fibres is added to that of the matrix (corrected appropriately for the addition of rubber). The process is then repeated for each additional increase in strain, resulting in a stress-strain response for the whole composite up to the defined yield strain.

6.4. INTERFACIAL SHEAR STRESS

The original model developed by Bader and Bowyer \(^1\) assumed a constant interfacial shear stress. This assumption is clearly an over simplification. Gupta and Mittal \(^3\) in later work found the interfacial shear stress to be related to the composite stress by a constant factor. The interfacial shear stress was also found to be very closely related to the response of the base polymer (matrix) stress strain profile. Applying the Bader-Bowyer and Gupta-Mittal interfacial shear stress hypothesis to the model gives significantly different results. Figure 6.3 gives stress strain responses from both models with identical initial data. The Bader and Bowyer assumption gives high stresses and moduli at low strains, but a more realistic load response at higher strains. Conversely, the Mittal and Gupta work tends to give lower more accurate stresses than the Bader and Bowyer model at low strains but overestimates the stress increase at higher strains.

Both of these models fail to predict the manner in which the interfacial shear stress changes with strain over the total
strain range of the composite. The Mittal and Gupta work is probably a very close approximation at low strains, but at higher strains, especially in a relatively ductile matrix, it seems more likely that the interfacial shear stress will level out, and possibly even peak as the matrix begins to yield. At these high strains the Bader Bowyer hypothesis is more likely. To enable both effects to be approximated, a power law fit can be used of the form:

$$\tau = \tau_{\text{max}} \cdot (\sigma_{\text{pp}}/\sigma_{\text{ppy}})^y$$

(6.16)

where:

- $\tau_{\text{max}}$ is the maximum interfacial shear stress (from the fibre fragmentation test);
- $\sigma_{\text{pp}}$ is the stress in the polypropylene base polymer at any composite strain, $\varepsilon_c$;
- $\sigma_{\text{ppy}}$ is the stress in the composite at matrix yield;
- $y$ is found to be 0.35 from data analysis.

The form of the interfacial shear stress from equation (6.16) is plotted against strain in figure 6.4. In this manner the interfacial shear stress rises rapidly at low strains, approaching the maximum value quickly. This is a purely empirical approach and appears to be a sensible compromise between the theories proposed previously.

The interfacial shear stress will also be influenced by the matrix composition. In chapter five the addition of low levels of rubber to the matrix was shown to reduce the interfacial shear strength significantly. It is reasonable therefore to assume that the interfacial shear stress will be reduced on the addition
of rubber in a similar manner to the elastic modulus of the matrix, i.e:

\[
\tau_{\text{matrix}} = \tau_{\text{PPmax}} \cdot \left( \frac{\sigma_{\text{PP}}}{\sigma_{\text{PPy}}} \right)^Y \cdot (1 - F_r)^X
\]  

(6.17)

In this way a single factor \((x = 0.69)\) accounts for the effect of the rubber on the mechanical properties of base polymer, the effect on the stress transferred at the fibre matrix interface, and hence on the critical fibre length at any strain.

From the analysis of this section it is possible now to account for the change in interfacial shear stress with increasing strain, and to allow also for the reduction in the interfacial shear stress caused by the addition of rubber to the matrix.

These factors are incorporated in both the composite stress/strain and following yield strength predictive processes. A listing of the computer programme developed to predict these properties is given in the appendices at the end of this chapter.
6.5. YIELD STRENGTH

Taking the Kelly and Tyson stress profile of a loaded short fibre it is a simple operation to calculate the average stress in a fibre whose length is shorter than the composite critical length:

\[ \sigma_f = \frac{1}{2l_c} \sigma_f^* \]  \hspace{1cm} (6.18)

The strength of the composite by a simple rule of mixtures analysis will be:

\[ \sigma_c^* = \left( \frac{1}{2l_c} \right) \sigma_f^* V_f + \sigma_m^*(1-V_f) \]  \hspace{1cm} (6.19)

For a composite containing fibres longer than \( l_c \) the strength is given by:

\[ \sigma_c^* = \left( \frac{1-1_c/2}{l} \right) \sigma_f^* V_f + \sigma_m^*(1-V_f) \]  \hspace{1cm} (6.20)

If some of the fibres are longer than the critical length, \( l_j \), and some are shorter, \( l_i \), combining equations (6.19) and (6.20) gives the (sometimes called modified rules-of-mixtures) strength of the composite as:

\[ \sigma_c^* = \Sigma \left( \frac{l_i \sigma_f^*/2l_c}{V_i} \right) + \Sigma \left( \frac{\sigma_f^* (l_j - l_c/2)}{l_j} \right) V_j + \sigma_m^*(1-V_f) \]  \hspace{1cm} (6.21)

where:

\[ \Sigma V_i + \Sigma V_j = V_f \]  \hspace{1cm} (6.22)
The stress in the matrix, \( \sigma_m' \), can be approximated from the stress in the rubber toughened polypropylene (equation (6.8)) where \( x \) is 0.69, thus producing equation 6.25. The fibre orientation factor, \( \eta_o \), evaluated in chapter 4, can be applied to equation 6.21 to account for the non-alignment of the fibres. The final equations are thus:

\[
\sigma^* = \eta_o \sigma_{(fibres)} + (1-\nu_f)\sigma_{(matrix)}
\]

(6.23)

where;

\[
\sigma_{(fibres)} = \sum (1_i \sigma_i^*/2l_c)V_i + \sum (\sigma_f^*(l_j-1)/l_j)V_j
\]

(6.24)

\[
\sigma_{(matrix)} = E_p(1-F_T)^{0.69}
\]

(6.25)

and;

\[
\sum V_i + \sum V_j = V_f
\]

(6.22)

A computer programme was developed to sum the strength contribution of the sub and supercritical fibres at the experimentally measured failure strain. The contribution of the matrix was added to this to give an approximation of the strength of the composite. A listing of the computer programme is given in the appendices at the end of this chapter.
6.6. PREDICTIVE CURVES

Within this section the predictive stress-strain curves of the rubber toughened and short fibre reinforced materials are presented.

6.6.1. Model Coefficients

To generate the model predictions it is necessary to assign reasonable values to the material variables:

- $E_f = 73 \times 10^9$ Pa
- $r_f = 7 \times 10^{-6}$ m

* $\tau_{\text{max}} = 25 \times 10^6$ Pa (Coupled)
* $\tau_{\text{max}} = 14.8 \times 10^6$ Pa (Uncoupled)

Polypropylene stress/strain response stored in data file FLD's stored in data files

Glass 15 Wt% = $V_f(5.40)$ : Orientation factor $\eta_0 = 0.804$
Glass 30 Wt% = $V_f(10.8)$ : Orientation factor $\eta_0 = 0.781$
Glass 40 Wt% = $V_f(14.4)$ : Orientation factor $\eta_0 = 0.765$

Rubber weight percent is equal to volume percent (0-40%)

* The maximum interfacial shear stress evaluated from the fibre fragmentation test was found to give low results when applied to the model. Both the coupled and uncoupled results were increased by the same factor to give values more consistent with other workers$^{3,4,5}$.

(7) The orientation factor is calculated from a through thickness average.
6.6.2. Model Results

Figures 6.2 and 6.5 to 6.12 give a range of the curves predicted by the modified Bader Bowyer stress/strain model along side the experimental curves. In general, it can be seen that the model and experiment are in very close agreement. The coupled grades give the best agreement for the largest strain range. The uncoupled grades give good agreement at low strains, but at higher strains the model overestimates the experimental data (figure 6.12). This is probably due to the frictional fibre/matrix interface of the uncoupled grades debonding at the higher strains. This effect would cause the interfacial shear stress profile to peak at much lower loads than is the situation for the coupled grades. Following the debonding, the interfacial shear stress may fall or will at best remain constant. The debonded fibres can carry no additional load and this results in the reduced yield strength and strain to yield.

Figures 6.13 to 6.15 give the modified rule-of-mixtures prediction for yield strength of the composite grades. For the fibre filled grades, figure 6.13, the model overestimates the yield strength substantially, particularly for the uncoupled grades. The model is deficient in that it does not predict the yield of the matrix around the fibres, more predominant in the coupled grades. Similarly, the model does not account for the internal notching effect of the fibres promoting failure, particularly in the uncoupled grades.
The addition of rubber to the unreinforced grades tends to extend the strain to yield of the polymer. This increases the relative strength of the polymer at yield. The model does not account for this and hence underestimates the yield strength, figure 6.14.

The model predicts that the addition of rubber to the fibre filled grades decreases the strength of the composite, figure 6.14. However, again the model overestimates the experimentally determined results especially for the uncoupled grades. Again, this is primarily as a result of the fibres debonding from the matrix and initiating failure.

In conclusion, prediction of composite strength is inherently difficult because the onset of yield is assisted by internal stress concentrations such as those caused by the notching effect of the fibres or the interfacial failure at the fibre ends. However, the stress/strain response of the composite can be predicted with confidence within the limitations of micro yield in the matrix. For the coupled grades, this extends almost to the yield point of the composite. In the uncoupled grades it is most probable that fibres debond at low strains reducing their efficiency and thus that of the composite. Debonded regions provide a fracture path for micro fracture inducing premature yield. A single power law exponent describes the effect of the rubber in the matrix and also on the fibre/matrix interface. This factor has a significant effect upon the calculated critical length at any particular strain, and hence upon the reinforcing efficiency of the fibres. It has been postulated that the
interfacial shear stress follows a power law relationship of the base polymer response. This provides a compromise between a rapidly increasing shear stress at low strains and a constant interfacial shear stress at higher strains when the matrix yields around the fibre.

References


CHAPTER SIX

Appendix A

Turbo Basic programme listing for composite stress/strain response evaluation.

REM *THIS PROGRAM CALCULATES THE STRESS STRAIN RESPONSE OF GLASS*  
REM ***FIBRE AND RUBBER TOUGHENED PLASTICS USING AN ITERATIVE****  
REM *****PROCESS WHICH SUMS THE CONTRIBUTION OF SUB AND SUPER*****  
REM **********CRITICAL FIBRES TO THE KELLY-TYSON MODEL **************

mfl=2280e-6  
$STACK 5000  
REM **mfl=maximum fibre length  
ss=40e-6  
REM **ss=step size  
DIM fld(2,mfl/ss)  
DIM strain(124)  
DIM stress&(124)  
retry:  
GOSUB systemdata  
GOSUB fld  
RESTORE  
GOSUB mainloop  
GOSUB presentation  
LOCATE 25,30  
PRINT "Do you wish to try another plot"  
A$ = INPUT$(1)  
IF a$="y" THEN  
GOTO retry  
ELSE  
END IF  
END

mainloop:  
REM ** MAIN LOOP **  
store=0  
FOR ec=0 TO (0.001+mstr/100) STEP 0.0005  
PRINT USING "###.####";ec  
fr=fro”.69  
READ sp  
t=tau*(sp^0.35)/(30.75e6^0.35)  
IF t>tau THEN t=tau  
IF t=0 THEN t=1  
l=rf*ef*ec/(t*(1-fr))  
GOSUB critical  
GOSUB subcrit  
GOSUB supercrit  
fra=(fr*(1-vf))  
z=(1-fra)*sp*(1-vf)  
sc=n*(x+y)+z  
GOSUB storedata  
NEXT ec
RETURN

173
fld:

REM ** THIS ROUTINE LOADS DATA OF THE RELEVANT **
REM ** FIBRE LENGTH DISTRIBUTION *******************
IF vf<.07 THEN RESTORE fldfifteen
IF vf>.07 AND vf<.12 THEN RESTORE fldthirty
IF vf>.12 THEN RESTORE fldforty
FOR a=0 TO (mfl/ss)
    READ fld(1,a)
    fld(2,a)=(a*ss)+(ss/2)
NEXT a
RETURN

REM ** DETERMINING THE PERCENT OF SUB/SUPERCRITICAL FIBRES******
critical:
a=0
DO
    a=a+1
    IF a>mfl/ss THEN RETURN
LOOP UNTIL fld(2,a)>li
b=a
RETURN

REM ** CALCULATE CONTRIBUTION OF SUBCRITICAL FIBRES ************
subcrit:
x=0
FOR a=1 TO b-1
    xi=t*(1-fr)*fld(2,a)*(fld(1,a)-fld(1,a-1))*vf/(2*rf*100)
    x=x+xi
NEXT a
RETURN

REM ** CALCULATE CONTRIBUTION OF SUPERCRITICAL FIBRES ************
supercrit:
y=0
FOR a=b TO (mfl/ss)
    yi=ef*ec*(1-(ef*ec*rf/(2*fld(2,a)*t*(1-fr))))*(fld(1,a)-(fld(1,a-1)))*vf/100
    y=y+yi
NEXT a
RETURN

REM ** INPUT DATA ON SYSTEM *****************************
systemdata:
INPUT"Youngs Modulus of Fibre";ef
INPUT"Orientation Factor";n
INPUT"Fibre Radius";rf
INPUT"Interfacial Shear Strength";tau
INPUT"Maximum Strain (%)";mstr
IF mstr>6 THEN mstr=6
INPUT"Volume Fraction of Glass in Composite";vf
INPUT"Volume fraction of Rubber in Matrix";fro
x=0:y=0:z=0:vi=0

REM ** STRESS STRAIN RESPONSE OF POLYMER ******************
00.000, 01.66e6, 02.66e6, 03.66e6, 04.66e6, 05.66e6, 06.66e6, 07.66e6, 08.66e6, 09.55e6
DATA
10.40e6, 11.20e6, 12.10e6, 13.00e6, 14.00e6, 14.66e6, 15.33e6, 16.00e6, 16.66e6, 17.00e6
DATA
17.83e6, 18.33e6, 18.66e6, 19.16e6, 19.66e6, 20.16e6, 20.66e6, 21.00e6, 21.33e6, 21.66e6
DATA
22.16e6, 22.50e6, 22.83e6, 23.06e6, 23.50e6, 23.73e6, 24.16e6, 24.43e6, 24.73e6, 25.00e6
DATA
25.33e6, 25.66e6, 25.93e6, 26.28e6, 26.50e6, 26.66e6, 26.93e6, 27.13e6, 27.33e6, 27.66e6
DATA
28.00e6, 28.27e6, 28.50e6, 28.66e6, 28.87e6, 29.07e6, 29.33e6, 29.57e6, 29.77e6, 30.00e6
DATA
30.23e6, 30.50e6, 30.75e6, 30.90e6, 31.10e6, 31.30e6, 31.40e6, 31.60e6, 31.80e6, 32.00e6
DATA
32.20e6, 32.30e6, 32.60e6, 32.80e6, 32.90e6, 33.00e6, 33.20e6, 33.30e6, 33.50e6, 33.60e6
DATA
33.80e6, 34.00e6, 34.10e6, 34.30e6, 34.40e6, 34.60e6, 34.70e6, 34.80e6, 35.00e6, 35.10e6
DATA
35.20e6, 35.30e6, 35.50e6, 35.60e6, 35.70e6, 35.80e6, 35.90e6, 36.00e6, 36.10e6, 36.30e6
DATA
36.40e6, 36.50e6, 36.60e6, 36.70e6, 36.80e6, 36.90e6, 37.00e6, 37.10e6, 37.20e6, 37.30e6
DATA
37.40e6, 37.50e6, 37.60e6, 37.65e6, 37.70e6, 37.80e6, 37.90e6, 38.00e6, 38.05e6, 38.10e6
DATA 38.15e6, 38.20e6, 38.25e6, 38.30e6, 38.33e6, 38.35e6, 38.36e6
RETURN

storedata:
strain(store)=ec
stress&(store)=sc
store=store+1
RETURN

presentation:
PRINT"Present data as a list of numbers (1)"
PRINT"as a file (2)"
INPUT"or as a graph (3)";pres
IF pres=1 THEN GOSUB numbers
IF pres=2 THEN GOSUB fyle
IF pres=3 THEN GOSUB graph
RETURN

numbers:
REM ** SEND DATA TO THE PRINTER *******************************
REM ** SEND DATA TO THE PRINTER ******************************************
LPRINT:LPRINT
p$ = \\
LPRINT USING p$,"Youngs Modulus of Fibre",ef
LPRINT USING p$,"Orientation Factor",n
LPRINT USING p$,"Fibre Radius",rf
LPRINT USING p$,"Interfacial Shear Strength",tau
LPRINT USING p$,"Volume Fraction of Glass",vf
LPRINT USING p$,"Volume fraction of Rubber",fro
LPRINT:LPRINT:
LPRINT "Strain Stress Modulus"
FOR a=0 TO (mstr/0.05) STEP 2
   IF strain(a)>0 THEN young=stress&(a)/strain(a) ELSE young=0
LPRINT USING ".#.#~#### .#.#~#### .#.#~####";strain(a),
stress&(a), young
LPRINT
NEXT a
RETURN

fyle:
REM *** OUTPUT TO FILE ***********************************************
INPUT "File name"; fylename$
OPEN fylename$ FOR OUTPUT AS #1
WRITE #1,"Youngs Modulus of Fibre",ef
WRITE #1,"Orientation Factor",n
WRITE #1,"Fibre Radius",rf
WRITE #1,"Interfacial Shear Strength",tau
WRITE #1,"Volume Fraction of Glass",vf
WRITE #1,"Volume fraction of Rubber",fro
WRITE #1,"Strain","Stress"
FOR a=0 TO (mstr/0.05)
   WRITE #1,strain(a),stress&(a)
NEXT a
CLOSE #1
RETURN

graph:
REM ** DISPLAY STRESS-STRAIN CURVE ON VDU ****************************
SCREEN 8
CLS
COLOR 9,1
LOCATE 3,1
PRINT "0.5% MODULUS ",
PRINT USING ".#.#~####"; stress&(10)/.005
LINE (50,150)-(50,50)
LINE (50,150)-(600,150)
LOCATE 6,1
PRINT USING "####.#~####"; stress&(mstr/0.05)
LOCATE 19,3
PRINT "0"
FOR a=0 TO mstr
   pt=(a/mstr)*68.75+6.25
   LOCATE 21,pt
   PRINT a
NEXT a
LOCATE 23,35
LOCATE 13,1
PRINT "STRESS"
FOR a=0 TO 30
   LINE (50+a*18.33,150)-(50+a*18.33,155)
   LINE (50,50+3.33*a)-(45,50+a*3.33)
NEXT a
fac=100/stress&(mstr/0.05)
rac=550/(mstr/0.05)
FOR a=1 TO (mstr/0.05)
   LINE (50+rac*(a-1),150-stress&(a-1)*fac)
      -(50+rac*a,150-stress&(a)*fac)
NEXT a
RETURN

REM ** QUANTIMAT FIBRE LENGTH DISTRIBUTIONS ******************************
fld fifteen:
   DATA 00.00,00.00,00.01,00.10,00.45,01.23,02.60,04.61,07.25,10.43,14.07
   DATA 18.03,22.22,26.54,30.88,35.21,39.44,43.54,47.48,51.25,54.83,58.20
   DATA 61.38,64.36,67.15,69.75,72.18,74.43,76.53,78.48,80.28,81.96,83.51
   DATA 84.95,86.28,87.51,88.65,89.71,90.69,91.59,92.44,93.21,93.94,94.61
   DATA 95.23,95.80,96.34,96.84,97.30,97.73,98.12,98.49,98.84,99.16,99.46
   DATA 99.74,100.0,100.0,100.0
fld thirty:
   DATA 00.00,00.00,00.00,00.04,00.32,01.21,03.11,06.27,10.67,16.13,22.35
   DATA 28.99,35.78,42.45,48.82,54.79,60.29,65.25,69.71,73.67,77.17,80.24
   DATA 82.92,85.26,87.29,89.04,90.56,91.87,93.00,93.97,94.81,95.54,96.16
   DATA 96.70,97.17,97.57,97.91,98.21,98.47,98.69,98.88,99.05,99.20,99.32
   DATA 100.0,100.0,100.0,100.0
fld forty:
   DATA 00.00,00.00,00.00,00.09,00.58,02.00,04.81,09.16,14.86,21.57,28.84
   DATA 36.29,43.59,50.52,56.93,62.74,67.93,72.51,76.52,79.92,82.99,85.57
   DATA 87.77,89.65,91.25,92.61,93.77,94.75,95.59,96.30,96.90,97.41,97.85
   DATA 100.0,100.0,100.0,100.0,100.0,100.0,100.0,100.0,100.0,100.0,100.0
Appendix B

Turbo Basic programme listing for composite strength prediction.

REM ****CALCULATE FAILURE STRESS**********
retry:
stressf=3.4e9
INPUT"Weight Fraction of Glass (%)";wf
vf=wf/277.78
INPUT"Volume Fraction of Rubber",vr
fr=vr^0.69
INPUT"Interfacial Shear Strength",tau
lc=7e-6*stressf/(tau*(1-fr))
INPUT"Fibre Orientation Factor",c
vm=(1-vf)*(1-fr)
stressm=39.73e6
GOSUB fibrefac
stressc=(vf*stressf*av*c)+(vm*stressm)
PRINT USING "M A
";stressc
PRINT av
PRINT "Another Grade?"
a$ = INPUT$(1)
IF a$="y" OR a$="Y" THEN
    GOTO retry
ELSE
    END IF
END

fibrefac:
tot=0 : fetot=0
IF wf=40 THEN fa=2.476131 : fb=0.084571
IF wf>20 AND wf<40 THEN fa=2.492912 : fb=0.084606
IF wf<20 THEN fa=2.5225 : fb=0.108072
FOR a=20 TO 3020 STEP 40
    vol=((1/(2*3.14159)^0.5)*EXP(-0.5*((fa-((LOG(a))^0.5))/fb)^2))
    IF a*1E-6<=lc THEN fe=(a*1e-6/(2*lc))
    IF a*1E-6>lc THEN fe=((a*1E-6-(0.5*lc))/(a*1E-6))
    PRINT a,fe
    fetot=fetot+(fe*vol)
    tot=tot+vol
NEXT a
av=fetot/tot
RETURN
Fig. 6.1. Particulate theory treats the rubber as a cube at the centre of a unit cube of matrix.
PREDICTIVE LOAD RESPONSE CURVES
10% & 30% RUBBER MODIFIED PP

Figure 6.2

PREDICTIVE LOAD RESPONSE CURVES
SHEAR STRESS CONSTANT & PROPORTIONAL

Figure 6.3
Figure 6.4

FIBRE/MATRIX INTERFACIAL SHEAR STRESS
A Power Law Fit For Tau

Relative Interfacial Shear Stress

Strain (%)

1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0

tau maximum

Figure 6.5

PREDICTIVE LOAD RESPONSE
15-40% COUPLED GLASS, 30% RUBBER & PP

Stress (Pa) (Millions)

90
80
70
60
50
40
30
20
10
0

15% Glass (Exp')
+
30% Glass (Exp')
*
40% Glass (Exp')
—
15% Glass (Theo')
—
30% Glass (Theo')
—
40% Glass (Theo')

Strain (%)

0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
PREDICTIVE STRESS STRAIN CURVES
15-40% Uncoupled Glass, 30% Rubber & PP

Figure 6.8

PREDICTIVE STRESS STRAIN CURVES
10-30% Rubber, Uncoupled 30% Glass & PP

Figure 6.9
PREDICTIVE STRESS STRAIN CURVES
0-40% Rubber, 30% Uncoupled Glass & PP

![Graph showing stress-strain curves for different coupling levels and rubber concentrations.]

PREDICTIVE STRESS STRAIN CURVES
Coupling Level x0.5, x1, x2

![Graph showing stress-strain curves for different coupling levels.]
PREDICTIVE LOAD RESPONSE CURVES
COUPLED & UNCOUPLED 30% GLASS GRADES

Stress (Pa) vs. Strain (%)

Coupled (Exp')
Uncoupled (Exp')
Coupled (Theo')
Uncoupled (Theo')

Tensile Yield Strength
Effect of Glass Content

Yield Strength (MPa) vs. Glass Content (%)

Polypropylene
PP & Coupling
PP & Rubber 30%
PP theoretical
PP & C theoretical
PP & R theoretical

Figure 6.12
Figure 6.13
**Figure 6.12**

**Tensile Yield Strength**

Effect of Glass Content

**Figure 6.13**
**Tensile Yield Strength**

**Effect of Rubber Content**

![Graph showing the effect of rubber content on tensile yield strength.](image1)

**Figure 6.14**

**Tensile Yield Strength**

**Coupling, 30% Rubber, 15-40% Glass & PP**

![Graph showing the effect of coupling content on tensile yield strength.](image2)

**Figure 6.15**
Tensile Yield Strength
Effect of Rubber Content

![Graph showing effect of rubber content on tensile yield strength.](Figure 6.14)

Tensile Yield Strength
Coupling, 30% Rubber, 15-40% Glass & PP

![Graph showing effect of coupling content on tensile yield strength.](Figure 6.15)
The series of fracture mechanics tests carried out during the course of this project followed a natural progression of testing techniques and methods. In the following chapter the results of these tests will be discussed to explain the reasons for selecting particular tests and the discrepancies in the results which were attained. The development of fracture mechanics testing in polymer and composite systems is an on going concern - some of the techniques discussed later in this chapter follow the most recent protocols.

A number of different tests were employed to investigate the fracture toughness of the composite grades over a range of strain rates. Impact tests, such as the Charpy and instrumented falling weight impact tests, were carried out to investigate the resistance to fracture at high strain rates. Compact tension and three point bend specimens were strained at slower rates to induce controlled crack growth and thus provide fracture toughness data. The visco-elastic nature of the matrix limits the applicability of linear elastic fracture mechanics to these composites and hence elastic-plastic fracture mechanics were employed to analyse them.

Fracture surface morphology was studied to gain an understanding of the toughening mechanisms which operate at the different strain rates and in different composite grades.
7.1. IMPACT TESTING

In the Charpy test, the notched three point bend specimen is struck by a swinging pendulum at the bottom of its arc. The impact energy is obtained from the loss in kinetic energy (speed) of the hammer. The advantage of this technique is that it is simple and quick. The test gives good qualitative comparisons between grades at impact speeds. Furthermore, the results of this test facilitated the selection of suitable grades for the more sophisticated elastic-plastic fracture testing (section 7.2) and the instrumented falling weight impact test (section 7.3).

Testing was performed using the miniature apparatus developed at the University of Surrey. Two specimen geometry test pieces were investigated: the differences between the specimen geometries being the thickness (3 and 10mm) and consequently the fibre orientation distribution. The 3mm thick Charpy test pieces were injection moulded without a notch. Notching was carried out according to ASTM D256-70 using a rotating Charpy notch cutter to give a notch depth of 2.5mm. As the notch was not moulded, there was no flow of fibres around the notch tip and thus crack propagation was perpendicular to the melt fill direction and predominant fibre orientation in the skin. The larger test pieces were machined from the 10mm thick plaques. Notching was performed in an identical manner to that of the 3mm plaques, with the crack running perpendicular to the melt fill direction.
The critical strain energy release rate for the 3mm thick specimens was obtained using the procedure proposed by Plati and Williams\(^1\) who derived the relationship:

\[
G_c = \frac{\text{Impact Energy}}{b \cdot d \cdot \phi}
\]  

(7.1)

where;

- \(G_c\) = critical strain energy release rate
- \(b\) = specimen breadth
- \(d\) = specimen thickness before notching
- \(\phi\) = a geometrical factor.

A series of sharply notched specimens with various ratios of notch depth to thickness were tested in the miniature impact machine, and the impact energy plotted against \(b \cdot d \cdot \phi\). The slope of the straight line fitted to the data gives \(G_c\).

This procedure was used to find the \(\phi\) factor for a standard notch depth. Samples from all the grades were prepared with this notch depth enabling \(G_c\) values to be measured. The critical stress intensity factor can be estimated using:

\[
K_c = \sqrt{E \cdot G_c}
\]  

(7.2)

and hence the radius of the plastic zone ahead of the crack tip, \(r_y\), can also be estimated with a knowledge of the yield stress, \(\sigma_{ys}\) since:

\[
r_y = \frac{1}{2 \pi} \cdot \left(\frac{K_c}{\sigma_{ys}}\right)^2
\]  

(7.3)
The 10mm thick specimens were tested more simply to provide a qualitative comparison of the relative energies absorbed at different strain rates. The Plati and Williams approach was not applied to these specimens.

7.2. ELASTIC PLASTIC FRACTURE MECHANICS TESTING

The results of the Charpy test programme are listed in Table 7.1 and discussed in section 7.5.1. Although it is not appropriate to discuss in great detail here the results of the Charpy test programme, it revealed that linear elastic fracture mechanics would not be valid under standard conditions for most of the material grades. The resultant plastic zone size at the crack tip would be a greater proportion of the thickness than is allowable for validity of the test. As a general rule, LEFM tests are only valid if the minimum dimension of the test specimen, $B$, is greater than the plastic zone radius by a factor of 2.5:

$$B > 2.5\left(\frac{K_c}{\sigma_y}\right)^2 \quad \text{(7.4)}$$

Elastic/plastic fracture mechanics was therefore employed to quantify the energy absorbing processes occurring at or around the crack tip. The data from the Charpy tests suggest that even for $J$ integral evaluation the specimen thickness (3mm) would be too small relative to the plastic zone size and it would therefore fail under conditions of predominantly plane stress. $K_c$ is related to $J_c$ through:
The size criterion for $J_C$ testing is:

$$B > 25 \frac{J_C}{\sigma_y}$$

The Charpy test, however, is not a precise fracture toughness test. The point of the tensile yield is not precise in a polymer, resulting in an associated uncertainty in the calculated plastic zone size. It was therefore decided to perform a limited number of $J$ integral evaluation tests on material grades believed to give conditions closest to plane strain at fracture, i.e. the stronger and more brittle materials. Material from batches $AH1F$ and $AH1I$ were selected for the series of tests.

This initial testing programme on the 3mm plaque material gave promising results and led to the production of the 10mm plaques purely for further fracture mechanics work.

7.2.1. J Integral Evaluation using 3mm Plaque

A rig was produced to ensure that the geometries machined from the coat hanger gated plaques were kept constant. The test specimen geometry was based upon the revised compact tension (CT) specimen devised originally by Lhymn and Schultz\textsuperscript{2}.

$$B = 3\text{mm}$$
$$a = 8.0\text{mm}$$
The dimensions were smaller than those specified by ASTM E399-74 (for metals) in order to eliminate the instability related mode III shear fracture. All specimens were cut from the 3mm thick plaques with the melt flow direction perpendicular to the central machined notch, figure 7.1. The notch was sharpened by running a scalpel through its apex.

Specimens were tested at a cross head speed of 0.5mm/min and taken to different loads, sufficiently high to cause limited amounts of stable crack growth, dependant on the maximum load. The energy absorbed by the specimen, $U_t$, was evaluated by integrating the area below the load displacement curve, figure 7.2. The vicinity of the crack was immersed in dye penetrant; the dye being drawn into the crack by capillary action. The specimens were then broken open to reveal a dyed region representing the fracture surface created during the initial loading. The average crack length increment, $b$, was taken as the average of nine such readings across the crack surface (see figures 7.3 and 7.5).

The $J$ integral is found from a method based on an approximate formulation developed by Rice et al.\(^3\):

$$J = \frac{2U_t}{B(W-a)}$$  \(7.7\)

where $a$ is the initial crack length, and $W$ is the distance between the centre of the loadings pins and the far uncracked face of the compact tension specimen.

Fracture resistance, $J$, is plotted against crack growth (da) and a theoretical blunting line added:
The intersection of the two lines gives, \( J_c \), the critical \( J \) integral.

\[
J = 2\sigma_y \text{da}
\]  \hfill (7.8)

The intersection of the two lines gives, \( J_c \), the critical \( J \) integral.

**7.2.2. \( J \) Integral Evaluation 10mm Thick Material**

\( J \) Integral evaluation tests were also performed upon specimens cut from 10mm thick plaque material. Specimens machined from this thicker plaque material more easily met thickness validation criteria (equation 7.6) and produced fracture conditions of predominantly plain strain.

During the course of this study the standards for \( J \) integral testing procedures have been changed. This is primarily due to the influence of the European Group on Fracture (EGF) protocols for evaluating \( J_c \) of polymeric materials. The protocols were developed to standardise accurate reproducible procedures for the \( J_c \) extrapolation and the measurement of the crack advance. Three different testing procedures have been applied to the 10mm thick material.

The first procedure (section 7.2.3a) uses a technique developed for ductile metals but with the CT specimen geometry altered to reduce mode III failure as described in the previous section for the 3mm thick material. The second technique (section 7.2.3b) follows an early EGF protocol\(^4\). The CT specimens are significantly smaller in size than in the previous technique, but the analytical blunting line extrapolation of \( J_c \) is basically the same. The final method (section 7.2.3c) follows
a later EGF protocol\textsuperscript{5}, this time utilising Single Edge Notched Bend (SEN'B) test specimens. The crack advance is measured using a novel specimen sectioning technique. The blunting line analysis is disregarded and a power law fit is applied to the data. $J_c$ is taken directly from the fit. The three variations of the test procedure are described below:

7.2.3a. $J_c$ Test I

An identical procedure to that discussed in section 7.2.1, but utilising material from the 10mm plaques.

Specimen dimensions based on the CT specimen, figure 7.1, were:

\begin{align*}
B &= 10.5\text{mm} \\
a &= 10.5\text{mm} \\
W &= 27\text{mm}
\end{align*}

7.2.3b $J_c$ Test II

The 'European Group on Fracture Protocol (Jan 1990)' uses a similar technique to that described in section 7.2.1 above. The main dissimilarity is again in the specimen size. The revised compact tension specimen dimensions were:

\begin{align*}
B &= 10.5\text{mm} \\
a &= 10.5\text{mm} \\
W &= 21\text{mm}
\end{align*}
A brief outline of the protocol testing procedure is given below:

1. Compact tension specimens are cut to the above dimensions from the 10mm thick plaques. The melt flow direction is perpendicular to the notch;
2. CT specimens are annealed at 130°C for 2 hours;
3. CT notches are sharpened by running a scalpel blade down their length;
4. CT specimens are loaded to different strains. Load is measured by the testing machine. Crack opening displacement is measured by an extensometer at the CT loading pins;
5. The correction for extraneous displacement etc., is calculated for the different grades by calibration with unnotched CT specimens;
6. The above correction is applied to the load displacement curves. The resultant area of the curves is calculated. This area is related to the work done, \( U \), figure 7.2;
7. The fibre reinforced specimens are subjected to a dye penetrant and broken open, figure 7.3;
8. The unreinforced CT specimens are cooled in liquid nitrogen and immediately broken open, figure 7.4;
9. Photographs are taken of the fracture surface;
10. From the photographs the average of nine measurements gives the average crack growth distance, figure 7.5;
11. The fracture resistance not allowing for crack growth, \( J_0 \), is given by:

\[
J_0 = \frac{nU}{B_N(W-a_0)}
\]  

(7.9)
where;
\[ n = 2 + 0.522 \left(1-a_0/W\right) \text{ for compact specimens} \]
\[ a_0 = \text{initial crack length} \]
\[ U = \text{area under load versus load-point displacement record} \]
\[ B_N = \text{specimen thickness}; \]

12. The above equation does not allow for crack growth during a test. Allowing for crack growth:
\[ J = J_0 \left\{1-(0.75n-1)da/(W-a_0)\right\} \quad (7.10) \]
where, \( da \), is the crack growth;

13. Fracture Resistance, \( J \), is plotted against Crack Growth;

14. Blunting line is added:
\[ J = 2\sigma_y da \quad (7.11) \]
where \( \sigma_y \) is the yield strength;

15. Exclusion lines are constructed between which the data are valid. The first of which is a 0.1mm offset parallel to the blunting line;

16. The maximum exclusion criterion is set by \( da_{\text{max}} \).
\[ da_{\text{max}} = 0.15b_0 \quad (7.12) \]
\[ b_0 = W-a_0 \quad (7.13) \]
An exclusion line parallel to the blunting line is drawn through \( da_{\text{max}} \);

17. A linear regression is fitted through the points within the exclusion lines;

18. \( J_{0.2/BL} \)
A parallel line offset to the blunting line at 0.2mm crack growth is drawn. The intersection of the regression line with the offset line defines $J_{0.2/BL}$, figure 7.6.

7.2.3c $J_c$ Test III

The 'E.G.F Testing Protocol for Conducting $J$-R Curve Tests on Plastics (Feb 1990)' used a new technique for measuring the crack advance, $da$, in the material, and in the manner in which the data is extrapolated to give $J_c$.

Single Edge Notched Bending (SENB) specimens were used in this series of tests as they are easier to machine accurately, figure 7.7. Dimensions were:

- $B = 10.5$mm
- $a = 10.5$mm
- $W = 21$mm

Specimens were produced from grades AH2A, 2B, 2C and 2D. Specimens from grades AH2C and AH2D were cut from both parallel ($2C_0$ & $2D_0$), and perpendicular ($2C_{90}$ & $2D_{90}$) to the melt fill direction so in all there were six batches of specimens. A brief description of the testing procedure is given below:

1. SENB specimens were cut to the above dimensions from the 10mm thick plaques. Non fibre filled grades were side notched to a depth of 0.7mm to encourage a planar fracture front;

2. The as machined SENB notches were sharpened by tapping a fresh scalpel blade into each specimen notch with a hammer. This produces a notch tip radius of less than 20µm;
3. SENB specimens were loaded in three point bending to different strains. Load and displacement were measured by the testing machine;

4. The correction for extraneous displacement etc, was calculated for the different grades by testing an unnotched SENB specimen;

5. The above correction was applied to the load displacement curves. The resultant areas of the curves were calculated. These areas were related to the work done $U$;

6. The specimens were cut in half perpendicular to the notch along their length. The vicinity of the crack was ground to 500 grit finish. This area was coated with a thin coat of silver paint. This helped to distinguish between the actual crack and the plastic zone as this can be very difficult to observe in translucent materials;

7. The specimens were placed in a hand operated three point bend rig below a video microscope. The load on the test piece was then increased to open the crack. The crack length was measured.

8. \[ J_0 = \frac{nU}{B_N(W-a_0)} \] (7.9)

where;

$J_0$ = fracture resistance not allowing for crack growth

$n = 2$ for SENB specimens

$U$ = area under load versus load-point displacement record

$B_N$ = specimen thickness

$a_0$ = initial crack length
9. The above equation does not allow for crack growth during a test. Allowing for crack growth:

\[ J = J_0 \left\{ \frac{1 - (0.75n - 1)da/(W-a_o)}{1} \right\} \]  \hspace{1cm} (7.10)

where \( da \), is the crack growth;

10. Fracture Resistance was plotted against Crack Growth;

11. A power law fit was applied to all those points whose crack extension lie between 0.1mm, and \( da_{max} \) where:

\[ da_{max} = 0.15(W-a_o) \]  \hspace{1cm} (7.12)

and \( W-a_o \) is the original uncracked ligament.

0.1mm is used as a lower limit as small amounts of crack growth are difficult to measure and hence are subject to error;

12. \( J_{0.2} \)

A vertical line at 0.2mm crack growth was drawn. The intersection of the power law line with the offset line defines \( J_{0.2} \), figure 7.8.
7.3. LINEAR ELASTIC FRACTURE MECHANICS TESTING

Validation criteria (equation 7.4) suggested that linear elastic fracture mechanics testing on specimens machined from the 10mm thick plaques would be valid at high strain rates due to the increased yield strength and also possibly at lower strain rates. Two testing procedures were applied to the 10mm thick specimens.

The instrumented falling weight impact test accurately measures the impact forces and deflections sustained by a notched three point bend specimen when struck by a falling weight at high speed.

The dynamic LEFM test measures the crack advance at the surface of a CT specimen under tensile load through a foil adhered to its surface. The test was performed at crosshead speeds of 1mm/min and 0.17m/s.

7.3.1. Instrumented Falling Weight Impact Testing

This method of measuring fracture mechanics parameters was performed on single edge notched bend specimens from a limited number of grades. SENB specimens from grades AH2A, 2B, 2C and 2D were cut from the 10 millimetre thick plaques. For the fibre filled grades, specimens were cut both parallel and perpendicular to the melt fill direction. Specimen geometry, notching and crack sharpening were identical to the $J$ integral specimens reported in section 7.2.3(c). Testing was performed in 3 point bending at 23°C and at a strain rate of 1 m/s. The average of five specimens per grade was taken. Similar specimens were cut
from the 3 mm plaque material for grades AH1D, 1F, 2B and 2C. The fracture mechanics measurements for these specimens were in SEN bending at -65°C and at 1 m/s in each case running the crack across the flow direction.

Specimens were supported over a span of 84mm by a pair of large diameter rollers (figure 7.9). A central roller was released from 51cm above the specimen so that it impacted the specimen directly above the notch, with a velocity of 1 m/s. The test was instrumented through the piezoelectric load cell situated in the falling weight. The impact forces sustained by the load cell were converted with very short delay times into electrical responses; the electrical impulses were amplified by a charge amplifier. The amplified signal was input to a storage oscilloscope. This device, which retains the voltage pulses, can resolve very small variations in voltage. These data were then logged to a computer where the force-time and force-deflection responses could be analysed. Since the impact energy was far in excess of that required for penetration of the specimens, it may be assumed that the falling weight velocity was unaltered by the impact. Thus the voltage (force)/time trace may be converted to force/displacement. The area below the force deflection response is then integrated (by the computer) and the crack initiation ($K_{IC}$) and crack propagation ($G_{IC}$) fracture parameters are found from:

$$K_{IC} = f \cdot \frac{F_c a}{B W^{3/2}}$$  \hspace{1cm} (7.14)

and;

$$G_{IC} = \frac{U_c}{B W \phi'}$$  \hspace{1cm} (7.15)
where $B$ is the thickness, $W$ is the width, $\phi'$ is a shape factor, $f$ is a geometrical factor dependent on the crack length, $a$, and $U_c$ is the elastic energy of the specimen at its peak load, $F_c$.

### 7.3.2. $K_c$ and $G_c$ at Different Strain Rates

The trends in the results from the fast and slow fracture mechanics testing suggested that different toughening mechanisms were predominant at different strain rates. So far, no single fracture mechanics measuring technique had been performed at a range of strain rates, and on identical material and specimen geometries. Linear elastic fracture mechanics tests were, thus, carried out on two material grades, with identical specimen geometries, at two extreme strain rates.

The basic testing procedure followed that detailed by the 'European Group on Fracture - A Linear Elastic Fracture Mechanics (LEFM) Standard for Determining $K_c$ and $G_c$ for Plastics (July 1989)'. In this series of tests a Compact Tension specimen, figure 7.1, was used:

- $B = 10.2\text{mm}$
- $a = 13.4\text{mm}$
- $W = 55.4\text{mm}$

This is similar in shape but larger than that used in the $J$ integral testing. The specimens were machined out of the $10\text{mm}$ thick plaques from grades AH2C and AH2D. The notch of the specimen was sharpened with a scalpel blade, and lay parallel to the melt fill direction. The resulting fracture path follows the path of least resistance across the width ($W$) of the specimen.
If the specimens were notched at right angles to the MFD, the fracture path would be heavily influenced by the predominant fibre orientation and would tend to lie away from the perpendicular to the loading axis, figure 7.10.

The CT specimen was supported by pins in an Instron loading frame and placed under tension in order to propagate a pre-initiated crack through the specimen. The load required to propagate the crack was measured, together with the displacement of the loading pins. The length of the crack was also measured using a metal foil which was bonded to the surface of the specimen using a high shear strength adhesive. The foil was connected to a bridge type amplifier, figure 7.11. As the crack propagated through the specimen it also tore the foil which was adhered to its surface. As the foil tore, its electrical resistance increased. The crack measurement was achieved by a TTI Fractomat Crack Amplifier. This amplifier converts the signal from a crack foil gauge into a voltage. Accurate measurements can be achieved for up to 30mm of crack growth. Specimens were strained at rates of 1mm/min and 10200mm/min (0.17m/s).

The stress intensity factor, $K$, at any stage of crack growth, can be calculated from:

$$K = \frac{f \cdot P a}{B \cdot W}$$

(7.16)

where:

- $P$ is the load in Newtons
- $B$ is the specimen thickness in m
- $W$ is the specimen width in m
\[ f \text{ is a geometry function which is calculated using:} \]

\[ f = \frac{(2+\alpha)[0.886+4.64\alpha-13.32\alpha^2+14.72\alpha^3-5.6\alpha^4]}{(1-\alpha)} \]  
(7.17)

where: \[ \alpha = \frac{a}{W} \]  
(7.18)

\[ G \] can also be calculated at initiation and propagation as indicated below.

Initiation -
For crack initiation \( G \) can be calculated using:

\[ G = \frac{U}{BW\phi'} \]  
(7.19)

where \( U \) is the energy under the force/deflection curve up to crack initiation, figure 7.12, and \( \phi' \) is a geometry function which is calculated using:

\[ \phi = \frac{(1.9+19.1\alpha-2.5\alpha^2-23.2\alpha^3+20.5\alpha^4)(1-\alpha)}{(19.1-5.0\alpha-69.7\alpha^2+82.16\alpha^3)(1-\alpha)+2(1.9+19.1\alpha-2.5\alpha^2-23.2\alpha^3+20.5\alpha^4)} \]  
(7.20)

where \( \alpha = a/W \)

Propagation -
During crack growth \( G \) can be calculated using the equation:

\[ G = \frac{\Delta U}{B\Delta a} \]  
(7.21)

where \( \Delta U \) is the change in energy under the force/deflection curve for a discrete crack length, figure 7.12.

\( K \) and \( G \) can be plotted against crack length or crack speed.
7.4. SCANNING ELECTRON MICROSCOPY

Fracture surfaces were studied using scanning electron microscopy, rather than optical microscopy, because of the excellent depth-of-field associated with the former technique. The technique also requires little specimen preparation. Fractured specimens were bonded to conductive aluminium studs. Because of the non-conducting nature of the materials under study, the specimens were first gold sputter coated for three minutes. A "Dag" conducting paint was used as a contact between the sputter coat and the aluminium stud which seat on the microscope stage, ensuring good electrical contact. The high electrical conductivity of the sputtered specimens promotes refined imaging in the microscope, and electrical charging of the specimen is avoided.

The microscope used most frequently during this work was a Cambridge Instruments S100 Stereoscan Microscope, at an accelerating voltage of 20kV.

Fracture specimens were examined in order to establish fracture mechanisms, specifically fibre/matrix interfacial failure mechanisms and matrix failure modes. Fracture surfaces from Charpy impact tests (1m/s), tensile tests (50mm/min), and \( J_c \) tests (1mm/min) were all prepared to investigate the effect of the strain rate on the various fracture morphologies. In a further series of tests, SENB specimens were impacted at a rate of 1m/s, to initiate and propagate a fast fracture in the specimen. The crack was not permitted to propagate through the full thickness of the specimen. This was achieved by limiting
the energy of the impacting hammer. The specimens were then broken open at a much slower strain rate of 0.5mm/min. This provided a means of observing the transition between fast and slow fracture mechanisms, as well as comparing the fracture surfaces, using a single specimen.

7.5. FRACTURE MECHANICS RESULTS

7.5.1. Charpy Impact Testing

Figures 7.13 - 7.15 show the Charpy impact resistance for the 3mm thick specimens as a function of material composition (table 3.1).

Addition of glass to a composite system generally increases the energy absorption of the system. Glass fibres act to hinder the crack growth because the crack front is forced to travel around fibres thus increasing the energy absorption. Glass fibres will also contribute to the overall fracture toughness through fibre pull-out as the fracture faces separate. The exception to the trend of increasing toughness with increasing fibre volume fraction is in the rubber reinforced uncoupled grades. For this system the weakly bonded fibres in a reduced modulus matrix show a general decrease in energy absorption with increasing glass content. Fibres in such a system act as stress raisers as they are so poorly bonded to the matrix, promoting crack formation within the matrix. An increase in glass content will reduce the average distance between crack initiators and promote easier crack path formation.
SEM photomicrographs showed that the fibres of uncoupled grades when pulled-out from the matrix were pulled cleanly from the matrix, figure 7.16. This pull-out morphology is clearly different to that exhibited by the coupled grades, figure 7.17. The pulled-out fibres from the coupled grades exhibited a heavy sheath of matrix material adhering to their surface. The low strength frictional bond of the uncoupled grades accounts for the adhesive failure at the interface as the fibre is pulled-out. The energy required to pull the fibre free of its encompassing matrix will be the energy required to overcome the interfacial shear strength and the subsequent friction of pulling the fibre out of the matrix. Both of these processes require fairly low levels of energy when compared with the similar processes for the coupled grades. In pulling a coupled fibre from the matrix the fracture path is no longer interfacial but is forced to propagate away from the fibre\matrix interface in a cohesive mode of failure. In this manner the shear strength of the matrix has to be overcome to allow the crack to run around the fibre. Further to this, energy is absorbed as the sheathed fibre (with its increased surface area due to the sheath), is pulled through the matrix causing plastic flow of the matrix around the fibre.

Under impact conditions, these sheathing effects were observed in all coupled grades. As the level of rubber was increased in the coupled grades, the sheathing started to become patchy. This is most clearly shown in the 40% rubber grade, figure 7.18. The coupling agent is specific to the polypropylene and does not react with the rubber. Any Rubber rich regions
around the fibre will not be strongly bonded, and could therefore lead to this effect.

The addition of rubber to the matrix increased the energy absorbing capacity of the matrix as demonstrated in the impact data of figure 7.14 and the fracture surface of figure 7.19. Possible processes enhancing the toughness may be microcavitation, strain-banding or crazing.

Coupling agent was added at set levels of the base polymer. Increasing the coupling level of the 30% and 40% glass filled grades increased the Charpy energy, figure 7.15. The increased levels of coupling improved the interfacial bond and thus probably increases the pull-out energy. The 15% glass grade exhibited a decrease in Charpy impact energy with the addition of coupling. The addition of the coupling agent slightly decreases the impact resistance of the matrix (compare AH1A and AH1B figure 7.13). At low fibre fractions the fibres will be fully bonded to the matrix at low levels of coupling agent. Further additions of the coupling agent will reduce the impact resistance of the matrix specifically and hence of the composite as a whole.

The critical strain energy release rate was approximated using the procedure proposed by Plati and Williams\textsuperscript{1}, outlined in section 7.1. The results of this analysis are given in table 7.1. The fracture parameters $G_c$, $K_c$ and the plastic zone radius, $r_y$, are approximations calculated within the limitations of uncontrolled crack growth at impact speeds. As a general rule, linear elastic fracture mechanics tests are only valid if the
specimens minimum dimensions, $B$, are greater than a multiple of the plastic zone radius leading to:

$$B > 2.5 \left( \frac{K}{\sigma_y} \right)^2$$

(7.22)

The results of this procedure are given in the final column of table 7.1. The calculated minimum dimensions, $B$, for fracture mechanics specimens for all of the grades are larger than the 3mm thickness limitation of the plaque material available at that time. Errors are associated with the technique, such as the incorporated measurement of extraneous energies, e.g., the kinetic energy of the samples after impact. The yield strength value used was measured at a strain rate of 50mm/min and will be an underestimate of the value at impact speeds. These factors may reduce $B$ to a level approximating the thickness of the plaque material. It was with this in mind that the limited sequence of 3mm thick J integral specimens was produced and tested.

Charpy type specimens were also machined at a later date from the 10mm thick plaques, from grades AH2A, B, C, D. These thicker specimens exhibited behaviour more typical of what was expected than did the same grades in the 3mm thick samples, table 7.2. The addition to the base polymer of glass and/or rubber substantially increased the energy absorbing capability of the system. The results from the 10mm thick Charpy specimens will be discussed in more detail in section 7.5.5 along with the results from the falling weight impact test performed on similar specimens. The falling weight impact test is essentially a more refined version of the Charpy test.
7.5.2. 3mm Thick $J_c$ Evaluation

The results of the Charpy impact programme, section 7.5.1, led to a small number of compact tension specimen being cut from the 3mm thick plaques to enable the possibility of evaluation of $J_c$ analysis. Grade AH1F (30% coupled glass in polypropylene) was selected for the tests as it was the fibre filled grade with properties closest to linear elastic fracture mechanics testing criteria (equation 7.22).

Figure 7.20 shows the results of the $J_c$ testing programme. A line of best fit was applied to the points and extrapolated to give a $J_{(0.2)}$ of 1388 J/m². The minimum dimension criterion for $J_c$ testing taken from ASTM E813-87 is

\[ B > \frac{J_Q}{25 \sigma_Y} \]  \hspace{1cm} (7.23)

where:

\[ J_Q = \frac{J_{0.2}}{BL} \text{ or } J_{0.2} \]

Using the above criterion $B_{min}$ is found to be 0.4mm. This is well below the specimen thickness, so that the criterion is met. However, it was acknowledged that this grade would always have the greatest possibility of meeting the criterion. In comparison, other grades would have a smaller possibility of fulfilling the criteria. As yet it has not been shown whether the same criterion which applies to metals (equation 7.23) also applies to polymers. It was with these reasons in mind that it was decided to produce the 10mm thick plaques to increase the error margin for validation.

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7.5.3. 10mm Thick J_c Evaluation - Methods I & II

Specimens from grades AH2A, B, C, D were prepared for J_c testing as described in section 7.2.3a. They were then loaded to produce a range of crack length increments. Great difficulty was found in breaking open samples of the plain polypropylene grade (AH2A) to reveal the fracture surfaces clearly. When the problem was finally overcome most of the AH2A samples from method I had been destroyed. It is for this reason that results from method I are only available for the three remaining grades.

The crack growth in the unreinforced material was measured from the edge of the razor notch to edge of the stress whitened region; and to the limits of the dye penetrant in the fibre filled grades. For both methods I and II, this technique of crack measurement proved to be unreliable, for the reasons given below.

When dealing with the unfilled grades the CT specimens were cooled in liquid nitrogen after loading and then broken open. The plastically deformed region produced during the initial crack formation could clearly be seen as a stress whitened moon adjacent to the colourless brittle fracture surface produced by breaking the specimen open, figure 7.21. The apparent crack extension was measured from the scalpel notch to the perimeter of the stress whitened zone. However, it was later found that the stress whitened region was a product of the plastic damage zone running ahead of the actual crack. This inability to distinguish between the two, produced an over estimate of the crack extension.
For the fibre filled grades, a dye penetrant technique was used. The penetrant was left on the samples for a number of hours. It was noticed that at the machined faces of the specimen the penetrant impregnated the specimen surfaces to varying depths depending on the localised fibre alignment. The possible impregnation of the penetrant past the crack front into the damage zone produced overestimates of the crack extension.

With these factors in mind, a discussion of the results can be made. The effect of overestimating the crack extension was to underestimate the toughness of the material. Figure 7.22 shows the results from the method I and figures 7.23-7.26 show the results from method II. The same general trends were exhibited by the curves from both methods although the values of $J_c$ do vary both in magnitude and ranking order of the materials, table 7.3. The curves from all four grades originate within a fairly narrow energy distribution, (figure 7.27) demonstrating, as we know from observation, that fracture initiation is matrix dominated. The surprising features of the curves are that the addition of glass and/or rubber to the polypropylene reduces the fracture toughness. It is possible that the glass fibres within the matrix may reduce the fracture toughness by acting as initiation sites for fracture. However it seems highly unlikely that the addition of rubber to the matrix will cause a similar effect, as it is added to promote energy absorbing processes during fracture. A possible reason is suggested below.

The level by which the crack extensions have been mismeasured will be related to the damage zone ahead of the
crack, and hence also to the plastic zone radius given in equation 7.3:

\[ r_y = \frac{1}{2\pi} \left( \frac{K_c}{\sigma_{ys}} \right)^2 \]  

(7.3)

The addition of rubber to the matrix will increase \( K_c \) and reduce \( \sigma_{ys} \) thus increasing \( r_y \) overall. The large increase in the size of the damage zone may account, in part, for the lower \( J_c \) measurements of the rubber toughened grades since, as discussed above, this may have been included in the estimate of the crack length increment. The observed reduction in fracture toughness on addition of glass will be discussed in greater detail in the following section.

7.5.4. 10mm Thick Jc Evaluation - Method III

The main difference between this technique and the previous ones is that the specimen geometry and crack measuring technique were changed to help overcome the problem realised in methods I and II. Single edge notched bend specimens were machined from the 10mm thick plaques and loaded in 3-point bending to grow small cracks. The resultant cracks were observed by cutting the specimens into halves down their length and measuring the crack in situ as described in section 7.2.3c. In this manner the possibility of incurring errors by including the damage process zone in the assessment of crack increment, which may occur in methods I and II, could be reduced. Having said that, a small error will be incurred by assuming that the measured crack length is the average for the whole thickness. It was seen in other
tests that the fracture front was arced. In this manner the fracture toughness may be slightly underestimated.

The results from the fracture mechanics $J_c$ testing using method III are given in figures 7.28 - 7.36 and table 7.3. In this series of tests the addition of rubber produces a small increase in toughness, as predicted in the previous section. However, in agreement with the results from methods I and II the addition of glass fibres to the matrix reduces the fracture toughness. The fibres appear to weaken the matrix by possibly aiding fracture initiation. This contradicts the results of the Charpy test programme which showed an increase in fracture toughness on the addition of glass.

To investigate this effect further, fracture surfaces from the $J_c$ tests and Charpy tests were observed in SEM. The fracture surface of coupled fibre filled Charpy test specimens exhibited the sheathed pulled-out fibres, figure 7.17, as discussed in section 7.5.1. The matrix fracture was predominantly brittle in nature, being mainly planar, figure 7.37. In rubber modified grades the rubber particles can be seen as nodular protrusions pulled out of the matrix, exhibiting little or no obvious plastic deformation, figure 7.38. In contrast, the $J_c$ specimen fracture surfaces show a completely different morphology, figure 7.39. The most noticeable feature is the lack of any matrix sheathing of the pulled-out coupled fibres, figure 7.40. This effect was general to all the pulled-out fibres from both rubber modified and unmodified grades. In addition to this the matrix fracture morphology was also different, figure 7.41. The matrix exhibited
large plastic deformation and shearing typical of polypropylene failure above $T_g$.

Specimens were also machined from the 10mm thick fibre filled grades with a perpendicular orientation (90°) to those previously prepared (0°). The crack in such specimens was grown parallel to the melt fill direction. The fracture toughness of these specimens (90°) was lower than that achieved by the 0° specimens. The fracture toughness is expected to be highest when the crack is run perpendicular to the fibre length direction. In this orientation the crack is forced to travel around the fibres in the most convoluted and energy absorbent route. The potential contribution from fibre pull-out will also be maximised. When the crack runs parallel to the fibre length direction the crack will easily propagate through the matrix between the fibres or along the fibre/matrix interface. The ratio of skin to core in the 10mm thick plaques was approximately 2:1 contributing greatly to the proportion of off axis fibres and hence toughness.

In parallel with the SENB $J_c$ testing programme, tests were also performed upon identical specimens to evaluate LEFM parameters.

7.5.5. Instrumented Falling Weight Impact Test Results

The IFWIT results are listed in tables 7.4 and 7.5. The tests performed upon the 3mm thick specimens were performed at -65°C to enable the thickness criteria to be met. The problem associated with reducing the test temperature was that it is
likely to alter the fracture morphology. At -65°C polypropylene will be well below its $T_g$ and presumably its fracture characteristics may be affected. However, the fracture toughnesses of the 3mm specimens are reasonably consistent with the results from the 10mm thick specimens tested at 23°C. As discussed in section 7.5.4, matrix fracture at impact speeds is brittle in nature. Reducing the temperature of the polymer to below its $T_g$ will have little effect upon an already brittle fracture morphology. The strain energy release rate of the 3mm specimens is lower than that of the 10mm thick specimens of identical grades. The strain energy release rate is greatly dominated by the fibre pull-out and rubber toughening processes. The reduction in temperature of the test will substantially reduce the matrix strength, the matrix shear contribution to the pull-out energy and the rubber toughening processes (molecular orientation and shear). Conversely, the reduction in temperature may increase the thermal contraction of the polymer around the fibres and hence increase the pull-out energy.

The effect of adding rubber to polypropylene, whether or not reinforced by glass fibres, is to increase $K_C$ and $G_C$. These observations are in agreement with the $J_C$ results performed on the CT specimens at 1mm/min. However, in this series of tests the addition of glass fibres also increased $K_C$ and $G_C$. These results are in agreement the results of the Charpy test programme on the 10mm thick specimens, table 7.2, as would be expected on the basis that the IFWIT is essentially a more sophisticated form of Charpy test.
It again appears that the fracture mechanics properties are strain rate dependent. When observed by SEM the fracture surfaces exhibited the same characteristics discussed in section 7.5.4, namely, brittle matrix fracture with sheathed pulled-out fibres at high strain rates, and ductile matrix fracture with clean pulled-out fibre surfaces at low strain rates.

It was with these results in mind that the final series of fracture mechanics tests were performed. It was hoped that with a dynamic LEFM test performed on identical specimens, but at two extremes of strain rate the changing fracture mechanics processes could be quantified.

7.5.6. Dynamic LEFM Tests at Different Strain Rates

The dynamic LEFM tests were performed on CT specimens at 1mm/min and 10200mm/min (0.17m/s - the fastest available strain rate from the hydraulic rig). The results given in this chapter were obtained from single specimens for the tests performed at 1mm/min. No problems were found with this technique at the low strain rate. Two specimens per grade were strained at 0.17m/s. Unfortunately the fractometer gauge from one specimen of each grade became unattached from the surface of its specimen after only limited crack growth during the higher strain rate tests. For these specimens the results are given for crack growth to the point when the gauges debonded. In addition, at the higher strain rate problems associated with inertial effects of the grips were encountered. Figures 7.42 and 7.43 show the force/deflection (time) profile for the two grades. The
underlying force/deflection curve is similar to that created by the slower rate test, figures 7.44 and 7.45. However, imposed on top of that curve is a sinusoidal resonant response. The CT specimens were held in position by pins supported by a specially made rig. The rig was clamped to the machine by large hydraulic grips. The mass of the whole clamping apparatus was quite substantial and much greater than that of the specimen. On commencement of straining, the inertia of the clamping apparatus caused a large force to be exerted upon the load cell. The apparatus was then forced to 'catch up' with the crosshead and a resonant 'bounce' was set up in the grips. The whole test only took 0.05 seconds, never allowing the 'bounce' to dampen out. This effect accounts for the resonant appearance of the overall force/deflection curve. For the high strain rate test, $K_C$ was calculated for all points on the curve, but the strain energy release rate $G_C$ was only calculated between the load peaks of the sinusoidal curve. Estimating how a crack will run under the influence of such a loading profile is highly complex. If a smoothing process were applied to the force curve for the $G_C$ calculation, lower values would be obtained.

In discussing the results from both the slow and fast strain rate tests, some interesting generalisations can be made from figures 7.42 to 7.57. Figures 7.42 - 7.45 show the force and crack length versus displacement. For all specimens the crack started growing before peak load. It is possible that the foil gauge was tearing slightly ahead of the actual crack due to the elasticity of the matrix. However, cracking of the matrix could be observed well before peak load. The crack growth profiles for
the tests were smooth with the exception of the coupled glass filled grade (AH2C) at the higher strain rate. Glass filled specimens subjected to these conditions exhibited a stepped crack growth rate, figure 7.42. Between each peak and trough of the force/displacement curve there appear to be steps in the crack growth profile, corresponding to the respective decrease or increase in cyclic loading. The cycling load caused the crack to 'pop' through the matrix in periods of rapid growth followed by periods of no growth. It is possible that the crack could be arrested by regions rich in fibres oriented as to hinder the advance of the cracks. Once the load is sufficient, the crack can drive around/through these regions until it is next stopped at a low point in the load cycle, and so on. In the slower strain rate test, the crack growth and loading is far more controlled thereby discouraging this effect. In the rubber toughened, fibre filled material, AH2D, at the higher strain rate the 'popping' effect was inhibited possibly due to the more compliant matrix or lower interfacial fibre/matrix shear strength.

Fracture toughness, \( K_c \), and the strain energy release rate, \( G_c \), are usually calculated at peak load. As already discussed, the crack starts growing before peak load, and, as can be seen from the figures 7.46 to 7.57, \( K_c \) and \( G_c \) do not stabilise until significant (10mm) crack growth has been achieved. The fracture mechanics parameters presented in table 7.6 were calculated at crack initiation and peak load. Peak load corresponds to the stabilisation of values for \( K_c \) and \( G_c \) at the lower strain rate. At the higher strain rate, \( K_c \) for both grades did not stabilise.
and is quoted at the point of peak load. Similarly, the value of $G_c$ for grade $AH2C$ at peak load is $6.40 \text{ MN/m}^{3/2}$ but at larger crack lengths stabilises at $11.80 \text{ MN/m}^{3/2}$.

Fracture toughness, $K_c$, represents the critical level of crack tip stress required to propagate an initially sharp crack through a medium, whatever that may be. At primary crack initiation, this entails the stress needed to start the crack to run from the scalpel sharpened notch. Once the primary initiation is overcome, the crack growth can be envisaged as a series of many initiations thus allowing the crack to run through the material. As the crack moves through the material it will induce a damage process zone ahead of the notch tip. The damage process zone will begin small in size and grow as the crack increases in size. The energy required to create the damage process zone will contribute to the fracture toughness. The fracture toughness will therefore increase with crack length and associated process zone size. Eventually the damage process zone will stabilise in size, as will the fracture toughness. This typical fracture toughness behaviour was demonstrated by the slower strained specimens. The specimens strained at the higher rate never achieved stable fracture toughness. At the high strain rates the process zone was not allowed to stabilise possibly due of insufficient time to allow for molecular orientation relaxation of the matrix and/or the inertial effects of the grips. A similar relationship was found between fracture toughness and crack speed. As the crack speed increases, the energy required to initiate further fracture increases, but at higher crack speeds the energy required levels out. Crack speed
increases with crack length. Once the damage process zone has stabilised, the crack speed remains fairly constant but can 'pop' through fibre deficient zones at higher speeds.

The strain energy release rate, \( G_c \), in its simplest form is the energy required to produce two new fracture surfaces. At crack initiation, energy is required to produce two fresh fracture surfaces. As the crack extends, energy is required to produce the new fracture area. Additionally, energy is also required to open the fracture faces left behind. In the case of fibre filled material this will include the energy needed to overcome fibre pull-out occurring behind the advancing crack front. As the crack length increases so will the contribution from the crack opening, thus increasing \( G_c \). Eventually the crack opening will completely free the fracture surfaces at a distance behind the crack front. At this point the strain energy release rate will stabilise. However, due to the non-homogeneous nature of short fibre filled composites, the energy required to pull-out fibres will vary from one region to the next, causing large variations in \( G_c \).

Figures 7.46, 47, 50, 51, 54 - 57 show that \( G_c \) responds in the manner described above with changing crack length for both strain rates. The variation in \( G_c \) is far greater than that determined for \( K_c \) - this being due to the crack opening effect, (figures 7.46, 50 - \( G_c \) : figures 7.48, 52 - \( K_c \)). There is little obvious correlation between \( G_c \) and crack speed, figures 7.47 and 7.51. At small crack lengths the crack speed is lowest, but at larger crack lengths the crack speed varies greatly with little correlation to the strain energy release rate.
The fracture surfaces from all the specimens were observed using SEM. The fracture surface of the specimens strained at 1mm/min, figures 7.58, exhibited the ductile matrix fracture surface and unsheathed pulled-out fibres identical to that of the $J_c$ specimens, figure 7.39. The fracture surfaces of the specimens loaded at 0.17m/s showed a fracture surface dissimilar to any seen previously. The matrix fracture, figure 7.59, exhibited a mainly brittle fracture surface, but with some evidence of ductile behaviour of and around the rubber particles, figure 7.60. The pulled-out fibres exhibited patchy matrix sheathing adhered to their surfaces, figure 7.61. The surface covering of sheathing was far less than that exhibited by the impact specimens, figure 7.17. From these observations it can be concluded that the strain rate achieved by the dynamic test was sufficient only in part to achieve the combination of a brittle matrix and sheathed fibre fracture surface akin to that of the impact tests. At these strain rates the matrix fracture morphology was brittle, but pulled out fibres exhibited poor sheathing more typical of that associated with a ductile matrix failure. The speed of the crack running through the matrix will be greater than the rate at which the fibres are pulled out of the matrix through the crack opening displacement. This difference in relative crack speeds may account for the difference in failure modes between matrix and pulled-out fibres.

An overview of the absolute fracture mechanics parameters and failure mechanisms from all the tests will be discussed in the next section.
7.5.7. Fracture Mechanics Parameter Results

This section will comprise a discussion of the results from fracture mechanics tests (Charpy, IFWIT, $J_c$, dynamic CT) performed on the 10mm thick coupons from grades AH2A, 2B, 2C and 2D. Table 7.7 summarises the results from all these tests.

The results can be broadly characterised into three groups depending upon the strain rate of the test. It is known from observation of the fracture surfaces that there are two general failure modes. At low strain rates (1mm/min) the matrix failure is primarily ductile in nature and pulled-out fibres are not sheathed. At impact speeds (1m/s) the matrix failure is brittle and pulled-out fibres are sheathed in a coat of polymer matrix. At intermediate strain rates (0.17m/s) a balance between the two processes is achieved.

The fracture morphology and relative contribution of the different toughening processes result in the difference in measured fracture toughness and their respective ranking order.

At the lowest strain rates (1mm/min) tests were performed on the SENB $J_c$ specimens and the dynamic LEFM CT specimens. The fracture mechanics parameters $J_c$ and $K_c$ are related through

$$K_c^2 = EJ_c$$

As $J_c$ is extrapolated at crack lengths of 0.2mm it is correct to use $K_c$ values at similar crack lengths. Initiation values are measured at zero crack length, and peak load values are calculated at crack lengths of 10 to 15mm. It is therefore only
appropriate to use values of $K_c$ recorded at crack initiation. Table 7.8 details the results of this relationship on the two grades for which $K_c$ values were obtained at initiation. The difference between $K_c$ calculated from $J_c$ and the measured $K_c$ is relatively small and can be attributed to the difference in crack length measuring technique. The CT technique measures the crack length at the surface, but the SENB test measures the crack length at the centre. As can be seen in figure 7.21 (a CT fracture surface), the fracture front grows as an arc. The different measuring techniques resulted in the crack lengths from the CT specimens being measured as being less than the SENB specimens. As discussed in section 7.5.3, an increased crack length measurement will result in a reduced fracture toughness, thus accounting for the slight discrepancy.

The general trend at these low strain rates is for the fracture toughness to be matrix dominated. Studies of the fracture surface exhibit large plastic deformation of the matrix associated with high toughness, but clean pulled-out fibres typical of low pull-out energy. These toughening processes manifest themselves in the resultant fracture parameters from the different grades. The addition of rubber to the polypropylene increases the fracture toughness of the matrix by the ductile toughening processes discussed earlier. However the addition of glass embrittles the matrix by adding discontinuities and initiating cracks at fibre ends and also contributes little through fibre pull-out.

The highest strain rate tests (IFWIT and Charpy) produced fracture mechanics parameters and ranking order quite different
to that produced at the lowest strain rate. The matrix fracture
surface at such strain rates is brittle, but pulled-out fibres
exhibit matrix sheathing typical of high pull-out energies. The
effect of these different processes is to alter the fracture
parameters significantly. It must be noted that at these high
strain rates the fracture parameters are measured at peak load,
and may therefore be higher by a factor of between 2 and 3 than
the same parameter measured at initiation. The fracture
toughness, $K_c$, of the unreinforced grades is much lower at the
higher strain rate, typical of a transition between ductile and
brittle matrix fracture behaviour. The addition of rubber has
little effect upon the fracture toughness. However, the addition
of glass increases the fracture toughness. This would be
expected as the sheathed fibres typify good interfacial bonding
thus reinforcing the matrix and not initiating cracks to the same
extent that the poorly bonded fibres would.

The strain energy release rate is dominated by (and to a
lesser extent $K_c$ is enhanced by) the energy absorbing process
associated with crack opening, namely, fibre pull-out, rubber
pull-out and matrix fracture surface creation. The energy
associated with the creation of the brittle matrix fracture is
considerably enhanced by the pull-out energies of the sheathed
fibres and rubber particles.

The dynamic LEFM tests performed on CT specimens at a strain
rate of 0.17m/s exhibit fracture morphology characteristics shown
by both higher and lower strain rate tests. The matrix fracture
is mainly brittle, but with some ductile regions especially in
the region of rubber particles. The fibres are pulled-out fairly
cleanly, but with some patchy matrix sheathing. As the strain rate of the dynamic CT tests was increased the fracture toughness, $K_c$, decreased for both initiation and peak load as the matrix fracture transitions from ductile to predominantly brittle behaviour. The measured strain energy release rate, $G_c$, under the same conditions did not alter significantly. This is possibly caused by an energy balance between the slight increase in fibre pull-out energy due to the patchy sheathing of the fibres, and the drop in surface energy due to the transition to a brittle matrix fracture surface.

Major discrepancies occur between $G_c$ calculated by the dynamic LEFM CT and IFWIT SENB tests. The results of the IFWIT are far lower than that of the dynamic CT test. One reason for this is the crack length at which a constant value of $G_c$ is obtained in the dynamic CT test. As explained in section 7.5.6, a constant value of $G_c$ was not obtained until the crack length was considerably greater than that produced at peak load. Therefore, $G_c$ under stable conditions is greater than the $G_c$ calculated at peak load of the cyclic force/displacement curve. In addition to this, as discussed in section 7.5.6 $G$ was calculated at the maximum values of the resonant force/displacement response from the machine (figure 7.42 and 7.43). This analysis produces maximum values for $G_c$. Smoothing the force/displacement curves will produce a lower value.
7.5.8. Additional Fracture Surface Observations

Figures 7.62 - 7.64 show fracture surfaces of specimens in which cracks were propagated at two different speeds and an associated transition in fracture morphology can be seen.

Figures 7.65 - 7.67 show specimens whose cracks were not grown through the full thickness of the specimen. Fibres can be seen partially pulled out of the matrix bridging the open crack. It is this type of process which gives rise to the high $G_c$ values of the fibre filled grades. Charpy impacts at lower speeds than previously used (and thus lower impact energies) caused cracks to propagate only partially through the thickness of the specimen. This gave rise to a large proportion of unsheathed fibres, similar to the CT test at 0.17m/s.

Fibres which were pulled out of the matrix exhibited some interesting features. The broken fibre ends were very seldom coated by matrix material, figure 7.68. The coupling agent probably bonds to the sizing on the surface of the glass fibres. As the fibres are broken into short lengths the fibre ends created by this process are pristine and not coated by sizing. There will be no chemical bond between the fibre end and the matrix. This effect has great influence upon the fracture toughness of the material. Fracture is initiated by penny shaped cracks which initiate at fibre ends. As fibre ends can debond easily, penny shaped cracks will also form quickly and thus contribute to a reduction in the fracture toughness. This process is generally obscured by other more dominant fibre toughening processes.
The broken fibre ends of pulled out fibres exhibited the oval 'thumb nail' fracture surface, typical of bending failures produced during extrusion, as discussed in chapter 4. At no point during the extensive fracture surface analysis was a pulled out fibre observed with a planar fibre end, typical of a tensile failure. This suggests that no pulled out fibres had lengths greater than the critical length. This would agree with the results from the fibre/matrix interface characterisation - that the majority of fibres in the composite have lengths shorter than the critical length.

As discussed in section 7.5.1, rubber particles do not bond with the coupling agent. In the high rubber loaded grades, rubber rich regions did not bond to fibre. This resulted in the patchy sheathing of pulled out fibres. Presumably, this effect will degrade the mechanical properties including the toughness and strain energy release rate.

References

1. Platti E. and Williams J.G.
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2. Lhymn C. and Schultz J.M.
Fracture Behaviour of Collimated Thermoplastic P.E.T Reinforced with Short E-Glass Fibres

Progress in Flow Growth and Fracture Toughness Testing.
ASTM STP 536, p 231. 1973

4. European Group on Fracture
August 1989

220
5. European Group on Fracture
   A Testing Protocol for Conducting J-R Tests on Plastics
   February 1990

   The Effect of Specimen Size on the Results of Plane-Strain
   Fracture-Toughness Tests.

7. ASTM E813-87
   Standard test method for $J_{IC}$, a measure of fracture
   toughness
Table 7.1

Charpy impact test results from 3mm thick moulded specimens.
Notch machined into moulded sample. Shape factor, $\phi$, determined as 1.79 for $G_c$ evaluation.

<table>
<thead>
<tr>
<th>Code</th>
<th>Charpy ($J$)</th>
<th>$G_c$ (kJ)</th>
<th>$E$ (GPa)</th>
<th>$K_{c_y}$ (MNm$^{-3/2}$)</th>
<th>$r_y$ (mm)</th>
<th>$\sigma_{ys}$ (MPa)</th>
<th>$B_{min}$ (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1A</td>
<td>0.19</td>
<td>2.68</td>
<td>1.87</td>
<td>2.24</td>
<td>0.53</td>
<td>38.70</td>
<td>8.37</td>
</tr>
<tr>
<td>1B</td>
<td>0.17</td>
<td>2.36</td>
<td>1.87</td>
<td>2.10</td>
<td>0.46</td>
<td>38.90</td>
<td>7.29</td>
</tr>
<tr>
<td>1C</td>
<td>0.23</td>
<td>3.15</td>
<td>1.20</td>
<td>1.94</td>
<td>0.54</td>
<td>33.40</td>
<td>8.48</td>
</tr>
<tr>
<td>1D</td>
<td>1.00</td>
<td>13.89</td>
<td>1.09</td>
<td>3.89</td>
<td>4.08</td>
<td>24.30</td>
<td>64.09</td>
</tr>
<tr>
<td>1E</td>
<td>0.31</td>
<td>4.28</td>
<td>6.75</td>
<td>5.37</td>
<td>1.88</td>
<td>49.50</td>
<td>29.46</td>
</tr>
<tr>
<td>1F</td>
<td>0.58</td>
<td>8.04</td>
<td>7.44</td>
<td>7.73</td>
<td>0.82</td>
<td>107.90</td>
<td>12.85</td>
</tr>
<tr>
<td>1G</td>
<td>0.34</td>
<td>4.76</td>
<td>5.96</td>
<td>5.33</td>
<td>2.91</td>
<td>39.40</td>
<td>45.72</td>
</tr>
<tr>
<td>1H</td>
<td>0.44</td>
<td>6.14</td>
<td>4.77</td>
<td>5.41</td>
<td>5.32</td>
<td>29.60</td>
<td>83.54</td>
</tr>
<tr>
<td>1I</td>
<td>0.61</td>
<td>8.50</td>
<td>7.05</td>
<td>7.74</td>
<td>0.98</td>
<td>98.80</td>
<td>15.35</td>
</tr>
<tr>
<td>1J</td>
<td>1.03</td>
<td>14.30</td>
<td>5.46</td>
<td>8.84</td>
<td>2.30</td>
<td>73.60</td>
<td>36.04</td>
</tr>
<tr>
<td>2A</td>
<td>0.17</td>
<td>2.33</td>
<td>2.06</td>
<td>2.19</td>
<td>0.48</td>
<td>39.73</td>
<td>7.61</td>
</tr>
<tr>
<td>2B</td>
<td>0.51</td>
<td>7.01</td>
<td>1.17</td>
<td>2.86</td>
<td>2.24</td>
<td>24.17</td>
<td>35.11</td>
</tr>
<tr>
<td>2C</td>
<td>0.34</td>
<td>4.72</td>
<td>7.34</td>
<td>5.89</td>
<td>0.83</td>
<td>81.77</td>
<td>12.96</td>
</tr>
<tr>
<td>2D</td>
<td>0.47</td>
<td>6.51</td>
<td>5.61</td>
<td>6.04</td>
<td>1.26</td>
<td>67.87</td>
<td>19.83</td>
</tr>
<tr>
<td>2E</td>
<td>0.46</td>
<td>6.44</td>
<td>3.52</td>
<td>4.76</td>
<td>1.18</td>
<td>55.40</td>
<td>18.48</td>
</tr>
<tr>
<td>2F</td>
<td>0.66</td>
<td>9.19</td>
<td>3.20</td>
<td>5.42</td>
<td>8.68</td>
<td>23.23</td>
<td>136.29</td>
</tr>
<tr>
<td>2G</td>
<td>0.66</td>
<td>9.14</td>
<td>7.05</td>
<td>8.03</td>
<td>1.63</td>
<td>79.43</td>
<td>25.53</td>
</tr>
<tr>
<td>2H</td>
<td>0.40</td>
<td>5.50</td>
<td>5.82</td>
<td>5.66</td>
<td>4.51</td>
<td>33.60</td>
<td>70.87</td>
</tr>
<tr>
<td>2I</td>
<td>0.43</td>
<td>5.97</td>
<td>6.07</td>
<td>6.02</td>
<td>0.90</td>
<td>80.03</td>
<td>14.15</td>
</tr>
<tr>
<td>2J</td>
<td>0.29</td>
<td>4.00</td>
<td>5.21</td>
<td>4.56</td>
<td>3.29</td>
<td>31.77</td>
<td>51.61</td>
</tr>
<tr>
<td>2K</td>
<td>0.75</td>
<td>10.44</td>
<td>5.00</td>
<td>7.23</td>
<td>1.95</td>
<td>65.37</td>
<td>30.55</td>
</tr>
<tr>
<td>2L</td>
<td>0.54</td>
<td>7.44</td>
<td>4.03</td>
<td>5.48</td>
<td>9.84</td>
<td>22.03</td>
<td>154.52</td>
</tr>
<tr>
<td>2M</td>
<td>0.29</td>
<td>4.01</td>
<td>5.67</td>
<td>4.77</td>
<td>1.36</td>
<td>51.57</td>
<td>21.39</td>
</tr>
<tr>
<td>2N</td>
<td>0.75</td>
<td>10.46</td>
<td>5.79</td>
<td>7.78</td>
<td>1.61</td>
<td>77.30</td>
<td>25.33</td>
</tr>
</tbody>
</table>

Table 7.2 Charpy results from the 10mm thick specimens

<table>
<thead>
<tr>
<th>Code</th>
<th>AH2A ($J$)</th>
<th>AH2B ($J$)</th>
<th>AH2C (0) ($J$)</th>
<th>AH2D (0) ($J$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average</td>
<td>0.559</td>
<td>1.545</td>
<td>2.280</td>
<td>2.960</td>
</tr>
</tbody>
</table>
Table 7.3 Elastic-Plastic fracture mechanics results for specimens cut from the 10mm thick plaques. (0) denotes that the crack was run perpendicular to the melt fill direction. (90) denotes that the crack was run parallel to the melt fill direction.

<table>
<thead>
<tr>
<th>Code</th>
<th>AH2A</th>
<th>AH2B</th>
<th>AH2C</th>
<th>AH2D</th>
</tr>
</thead>
<tbody>
<tr>
<td>Method</td>
<td>$J(0.2)$</td>
<td>$J(0.2)$</td>
<td>$J(0.2)$</td>
<td>$J(0.2)$</td>
</tr>
<tr>
<td></td>
<td>(J/m²)</td>
<td>(J/m²)</td>
<td>(J/m²)</td>
<td>(J/m²)</td>
</tr>
<tr>
<td>CT¹ (0)</td>
<td>----</td>
<td>586</td>
<td>485</td>
<td>979</td>
</tr>
<tr>
<td>CT² (0)</td>
<td>1465</td>
<td>365</td>
<td>891</td>
<td>695</td>
</tr>
<tr>
<td>SENB³ (0)</td>
<td>2100</td>
<td>2270</td>
<td>1400</td>
<td>1840</td>
</tr>
</tbody>
</table>

Table 7.4 Linear elastic fracture mechanics test results from specimens cut from 3mm thick plaques

<table>
<thead>
<tr>
<th>Code</th>
<th>AH1F (0)</th>
<th>AH2C (0)</th>
<th>AH1D</th>
<th>AH2B</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K_{IC}$</td>
<td>5.4</td>
<td>4.5</td>
<td>1.6</td>
<td>1.4</td>
</tr>
<tr>
<td>(MN/m³²)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$G_{IC}$</td>
<td>6.0</td>
<td>4.7</td>
<td>1.3</td>
<td>0.9</td>
</tr>
<tr>
<td>(kJ/m²)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 7.5 Linear elastic fracture mechanics test results from 10mm thick single edge notched bend specimens. Specimens tested at impact speeds of 1m/s. $K_{IC}$ and $G_{IC}$ calculated at peak load.

<table>
<thead>
<tr>
<th>Code</th>
<th>AH2A</th>
<th>AH2B</th>
<th>AH2C (0)</th>
<th>AH2C (90)</th>
<th>AH2D (0)</th>
<th>AH2D (90)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K_{IC}$</td>
<td>1.82</td>
<td>1.97</td>
<td>4.01</td>
<td>3.09</td>
<td>4.02</td>
<td>2.99</td>
</tr>
<tr>
<td>(MN/m³²)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$G_{IC}$</td>
<td>1.11</td>
<td>2.90</td>
<td>6.46</td>
<td>3.63</td>
<td>8.03</td>
<td>5.29</td>
</tr>
<tr>
<td>(kJ/m²)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 7.6 Dynamic linear elastic fracture mechanics test results from 10mm thick compact tension specimens.

<table>
<thead>
<tr>
<th>Code</th>
<th>( K_{IC} ) (1\text{mm/min}) (MN/m(^3)/2)</th>
<th>( K_{IC} ) (0.17\text{m/s}) (MN/m(^3)/2)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Init</td>
<td>Peak</td>
</tr>
<tr>
<td>Calc' at</td>
<td></td>
<td></td>
</tr>
<tr>
<td>AH2C (90)</td>
<td>2.41</td>
<td>6.04</td>
</tr>
<tr>
<td>AH2D (90)</td>
<td>1.96</td>
<td>4.18</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>( G_{IC} ) (1\text{mm/min}) (kJ/m(^2))</th>
<th>( G_{IC} ) (0.17\text{m/s}) (kJ/m(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>AH2C (90)</td>
<td>3.44</td>
</tr>
<tr>
<td>AH2D (90)</td>
<td>3.45</td>
</tr>
</tbody>
</table>

Table 7.7 Comparing the results from the fracture mechanics tests on specimens produced from the 10mm thick plaques.

<table>
<thead>
<tr>
<th>Code</th>
<th>( J_{c} ) (kJ/m(^2))</th>
<th>( K_{C} ) (MN/m(^3)/2)</th>
<th>( G_{c} ) (kJ/m(^2))</th>
<th>Charpy (J)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crack Length</td>
<td>0.2mm</td>
<td>Init'n</td>
<td>Peak Load</td>
<td>Init'n</td>
</tr>
<tr>
<td>AH2A</td>
<td>2.10</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AH2B</td>
<td>2.27</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AH2Cn</td>
<td>1.41</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AH2C90</td>
<td>1.33</td>
<td>2.41</td>
<td>6.04</td>
<td>0.70</td>
</tr>
<tr>
<td>AH2Dn</td>
<td>1.84</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AH2D90</td>
<td>1.14</td>
<td>1.96</td>
<td>4.18</td>
<td>0.45</td>
</tr>
</tbody>
</table>
**Table 7.8** Theoretical relationship between $J_c$ and $K_c$. $J_c$ is extrapolated at crack lengths of 0.2mm. $K_c$ is taken at initiation from the dynamic CT tests. The IFWIT produces a $K_c$ value at crack lengths of between 1mm and 1.5mm, and is therefore not applicable due to the observed increase in $K_c$ with crack length.

<table>
<thead>
<tr>
<th>Code</th>
<th>$J_c(0.2)$ (kJ/m²)</th>
<th>$(E/1-\nu^2)_{90}$ (GPa)</th>
<th>Calc' $K_c$ ($K_c^2 = E.J_c$ (MN/m³/²))</th>
<th>Exp' $K_c$ (MN/m³/²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AH2C₉₀</td>
<td>1.33</td>
<td>2.64</td>
<td>1.87</td>
<td>2.41</td>
</tr>
<tr>
<td>AH2D₉₀</td>
<td>1.14</td>
<td>2.02</td>
<td>1.52</td>
<td>1.96</td>
</tr>
</tbody>
</table>
Fig 7.1  Compact Tension Specimen Configuration

Fig 7.2  Definition of energy absorbed, $U_t$
Figure 7.3 Compact tension specimen fracture surface of fibre filled grade. A dye penetrant is used to reveal the crack path.

Figure 7.4 Unreinforced CT specimens are broken open at liquid $N_2$ temperatures. A difference in fracture morphology reveals the extent of the crack growth.
Fig 7.5 Measurement of crack lengths on compact tension specimens

Fig 7.6 Derivation of $J_{0.2/BL}$
Fig 7.7  Single edge notched bend specimen

Fig 7.8  Derivation of $J_{0.2}$
Fig 7.9  Schematic representation of the falling weight impact test performed upon a single edge notched bend specimen

Fig 7.10  The dynamic CT specimens were cut from the plaques with their notch parallel with the melt fill direction. This orientation produced a crack which advanced perpendicularly to the load direction.
Fig 7.11 Fractomat foil bonded to the surface of a CT specimen. When the specimen is loaded the crack will propagate through the specimen and foil. The crack length can be measured electronically in situ.
Interpretation of strain energy release rate, $G$, at crack initiation

Fig 7.12  Interpretation of strain energy release rate for a propagating crack
Charpy Impact Test Results
3mm Thick Moulded Specimens

**Fig 7.13**

**Fig 7.14**
Charpy Impact Test Results
3mm Thick Moulded Specimens

Figure 7.16 Uncoupled pulled-out fibres from a charpy impact test fracture surface. Fibres are pulled-out clean.
Figure 7.17  Charpy impact test specimen. Coupled pulled-out fibres exhibit matrix sheathing.

Figure 7.18  40% rubber samples exhibited patchy sheathing of pulled-out coupled fibres.
Figure 7.19  Rubber toughens the matrix by inducing plastic flow and other energy absorbing processes.

Jc Evaluation
30% Glass & PP - 3mm Thick

![Graph showing Jc Evaluation](Fig 7.20)
The stress whitened region of CT fracture surface represents the process zone of the crack.

**Jc Evaluation - Method I**

10mm Thick CT Specimens

![Graph](image)
J INTEGRAL EVALUATION
AH2C 10mm CT - Method II

J INTEGRAL EVALUATION
AH2D 10mm CT - Method II
J INTEGRAL EVALUATION
AH2A,B,C & D 10mm CT - Method II

FRACTURE RESISTANCE, J (J/m^2)

CRACK GROWTH, dA (m)

J Integral Evaluation
AH2A  SENB Test

J(0.2) = 2096 J/m^2
J Integral Evaluation
AH2B  SENB Test

\[ J(0.2) = 2270 \text{ J/m}^2 \]

Crack Extension (mm)

J Integral Evaluation
AH2C (0)  SENB Test

\[ J(0.2) = 1406 \text{ J/m}^2 \]

Crack Extension (mm)
**J Integral Evaluation**

**AH2C(90) SENB Test**

![Graph showing J Integral Evaluation for AH2C(90) SENB Test]

- $J(0.2) = 1333 \text{ J/m}^2$

**AH2D(0) SENB Test**

![Graph showing J Integral Evaluation for AH2D(0) SENB Test]

- $J(0.2) = 1843 \text{ J/m}^2$
J Integral Evaluation
AH2D (90)  SENB Test

J(0.2) = 1138 J/m^2

J Integral Evaluation
AH2A & AH2B  SENB Test

Fig 7.33

Fig 7.34
J Integral Evaluation
AH2C(0) & AH2C(90) SENB Test

![Graph of J Integral Evaluation for AH2C(0) & AH2C(90) SENB Test]

J Integral Evaluation
AH2D(0) & AH2D(90) SENB Test

![Graph of J Integral Evaluation for AH2D(0) & AH2D(90) SENB Test]
Figure 7.37  Planar brittle fracture surface of polypropylene fractured at 1m/s.

Figure 7.38  Rubber particles are pulled-out during a charpy impact test (1m/s) and observed on the fracture surface.
Figure 7.39 Compact tension J integral fracture surface (1mm/min). Fibres are pulled-out clean and the matrix fracture is ductile.

Figure 7.40 Fibres pulled-out at 1mm/min are clean of any adherent material.
Matrix failure at 1mm/min is ductile showing large cold drawing and plastic deformation.

AH2C Dynamic LEFM - 0.17m/s
Force & Crack Length vs Deflection

Fig 7.42
AH2D Dynamic LEFM - 0.17m/s
Force & Crack Length vs Deflection

AH2C Dynamic LEFM - 1mm/min
Force & Crack Length vs Deflection
AH2D Dynamic LEFM - 1mm/min
Force & Crack Length vs Deflection

AH2C Dynamic LEFM - 1mm/min
$G_c$ vs Crack Extension
AH2C Dynamic LEFM - 1mm/min
Gc vs Crack Speed

![Fig 7.47](image1)

AH2C Dynamic LEFM - 1mm/min
Kc vs Crack Extension

![Fig 7.48](image2)
AH2C Dynamic LEFM - 1mm/min
\( K_c \) vs Crack Speed

\[ \begin{array}{c}
K_c \text{ (MN/m}^{3/2}\text{)} \\
3.5 \\
3 \\
2.5 \\
2 \\
0.5 \\
0.05 \\
0.1 \\
0.15 \\
0.2 \\
0.25 \\
0.3 \\
0.35 \\
0.4 \\
0.45 \\
0.5 \\
\end{array} \]

Crack Speed (mm/sec)

Fig 7.49

AH2D Dynamic LEFM - 1mm/min
\( G_c \) vs Crack Extension

\[ \begin{array}{c}
G_c \text{ (kJ/m}^2\text{)} \\
25 \\
20 \\
15 \\
10 \\
5 \\
0 \\
\end{array} \]

Crack Extension (mm)

Fig 7.50
AH2D Dynamic LEFM - 1mm/min

$G_c$ vs Crack Speed

Fig 7.51

AH2D Dynamic LEFM - 1mm/min

$K_c$ vs Crack Extension

Fig 7.52
AH2D Dynamic LEFM - 1mm/min

$K_c$ vs Crack Speed

![Graph showing $K_c$ vs Crack Speed](image)

$K_c$ (MN/m$^{3/2}$)

Crack Speed (mm/sec)

Fig 7.53

K & G vs Crack Length

AH2C 0.17 m/s

![Graph showing K & G vs Crack Length](image)

Crack Length (mm)

$K$ (N/m$^{3/2}$) (Millions)

G (J/m$^2$)

The crack measuring foil failed after 15mm crack growth.

Fig 7.54
The foil on this specimen failed after 15mm.

This foil tore O.K.

---

K & G vs Crack Length

AH2C 0.17 m/s

K (N/m^3/2) vs Crack Length

AH2D 0.17 m/s

K (N/m^3/2) vs Crack Length

---

The foil on this specimen failed after 15mm.
Figure 7.58  Dynamic CT at 1mm/min exhibits cleanly pulled-out fibres and ductile matrix failure.
**Figure 7.59** Dynamic CT at 0.17m/s (10200mm/min). Fracture surface is predominately brittle with ductile rubber components.

**Figure 7.60** Dynamic CT at 0.17m/s. Rubber particles are sheared from the brittle matrix.
**Figure 7.61** Dynamic CT at 0.17m/s. Pulled-out fibres exhibit patchy sheathing.

**Figure 7.62** Two speed fracture surface of polypropylene. Brittle to the left, ductile to the right.
Figure 7.63 Two speed fracture surface showing the ductile (left)/brittle (right) transition of a rubber toughened polypropylene.

Figure 7.64 Two speed fracture surface of a rubber toughened, glass reinforced polypropylene. Brittle to the right, ductile to the left.
Figure 7.65 3-point bend specimens were partially broken open at different speeds to observe crack opening phenomena.

Figure 7.66 Crack opening at low strain rate (1mm/min). Unsheathed fibres bridge the crack.
Figure 7.67 Crack opening at 1m/s causes sheathed fibres to bridge the brittle fracture surfaces.

Figure 7.68 Pulled-out fibres always show fibre ends caused by bending failures. Fibre ends are usually not sheathed.
8. DISCUSSION

The course of this project has followed a natural progression from material production, through characterisation, to predictive mechanical modelling. This chapter draws together results, observations and discussion from the previous chapters in a general overview of the materials microstructural characteristics and properties.

8.1. PRODUCTION OF MATERIALS

Despite the care taken to keep the composite constituent elements (polypropylene, rubber, glass, coupling) and processing conditions as constant as possible, there was a significant difference in the mechanical properties of the nominally identical grades extruded in the two campaigns. These differences in composite properties are believed to be due to changes in the mechanical properties of the matrix and, more specifically, of the polypropylene base polymer. Polypropylene used in the first campaign \((AH1\_\text{)}\) was less stiff than that used in the second campaign \((AH2\_\text{)}\), but realised a much higher strain-to-yield and yield stress. The polypropylene for the two campaigns originated from different batches. These characteristics of the base polymer were then repeated through the resulting composite grades. The addition of rubber to the matrix compounded this effect due to the manner in which the rubber alters the matrix properties and the fibre/matrix
interface properties. These properties will be discussed in the next section.

8.2. CONSTITUENT PROPERTIES

8.2.1. Rubber Modification

Rubber particles were found to be distributed uniformly as a separate phase within the polypropylene matrix. In all grades the particles were oval in shape, extended in the melt fill direction, and approximately 1 to 3 microns in diameter. The rubber particles were pulled out of the matrix at high strain rates and/or low temperatures, figure 7.38, demonstrating that there was only limited bonding between the rubber and the polypropylene. The coupling agent did not appear to promote bonding between the rubber and the fibre. This effect becomes particularly significant as a disproportionate reduction in the mechanical properties and fracture toughness of composite grades in which the rubber content becomes a high proportion of the total matrix, section 7.5.8.

The addition of rubber to the matrix has two main effects: it alters the load response of the base polymer, and it affects the properties of the fibre/matrix interface in the fibre filled grades. Fortunately, at least from a modelling viewpoint, it appears that both properties are affected in a similar manner.

A small addition of rubber to the polypropylene reduces the matrix stiffness appreciably. Subsequent equivalent additions of rubber decrease the stiffness of the matrix further, but to
a reduced extent. The classical rule-of-mixtures particulate model does not predict the observed reduction in matrix stiffness accurately. It has been shown in chapter 6 that an empirical power law fit is more appropriate:

\[ \sigma_m = \sigma_p (1 - F_r)^x \]  

(6.7)

where;

- \( \sigma_m \) is the stress in the matrix at any specific strain \( \varepsilon_m \);
- \( \sigma_p \) is the stress in the polypropylene at strain \( \varepsilon_m \);
- \( F_r \) is the volume fraction of rubber in the matrix;
- and \( x \) is a constant exponent which describes the rate at which the rubber softens the matrix. The constant is found to be 0.69 for the AH2 grade materials and 0.56 for the AH1 grade materials. This model describes the observed mechanical properties of the rubber modified polypropylene grades well, figure 6.2.

The effect of the rubber on the fibre/matrix interface will be discussed in the following section.

8.2.2. Interfacial Properties

A number of attempts to measure the fibre/matrix interfacial shear strength were made. Unfortunately, to create a test specimen with a fibre/matrix interface identical to that of an injection moulded component proved impossible with the equipment available. The specimens which were produced showed, in a qualitative manner, that the interfacial shear strength was enhanced greatly by the addition of a small quantity of the
coupling agent, but weakened substantially by small additions of rubber. The interfacial shear strengths which were evaluated, chapter 5, are lower than those proposed by other workers\(^1,2,3\). Also, when these values were incorporated into recognised models the resulting predictions underestimated actual material properties. It was for these reasons that the maximum interfacial shear strength of the interface was estimated as being larger, by a constant proportion, than the measured values.

In a similar way to that in which the rubber decreases the strength of the matrix, the interfacial shear strength might be expected to be equally affected. This was shown to be the case in the measured interfacial shear strengths, chapter 5, and composite stress/strain predictions, chapter 6. It is proposed that rubber decreases the interfacial shear strength in a power law manner, using an identical exponent, \(x\), to that found for the matrix stiffness:

\[
\tau_{\text{Mmax}} = \tau_{\text{PPmax}} \cdot (1 - F_r)^x
\]  

(8.1)

where:

\(\tau_{\text{Mmax}}\) is the fibre/matrix interfacial shear strength;

\(\tau_{\text{PPmax}}\) is the fibre/polypropylene interfacial shear strength.

It is proposed that for thermoplastic composites, where plasticity in the matrix is extensive, the interfacial shear stress changes with strain. The relationship can be modelled through a power law fit based on the polypropylene response, and limited to the maximum shear strength of the interface. This
proposal is a compromise between the models of Mittal and Gupta\(^1\), and Bader and Bowyer\(^4\). The former suggest a linear relationship between the composite (base polymer) stress and the fibre/matrix interfacial shear stress; the latter, a constant interfacial shear stress.

The proposed relationship is:

\[ \tau = \tau_{\text{max}} \left( \frac{\sigma_{PP}}{\sigma_{PPY}} \right)^{\gamma} \]  

(6.21)

where:

- \( \gamma \) is found to be 0.35 by data analysis;
- \( \sigma_{PP} \) is the stress in the polypropylene base polymer at any composite strain, \( \varepsilon_c \);
- \( \sigma_{PPY} \) is the stress in the composite at matrix yield.

Equations (6.21) and (8.1) are combined to develop a relationship expressing the fibre/matrix interfacial shear stress at any strain in a rubber toughened polypropylene:

\[ \tau = \tau_{\text{PPmax}} \left( \frac{\sigma_{PP}}{\sigma_{PPY}} \right)^{\gamma} (1-F_r)^x \]  

(6.22)

Equation (6.22) is fundamental to the calculation of the load carried by any fibre, at any strain, embedded in a rubber modified polypropylene matrix. The critical fibre length can be calculated at any strain from\(^5\):

\[ l_c = r \sigma_{f}^{x}/\tau \]  

(6.8)
and subsequently the contribution to the composite stiffness from subcritical and supercritical fibres can be determined:

\[ X = \sum_{(\text{subcritical})} t_i \frac{V_i}{2r_f} \tag{6.18} \]

\[ Y = \sum_{(\text{supercritical})} E_f \varepsilon_c \left(1 - \frac{E_f \varepsilon_c r_f}{2l_i \tau} \right) V_j \tag{6.19} \]

8.2.3. Fibre Reinforcement

Apart from the interfacial characteristics, two other factors contribute to the reinforcing efficiency of the fibres: namely the fibre length, and orientation distributions. Both of these parameters have been measured for a range of grades, using automated image analysis techniques, chapter 4.

The fibre length measurements exhibit log-normal distributions. Fibre lengths vary from fibre fragments of less than 14 microns (fibres of length shorter than the fibre diameter) to fibres approaching the dimensions of the extruded granule, although these were exceptionally rare. As the level of fibre reinforcement was increased, the fibre length distribution narrowed and the average fibre length became less. It is envisaged that increasing the fibre loading within the extruder increases the shear rate and the probability of fibre/fibre interactions. This results in increased fibre attrition and hence a shorter average fibre length.

On closer inspection the broken fibre ends were observed to exhibit the thumb nail fracture surface typical of a high
curvature bending failure - a process consistent with fibre attrition in extruder compounding. The short fibre fragments were found to be glass shards created at the fracture face during these bending failures.

Fibre length distributions were evaluated from extruded granules as well as the final moulded components. There was seen to be only a small difference in the FLD from the two samples, implying that the majority of the fibre attrition occurs during compounding.

Fibre orientation distributions were evaluated for a range of fibre reinforced grades. Through both the thickness and the breadth, the samples were envisaged as being comprised of three symmetric layers. Adjacent to the surface of the mould, a layer of fairly randomly oriented fibres formed, created by turbulent flow of the melt front freezing on contact with the chilled mould wall. This outer shell of material is less than one tenth of a millimetre thick. Within this shell lies a thicker concentric layer of fibres oriented in the melt fill direction. The core of the composite is made up of fibres oriented away from the melt fill direction and towards the perpendicular. The relative thicknesses of these layers depends on the constraining forces and proximity of the surfaces of the mould. Through the thickness of the sample, the vast proportion of the section is dominated with fibres well aligned to the melt fill direction. Across the central breadth of the sample the core dominates with fibres oriented away from the melt flow direction. Travelling
away from the central core through the thickness of the specimen, the proportion of off-axis fibres decreases.

The Krenchel orientation factor was evaluated for the central through thickness section (the significant thickness section with the greatest proportion of off-axis fibres). This was calculated as being 0.9, implying that the majority of fibres were oriented in just one plane across the breadth of the sample. The composite was thus treated as a laminate made up of layers containing fibres oriented in just 2-dimensions. The relative thickness and average Krenchel orientation factor was evaluated for each of the layers and combined to create an orientation factor for the composite as a whole.

Increasing the volume fraction of glass increases the degree of misorientation within the composite. This is because adjusting the volume fraction of glass in the melt will alter its flow, viscosity and shear properties and hence affects the final orientation distribution.

8.3. COMPOSITE PROPERTIES

8.3.1. Tensile Properties

Composite stiffness increases with the addition of glass and decreases with the addition of rubber. The model developed by Bader and Bowyer\(^4\) can be modified in the manner summarised above, to account for a rubber modified matrix, and a fibre/matrix interfacial shear stress affected by composite strain and rubber content.
Predicting composite strength is more difficult due to the complex nature of yield initiation. The modified rule-of-mixtures overestimates the yield strengths of all the fibre reinforced composite grades. Coupled grades exhibit the best agreement, but the strengths of the uncoupled grade are overestimated significantly. The model does not allow for the premature matrix yield initiated by the fibres. Yield is initiated from cracks developed at fibre ends, particularly when the fibre end debonds from the matrix inducing a high localised stress in that region. In all fibre filled grades, the fibre fragmentation test showed that fibres partially debond at strains well before the strain-to-yield of the matrix. However, the strains at which the coupled fibres (chemically bonded to the matrix) debond from the matrix are well in excess of those at which the uncoupled (frictionally bonded) fibres debond. This accounts for the large reduction in the strain-to-yield of the uncoupled grades when compared with the coupled grades, and the differences between the predicted and actual yield strengths.

8.3.2. Toughness

The quantitative evaluation of toughness of a short fibre reinforced thermoplastic has proven to be highly complex. Not only is it difficult to select an appropriate fracture mechanics parameter, but, due to the nature of the damage zone ahead of the crack tip, it is very difficult to evaluate the crack length and/or initiation point. The effect of strain rate on fracture morphology makes the situation even more complex.
Two main approaches were used to evaluate fracture mechanics parameters of the coupled composite grades, namely linear elastic fracture mechanics and J integral elastic/plastic fracture mechanics.

Linear Elastic Fracture Mechanics has limited applicability to SFRTF materials. Extensive plasticity at the crack tip is not accounted for. The applicability of the test to an advancing crack tip impinging upon brittle fibres within the ductile matrix is, as yet, not fully understood. However, within these limitations, quantitative testing was performed on a range of specimen geometries (Dynamic CT, IFWIT SENB) at room temperature. The values obtained for LEFM parameters were strain rate dependent. Testing strain rates ranged from 1mm/min to 1m/s. More specifically, the fracture surfaces of the failed test specimens exhibited a radical change in fracture morphology at the extremes of strain rate of the tests.

The dynamic CT specimens with the fractometer foil attached indicated that the crack initiated before peak load. $K_{IC}$ and $G_{IC}$ were evaluated at crack initiation (Init') and at peak load where crack lengths were approximately 10 to 15mm beyond the original razor notch. At these crack lengths, conditions approximating stable crack growth were achieved. The IFWIT results from SENB test specimens were calculated at peak load only.

Elastic/plastic fracture mechanics tests are more applicable to this range of composite materials because the presence of plasticity at the crack tip is recognised. The major problem encountered with the J integral evaluation is the accurate measurement of the crack tip advance. This problem has been
overcome by measuring the crack advance 'in situ' using a specimen sectioning technique, section 7.2.3c. Tests were performed at room temperature at a strain rate of 1mm/min. The $J$ integral values were extrapolated from a power law fit and produced an elastic-plastic value of fracture toughness at crack initiation.

Results from the two fracture mechanics approaches were compared and related to the observed fracture surface morphology.

Table 7.7 indicates that the addition of rubber to the matrix, increases consistently the toughness of the material at impact speeds. Energy absorbing processes occurring during loading of ductile polymers at temperatures at and above the $T_g$ of the matrix components are shear flow and craze formation.

Such mechanisms would help to account for the increased toughness of rubber modified grades at impact strain rates. At the lower straining rates the addition of rubber decreases the fracture toughness of fibre filled grades. The reduction in the modulus and fibre/matrix shear strength of the matrix caused by the rubber reduces the reinforcing ability of the fibres and their associated pull-out energy. The elastic-plastic fracture mechanics and high strain rate LEFM testing show opposite trends. The elastic-plastic results suggest that the addition of glass fibres to the matrix reduces the energy required to initiate fracture because they act as stress concentrators. At the higher strain rate of the IFWIT this effect is reversed, implying that the fibres toughen the material by increasing the energy required to initiate and propagate fracture.

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When fracture surfaces were observed in SEM, a clear difference in fracture morphology as a function of strain rate could be seen. This manifested itself as a change in the modes of the matrix failure and fibre pull-out. At low strain rates the matrix fracture surface exhibits large shear and plastic deformation, figure 7.41. Under impact conditions the fracture surface is planar in nature, figure 7.37. There is little or no plastic deformation. Rubber particles are pulled-out of the matrix and can be seen clearly in figure 7.38 as spheres protruding from the fracture surfaces. Such polymeric materials are viscoelastic solids. Their propensity to anelastic and plastic deformation is reduced when they are tested at high strain rates and/or at low temperatures. This reduced deformability causes a formerly tough polymer to respond in a brittle manner. It is reasonable to expect that the toughening effect of the rubber in the matrix will be greater at low strain rates.

At low strain rates the surfaces of pulled-out fibres were clean of any additional material, figure 7.39. The surfaces of pulled-out fibres at higher strain rates exhibited a sheath of adherent matrix material, figure 7.17. Bailey and Bader\(^7\) observed a similar process with short glass fibre reinforced nylon and suggested that the cohesive failure of the sheathed fibre absorbed far more energy than the adhesive failure of the unsheathed fibre. At low strain rates the fibres acted as stress raisers and debonded easily from the matrix, and this accounts for the \(J\) integral results. At higher strain rates the fibres toughened the matrix through the sheathing effect.
The purpose of the compact tension LEFM testing was to investigate this process further at two significantly different strain rates under more controlled conditions. At both strain rates similar trends to the $J$ integral behaviour were produced. The higher strain rate was limited by the cross head speed of a hydraulic load frame. The maximum obtainable speed was 0.17m/s. At this speed the fracture surfaces of the specimens exhibited only slight patchy sheathing of pulled-out fibres, figure 7.61. Fibre sheathing is very strain rate dependent. The recorded speeds of the impact tests were the speeds of the impactors on contact with the specimen. The elastic nature of the specimen causes it to deflect upon impact. When the crack finally initiates and begins to propagate the crack speed may be many times greater than the speed of the impactor. The fastest strain rate obtained in the CT test was not sufficient to reproduce fully the results obtained from impact testing.

In summary, at low strain rates the plastic deformation of the matrix and rubber particles are the predominant forms of toughening. Under such conditions the fibres weaken the system by introducing stress raisers and discontinuities to the matrix. At impact speeds the viscoelastic polymer matrix cannot respond fast enough to the loading conditions and fractures in a brittle manner. However, at such speeds the fibres act to toughen the material by increasing the pull-out energy. This is due to an adherent sheath of matrix on the fibre surface which is not present at the lower strain rates.
8.4. CONCLUDING REMARKS

The mechanical properties and impact resistance of thermoplastics can be enhanced greatly by the selective addition of fibre, rubber and coupling components. Predicting the relative contributions from these components individually and combined is complex, but to a limited extent can be achieved through modified models and reasonable assumptions. However, none of this would be possible without the preliminary determination of the microstructural characteristics of the composite grades.

Microstructural characterisation of SFRTP's is highly complex, especially in a three-phase system. The combination of image analysis techniques for recording large numbers of fibres and a computer simulation and correction program to allow for the limitations of the analyser when dealing with a large aspect ratio range, can produce sensible results. Fortunately, in this series of tests the fibres within the mouldings were mainly oriented within a single plane due to the mould surface constraints. Thus the moulded material could be modelled as being composed of plies of varying average orientation. The rubber was distributed uniformly throughout the specimens. The main effect of the rubber was to reduce the stiffness of the matrix and hence the shear stress at the fibre/matrix interface while at the same time improving the fracture toughness and critical strain energy release rate of the matrix.

The fracture mechanics properties of coupled short glass fibre reinforced polypropylene toughened with rubber were highly
strain rate dependent. At low strain rates the addition of glass fibres degrades the toughness of the material. The fibres act as discontinuities within the matrix, aiding initiation and propagation of a crack. At higher strain rates the fibres toughen the material by increasing the energy dissipation associated with fibre pull-out. These effects resulted in changes in the fracture surface morphology. Fibres pulled-out at low strain rates had clean surfaces. At higher strain rates the surface of pulled-out fibres was coated in an adherent sheath of matrix material. These effects are a direct consequence of the viscoelastic nature of the matrix. At low strain rates the matrix deforms plastically. At impact speeds the matrix responds in a predominanantly brittle manner.

Careful selection of material grades can thus lead to relatively inexpensive, easily fabricated, mass produced composite components exhibiting high toughness and reasonable strength.
8.5. **FUTURE WORK**

This project has highlighted a number of areas which may merit further useful work:

1. **Experimental evaluation of the manner in which the fibre/matrix interfacial shear stress varies with strain in ductile matrices.**

2. **The possible development of a method for evaluating complex three dimensional orientation distributions from essentially two dimensional planar sections.**

3. **Chemical modification of the rubber used in this series of tests may promote greater adhesion between the rubber and the fibre, thus increasing the toughness of the material.**

4. **The application of the mechanical modelling used in the present work to the Cox model for the stress build up within a short fibre.**

5. **A specific investigation of the matrix sheathing observed around fibres drawn at high strain rates from ductile thermoplastics under impact conditions.**
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