FIBRILLATED POLYALKENE FILMS

IN CEMENT

by

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SUMMARY

A review is made of existing theories for predicting the tensile stress-strain behaviour and the mechanisms of fibre-matrix stress transfer for fibre reinforced cements. The applicability of the inherent assumptions in the theories to practical fibre cements and polyalkene cements in particular is discussed. A description is given of the structure, mechanical properties and production techniques of fibrillated polyalkene films in order that their properties may be related to those of the composites.

Five types of fibrillated polyalkene film and a monofilament were incorporated in a high strength cement based matrix. The general composite tensile behaviour was examined with particular reference to the "first cracking" strain of the composite and the fibre-matrix stress transfer mechanisms. The films were mainly opened to four times their unopened width but two composites were made with unopened film to increase the axial alignment of the fibrils. The majority of testing was after 28 days water curing with two composites also being cured for 1 year. Composites were monitored for load, strain and acoustic emission measurements. A small number pull out tests were performed on narrow unfibrillated strips of film.

It is shown that the general behaviour of the composite complies with existing theoretical predictions. However, a new model is proposed for predicting the "first cracking" strain of the matrix. The fundamental mechanism of fibre-matrix stress transfer is attributed to misfit resulting from the non-uniform cross section of the individual fibrils.

The implications of this work are assessed and a tentative film specification is proposed for the production of improved composites.
TO MY FRIENDS

...but the speculatist, who is not content with superficial views, harasses himself with fruitless curiosity, and still, as he inquires more, perceives only that he knows less.

Samuel Johnson
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This project forms part of the continuing development of a new material from laboratory bench to commercial production. As such, discussions have been held with many representatives of both the academic and commercial worlds. I would like to acknowledge the following - Dr. A. Kelly of the University of Surrey, Professor A. Keller and Dr. P. Barham of Bristol University, Dr. J. Bijen and Dr. M. Jacobs of DSM, Holland and Dr. A. Vittone of Montedison, Italy.

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Note on Statistical Analysis

All significance testing used the student t-test with a significance level of 95%.
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CHAPTER 1

INTRODUCTION

The structural use of concrete is restricted to those applications where the stress field is purely compressive, unless reinforcement is included to carry the tensile loads. This is because of the dual effect of the low tensile strength and low work of fracture of cementitious materials. The invention of the wet process-sieve cylinder by Hatshek in 1900 (Austrian Patent, 5970) presented, what has become to date, the most successful commercial means of overcoming one of these limitations for thin sheet applications, namely asbestos-cement.

However, while asbestos-cement has a higher strength than ordinary cement pastes it remains a brittle material. Some countries insist on a "safety net" being provided under asbestos-cement roofs to prevent injury should anyone fall through the roof. Also, the environmental pressures on asbestos fibre, suitable supplies of which may be limited (Krenchel and Hejgaard, 1975), have been increasing since the mid 1970's as a result of the long suspected health hazards associated with it (Annual Report of the Chief Inspector of Factories and Workshops, 1898). As a result, much effort has been expended to find a suitable material as a replacement for asbestos cement. Some potential replacements for flat sheet have been a success, e.g., Masterboard produced by Cape Boards and Panels Limited, whilst others appear to have been taken off the market, e.g., TAC Board produced by TAC Building Products. A general replacement has still to be found for the vast European market of corrugated cladding panels (400 x 10⁶ m² per annum) and it is to this market that the material discussed in this thesis is mainly directed.
The range of fibres which may be incorporated within a cement matrix is wide, however, each has its disadvantages. Natural fibres such as coconut, various grasses or cellulose have been suggested, but all may swell or rot if kept moist for periods of time due to their hygroscopic nature and they may be affected by the alkaline nature of the matrix. Carbon and Kevlar\(^{(R)}\) fibres produce high strength composites but at such a cost as to make their use uneconomical. The development of alkali-resistant glass fibres has increased the potential of glass reinforced cement, but doubts still remain about its long term strength and its high cost may restrict its use to architectural cladding units rather than the corrugated sheet market. Combinations of glass and cellulose fibres are also under investigation. Ferrocement, with its fine sheet meshes, has been used in boat and shell construction but has not penetrated the corrugated sheet market, probably due to the high labour costs involved in its lay-up.

Another major group of fibres to be considered are the low modulus man made organic fibres such as polypropylene. In recent years interest in these fibres has increased following work at the University of Surrey (Hannant, Zonsveld and Hughes, 1978) and its licensees (Bijen and Geurts, 1980; Vittone, Camprincoli, Rosati and Maltese, 1982) and also by Krenchel and Dansk-Etermit (Krenchel and Jensen, 1980). Chemical inertness and low cost are among the advantages claimed for these polyalkene fibres (unsaturated hydrocarbons containing one double carbon-carbon bond, the simplest being ethylene, \( \text{CH}_2 = \text{CH}_2 \)).

A disadvantage often mentioned is the low fire resistance of materials having at least 5% by volume of organic matter. However, farm and single storey industrial buildings more than 1 metre from a boundary have low fire requirements and also account for a large

\(^{(R)}\) Registered Trademark of Dupont.
In the early 1970's it was suggested that, due to their low moduli, polyalkenes would not reinforce cement (Thomas, 1972). However, Hughes and Hannant (1982) showed experimentally that, using fibrillated polypropylene films, apparent moduli of rupture of at least 50 MPa could be achieved and that the composite exhibited substantial ductility resulting from the development of multiple fracture of the matrix. The composite appears to have commercial prospects and has been patented (GB Patent Specification No. 1, 582, 945) and licensed by the University of Surrey. The material is known as NETCEM(R).

In order for a pseudo-ductile material, such as NETCEM, to be commercially viable it is necessary for it to show a high first cracking strength in relation to the working stresses. Also, it is necessary that the widths of any cracks, which may form during transient overloads, are very small and self-sealing so that the corrugated sheet does not permit the ingress of water. This thesis is aimed at developing an understanding of how to achieve these properties using fibrillated polyalkene films and to test the applicability of existing composite theories to polyalkene cements.

Five fibrillated films and a monofilament, which were known to produce composites exhibiting properties ranging from poor to the best available in 1978, were incorporated in a high strength, cement based matrix and tested in direct tension. This mode of testing was chosen in preference to flexural tests since the analysis of the experimental results is of a more fundamental nature and fewer assumptions have to be made about the distribution of stresses.

A tentative specification is proposed for the production of a fibrillated polyalkene film which may yield an improved composite.
2.1. Early Research and its Relation to Recent Work

The concept of reinforcing cement and concrete with fibrous inclusions is not new; indeed, asbestos cement has been known since 1879 (Klos, 1975). Also Porter (1910) tells of including "a certain number of cut nails or spikes per cu. ft. of concrete to increase the compressive and tensile strength of the concrete by up to 8 times". However, it was not until Romualdi and Batson (1963) published their first paper that the impetus was provided for the large research effort expended in subsequent years.

The latter authors claimed a reinforcement of concrete by the use of fine steel fibres (i.e., an increase in strain at first cracking). Provided that the fibre spacing was less than 8 mm it was proposed that the tensile cracking strain of the matrix would be enhanced by a function inversely proportional to the square root of the fibre spacing, at a given fibre volume fraction. The theory was based upon a fracture mechanics approach assuming elastic continuity across the fibre-matrix interface and was apparently verified by the results of flexural tests.

It was not until the late 1960's that results were published of direct tensile tests which refuted the early claims (Shah and Rangan, 1969). Further work (Edgington, 1973; Johnston and Coleman, 1974) supported the experimental evidence, showing that the matrix failure strain was only marginally increased and that the Laws of Mixtures appeared to apply.
Aveston and Kelly (1973), using another fracture mechanics approach produced a similar relationship (to Romualdi and Batson) between matrix failure strain and fibre spacing. However, they concluded that the shear stresses at the fibre-matrix interface were such that elastic continuity could not be maintained in close proximity to a crack. Thus, the interface would debond producing a frictional stress transfer, and, hence, such large increases in matrix failure strain could not be recognised. They also analysed the case where partial debonding occurred but concluded that, for practical purposes, the assumption of frictional stress transfer should describe the behaviour of fibre cements unless the fibre-matrix bond strength was greater than the matrix strength.

Thus, in relation to further discussion, the stress transfer between polyalkene fibre and matrix is considered to be purely frictional (Zonsveld, 1975; Majumdar, 1975). It is not intended to present a comprehensive review of fibre cements and concretes (see Hannant, 1978) but rather a selection of the literature which has particular relevance to thin sheet fibre cements.

2.2. Single and Multiple Fracture of Brittle Matrix Composites

The practical composite is usually a combination of fibres of strength, \( \sigma_{fu} \), greater than that of the matrix, \( \sigma_{mu} \). Should the fibres fail at a lower strain, \( \varepsilon_{fu} \), than the matrix, \( \varepsilon_{mu} \), single fracture will occur if the matrix is unable to sustain the extra load thrown upon it:

\[
i.e., \quad \sigma_{fu} V_f > \sigma_{mu} V_m - \sigma'_m \ V_m \quad \ldots \quad (2.1)
\]

where \( \sigma'_m \) = stress in matrix at \( \varepsilon_{fu} \),
\( V \) = volume fraction, subscripts \( m \) and \( f \) refer to matrix and fibre respectively.
If equation 2.1. is not satisfied the fibres will continue
to fracture into discrete lengths and the matrix strain will
increase until $\epsilon_{mu}$ is exceeded.

The case of more current interest is that in which
$\epsilon_{fu} >> \epsilon_{mu}$, where multiple fracture of the matrix will occur
when

$$\sigma_{fu} V_f > \sigma_{mu} V_m + \sigma'_f V_f$$ \hspace{1cm} (2.2.)

where $\sigma'_f = \text{stress in fibre at } \epsilon_{mu}$.

The matrix will continue to fracture into blocks of length
between $x'$ and $2x'$ (Robinson, 1962-63).

The theoretical description of the tensile behaviour of
brittle matrix composites which fail by multiple fracture was
significantly advanced by the work of Aveston, Cooper and Kelly
(1971). This paper is extremely important for its presentation
of a unified theory covering many aspects of composite performance.
The theory was developed at the National Physical Laboratory
and the contributions of Hale, Mercer and Sillwood are recognised,
although, in keeping with the literature, it will be henceforth
referred to as the ACK theory. The theoretical development
of the subject was aided by previous work on reinforced concrete,
metallic and polymeric matrix composites, ferrocement and glass
reinforced cement (e.g., Efsen and Krenchel, 1957; Krenchel,

2.3. Tensile Behaviour of Brittle Matrix Composites - Theoretical
Assumptions and the Idealised Stress-strain curve

The ACK theory is based upon the following assumptions:

(a) The fibres are continuous and aligned;
(b) The matrix exhibits a well defined, single valued
failure strain;
(c) The fibres are linear elastic;
(d) Poisson's ratio of matrix and fibre is zero;
(e) Fibre-matrix stress transfer is frictional and linear.

Although the limitations of these assumptions will be discussed in greater detail in later sections the following should be noted. Cement matrices are known to be highly variable in terms of strength (Aveston, Mercer and Sillwood, 1974) and elastic modulus (Allen, 1975). The assumption of linear elastic fibres for steel and glass may be adequate but is inapplicable for polyalkenes. Not only is Poisson's ratio non zero for these polymers, they also exhibit a different value from that of cement; typical values being 0.23 for cement and 0.3 for polypropylene (Kelly and Zweben, 1976). Values as high as 0.4 have been reported for high modulus polyethylene (Zihlif, Duckett and Ward, 1978). The assumption of linear fibre-matrix stress transfer simplifies the analysis.

The idealised tensile stress-strain curve is shown in Figure 2.1. The composite is initially stiff, OA, the matrix being dominant until cracking occurs at a constant stress. The zone of multiple cracking, AB, is followed by a less stiff region, BC, in which the additional load is carried by the fibres.
In the following sections it is intended to critically examine all regions of the curve with relation to practical composites.

2.4. Tensile Behaviour of Brittle Matrix Composites - First Crack Performance

2.4.1. Uncracked Composite

Prior to matrix cracking the mechanical behaviour of the composite is approximated by the Laws of Mixtures which relate the mechanical properties of the individual phases and their volume fractions. Hence, the modulus of the uncracked composite, $E_c$, is given by,

$$ E_c = E_m V_m + E_f V_f $$  (2.3.)
This term may be modified to account for composites containing short, non-aligned fibres such that,

\[ E_c = E_m V_m + \eta_1 \eta_2 E_f V_f \]  

\[ \ldots \ldots (2.4.) \]

Laws (1971) has suggested that the efficiency factor \( \eta_1 \) for short, aligned fibre composites is approximately unity (0.98). In contrast, Allen (1975) related the fibre length, \( l \), to its critical length, \( l_c \), where \( l_c \) is defined as twice the shortest length of embedment to produce fibre fracture in a pull out test.

\[ \eta_1 = \frac{1}{2^{l_c}} \quad 1 < l_c \]  

\[ \ldots \ldots (2.5.) \]

\[ \eta_2 = 1 - \frac{l_c}{2^{l}} \quad 1 > l_c \]  

\[ \ldots \ldots (2.6.) \]

The difference in approach lies in the assumptions made of the nature of the fibre-matrix interface. Where as Laws assumes the interfacial stress transfer to be frictional, Allen assumes elastic continuity to be maintained.

The second efficiency factor, \( \eta_2 \), compensates for fibre orientation as shown by the typical values in Table 2.1.

<table>
<thead>
<tr>
<th>Fibre Orientation</th>
<th>( \eta_2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cox (1952)</td>
</tr>
<tr>
<td>1D</td>
<td>1</td>
</tr>
<tr>
<td>2D Random</td>
<td>1/3</td>
</tr>
<tr>
<td>3D Random</td>
<td>1/6</td>
</tr>
</tbody>
</table>

Table 2.1. Typical Fibre Orientation Efficiency Factors
Again, the small difference in value results from the method of analysis. For practical purposes there is not much to be gained in precisely evaluating these factors since they are likely to be masked by the inherent variability of the matrix properties. Further, there is no direct relationship between fibre pull out behaviour and that which occurs during a composite test (Kelly, 1980), hence, the applicability of $I_C$ to a composite test is uncertain.

2.4.2. Matrix Fracture Assuming Laws of Mixtures to Apply

Before progressing further it should be noted that the stress-strain behaviour for cement mortars is inelastic. This results from the development of stable cracks prior to catastrophic propagation of a critical flaw. Such cracks may be present even before load is applied, e.g., shrinkage cracks, interfacial cracks at inclusions, etc. In the ensuing discussion "first crack" is taken to be the crack which catastrophically propagates across the complete section.

It is further assumed that after first cracking of the composite, continued cracking will occur (failure by multiple fracture). Equation 2.2. may be simplified to evaluate the critical fibre volume fraction $V_f(crit)$, which needs to be incorporated to achieve multiple cracking.

$$V_f(crit) = \frac{E_c \epsilon_{mu}}{\sigma_{fu}} \quad \ldots \quad (2.7.)$$

The qualification must be made that the fibre-matrix stress transfer is sufficient to transfer the extra load back into the matrix; without it, single fracture would occur.
The Laws of Mixtures assume that the composite will crack when the strain in the matrix reaches the failure strain of the unreinforced matrix, and is given by

$$\varepsilon_{\text{mu}} = \frac{\sigma_c}{E_m (1 + V_f (\frac{E_f}{E_m} - 1))}$$

hence, the composite cracking stress, $\sigma_c$, may be calculated.

However, this is an oversimplification ignoring any fibre-matrix interaction during the fracture process. A more comprehensive analysis is one which accounts for the various energy dissipating mechanisms which operate during crack growth.

2.4.3.(a) Matrix Fracture Predicted using Energy Concepts.

Since the work of Griffith (1920) it has been recognised that structural design cannot be universally based upon the elastic limit or yield strength of the material. Instead, design may account for the flaws present in a body and the energy which is released for incremental extension of that flaw.

Griffith showed that for conditions of plane stress (i.e., a thin plate of small thickness in comparison to the length of flaw, $2c$) the failure stress of a material is given by

$$\sigma = \left( \frac{2E\gamma}{\pi c} \right)^{\frac{1}{2}}$$

where $\gamma$ = surface energy or surface tension for an ideal brittle material.
Many results have confirmed this relationship for ideal brittle, elastic materials which show minimal plastic deformation at the crack tip. This criterion may not be applicable to cement and concrete as it over-simplifies the geometry of the extending crack. During Scanning Electron Microscopy studies of crack growth in a sand-cement mortar Mindess and Diamond (1980) noted that,

(i) the crack is tortuous
(ii) discontinuous branch cracking occurs
(iii) discontinuities or 'jumps' in the main crack were apparent
(iv) there may be significant relative lateral displacements between crack faces.

Higgins and Bailey (1976a) noted similar features in an optical study of cement paste. They concluded that failure was by the coalescence of many microcracks. In wedge loaded specimens they noted that approximately 25% of the length of micro cracks did not join up perfectly and did not contribute to the fracture surface (Higgins and Bailey, 1976b). Extending these observations they suggested that for crack separations of approximately 1\mu m-2\mu m a tied crack exists such that attractive forces exist across the crack (Figure 2.2).

![Stress-Crack Separation for the Tied Crack Model](after Higgins, 1980)
A similar approach has also been adopted in the Fictitious Crack Model of Petersson (1981).

Hence, to be applicable to a material showing limited plastic deformation equation 2.8. must be modified thus,

\[
\sigma = \left( \frac{2E(\gamma + \gamma_p)}{\pi c} \right)^{\frac{1}{2}}
\]

\[\text{..... (2.9.)}\]

where, \(\gamma_p = \) work of plastic deformation.

In the energy approach the criterion for crack propagation is expressed in terms of the rate of energy release and absorption, \(dU/dA\), with respect to incremental crack extension. The symbol, \(G\), is assigned to \(dU/dA\) and is referred to as the "strain energy release rate", it may also be called the "crack driving (or extension) force".

In cases of stable crack propagation, \(G\) is absorbed by the various energy dissipating mechanisms until it reaches a critical value, \(G_c\), when instability occurs. It can be shown (e.g., Swamy, 1979) that

\[
G_c = 2(\gamma + \gamma_p)
\]

\[\text{..... (2.10.)}\]

Values for \((\gamma + \gamma_p)\) may be found from work of fracture tests in which, for example, notched beams are loaded in a controlled manner and is termed the work of fracture, \(\gamma_F\).

\[
\sigma = \left( \frac{2E_\gamma_F}{\pi c} \right)^{\frac{1}{2}}
\]

\[\text{..... (2.11.)}\]
Mindess, Laurence and Kesler (1977) and Hillemeier and Hilsdorf (1977) have stated that $G_c$ is an appropriate parameter to be considered for cement pastes. Therefore, further discussion of matrix fracture will assume the validity of this term within the limitations of the assumptions already made (2.3.).

2.4.3.(b) Fracture Mechanisms of Fibre Cements.

The toughness of a fibre cement may be derived from,

(i) work of fracture of the matrix,
(ii) work of fracture of the fibre,
(iii) energy required to debond the fibre-matrix interface,
(iv) strain energy released from the matrix,
(v) increase in strain energy of the fibres,
(vi) fibre pull out,
(vii) plastic shearing of fibres which do not lie normal to the crack face,
(viii) delamination,
(ix) multiple cracking,
(x) additional work due to increase in compliance of cracked composite.

(e.g., see Aveston, 1971; Harris, 1972; Hale and Kelly, 1972; Beaumont and Aleska, 1978).

In order to predict the failure criteria of a fibre cement it is necessary to examine in some detail the development of a crack through a brittle matrix.
The idealised composite shown in Figure 2.3. is one in which the fibres are discontinuous or exhibit a large degree of strength/length variation. In region B, fibre debonding and small irreversible displacements occur, in contrast to region A where fibre pull out may occur over relatively large distances. Region C occurs ahead of the crack tip in which debonding of a different nature may occur.

The debonding in region B was first proposed as an energy dissipator by Outwater and Carnes (1967) although the model is generally known by the name, Outwater and Murphy (1969). They showed that small fibre diameter, high bond strength and low fibre volume fraction would yield a brittle composite. However, the assumption of frictional bond does not require a debonding mechanism of this type to be considered.

Whilst Outwater/Murphy debonding may only occur after the crack tip has passed the fibre, Cook and Gordon (1964) proposed another model considering the stress distribution ahead of the crack tip. They showed that in an infinite, homogeneous and elastic body a tensile stress, $\sigma_x$, normal to the applied stress, $\sigma_y$, attains a maximum, a small distance ahead of the crack tip.
Cook and Gordon suggested that if a plane of weakness (of strength less than 0.2 times the cohesive strength of the material) was introduced ahead of the crack then the crack would be deflected into a non critical path or blunted.
Almond, Embury and Wright (1969) also describe the use of interfaces in mild steel laminates as a means to increase fracture toughness.

The Cook/Gordon interfaces cannot be equated directly with the fibre-matrix interface, since, in the latter material the bulk strength of the matrix also contributes to the transverse strength (Cooper and Kelly, 1969). Kelly (1970) has suggested that a ratio of longitudinal to transverse strength of at least 50 is required in a 50% $V_f$ carbon fibre-epoxy and a ratio of 13.2 for a copper-tungsten composite (Cooper and Kelly, 1967).
The magnitude of the tensile bond between polyalkenes and cement is not known but may be minimal (< 0.1 MPa), polyalkenes being commonly used as release agents. Further, in a composite which has been cured such that any bleeding water may be trapped under the hydrophobic fibres, it may be imagined that a crack might run into a severely weakened, porous interface, so impeding its progress.

Accommodating some of these ideas two models have been proposed which predict matrix failure strain. The first is encompassed in the ACK theory (Aveston et al., 1971; Hale and Kelly, 1972; Cooper and Sillwood, 1972; Kelly, 1976a) and the second was developed at Nottingham University (Morley and McColl, 1975; McColl and Morley, 1977a; McColl and Morley, 1977b; Korczynskyj, Harris and Morley, 1981).

2.4.4. ACK Model

The model considers two extreme cases, i.e., before matrix cracking has occurred and after complete, instantaneous propagation of the crack under conditions of constant load. The energy balance is considered in terms of work done by the applied load, release of strain energy from the matrix, matrix work of fracture, frictional work and increase of strain energy in the fibres. The ultimate enhanced matrix failure strain, $\varepsilon_{mu}^{\ast}$, is thus given by,

$$\varepsilon_{mu}^{\ast} = \left( \frac{12\tau \gamma_{m} E_{f} V_{f}^{2}}{E_{c} E_{m}^{2} r V_{m}} \right)^{1/3}$$

where, $\tau$ = interfacial frictional shear bond.
$\gamma_{m}$ = matrix work of fracture.
$r$ = fibre radius.
If \( \varepsilon_{\text{mu}} \) is less than \( \varepsilon_{\text{mu}} \), it is assumed that the composite will not crack until \( \varepsilon_{\text{mu}} \), where \( \varepsilon_{\text{mu}} \) is the failure strain of the matrix in the absence of fibres.

The derivation of this formula is given by Aveston et al. (1971) in which it can be seen that the initial equations were built up in terms of crack spacing, \( x' \). Equation 2.12. is derived by assuming,

\[
x' = \frac{\sigma_{\text{mu}} V_m r}{\tau V^2_f}
\]

Thus, using the initial equations, \( \varepsilon_{\text{mu}} \) may be expressed as

\[
\varepsilon_{\text{mu}} = \left\{ \frac{6 \gamma_m}{E_m x'(1 + \alpha)} \right\}^{\frac{1}{2}}
\]

where, \( \alpha = \frac{E_m V_m}{E_f V_f} \)

which Kelly (1976) has shown to be a form of the Griffith Equation.

Equation 2.14. implies that as \( x' \) is reduced (at constant \( \alpha \)) then \( \varepsilon_{\text{mu}} \) may increase.

Aveston et al. (1974) have published results which appear to confirm the ACK theory for steel and carbon fibre cements. Commenting in the same paper, Laws reports that GRC shows an increased \( \varepsilon_{\text{mu}} \) with increased fibre content. However, in Law's case possible
moisture loss during fabrication confuses the theoretical verification and further, she did not indicate what criteria were used for experimentally determining $\varepsilon_m$.

2.4.5. Nottingham Model

This model was originally developed for metallic composites and later extended to carbon fibre reinforced glass. In contrast to the ACK approach, the Nottingham model considers the incremental growth of a crack under conditions of fixed grip. It is assumed that the critical Griffith crack length remains the same as that for the unreinforced matrix. As before, the fibre-matrix stress transfer is assumed to be frictional; also the fibres are assumed to be circular in section throughout.

An elliptical zone of strain relaxation, $L_3$, of major axis 3 times the crack length, is assumed to exist in the fibre and matrix as shown in Figure 2.6. where $\varepsilon_\beta$ is the strain in the uncracked composite remote from the crack.

![Figure 2.6. Assumed Strain Distribution in Fibre and Matrix in Section A-A Perpendicular to the Crack Face](image)
Equation 2.15. allows the complete strain distribution along a section to be defined.

\[ \varepsilon_r = \left( \frac{\varepsilon \beta L_3}{Q (P + \frac{\varepsilon \beta}{L_3})^2 + \frac{L_3}{\varepsilon \beta}} \right)^{\frac{1}{2}} \]

\[ \varepsilon_{f\text{max}} = L_1 (P + Q + \frac{\varepsilon \beta}{L_3}) \]

\[ L_1 = \varepsilon_r (P + \frac{\varepsilon \beta}{L_3}) \]

\[ L_2 = L_3 \frac{\varepsilon_r}{\varepsilon \beta} \]

\[ L_3 = 3(a^2 - y^2)^{\frac{1}{2}} \]

where,

\[ P = \frac{2V_f \tau}{E_m V m r} \]

\[ Q = \frac{2\tau}{E_f r} \]

\[ y = \text{distance from the centre of the crack to the section considered.} \]
The system is analysed by dividing the length of the crack into a number of segments and using equation 2.15 the strain energy released, \( \delta W_{R_y} \), per segment of width, \( \delta y \), and unit thickness is given by,

\[
\delta W_{R_y} = \left[ \frac{E_c \varepsilon_\beta^2 L_3}{2} - \frac{E_c \varepsilon_r^2 (L_3^2 - L_2^2)}{6 L_3^2} \right. \\
- \frac{E_c \varepsilon_r^2 (L_2 - L_1)}{2} - \frac{V_m E_m \varepsilon_r^2 L_1}{6} \\
- \frac{V_f \tau \varepsilon_{f_{\text{max}}}^2 + \varepsilon_{f_{\text{max}}} \varepsilon_r + \varepsilon_r^2 L_1}{6} \right] \delta y \quad \ldots \ldots (2.16.)
\]

The energy absorbed by frictional work, per segment \( \delta W_{A_y} \), is given by,

\[
\delta W_{A_y} = V_f \tau (P + \frac{\varepsilon_\beta}{L_3} \frac{L_1^3}{r} + Q) \delta y \quad \ldots \ldots (2.17.)
\]

The total energy released and absorbed across the complete crack is obtained by numerical integration and the rate is obtained by differentiation for incremental crack growth. The fracture surfaces are able to absorb energy as in the ACK model and the work of fracture is again assumed to control matrix failure. Unstable
crack growth will occur when the rate of release of strain energy is greater than the combined rate of absorption.

2.4.6. Comparison Between ACK and Nottingham Models

In this section typical values of parameters (Table 2.2.) controlling the first crack strain will be inserted into both models and a comparison of the predicted enhanced matrix failure strain obtained (Figures 2.7. - 2.10.).

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\gamma_m$</td>
<td>4, 15 J/m$^2$</td>
</tr>
<tr>
<td>$E_f$</td>
<td>8, 30 GPa</td>
</tr>
<tr>
<td>$E_m$</td>
<td>30 GPa</td>
</tr>
<tr>
<td>$\varepsilon_{\text{mu}}$</td>
<td>$200 \times 10^{-6}$</td>
</tr>
<tr>
<td>$r$</td>
<td>1, 10, 50 (\mu)m</td>
</tr>
<tr>
<td>$\tau$</td>
<td>0.5, 1.0 MPa</td>
</tr>
</tbody>
</table>

Table 2.2. Typical Parameters Controlling First Crack Performance of Brittle Matrix Composites

It is apparent in Figures 2.7. - 2.10. that in all comparisons -

(a) The Nottingham Model yields the higher estimate; although, if higher (but impractical) fibre volume fractions were illustrated, the estimated $\varepsilon_{\text{mu}}$ would be shown to converge.

(b) The Nottingham Model shows a continuous enhancement of $\varepsilon_{\text{mu}}$ whereas that of ACK does not predict enhancement until a certain $V_f$ is present.
Figure 2.7. Predictions of Matrix Failure Strain for Composites Containing Fibres of Various Radii. ($\gamma_m = 4 \text{ J/m}^2$, $\tau = 0.5 \text{ MPa}$, $E_f = 8 \text{ GPa}$)
Figure 2.8. Prediction of Matrix Failure Strain for Composite Containing Film of 30 GPa Elastic Modulus
\( \gamma_m = 4 \text{ J/m}^2, \tau = 0.5 \text{ MPa}, r = 10 \mu\text{m} \)
Figure 2.9. Prediction of Matrix Failure Strain for composites with a Matrix Work of Fracture $= 15 \, \text{J/m}^2$ ($\tau = 0.5 \, \text{MPa}, E_f = 8 \, \text{GPa}, r = 10\mu\text{m}$)
Figure 2.10. Prediction of Matrix Failure Strain for Composites with a fibre-Matrix Bond Strength of 1 MPa ($\gamma_m = 4 \text{ J/m}^2$, $E_f = 8 \text{ GPa}$, $r = 10\mu\text{m}$).
From Figures 2.7 - 2.10, it can be seen that $\varepsilon_{\text{mu}}$ is enhanced by increasing $V_f$ and that enhancement is greater for finer fibres (Figure 2.7). The critical $V_f$ for enhancement (ACK) increases with fibre radius such that no enhancement is predicted at 15% $V_f$ for a fibre radius of 50\textmu m.

Figure 2.8 shows that if fibres of a higher modulus are incorporated (30 GPa in contrast to 8 GPa in Figure 2.7) a greater enhancement of $\varepsilon_{\text{mu}}$ is predicted. This behaviour has already been reported for polyalkene films in cement matrices by Hughes and Hannant (1982). Although they did not report values of $\varepsilon_{\text{mu}}$, their results showed increases in average matrix failure stress for 10% $V_f$ composites with films of moduli of 2.5 to 30 GPa.

Values of work of fracture for cement pastes quoted in the literature vary up to 15 J/m$^2$ (Kelly, 1976). Using this value, $E_f = 8$ GPa, $\tau = 0.5$ MPa and $r = 10\textmu$m, it can be seen (Figure 2.9) that $\varepsilon_{\text{mu}}$ has been increased in comparison to $\gamma_m = 4$ J/m$^2$ (Figure 2.7).

The bond between polyalkenes and cement is a complex mechanism which is not fully understood (Laws, 1982). However, if systems can be devised with increased bond then $\varepsilon_{\text{mu}}$ may be expected to be further enhanced. Figure 2.10 shows that if $\tau$ is increased from 0.5 MPa to 1 MPa, an increased $\varepsilon_{\text{mu}}$ is predicted. It is interesting to note that both analyses show that the same increase would be achieved by alternatively reducing the fibre radius from 10 \textmu m to 5 \textmu m.

Further discussion of these models will be found in Chapter 6 when the detailed experimental results are presented. Then, the models may be discussed with particular reference to the polyalkene cements being examined.
2.4.7. Problems of Comparing Experimental Values for "First Crack" with Theoretical Predictions.

From the previous sections it is apparent that predictions of $\varepsilon_{mu}$ will depend in part, upon the loading configuration, fixed grip and constant load. However, most testing is performed on Instron type machines utilizing constant rate of crosshead movement (This does not imply a constant rate of strain increase, (Singh, Walton and Stucke, 1978)). Several authors have reported that significant inelastic behaviour is encountered at a lower stress/strain than at which the first crack catastrophically propagates (Laws, Lawrence and Nurse, 1973; Walton and Majundar, 1977; Hughes and Hannant, 1982) such that the Nottingham model failure mode may be more appropriate. It has been suggested (Chan and Patterson, 1972) that the maximum crack length is restricted to the fibre spacing. In such a case, any surface scratch of a greater length than the fibre spacing would result in a severe reduction in $\varepsilon_{mu}$ and this is not observed.

Great problems are presented in the interpretation of the stress-strain data obtained from real composites. Both models assume that a uniform stress/strain distribution occurs across the specimen section at a point remote from the crack; yet this is the exception rather than the rule. Most specimens are bowed and merely clamping them in the machine induces significant strain (up to $500 \times 10^{-6}$) without a load being recorded from the load cell. Unless individual strains are monitored on opposite faces this affect may be overlooked since clamping strains are usually of approximately equal but opposite magnitude. In such a system only a proportion of the specimen volume will be stressed in tension at the highest level and any crack which formed within this region may run into a lower stressed zone to be arrested. It is also known that higher strengths are recorded for cementitious materials from small specimens. Therefore, it may be that an artificially high value of strain at crack initiation and propagation would be achieved.
Interpretation of the initial tensile stress-strain curve to identify cracking stress or strain is particularly difficult. Many researchers produce results for stress at Limit of Proportionality (LOP) and/or Bend Over Point (BOP) without adequate attention to detail. In an ideal elastic material the LOP and BOP would coincide and represent the first deviation from linear elasticity. Brittle matrix composites are distinctly non-linear in regions where micro-cracking has been initiated but not completely propagated. The determination of the LOP is highly prone to both operator variability and sensitivity of the measuring and recording equipment and also the micro mechanical features associated with it are ill defined. The BOP may appear a more attractive and more easily defined point, yet it is also dependent upon experimental recording equipment; there is also no consistent standard for its determination to allow accurate comparison from one laboratory to another.

To take account of these criticisms it is necessary to determine $\varepsilon_{\text{mu}}$ at a readily definable point on the stress-strain curve at a stage of fracture when bending effects have been minimised. This may occur after significant cracking has already taken place but before multiple cracking has been completed. Aveston et al (1974) monitored the stress-strain curve and acoustic emission (AE) and assumed that maximum AE coincided with maximum crack propagation. The average matrix failure strain, $\overline{\varepsilon}_{\text{mu}}$, is defined as $\overline{\sigma_c} / E_c$ where $\overline{\sigma_c}$ is the composite stress at the point of maximum AE. The interpretation of AE results is complicated by attenuation of the signal from a distant source as it travels through the composite and across numerous cracks. Nevertheless, this approach is an improvement on previous techniques.

For the very limited curves shown in the paper it appears that maximum AE occurs at a strain approximately in the middle of the multiple cracking region. For Polyalkene reinforced cement $\varepsilon_{\text{mc}} \gg \varepsilon_{\text{mu}}$ (see Fig 2.1) hence, it will be assumed that the average matrix cracking strain, $\varepsilon_{\text{mu}}$ occurs at a strain $\varepsilon_{\text{mc}}/2$. 
It is apparent that $\varepsilon_{\text{mu}}$ and $\bar{\varepsilon}_{\text{mu}}$ are not strictly the same parameter. However, since it is impossible to directly measure $\varepsilon_{\text{mu}}$ from the typical flat strip composite specimen, $\bar{\varepsilon}_{\text{mu}}$ provides a reasonable approximation of the same matrix failure mechanisms.

2.5. Tensile Behaviour of Brittle Matrix Composites - Multiple Cracking Region

2.5.1. Theoretical Composite

This topic was briefly introduced in section 2.4. If the fibre volume fraction is such that it is able to withstand the additional load thrown upon it at the first crack and the fibre-matrix interface is capable of transferring this load within the length of the specimen, then subsequent cracking of the matrix will occur. Crack spacing is governed by equation 2.13.

\[
\varepsilon_{\text{mu}} \approx \frac{E_m \bar{\varepsilon}_{\text{mu}} V_m r}{\tau V_f 2}
\]

It has already been stated that the spacing, $\bar{x}$, will be in the range $x' < x < 2x'$. Aveston et. al., (1974) have suggested that $\bar{x}$ is equal to $1.364 x'$. Kimber and Keer (1982) have suggested that $\bar{x}$ is equal to $1.337 x'$. However, the difference is minimal and the former figure will be assumed to apply. The additional strain in a composite at the completion of multiple cracking is given as $0.659 \varepsilon_{\text{mu}} (\bar{x} = 1.364 x')$. Hence, $\varepsilon_{\text{mc}}$, is given by,

\[
\varepsilon_{\text{mc}} = (1 + 0.659 \alpha) \varepsilon_{\text{mu}} \quad \cdots \quad (2.18)
\]

Equation 2.18. is derived assuming linear stress transfer between fibre and matrix. If the transfer is more complex this equation will not apply.
2.5.2 Practical Composite

The theoretical composite assumes a single valued failure stress for the matrix, however, cement matrices show a variable strength/length relationship. Provided that energy is not imparted to the specimen upon propagation of the first crack, subsequent cracking is likely to occur at increasing stress levels. This results in a rising multiple cracking region and the repercussions on attempts to relate crack spacing and bond strength are profound.

Associated with an increasing matrix strength, \( \sigma_{mu} \), is the related minimum crack spacing, \( x' \). Therefore, although the measured crack spacing, \( \bar{x} \), is related to \( x' \), \( x' \) itself is increasing such that it is impossible to relate the final crack spacing to a particular value of matrix strength. The adopted solution is a compromise in which the average matrix strength, \( \bar{\sigma}_{mu} \), is determined at \( \varepsilon_{mc}/2 \) (c.f. \( \bar{\varepsilon}_{mu} \)) and the calculated bond strength can only be termed notional.

If the rate of fibre-matrix stress transfer increases with relative displacement then cracking may occur on the "post-crack" curve, further complicating the calculation of bond strength. Further discussion of this point may be found in Chapter 7.

2.6 Tensile Behaviour of Brittle Matrix Composites - Post Crack Curve.

Upon completion of multiple cracking the increase in load is accompanied by extension of the fibres alone since the matrix is unable to carry any extra load. The composite modulus is given by \( E_f V_f \) and fracture occurs at a composite stress, \( \sigma_{fu} V_f \).

The length of the multiple cracking region and the post-crack slope are influenced by the elastic modulus of the fibre. The basic theory assumes linear elastic fibres which is clearly inapplicable for polyalkenes. Once cracking has commenced \( E_f \) will decrease (at a variable rate) with extension of the fibre. It is unlikely that a tangent modulus
of the fibre at a specified strain will be an appropriate parameter for determining \( \epsilon_{mc} \), although a secant modulus may be more appropriate. Another problem is that the fibre strain at a crack will be very much higher than the average composite strain. Consequently, choosing an appropriate single value of fibre modulus may well be arbitrary.

The failure strain of the composite, \( \epsilon_{cu} \), will always be less than that of the fibre alone and is related to \( \alpha \epsilon'_{mu} \) such that

\[
\epsilon_{cu} = \epsilon_{fu} - 0.341 \alpha \epsilon'_{mu}
\]  

(2.19)

where \( \bar{x} = 1.364x' \).

2.7 Theoretical Tensile Stress-Strain Curve

The preceding discussion allows the theoretical tensile stress-strain curve to be plotted using the assumptions of section 2.3.

Figure 2.11. Theoretical Tensile Stress-Strain Curve (\( \bar{x} = 1.364x' \))
It will be noted that monotonic loading only has been considered. For a comprehensive review of the application of cyclic loading to polyalkene reinforced cements see Keer (1983).

2.8 Stress Transfer Between Polyalkene Fibre and Cement

2.8.1 Stress Transfer and Multiple Cracking in Polyalkene Reinforced Cements.

In sections 2.3 - 2.6 it was conveniently assumed that a frictional bond existed between polyalkene fibres and cement matrices such that multiple cracking of the matrix would occur.

Theoretical doubts about frictional bond between polypropylene fibres and cement were first raised by Kelly (1976b). In a later paper, Kelly and Zweben (1976) concluded that for aligned, uniform polypropylene fibres, debonding would be immediately followed by pull out due to the combination of low elastic modulus and high Poisson's ratio such that multiple cracking could not occur. Using different analyses both Pinchin (1976) and Baggott and Gandhi (1981) reached the same conclusion. However, it is known that crack spacings as low as 0.6 mm may be achieved using fibrillated polyethylene films (Hughes and Hannant 1982). Further, Baggott and Gandhi achieved multiple cracking in a carefully prepared aligned polypropylene (monofilament) composite. Krenchel has claimed to have produced composite using Perlon and polypropylene fibres, in which no discrete cracks are formed; rather, pseudo-ductility is achieved via a general loosening of the matrix structure (Krenchel, 1976; Krenchel and Jensen, 1980; International Patent Publication, No. WO 80/00960).

Walton and Majumdar (1975) reported bond strengths in the order of MPa from pull out tests on 150 μm diameter polypropylene monofilaments. It is interesting to note that in a significant number of cases an increased load was carried after slip had occurred although neither Walton and Majumdar nor Laws (1982) were able to offer any explanation. A similar
behaviour may be inferred from the results presented by Hughes and Fattuhi (1975). This phenomenon is not generally encountered in a pull out of inorganic fibres from cement and gypsum matrices (e.g. Majumdar, 1974; Pinchin and Tabor, 1978b).

Possible explanations such as matrix shrinkage, fibre asperities, lateral displacement of crack faces and fibres being bent across the crack creating pulley forces have been suggested to account for the development of multiple cracking. The pulley force is proportional to \((1-\sin \theta)\) where \(\theta\) is the angle between the fibre and the crack surface; hence for the aligned composite this effect may be expected to be minimal. The experiments of both Hughes and Hannant and of Baggott and Gandhi were conducted on wet, water cured samples such that shrinkage may be largely discounted. The asperities illustrated by Baggott and Gandhi were apparently sparsely distributed such that their contribution may be anticipated to be small, especially since their maximum height was 10\(\mu\)m compared to an approximate fibre poisson contraction of 10 \(\mu\)m.

It would appear, therefore, that these explanations are inadequate to describe the phenomenon of multiple cracking and that attention should be directed to the interfacial zone. It is surprising to note that in his recent review of bond, Bartos (1981) specifically did not include this feature. The ensuing discussion is highly selective. The majority of the literature concerns itself with magnitude of bond, elastic and frictional, rather than with the micromechanical mechanisms of fibre-matrix interaction. It is the current author's view that until the mechanism of stress transfer is reasonably described, the calculation of a 'bond strength' is not helpful in understanding complete composite performance.

2.8.2 Types of Bond.

2.8.2 (a) Tensile Bond.

The tensile bond between fibre and matrix acts perpendicularly to the interface. It is particularly relevant to the strength of a composite in which the fibres are not perfectly aligned. In the extreme, (the transverse strength of an aligned fibre composite) a weak tensile bond may be considered as creating a void within the matrix, thus reducing the composite cracking stress, \(\sigma_{co}\)
\[
\sigma_{CO} = \sigma_m (1 - \sqrt{\frac{4V_f}{H}})
\]  
...... (2.20)

(after Cooper & Kelly, 1969)

The role of the tensile bond as a crack arrestor has been previously discussed. In the case of relative lateral contraction of a stressed fibre, the tensile bond operates in conjunction with the shear bond.

2.8.2 (b) Shear Bond.

The shear bond is that which acts parallel to the interface and controls the rate of stress transfer between fibre and matrix. If continuity is maintained across the interface then an elastic shear bond is said to operate. If the interface debonds, transfer is via static and dynamic frictional shear. The static frictional bond governs the shear stress which must be exceeded for relative displacement to occur and the dynamic frictional bond the stress which must be exceeded for slippage to be maintained.

Failure of the elastic shear bond (in the uncracked composite) may lead to premature fracture of the matrix if the subsequent static frictional bond is of a lower magnitude, since the fibres would be utilised less efficiently. The dynamic frictional bond has been shown to effect \( \epsilon_{mu} \) (2.4) and the average crack spacing in those composites which develop multiple cracking prior to failure by fibre fracture or pull out. In the latter, the dynamic bond also dictates the ultimate composite strength and toughness, although in a contradictory manner. As bond strength is increased so is the ultimate strength, but at the expense of toughness. Compromises have been sought to maximise both parameters for example, by the incorporation of fibres exhibiting intermittent bonding (Atkins, 1975).
2.8.3 Microstructure of the Interfacial Zone.

2.8.3 (a) Matrix.

The microstructure of hydrating cement is highly complex and heterogeneous in nature and is dependent upon mix proportions, curing conditions and age, especially when fly ash is a component (Montgomery, Hughes and Williams, 1981). The products of hydration in the bulk paste differ from those at the interface with a substrate. Barnes, Diamond and Dolch (1978) observed the interfacial zone with a glass slide as the substrate and the initial microstructure may be summarised as shown in Figure 2.12.

Supersaturation of calcium hydroxide in solution \([\text{Ca(OH)}_2]\) results in the deposition of a layer of \(\text{Ca(OH)}_2\) showing no indication of individual crystal outlines or boundaries. Rod like calcium silicate hydrates, type I, \([\text{CSH I}]\) in Diamond's classification (1976), is then deposited upon the \(\text{Ca(OH)}_2\). Such a structure has been termed a duplex film and is approximately \(1\ \mu\text{m}\) thick. Similar films have been reported on glass fibres (Cohen and Diamond, 1975) and on fly ash particles (Diamond, Ravina and Lovell, 1980). Adhering to the CSH I are large impure \(\text{Ca(OH)}_2\) crystals with their c-axes parallel to the interface and hydrating cement grains, some of which are hollow shell 'Hadley grains'.

---

Figure 2.12. Initial Microstructure of Interfacial Zone

- Large \(\text{Ca(OH)}_2\) crystals
- \(\text{CSH I}\)
- \(\text{Ca(OH)}_2\)
- \(\approx 1\ \mu\text{m}\)
- SUBSTRATE
After a few days hydration the CSH undergoes a morphological change to a reticular structure (CSH II). Also, at this stage, pure Ca(OH)$_2$ of a platy structure is laid down to form a continuity between the interfacial zone and the bulk paste.

Similar structures have been reported elsewhere. Calcium enrichment of the interfacial zone of steel fibre cements has been reported by Pinchin and Tabor (1978a) and Khalaf and Page (1979) although the degree of continuity of the duplex film is debated. Both latter papers report the interface to be voided at 3 days, yet substantially continuous by 7 days with the voids having been filled with Ca(OH)$_2$. The morphology of this Ca(OH)$_2$ has not been described but is likely to be that of the platy structure. Khalaf and Page and also Majumdar (1974) noted that CSH of varying morphology was itself nucleated directly upon the substrate.

Pinchin and Tabor (1978a) observed an increase in Vickers Hardness and a decrease in porosity (25% to 16%) as a function of distance from the interface. With the increased porosity at the interface is an accompanying decrease in strength (115 MPa to 45 MPa). Their techniques only allowed measurements to within approximately 200μm of the interface and they anticipated an increase in hardness at the interface due to the deposition of Ca(OH)$_2$. They observed that this relationship could be eliminated by the use of a plasticiser to obtain compaction of the paste rather than vibration. The micro-structure will also depend upon the orientation of the fibre during casting, with bleed water likely to be trapped under a horizontal fibre. Gray and Johnston (1978) have suggested that the larger coefficient of variation obtained with vertically cast fibres may be due to a larger variability of the matrix at the entry-point of the fibre into the matrix.

The locus of failure at the interface is known to change with time. Barnes et al (1978) induced a shrinkage failure and Khalaf and Page a tensile failure and noted that at early ages failure occurred at the interface and in the bulk paste. By 7 days the large majority of failure was at the interface. However, Pinchin and Tabor (1978b) reported that at 4 weeks large areas of steel fibre were coated with adhering paste immediately after debonding in a pull out test. The different failure modes are likely to effect the locus of failure and the appropriate microstructure should be considered when interpreting test results.
From this brief review it can be seen that the interfacial zone is heterogeneous and complex in nature, changing morphology with time. It will be noted that the substrates investigation were relatively hydrophillic. The morphology of the interface in the presence of a hydrophobic substrate, such as polypropylene, is currently unknown.

2.8.3 (b) Fibre

The structure of the fibre may be considered in 5 categories:-

(i) Smooth, uniform cross section.
(ii) mechanically deformed.
(iii) surface roughened.
(iv) surface chemically treated.
(v) shaped fibres.

Of particular interest to the study of polyalkene-cement bonding are single fibre pull out results from smooth, surface roughened and shaped steel fibres, although the latter have only really been treated theoretically. This topic formed the basis of the thesis work by Pinchin (1977). He has reported results showing the effect of surface roughness on both static and dynamic bond (Pinchin and Tabor, 1978b) - details of the fibre preparation and roughness profile may be found in Pinchin and Tabor (1978a). Whilst an increase in surface roughness resulted in a significant increase in the debonding load (load at first slip), no unique relationship was exhibited for the dynamic frictional load subsequent to debonding. In some cases the smooth fibres showed the highest dynamic load and in others the reverse was observed. The important factor in Pinchin's work is the examination of the interface and the subsequent experimental work.

Microscopic examination showed that immediately after debonding the interface exhibited adhering paste over a large area combined with voided regions. After further pull through the smooth fibres appeared to be striated. However, this turned out only to be within a thin coating of cement and the fibre beneath remained undamaged. In the case of the rough fibre the transfer of material to the fibre, upon displacement, was more pronounced. The shear plane was not across the tips of the asperities as had been previously suggested (Plowman, 1963) but followed a more tortuous path.
The increased debonding load was explained by increased mechanical keying between the asperities and matrix; when this was overcome, a dynamic frictional stress transfer resulted. The decrease in load within the first millimetre of relative displacement was the result of a reduction in interfacial contact pressure due to compaction of the matrix at the interface, rather than any changes in coefficient of friction (Pinchin and Tabor, 1978b).

In a subsequent paper (Pinchin and Tabor, 1978c) pull out tests are described in which the interfacial pressure was increased and the effect on pull out load observed. A very simple analysis was used to relate fibre stress, \( \sigma_f \), to the interfacial pressure, \( P \), in a shrink fit configuration.

\[
\tau = \mu P 
\]  

\[ \tau \]  

\[ (2.21) \]

where, \( P = \frac{\varepsilon_0}{1 + \nu_m \left( \frac{1 - \nu_f}{E_m} \right) + \left( \frac{1 - \nu_f}{E_f} \right)} \) (after Timoshenko, 1976)

and \( \mu \) is the coefficient of friction.

This expression is valid when the radius of the enclosing matrix cylinder is much larger than the fibre radius and the fibre remains unloaded.

\( \varepsilon_0 \) is defined as the strain in the shrink fit configuration and may be due to either matrix strinking or applied confining pressure or a combination of both.

\( \varepsilon_0 \) may be expressed at \( \delta/r \) where \( \delta \) is the fibre-matrix misfit, that is the difference between the fibre radius and the radius of the hole in the absence of the fibre. When the fibre is loaded Poisson contraction will reduce the misfit such that,
\[ \varepsilon'^*_0 = \frac{\delta - \frac{\nu_f \sigma_f}{E_f}}{r} \quad \text{...... (2.22.)} \]

where, \( \varepsilon'^*_0 \) is a potential misfit strain.

This is the case at the point of entry of the fibre into the matrix; the general case for any point on the fibre has been given by Hale (1975).

\[
\frac{\delta}{r} = P \left\{ \frac{(1 - \nu_f)}{E_f} - \frac{(1 - \nu_m)}{E_m} + \frac{2}{E_m V_m} \right\} + \frac{\nu_f \sigma_f}{E_f} - \frac{\nu_m \sigma_m}{E_m} \quad \text{...... (2.23.)}
\]

It can be seen from equation 2.23. that, in the composite, the greater the distance from the crack face, the larger will be the value of \( P \) for a given value of \( \delta/r \) and hence the larger will be the local fibre-matrix stress transfer.

Using equation 2.22. Pinchin and Tabor developed an equation for the fibre stress at debond, \( \sigma_f \), thus,

\[
\sigma_f = k\delta \quad \text{...... (2.24.)}
\]

where \( k = \frac{E_f}{r \nu_f} \left[ 1 - \exp \left( \frac{-2\nu_f \mu}{E_f} \frac{x}{1 + \nu_m} \right) \right] \) \left\{ \frac{1 - \nu_f}{E_f} \right\} \left\{ \frac{1}{E_m} + \frac{1 - \nu_f}{E_f} \right\}

and \( x \) is the embedment length.
Their experimental results showed that for a smooth fibre the debonding load increased rapidly with confining pressure, in fact, greater than predicted by equation 2.24. The dynamic load reduced with pull out indicating a drop in misfit (or changing nature of the interface with pull out). In the case of rough fibres the rate of increase in debonding load with confining pressure was much lower than with the smooth fibre and the dynamic load was very low, corresponding to misfits much lower than the predicted values. It was proposed that the asperities cause a more rapid compaction of the matrix interfacial material. Bowden (1970) and Hadjis and Piggott (1977) have also reported increased stress transfer with confining pressure from pull out tests from resin matrices.

If it were possible to introduce a steel fibre of radius 1% greater than the matrix hole, the interfacial pressure would be of the order 250 MPa. This value may be in excess of the local matrix triaxial strength and compaction of the interface would be expected. This approach may explain the poor performance of steel fibre concretes containing such surface modified fibres as opposed to those which give a positive mechanical anchorage.

It is known that the shrinkage of resin matrices onto fibres increases the interfacial contact pressure and hence bond strength (Bowden, 1970). However, the same is not true for cement matrices. de Vekey and Majumdar (1968), Tattersall and Urbanowicz (1974) and Pinchin and Tabor (1978b) report results showing lower bond strengths for curing regimes where shrinkage is a factor to be considered; thus, cement matrices do not behave elastically. It is well known that shrinkage of cement is accompanied by the formation of microcracks. It is likely that these cracks disrupt the fibre-matrix interface resulting in inelastic behaviour such that achieving good bond by matrix shrinkage is not reliable.

Throughout the literature there are many references to pull out stress increasing less than linearly with increases in embedment length (e.g., Takaku and Arridge, 1973; Aveston, Mercer and Sillwood, 1975; Bowling and Groves, 1979). Equation 2.22. shows simply that as pull out stress increases (increase in embedment length) $\varepsilon_0$ decreases with a subsequent decrease in interfacial pressure and
hence bond. It is surprising that there has been apparently no comment in the literature as to the relationship of this phenomenon to the 'bond strength' derived from multiple cracking experiments.

As $V_f$ is increased the stress in each fibre across a crack is rapidly reduced even allowing for enhanced $\varepsilon_{\text{mu}}$. Thus, in light of the pull out results, it may be expected that the calculated 'bond strength' from multiple cracking experiments would show an increase with $V_f$. It is common for a plot of $\bar{x}$ against $(1 - V_f)/V_f$ to be illustrated and a 'bond strength' derived (according to ACK theory this should be a linear relationship - hence its popularity). However, the term $(1 - V_f)/V_f$ is relatively insensitive to changes in $V_f$ above approximately 6%, and so it is likely that any changes in 'bond strength' with $V_f$ would be masked. If it is required to calculate 'bond strength' from such a test, sufficient specimens must be tested at each $V_f$ in order that any $V_f$ dependency is illustrated.

2.8.4. Relevance of Fibre-Matrix Misfit to Polyalkene Reinforced Cement

Three important results from Pinchin's work may be summarized thus,

(i) stress transfer is increased with interfacial contact pressure.
(ii) interfacial contact pressure is related to the misfit between fibre and matrix.
(iii) interfacial contact pressure is reduced by compaction of the matrix during pull out, reducing misfit.

How these findings may be related to polyalkene cements will be briefly stated here but will be developed in detail in Chapter 7.
It has been stated that fibre-matrix misfit may arise from matrix shrinkage or confining pressure. Yet, multiple cracking of polyalkene cements does not appear to be dependent upon these parameters. A third source of misfit will occur as a fibre is drawn through the matrix. Previous workers have proposed surface asperities as a source of fibre-matrix interaction, but without adequate experimental justification. If the fibre is viewed at the microscopic level and is shown to exhibit a non-uniform cross section profile, then, as it is drawn through the matrix it would become deformed as certain regions were squeezed into smaller channels. Thus, along the fibre length, a varying interfacial contact pressure would exist (small cross sections of fibre drawn into larger matrix channels would be completely debonded). It was previously shown that for 1\% misfit of a steel fibre, a contact pressure of approximately 250 MPa may lead to matrix compaction. For a typical polyalkene the pressure, for similar misfit, is calculated to be 55 MPa and the accompanying compaction is likely to be negligible for the matrix considered in this thesis.

A hypothetical steel fibre of constantly changing cross section was proposed by Hale (1975) as a means of increasing bond with pull out, in order to obtain large composite failure strains. It was based upon the assumptions that matrix splitting would not occur and that there would be elastic behaviour at the interface. However, no such fibre has been produced and the approach not subsequently considered seriously.

Further details of the fibre used by Krenchel and Jensen (1980) have been published (UK Patent Specification, 1 605 004). The fibre is said to vary in cross section along its length as a function of the fibre production techniques. Consideration of this fibre will be reserved until these techniques have been described in Chapter 3, since its performance may be determined by the microstructure of the fibre.

In order to provide a basis for discussion of the relation between fibre structure and composite properties the next chapter outlines the production, structure and properties of fibrillated polyalkenes.
"Today we made polypropylene"

So wrote Giulio Natta (Director of the Institute of Industrial Chemistry, Milan Polytechnic) in his private notebook on 11 March 1954. Independently and contemporaneously, Karl Ziegler (Director of the Max Planck Institute, Mulheim) also prepared polyproplene. Both scientists used the catalysts identified by Ziegler in 1953 in his pioneering discovery of linear polyethylene. Thus, within 12 months, two new types of polyalkene of major interest for cement composite production were discovered. The story of these discoveries and their exploitation has recently been published (McMillan, 1979) and provides a sobering insight into the development of an innovation.

This chapter presents a brief synopsis of polymer structure and properties, long term stability and of possible industrial production techniques for fibrillated film in a form suitable for reinforcing cement matrices. For those requiring a greater depth of understanding, attention is drawn to relevant publications which may be used as source literature. Finally, a simplified model is proposed which describes the mode of action of fibrillated film in reinforcing cement.

3.1 Polymer Structure and Properties

3.1.1 Molecular structure

The action of Ziegler's catalysts was to produce the polymerisation of linear, crystalline polyethylene which may be shown thus:

\[
\begin{array}{c}
\text{H} \\
\text{H} \\
\text{C - C -} \\
\text{H} \\
\text{H}
\end{array}
\]

\(n\)
where \( n \) is measured in thousands. Prior to 1953 the polymerisation reaction required high pressures and produced a branched, low density polyethylene of low crystallinity and strength.

Natta's claim to have discovered polypropylene is legitimised by his ability to verify the stereo-regular nature of the polymer in the isotactic form, i.e. all the methyl groups are located on one side of the carbon-carbon bond thus:-

\[
\begin{align*}
\text{H} & \quad \text{CH}_3 \\
\text{H} & \quad \text{C} - \text{C} - \\
\text{H} & \quad \text{H} \\
\end{align*}
\]

It is apparent that the polypropylene molecule is not as symmetrical as that of polyethylene due to the alternating presence of hydrogen and methyl groups. Hence, to alleviate the steric interaction, successive rotation of the carbon-carbon bond forms a three fold helical spiral corresponding to the minimum steric strain.

3.1.2 Morphology of Drawn Films and Fibres.

It is not appropriate to present an exhaustive review of the physical structure of polymer fibres and its relation to their mechanical properties. Hence, the following is necessarily selective but provides a starting point for those who require more detailed description.

The fundamental crystalline morphology of polyalkenes is that of chain folded lamellae (Keller, 1962) as shown in Fig 3.1
Fig 3.1 Sketch Showing Folded Chains Within Lamellar Crystals.

The molecules are aligned perpendicularly to the lamellar surface with a folded length of approximately 100Å, the major crystal dimensions being 3 orders of magnitude greater. In a polyethylene crystal there are less than 100 \( \text{C}_2\text{H}_4 \) units between each fold and maybe \( 10^8 \) molecules in a single crystal.

Within the undrawn polymer the lamellae are usually aggregated into spherulites in densely packed parallel stacks (Williams, 1973), themselves randomly oriented as a consequence of the random orientation of the large numbers of primary nuclei produced by quenching (Peterlin, 1977). The lamellae are separated by amorphous regions comprising chain folds, chain ends and importantly, tie molecules which constitute the fundamental source of elastic modulus and tensile strength.

It is known that drawing a polymer into fibrous form increases both elastic modulus and tensile strength. Morphologically, drawing is accompanied by a transformation from a lamellar to microfibrillar structure. The concomitant plastic deformation of the lamellae comprises rotation and sliding of individual lamellae and chain tilting and shearing within lamellae (Peterlin, 1977). The microfibrils themselves are
aggregated to form a fibrillar structure which is visible by electron microscopic examination (Capaccio and Ward, 1977). Hence, within the bulk polymer a preferential molecular orientation is noted; however, this is not to say that the molecules are also fully extended and capable of yielding mechanical properties approaching theoretical values.

Taylor & Clark (1978) have proposed morphologies of natural drawn fibres (draw ratio approximately 5) and of superdrawn fibres (draw ratio >5). This is necessary since the increases in mechanical properties above draw ratios of approximately 5 cannot be accounted for by simple molecular re-orientation (Williams, 1973). It is proposed that the natural drawn fibre comprises alternating crystalline and amorphous zones, the lamellae having been broken into chain folded blocks of approximate width, 20 nm, i.e. the width of the previously defined microfibril (see Fig 3.2(a)).

![Diagram](attachment:image.png)

**Fig 3.2 Proposed Morphology of (a) Natural Drawn Fibre and (b) Superdrawn Fibre.**
It can be seen that within the superdrawn fibre the amorphous regions have become dispersed within a more crystalline matrix. Thus, a path has been created for the direct transfer of stress within the fibril by means of the covalent bonds of the increased fraction of tie molecules.

It is apparent from this structure that the drawn polymer is highly anisotropic. The transverse tensile strength is much lower than the longitudinal strength and it is this property which accounts for the ease of fibrillation. Similarly, the longitudinal modulus is higher than the transverse modulus (Owen and Ward, 1973).

From this brief review it may appear that the best mechanical properties are simply obtained by drawing the polymer. However, attaining optimum properties also requires consideration of molecular weight, extrusion, quenching and draw temperatures, annealing etc. Such a complex topic is beyond the scope of this thesis and attention is drawn to the detailed work of Sheehan and Cole (1964).

3.1.3 Mechanical Properties of Drawn Films and Fibres.

Research to optimise mechanical properties has concentrated on polyethylene with its higher theoretical modulus of 220-380 GPa compared with 35-42 GPa of polypropylene.

Typically, moduli of 22 GPa (Taylor and Clark, 1978) and 19 GPa (Cansfield, Capaccio & Ward, 1976) have been obtained for polypropylene, the former exhibiting a tensile strength of 930 MPa (a more typical strength being 300-400MPa). If these values are assumed to approach the maximum obtainable under laboratory conditions, then the films which have been commercially drawn and sent to this laboratory may be placed in perspective. During the term of NETCEM development (from 1976 onwards) the film modulus, of commercially produced film, has increased from 4GPa to approximately 15GPa, and hence it is unlikely that any further dramatic increases may be expected from commercial film.
Inevitably, the modulus obtained from polyethylene has been greater, 67GPa (Capiati and Porter, 1975), approximately 80GPa (Barham and Keller, 1976), 70GPa (Ward, 1980) and 46 GPa for a hydrostatically extruded polymer (Gibson, Ward, Cole and Parsons, 1974). The techniques developed by Ward at Leeds University have been patented through the NRDC (now the National Engineering Research and Development Corporation) and the technology has been licensed by Metal Box Ltd. During the transformation from a laboratory technique to the commercial scale a loss of property has resulted. Nevertheless, 25mm wide tape has been produced (with a modulus of 30GPa) and subsequently fibrillated at Brunel University. The material, classified E3H, has been used in the work reported herein.

3.1.4 Mechanical Properties of Fibres Produced by Crystallisation from the Melt or Solution.

It is important to note that, another major technique exists for producing ultra-high modulus polyethylene (262GPa measured at 77°k (Barham and Keller, 1979)). However, the techniques, involving the growth of crystals from an initiator within a solution or melt of the polymer, are currently restricted to the laboratory and are of academic interest. Further, the material produced is generally monofilament which is inconvenient for incorporation within a cement matrix. Should it be possible to produce a spun bonded web of such a material, it would be of considerable interest.

Attention is drawn to a highly readable account of the history of seed grown fibres (Keller and Barham 1981) covering their own work at Bristol University and that of Pennings et al at Groningen University, Holland.

3.2 Long Term Stability of Polypropylene

3.2.1 Photo Thermal Oxydation and its Stabilisation

The introduction of any new material into the building industry must be accompanied by guarantees of its durability. In comparison to cement, polypropylene is highly resistant to aggressive chemicals and hence, it is likely that the cement would deteriorate first.
Polypropylene has a large number of tertiary carbon atoms, (1 in 3), a tertiary carbon being one bonded to three other carbons atoms. The associated tertiary carbon hydrogen bond is very weak and is very easily broken by the action of heat and light. Unless controlled by use of stabilisers, the degradation may be rapid. Indeed, degradation can never be eradicated but only substantially delayed before the reactions become autocatalytic.

The precise mechanism of degradation and its stabilisation is beyond the scope of this thesis, however, more detailed information concerning polymer stability is available in the literature (e.g. see Scott, 1979 & 1980; De Paolo & Smith, 1968) and for particular reference to polyalkene cements see Gardiner (1982).

3.2.2 Accelerated Ageing Tests on Polypropylene Fibre Cements.

The degradation process may be accelerated by subjecting samples to elevated temperatures and UV light. Use of the Arrhenius equation, relating the rate of degradation to the absolute temperature, allows an estimation of the useful product life at ambient temperatures. Whilst it is known that special combinations of stabilisers produce composite with superior qualities than is possible with general purpose stabilisers, the subject is shrouded in commercial secrecy. This problem was encountered by Mai et al (1980) who tested autoclaved composite only to discover that the unknown stabilisers were steam soluble.

Present indications from commercial sources are that suitable stabilisers are available and that photo thermal oxidation is not a serious problem. Independent research is in progress at the University of Surrey using Chimassorb 944 manufactured by Ciba Geigy, Ltd., and also Arbestab Z (Robinson Brothers Ltd.) and this data will be published in due course.

3.3 Industrial Production of Fibrillated Polypropylene Film

The production of a fibrillated polypropylene film in a form suitable for direct incorporation in a cement matrix is a recent development and details may only be found by reference to the patent literature. The basic procedure is one of extrusion, quenching, drawing, fibrillation, opening, stabilisation and collection.
The constituents of the mix may be varied but may typically be polypropylene (melt index* 3-12; residue at heptanic extraction**, >95%), up to 20% by weight of low density polyethylene and/or ethylene - propylene copolymer, up to 0.5% by weight of an expanding agent such as azo-dicarbonamide, sodium bicarbonate, etc. (creating bubbles within the film which may or may not completely penetrate the film) and up to 20% of a cement based material (European Patent Application, 0 027 273; International Patent Application, WO 80/00960). Processing and photo-thermal oxidation stabilisers would also be included.

The mix is extruded, at a temperature between 180°C and 220°C, into either a flat or tubular film and quenched (in air or water). The film is led over a pair of rollers, the second rotating faster than the first, such that the film is longitudinally drawn up to 25 times its original length (drawing temperature, typically 130-155°C). At this stage additional treatment may be introduced in the form of superficial abrasion by brushes, abrasive paper, sand blasting etc. to produce a film with a fine, hairy surface texture (European Patent Application, 0 021 017).

The film (say 1m wide) may be slit into narrower tapes, if desired, before passing over a fibrillating roller, incorporating a desired pattern of pins or blades. The pattern of the pins and the relative speed of the roller with respect to the film produce predetermined slits in the film (see 3.4.1). A fibrillation of a regular form or a random nature may be produced (Fig 3.3).

Although the film is now in a form suitable to reinforce cement on a laboratory scale, it is not suitable for introduction into the machinery currently undergoing development for producing NETCEM composite (e.g. US Patent 4,242,407). After fibrillation the films may be superimposed to form a pack of films and sealed at the edges by rotary or spot welding techniques. Specially designed grips, which allow for the longitudinal shrinkage of the film (see 3.4.1), open the pack to the required width prior to passage through an oven or on a calendar with heated pressure rollers, mechanically stabilising the film (European Patent Application 0 021 017).

*function of molecular weight.
**proportion of isotactic polypropylene.
The packs may contain films either uni-axially or bi-axially oriented which if desired, may be made more wettable to aid incorporation with the cement slurry.

It is envisaged that the film packs would be packaged on reels for despatch to the composite manufacturer who would have the flexibility to incorporate film and matrix according to needs.

3.4 Physical Description of a Fibrillated Film

3.4.1 Control of the Fibrillated Pattern

As is shown in Fig 3.3 it is possible to produce a wide range of fibrillation patterns, ranging from the precise, regular fibrillation of Fig 3.3(a) through to the random configuration of Fig 3.3(b). The technique of achieving either type of fibrillation is similar and hence, for simplicity, we shall consider the case of regular fibrillation.

Fig 3.4 shows (a) the formation of the pin pattern on the roller and (b) the slit pattern consequently developed in the film. The features of the roller and the film are described thus:-

(a) - distance between two pins arranged on a generating line of the cylindrical roller.

(b) - pin position.

(c) - distance between two pins arranged on adjacent circumferences of pins, and also, the distance between adjacent slits.

(d) - distance between adjacent generating lines of pins.

(e) - slit length.

(f) - unslit length between two successive slits.

(g) - stagger between adjacent slits.
Figure 3.3. (a) Regular and (b) Random Fibrillation Patterns
Figure 3.4. (a) Pattern of Pins on the Fibrillating Pin Roller and (b) Slit Pattern in the Fibrillated Film
(\beta_0) - obliquity of the unfibrillated band comprising the oblique succession of lengths (f).
i.e. \beta_0 = \tan^{-1} \frac{c}{g}.

Fig 3.5 - Development of Slit Pattern Shown in Fig.3.4(b) Upon Opening of the Film (m/c = 2)

Fig 3.5 shows the development of the slit pattern of Fig 3.4 and to aid interpretation three further terms are introduced, namely, primary and secondary fibrils and nodes as indicated in Fig 3.5. It can be seen that the length of the secondary fibril is given by (e-g) and the width of the primary fibril as 2c, in this particular case (the width of the secondary fibril is always c). The width of the primary fibril is indicated in Fig 3.4 by the distance between two slits overlapping across the oblique band, i.e. m.
As stated in 3.3 the film contracts upon opening and this is demonstrated in Fig 3.5. The slit, $s$, was originally longitudinally oriented but after opening, the longitudinal component of the slit has been reduced to $s'$. Thus, the film may contract by a ratio of approximately $(1 - e'/e)$.

3.4.2 Proposed Structural Model of a Fibrillated Film.

In 3.1 it was proposed that, upon drawing, a film assumes a fibrillar structure. It is now suggested that this concept be expanded such that a fibrillated film is viewed as a series of parallel, aligned and continuous elements of width, $c$. One such element is illustrated, shaded, in Fig 3.5 in the configuration dictated by the film opening. It is apparent that within the primary fibrils several elements may exist, the number of which is determined by the ratio, $m/c$. Thus the primary fibrils may be assumed to comprise a series of staggered parallel elements connected by a shear link. It is important to remember that, since a fibrillated film is derived from a uni-axially oriented film, repeating points of each fibrillar element (i.e. points A, Fig 3.5) must maintain a line parallel to the unopened molecular orientation. Thus, assuming an even opening of the film, each element approximates to a saw-tooth form oscillating about an arbitrary datum parallel to the edge of the film (Fig 3.6)

![Fig 3.6 Representation of the Configuration of a Single Element in a Fibrillated Film.](image)
3.4.3 Implications for Composite Behaviour

With this simplified model it is apparent that although a fibrillated film may be opened such that both primary and secondary fibrils appear oriented at approximately 45° to the longitudinal direction, in fact, the fibrillar elements are merely zig-zagging through the film. Hence, any such film, embodied in a cement matrix, may be expected to yield different characteristics when tested longitudinally and transversely.

(a) Longitudinal testing:

Upon formation of the first crack, the load thrown onto the fibre can be completely transferred back into the matrix (given adequate bond). The crack can only cross each element once and traditional mechanics apply to the system.

(b) Transverse testing:

In this case the load transfer is of a more complex nature since the crack may intersect an element repeatedly.
The secondary fibril is only capable of transferring load in the distance between the crack and the node. The capability of the primary fibril is altogether more complicated. It should be remembered that, unlike the secondary fibril, it consists of a multiplicity of fibrillar elements. Hence, whilst each individual element may only transfer load in the distance between the crack and its node, there is the possibility of transferring load between fibrillar elements by way of the shear link connecting adjacent elements. In this manner load in the primary fibril could be transferred to elements remote from the crack.

Tensile tests on composite made in the laboratory have yet to yield adequate two dimensional strength by widely opening the film network. It has been observed that the films tend to tear at the nodes after the formation of the first crack (transverse testing); also, the crack path is in some part influenced by the angle of the fibrils to the direction of stress, making precise analysis difficult.

It is felt that this simple model begins to offer an explanation for the poor two dimensional strength of composites incorporating widely opened film. Adequate two dimensional strength may be obtained by orienting films orthogonally but orienting films in this manner is more difficult.
to achieve on a commercial scale. However, the latter approach offers a more immediate solution.

3.5 **Fibre-Matrix Misfit as Related to Film Production.**

We are now in a position to begin to consider the possible mechanisms of interaction between polyalkene films and cement. Chapter 2 illustrated the properties of fibre cements and the manner in which they are controlled by the properties of the matrix and fibre. Previous authors have stated that multiple cracking would not occur in a polyalkene reinforced cement containing perfectly aligned and parallel sided fibres and yet, practical composites do show this behaviour. It was suggested that the structure of the fibre should be examined to show features which would promote fibre-matrix misfit. Chapter 3 described the microstructure and properties of drawn polyalkenes and Fig 3.3 shows the extremes of fibrillated films produced i.e. clean, regular networks and hairy, random networks.

The recent patent of Dansk Eternit (UK Patent Publication No. 1 605 004) claims that excellent fibre-matrix interaction is achieved with fibres "having cross sections which vary along its length and polyolefin fibrils from surface portions thereof...". Presumably, this may be taken to mean films of a hairy texture such as those shown in Fig 3.3(b). The patent claims that known fibrillating techniques produce fibres of "rectangular cross-sections, always completely smooth surfaces, and constant cross-section throughout their length". Consequently, the claimed fibre-matrix interaction must be purely an edge effect.

It is known in the plastics industry that the drawing procedure is not uniform.* The drawn films show an increased thickness at the edge compared with the middle of the film (maybe up to 20%). It is therefore, conceivable that the heterogeneous structure of the film also leads to a changing thickness over the length of the film. Early measurements on fibrillated film, conducted in this laboratory, showed that the weight of 1 metre lengths of film did indeed change along the length of the film. However, there are no published details on the change in thickness over much smaller lengths (100-500µm).

*(Schuur and Van der Vegt, 1975)
The distinction between changes in cross-section attributable to thickness and width variations may be important since the mode of fibre matrix interaction is different. Fig 3.9 shows (a) a fibre of variable thickness and (b) variable width.

As the fibre in Fig 3.9(a) is drawn through the matrix the fibre will become compressed and the interfacial contact pressures increased. A similar behaviour may occur initially with the fibre shown in Fig 3.9(b) but with increased deformation a tearing action may possibly occur as the protruding fibrils are peeled from the main fibre. In such a case the stress transfer is most complex but may be of a less efficient nature than simple fibre compression.

Therefore, the changing cross-section described in the Dansk Eternit patent is not the fundamental type considered in 2.7.4 but is supplementary, both types of misfit may occur with the fibrillated films chosen for this current programme and are considered in Chapter 7.

The next Chapter details the films used in this investigation, their incorporation in a matrix and the subsequent testing.
CHAPTER 4

EXPERIMENTAL DETAILS

4.1 Choice of Films and Fibre

The first successful production of NETCEM composite in 1976 used a lenoweave woven fabric. It was soon realised that, based on materials costs alone, an economic composite would have to use the cheaper form of non-woven fibrillated film. Following a Press Release (April 1978) many polypropylene film manufactureres sent samples of their films to the University of Surrey for evaluation. Of these companies, Bridon Fibres and Plastics Ltd. and H. & A. Scott Ltd were very helpful in producing films of various fibrillation patterns and properties. From these films, four were chosen such that the quality of the composites produced using them, ranged from poor to the best available at the time (in terms of crack spacing and load carrying capacity).

The experimental high modulus polyethylene of Ward (1980) appeared to be an excellent prospect for achieving high load carrying capacity in the composite at low strain. Metal Box Company Ltd produced, for this investigation, a narrow tape (25mm wide) of X30 polyethylene film which was subsequently fibrillated at Brunei University. Professor Keller, at Bristol University, was also approached with a view to production of samples of high modulus polyethylene from the solution process. However, whilst he thought film could be produced, the intiative was not pursued since their experiments manufactured monofilament and at too slow a rate for the needs of composite production.

Also used was a polypropylene monofilament supplied in a hank, the filaments within which varied in cross sectional area between filaments and along the length of the individual filaments.
A description of the films is given in Table 4.1. It will be noted that films BAR 112, S8 and E3H are termed voided. This designation was given after examination of the fibrillated film in a Cambridge Stereoscan Scanning Electron Microscope. Voiding refers to the presence of micron size voids which completely penetrate the film (Fig 4.1). Voiding may be created either by the inclusion of an expanding agent in the polymer production stage or accidently during the fibrillation process. It is thought, that of the voided films, only BAR 112 was voided as a consequence of an expanding agent.

The experimental programme was designed using films BAR 112 and BAR 113 as the basis. In specifying these films to Bridon Fibres and Plastics Ltd., it was requested that their properties be similar apart from the voiding to be generated in BAR 112. It can be seen from Table 4.1 and Figs 4.2 and 4.3 that this requirement was achieved (the elastic moduli also being comparable - see 5.1). BAR 21 was chosen because of its finer structure which yields a higher specific surface area generated by the perimeter of the fibrils rather than by the presence of voiding. S8 represented the highest modulus then available from a polypropylene film and it also possessed a lower specific surface area than BAR 112. E3H was chosen for its greater elastic modulus. The monofilament was included to investigate whether mechanical anchorage of the nodes of a fibrillated film (see Fig 3.5) is a major factor in fibre-matrix stress transfer or that the basic mechanisms of stress transfer are similar for a fibrillated film and monofilament.

The films are shown in Figs 4.2 - 4.6.

4.2 Determination of Film Properties

4.2.1 Elastic Modulus

The elastic modulus of the film was measured on strips cut from unfibrillated samples. Five strips, 600mm x 3mm wide, were cut using a common datum line such that all strips were of similar orientation,
<table>
<thead>
<tr>
<th>Designation</th>
<th>Manufacturer</th>
<th>Material</th>
<th>Film Thickness (µm)</th>
<th>Film Width (mm)</th>
<th>Fibril Width (µm)</th>
<th>Slit Length (mm)</th>
<th>Specific Fibre Surface - (SFS) 2 3 (mm/mm)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>BAR 21</td>
<td>BRIDON</td>
<td>PP</td>
<td>15</td>
<td>75</td>
<td>100-500</td>
<td>10-15</td>
<td>171 (24%)</td>
<td>-</td>
</tr>
<tr>
<td>BAR 112</td>
<td>BRIDON</td>
<td>PP</td>
<td>70</td>
<td>70</td>
<td>200-400</td>
<td>10-20</td>
<td>311 (14%)</td>
<td>Voided</td>
</tr>
<tr>
<td>BAR 113</td>
<td>BRIDON</td>
<td>PP</td>
<td>70</td>
<td>70</td>
<td>200-400</td>
<td>10-20</td>
<td>86 (35%)</td>
<td>-</td>
</tr>
<tr>
<td>S8</td>
<td>SCOTT</td>
<td>PP</td>
<td>75</td>
<td>220</td>
<td>200-600</td>
<td>10-15</td>
<td>231 (16%)</td>
<td>Voiled</td>
</tr>
<tr>
<td>E3H</td>
<td>METAL BOX</td>
<td>PE</td>
<td>35</td>
<td>25</td>
<td>100-300</td>
<td>10-20</td>
<td>320 (51%)</td>
<td>Voiled</td>
</tr>
<tr>
<td>MONOFILAMENT</td>
<td>-</td>
<td>PP</td>
<td>r = 83</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>24</td>
<td>-</td>
</tr>
</tbody>
</table>

PP - polypropylene  
PE - polyethylene  
Values in parentheses are coefficients of variation.

Table 4.1 Description of Fibrillated Films and Monofilament.
Figure 4.1. Example of voids in a fibril of BAR 112
Figure 4.2. Film BAR 112 Opened to Four Times its Original Width
Figure 4.3. Film BAR 113 Opened to Four Times its Original Width
Figure 4.4. Film BAR 21 Opened to Four Times its Original Width
Figure 4.5. Film S8 Opened to Four Times its Original Width
Figure 4.6. Film E3H Opened to Four Times its Original Width
i.e. parallel to the edge of the film. The choice of length was such that the aspect ratio was well in excess of 100, above which end affects are minimised (Arridge, Barham, Farrell and Keller, 1976).

The strip was accurately aligned centrally in specially made grips as shown in Fig 4.7. Initial location was achieved with double sided tape, masking tape being placed on top of the located strip. This preparation prevented premature fibrillation of the film during the test. Use of these special grips allowed more accurate alignment of the strip than was possible with the standard wedge grips.

The mounted strip was placed in the Instron 1122 and tested at a rate of 28mm/min (approximately 5%/min). The strain was taken to be the cross head displacement divided by the original distance between the grip faces. The cross sectional area of the strip was determined by weighing the strip (of known length) to 0.0001g and assuming a specific gravity of 0.91 for polypropylene and 0.96 for polyethylene.

All films except BAR 21 were supplied in both fibrillated and unfibrillated forms - although the fibrillated and unfibrillated S8 came from different batches of identically drawn film. In order to obtain an initial modulus for BAR 21, the fibrillated film (600mm long) was carefully folded to 25mm width and masking tape wrapped over each end. The film was placed in the Instron wedge grips and tested as previously described.

The latter method will yield a lower bound to the true modulus since any discontinuous fibrils (hairs) attached to the primary network are included in the determination of cross sectional area yet are non load bearing. Further, alignment of all fibrils in the direction of drawing cannot be accurately controlled. Fortunately BAR 21 was not a particularly hairy film (Fig 4.4), consequently the modulus, so determined, was considered a reasonable lower bound estimate.
Figure 4.7. Film Strip in Partially Assembled Crips for Modulus Evaluation
4.2.2 Specific Film Surface

The specific film surface (SFS) is defined as the surface area of film per unit volume of film. For a simple monofilament SFS may be calculated from the perimeter, $P_f$, and the cross sectional area, $A_f$, of the fibre.

\[
SFS = \frac{P_f}{A_f} \quad \ldots \ldots \ (4.1)
\]

However, this is not possible for the complex structure of a fibrillated film. Although the fibril width was stated to be 200 - 400\(\mu\)m (BAR 112 - Table 4.1) Fig 4.8 shows that much finer fibrils also exist, such that it is impossible to define even an average fibril width and hence calculate the specific film surface.

No technique was available within the University of Surrey to experimentally determine the specific film surface and hence the Shirley Institute, Manchester was contacted with a view to supplying the data. Unfortunately, this approach was not fruitful and finally Montedison, SpA, Italy offered to measure the specific film surface using a technique involving measuring the adsorption of krypton onto the film (approximately 20g sample) at liquid nitrogen temperatures. The SFS was determined thus

\[
SFS = \frac{X_m N A_m x 10^{-20}}{M} \quad \ldots \ldots \ (4.2)
\]

where SFS is in m\(^2\)/g.

- $X_m$ - monolayer capacity (g) of adsorbate per unit weight of film.
- $A_m$ - molecular cross sectional area of adsorbate (21 \(\AA^2\)).
- $N$ - Avogadro's Constant (6.02 x 10\(^{23}\) molecules per mole).
- $M$ - molecular weight of adsorbate.

The results reported in Table 4.1 are the average of 3 runs over a period of 18 months.
Figure 4.8. Micrograph of Film BAR 112 showing Complex Structure of a Fibrillated Film
4.3. Production of Composite for Tensile Testing

4.3.1 Matrix

The same quantity of matrix was prepared for each composite sheet, regardless of sheet size. The mix quantities are given below:

<table>
<thead>
<tr>
<th>Material</th>
<th>Quantity</th>
<th>Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement</td>
<td>4.00 kg</td>
<td>1.0</td>
</tr>
<tr>
<td>Fly Ash</td>
<td>1.00 kg</td>
<td>0.25</td>
</tr>
<tr>
<td>Sand</td>
<td>0.76 kg</td>
<td>0.19</td>
</tr>
<tr>
<td>Total Water</td>
<td>1.36 kg</td>
<td>0.34</td>
</tr>
<tr>
<td>Melment L10</td>
<td>0.072 kg</td>
<td>0.018</td>
</tr>
</tbody>
</table>

The cement (ordinary typical cement, Batch 103; see appendix for chemical analysis) and fly ash (Pozzolan Ltd) were from single batches. The fly ash tended to be compacted within the bag, and was placed over a No. 25 sieve to break up the lumps. In order to obtain a highly workable high strength mix at low w/c ratio a melamine formaldehyde superplasticiser, Melment L10 (Hoechst Chemicals, Ltd.) was used. The quantity of Melment L10 was experimentally determined such that mix segregation was minimised. The sand was a bagged single size (100-300μm) Leighton Buzzard sand (Curtis Onx, Ltd.) and was used in the as supplied condition.

A 3 speed Peerless planetary action mixer was used. The bowl and paddle were moistened to minimise water loss from the mix. The cement, fly ash and sand were dry mixed at low speed (speed 1) and then the water added. Mixing was continued until a homogeneous stiff paste was achieved, at which stage the Melment L10 was added. After approximately 1 minute the speed was increased (speed 2) and the mixing continued for a further 2 minutes. The mix was then transferred to a polyethylene bin since sheet manufacture could take up to 3 hours. If the mix stiffened appreciably, further hand mixing restored workability.

4.3.2 Composite Manufacture

Achieving a uniform film distribution, through the sheet thickness, was relatively easy at a film volume fraction greater than 8%, but was
increasingly more difficult at lower film volume. Hence, slightly different manufacturing techniques were required. The basic technique was a hand lay up process in which opened layers of film were impregnated with an appropriate quantity of matrix until the correct thickness and film volume fraction were attained. Obviously the number of layers of film for a specified volume of film depended upon the film thickness and degree to which the film was opened. For the work reported herein, all films were opened to a width of 4 times their unopened or unfibrillated width unless otherwise specified.

The basic procedure was as follows:

(i) A release layer of polyethylene sheeting was placed on a wooden base board, which had been previously moistened. The polyethylene was firmly pressed onto the board, the moisture ensuring intimate contact. Two parallel rows of nails were placed in the board at the required spacing.

(ii) Three or four layers of film were hooked over the nails before the first matrix was applied and well worked through the films. The subsequent procedure depended upon the target film volume fraction.

(iii) Low film volume fraction (<5%) - a copious quantity of matrix was applied to the sheet and 1 or 2 layers of film "sunk" into it. This was repeated until all the films had been incorporated.

(iv) High film volume fraction (>9%) - 3 or 4 layers of film were worked into the sheet until no more could be introduced. Sufficient matrix was added until the last layer of film was barely coated. More film was added and the procedure repeated.

(v) The above procedures were perfected with experience and sheets of intermediate film content could be produced with subtle adaptions to the procedures.

(vi) Generally, layers were worked by hand, however, at sheet thickness of 2, 4 and 6mm the top surface was trowelled to achieve a more uniform thickness across the sheet. When the sheet was complete,
(target thickness - 6mm) the nails were removed and a second layer of polyethylene sheeting was trowelled into the top surface, ensuring that no air pockets remained trapped and that any excess matrix was squeezed out.

(vii) The sheet was removed from the board and placed on a ground steel bed and again trowelled flat. It was left overnight on the bed in a constant temperature room (20°C). Upon demoulding, curing was under water at approximately 20°C until testing at 28 days.

Halfway through the manufacture of each sheet, 4 coupons were made from the matrix alone for flexural testing as a control on the mix proportions and were placed in the fog room overnight prior to water curing for a further 27 days.

At each target film content, 2 sheets were manufactured on different days to partly average the effects of variation between matrix batches.

The choice of 4 times film opening ratio was a convenient but arbitrary choice. Two further sheets were manufactured (BAR 113) in which the film was not opened. The film was unreeled straight onto the sheet and the matrix wiped through the unopened film. One sheet was made to a target film volume fraction of 15 - 17% and the other to the maximum achievable. Production and curing was otherwise as previously described.

The manufacturing procedure for the monofilament composite was similar to that for the fibrillated film except in the method of introduction of the fibre. The monofilament was packaged in a hank on a drum. The hank was unreeled and distributed evenly across the sheet (approximately parallel to the edge of the sheet) and cut to length. Sheet preparation was otherwise as that for a fibrillated film.

4.3.3 Tensile Strip Preparation

After curing, each sheet was cut into 10 strips, 300mm x 25mm wide (only 9 could be cut from the E3H composites and the unopened BAR 113 composites). The strips were cut so that they were parallel to the direction of film alignment. Material within 50mm of the edge of the sheet was discarded (with the same exceptions as previously mentioned where the sheets were made with greater attention to the edge and 15 mm discarded). The strips were kept wet during cutting and were subsequently returned to the curing tank.
4.4 Instrumentation for Tensile Testing of Composite

4.4.1 Strain Measurement

The requirements for strain measurement were particularly rigorous. From each test not only was the complete load-strain curve required (up to 10% strain) but also the initial load-strain curve to 0.2% strain with good resolution in the range 0-0.2%. The technique adopted was based upon the pivotting extensometer developed at BRE for work on GRC (de Vekey, 1974).

The extensometer was manufactured in the departmental workshop with the sole modification of an increased gauge length of 98mm. The extensometer is shown in Fig 4.9. With a gauge length of 98mm, and 100mm between the fixed knife edge and the pivot point, the first 4mm of extension was measured ±2mm either side of the pivot, this being the most linear region of the extensometer.

Extension was measured using 2 linear variable differential transformers (LVDT's) which had to satisfy 3 requirements:-

(i) high degree of linearity.

(ii) ±5mm travel about electrical zero.

(iii) generate highly stable and sensitive signals at small extensions.

The system which met these requirements used RDP D5/200 AG ac. LVDT's in conjunction with RDP Type D7M Signal Conditioning Modules.

In order to measure clamping strains, due to specimen warping, the individual signals from the 2 LVDT's were displayed on the Y channels of a Bryans series 26000 A3 chart recorder (calibrated to 0.2% strain full scale). The signals were then electronically summed and averaged (unit constructed by Sands-Whitely R & D Ltd.) prior to display on a second recorder (XY recorder of Instron 1122) calibrated to approximately 9% strain. The load signal was taken from the Instron 1122 load cell, being tapped from the controller after amplification.
Figure 4.9. Extensometer shown Mounted on Calibration Bench - The Locking Pins are shown in Position.
The extensometer was calibrated in a micrometer bench before each day's testing (Fig 4.9). The LVDT's were positioned such that electrical zero was at approximately half the anticipated failure extension.

4.4.2 Acoustic Emission Detection

Preliminary tests indicated that detection of initial cracking and the completion of multiple cracking could be difficult from the stress-strain record alone, especially with high $V_f$ composites. It was thus decided that more information might be forthcoming by monitoring the acoustic emissions (AE) emanating from the strip under test.

Some materials give audible emissions as a sign of distress; eg., tin cry, martensitic phase transformation in steels or creaking of wooden pit props. However, most materials yield inaudible signals. Detection of these emissions is the basis of AE monitoring.

The AE system essentially comprises a piezo-electric transducer which converts the elastic stress waves into an electrical signal. The signal is fed, via a preamplifier to a processing unit comprising a bandpass filter, variable gain amplifier, comparator and counter.

The electrical signal from a single acoustic "event" may be approximated by a decaying sine wave (Fig 4.10).

![Figure 4.10. Electrical Signal Produced by an Acoustic Event](image)
A common method of "analysing" these signals is to simply count the number of times the signal exceeds a specified datum or threshold voltage. The data may be utilised in the form of a continuous summation of counts or the number of counts in a specified time (rate count). The cumulative count (rate count) will have an arbitrary scale.

Limited use has been made of AE to monitor deterioration of composite materials. In 2.4.7 it was reported that Aveston et al (1974) used the cumulative count to identify the stress at which maximum cracking occurred in a fibre cement. Others have used AE to determine the number of fibre fractures in Boron-Epoxy composites (Fitz-Randolph, Phillips, Beaumont and Tetelman, 1971), crack area in GRP (Adams and Flitcroft, 1976) and degradation of CFRP (Fuwa, Bunsell and Harris, 1976).

AE sources within fibrous composites may include (Williams and Lee, 1978);-

(i) matrix and fibre fracture
(ii) interfacial debonding
(iii) fibre relaxation upon fracture
(iv) pull out friction
(v) intralaminar and interlaminar cracks

It is apparent that these different mechanisms may produce signals of varying amplitudes and frequencies. During its passage through the material the wave amplitude will be attenuated, being affected by the source of emission (internal or surface), wave frequency and specimen size (Dunegan and Green, 1971; Swindlehurst, 1973). Reflection of the wave from fibre-matrix interfaces, crack faces etc, further complicates eventual signal analysis (Fuwa et al, 1976)

Jolly (1980) has stated that both the cumulative count and rate count will be similar in a series of tests provided that:-
(i) the specimens are geometrically similar.

(ii) the failure mechanisms of each specimen are similar.

(iii) the distribution of the failure mechanisms in each specimen is similar.

The amplitude of emissions during the multiple cracking region was expected to be larger than those both preceding and following multiple cracking. Thus, AE was considered a useful tool for the purposes intended.

The system used in this current work is described below:-

System type       AECL 105
Band Pass Filter  100-300 Hz
Gain              86 db
Threshold voltage 1v
Acoustic couplant sc-6 (from AECL)
Sensor attachment Coiled spring clip

In order to maximize the AE information the cumulative count was plotted against load to yield first cracking data (5 strips out of each batch of 10) and also against strain and the rate count against strain to yield multiple cracking and post cracking data (remaining 5 strips).

The AE equipment was calibrated to show $10^6$ counts (cumulative count) and $10^4$ counts resetting every 100 milliseconds (rate count).

A block diagram of the complete instrumentation is shown in Fig 4.11 and a strip under test is shown in Fig 4.12.
Figure 4.11. Block Diagram of Instrumented Tensile Test
Figure 4.12a Complete Instrumentation for Direct Tensile Testing of Composite
Figure 4.12b. Detail of Tensile Strip Under Test (note: the grips were used in this orientation for greater degree of alignment).
4.5 Method of Test for Composite Strips

4.5.1 Load/AE/Strain

The procedure adopted was the same for all composite strips. The strips were kept under water until required for testing. Prior to placing in the Instron 1122 the width and thickness were measured at 3 points within the central third of the strip. The strip was clamped in the top wedge grip with lead flashing (approx 30mm x 1.2mm) between the strip and the faces of the grip. The lead (acoustically silent at 86 db) was used to reduce stress concentrations within the gripped portions of the strip preventing premature failure within the grips. The AE transducer and extensometer were then placed in position. With the AE on "hold", the locking pins were withdrawn from the extensometer and the bottom grip tightened, thus allowing measurement of the strains induced by clamping. The AE "hold" was released and the crosshead started (10mm/min) in order to apply tensile load to the strip. No AE was detected when the crosshead was started.

At 5% strain (3% for S8 and E3H composites) the strip was unloaded and immediately reloaded to failure. This procedure was only followed on alternate strips from each batch.

4.5.2 Crack Spacing/Film Volume Fraction

After testing, the number of cracks were counted within a given length (chosen so that at least 30-40 cracks were counted). Detection of the cracks in composites with high film volume fractions was difficult and hence these were highlighted by wiping the strip with ink. The ink soaked rapidly into the cracks and, after the excess had been removed from the surface, the cracks were clearly visible.

Approximately 50mm was cut from one end of the strip for determination of film volume fraction by the following method:-

(i) Strip weighed in air (SSD) - \( W_1 \)

(ii) Beaker of water weighed - \( W_2 \)
(iii) Strip suspended in beaker of water and combined weight noted $- W_3$

(iv) Strip placed in 50/50 water-Hydrochloric acid (Assay 35-38%) solution for more than 7 days.

(v) Film extracted and weighed after 24 hours at $105^\circ\text{C} - W_4$

The specific gravity of the composite was determined by $\frac{W_1}{(W_3 - W_2)}$ and the film volume fraction by $\frac{W_4}{A(W_2 - W_3)} \times 100\%$ where $A$ is the assumed specific gravity of the film.

4.5.3 Matrix Control Specimens

The matrix controls were tested in a flexural rig (Ali and Grimer, 1969) at a crosshead speed of 10mm/min and the maximum load noted.

4.6 Pull Out Testing of Narrow Film Strips

4.6.1 Preparation of Specimens

The value of single fibre pull out tests has been previously discussed (2.8). The approach adopted herein was similar to that of Pinchin, to obtain "some idea" of the mechanisms involved in voided (BAR 112) and non voided (BAR 113) films.

In testing steel, glass or polymer monofilament the test specimen may be a representative sample of the "as used" fibre. This is not possible for the structurally more complex fibrillated film, hence, a special sample must be prepared. Three millimetre wide strips were stamped out from the unfibrillated film with an accurately machined die so that the edges were parallel and all specimens comparable. It was found that 3mm strips were the narrowest which could be prepared by this technique without longitudinal splitting of the strip.

The strips were embedded in the matrix for a depth of approximately 2mm; the mould is shown schematically in Fig 4.13 and partially assembled in Fig 4.14. The mould has the versatility of being able
Figure 4.13. Schematic Diagram of Assembly of Narrow Strip Pull Out Mould
Figure 4.14. Partially Assembled Mould for Narrow Strip Pull Out Samples
to accommodate embedment lengths of up to 25mm. An embedment depth of 2mm was chosen as being the maximum possible to allow pull out rather than breakage of film BAR 112.

Particular attention was paid to sealing the top of the threaded tube so that paste did not run down the fibre protruding through the tube. The seal (Fig 4.13b) comprised a thin copper disc stamped to the same diameter as the coupling thread and a central slot, approximately 4x1mm was cut in it with a scalpel. The bottom of the disc was covered with an adhesive insulating tape and a fine cut, 3mm long, made in the tape. A thin layer of plasticine was applied to the top of the coupling thread to complete the seal. The method of assembly was to fit the fibre through the slotted disc and coupling thread and to clamp the fibre under a slight tension to ensure alignment. The disc was then gently pushed onto the plasticine ensuring that no fibre misalignment occurred.

Since the interest in these tests lay in the comparative behaviour of voided and a non-voided film the exact nature of the matrix was not considered to be critical. Consequently, a superplasticised cement paste, of the same water-cement ratio as the composite sheets, was used as detailed below. Mixing was by hand in a polyethylene bag.

Cement (Batch 103) - 200g
Water - 68g
Melment Ig - 1g

The paste was placed in the moulds using a syringe and the moulds gently vibrated. If necessary, the fibres were re-tensioned before the moulds were placed in the fog room overnight. Curing was under water until testing.

From each batch of paste 5 voided and 5 non-voided fibre pull out specimens were made. Three paste batches were made in all.

4.6.2 Method of Test

The test rig is shown in Fig 4.15 and is based upon that of Gray and Johnston (1978). Upon removal from water the specimen was wrapped in clingfilm to prevent moisture loss whilst the fibre was assembled in the grip as previously described in 4.2.1. The distance between the
Figure 4.15. Pull Out Sample Under Test
grip face and the cement paste was only ~15mm and great care was taken not to stress the fibre-matrix interface. The cling film was removed before securing the specimen in the test rig. Testing was at a crosshead displacement of 10 mm/min and a load cross-head displacement record obtained.

After test the specimen was split open and the embedment length measured to 0.1mm.

4.7 Matrix Work of Fracture

4.7.1 Preparation of Specimens

Three blocks of matrix (4.3.1) 200mm x 50mm x 30mm deep were cast and cured as before. From each block 2 beams were sawn, 200mm x 25mm x 20mm deep, such that material which had been in contact with the sides of the mould and the atmosphere were discarded. Further, the horizontal plane, as cast, became the vertical plane, as tested.

An initial notch, 3mm wide, was sawn across the centre of each beam, to a depth of 10mm. This was extended a further 3mm, using a saw blade of 150µm thickness, such that the notch/depth ratio was approximately 0.65. All cutting was performed under a copious flow of water.

4.7.2 Method of Test

The test was performed under water using an Instron 1195 as the reaction frame. The load was measured using a high stiffness (6KN/µm) load cell (Kistler piezo electric load washer, 9031 and charge amplifier, 5007). The deflection was measured with a RDP D5/40 a.c. LVDT using an RDP Type D7M Signal Conditioning Module. The signals were recorded on a Bryans 29000 series A3 XY recorder. The rate of crosshead displacement was 0.05mm/min. The test configuration is shown in Fig 4.16 and 4.17.
Figure 4.16. Test Configuration for Matrix Work of Fracture (Span = 180 mm)

Figure 4.17. Detail of End Roller Support Showing Spherical Seating to Accommodate Specimen Distortion
The LVDT was strapped to the loading column above the load washer and measured the deflection to the base plate. Hence any slight displacement of the supports would be included. However, this had been previously checked to be negligible.

4.8 Scope of the Composite Test Programme

The scope of the test programme for composites produced at 4 times film opening is detailed in Table 4.2.

<table>
<thead>
<tr>
<th>Film Type</th>
<th>Target Film Volume Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>4%</td>
</tr>
<tr>
<td>BAR 21</td>
<td>✓</td>
</tr>
<tr>
<td>BAR 112</td>
<td>✓</td>
</tr>
<tr>
<td>BAR 113</td>
<td>✓</td>
</tr>
<tr>
<td>S8</td>
<td>✓</td>
</tr>
<tr>
<td>E3H</td>
<td>✓</td>
</tr>
</tbody>
</table>

Table 4.2 Test Programme Showing Pairs of Sheets Made at 4 Times Film Opening.

Single Sheets were also made at film volume fractions of 15% and 25% with BAR 113 (unopened film) and a monofilament sheet at an approximate film volume fraction of 15%.

All the above mentioned sheets were tested at 28 days. However, the 6.5% BAR 112 and BAR 113 sheets were made large enough for 10 strips to be tested at 1 year in addition to the 28 day tests.

Chapter 5 presents typical results achieved using the test procedures described herein. Chapters 6 and 7 examine in greater detail the initial cracking behaviour and fibre-matrix stress transfer respectively.
CHAPTER 5

PRESENTATION OF TYPICAL RESULTS

Chapter 5 presents the results of the film modulus tests, typical load/strain/acoustic emission data for the composites, typical load/crosshead displacement data for the narrow strip pull out specimens and results of the matrix work of fracture tests. Additional discussion of enhanced matrix failure strain and fibre matrix stress transfer follows in Chapters 6 and 7 respectively.

5.1. Film Modulus Tests

5.1.1. Results

The results in the form of tangent and secant moduli are presented in Figures 5.1. and 5.2 respectively. The coefficient of variation for each determination was typically less than 2%. Since BAR 21 was only available in the fibrillated form a strict comparison with the other films is not possible and only the initial modulus (0.1% strain) is presented so that the film may be approximately ranked.

Failure of the films was by progressive fibrillation and fracture which prevented an accurate determination of ultimate strength and strain. The moduli curves are plotted to the approximate strain which all five strips attained before failure was initiated, e.g., BAR 112 exhibited failure initiation at strains between 3.5% and 4.3% and is only reported to 3% strain.
Figure 5.1. Elastic Modulus (tangent) of the Films as a function of Strain
Figure 5.2. Elastic Modulus (secant) of the Films as a Function of Strain
A general feature of film behaviour, illustrated in Figures 5.1 and 5.2, is the markedly non-linear stress/strain characteristic of polyalkene films. Films E3H and S8 showed a linear relationship to only approximately 0.1% strain whereas BAR 112 and BAR 113 were linear to strains greater than 0.25% before the modulus decreased. The rate of decrease is dependant upon the initial modulus, e.g., BAR 113 loses 17% of its 0.1% strain tangent modulus when measured at 0.5% strain whereas E3H decreases by 45% by the time 0.5% strain is reached (See Table 5.1). Previously published results (Hughes and Hannant, 1982) also follow this trend in which a low modulus polypropylene film (2.5 GPa) maintained linearity to approximately 1% strain.

<table>
<thead>
<tr>
<th>Strain</th>
<th>Tangent Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>BAR 112</td>
</tr>
<tr>
<td>0.1 %</td>
<td>8.7</td>
</tr>
<tr>
<td>0.25 %</td>
<td>8.7</td>
</tr>
<tr>
<td>0.5 %</td>
<td>7.7</td>
</tr>
</tbody>
</table>

Table 5.1. Tangent Moduli at Low Strains

Interestingly, S8 and BAR 113 show small increases in tangent modulus of 0.2 GPa and 0.3 GPa respectively at strains in excess of 1% (occurring on all five strips tested). This may possibly be due to the further drawing and restructuring of the film during the test. This behaviour was not noted for E3H although a 10% increase in the post crack curve has been reported to occur at a composite strain of 1.9% in composites made from a previous batch of E3H (Hughes and Hannant, 1982).
It will be remembered that the unfibrillated film that was tested and reported as S8 was, in fact, a duplicate film (laboratory designation S9), manufactured at the same draw ratio of 18:1 as a replacement for exhausted stocks of S8. The modulus curve for fibrillated S8 has already been reported (Hughes and Hannant, 1982) for which the tangent moduli at 0.1%, 1.0% and 2.0% strain are 10.2 GPa, 8.4 GPa and 5.6 GPa respectively. The corresponding values for the unfibrillated film tested are 12.0 GPa, 6.2 GPa and 6.4 GPa respectively. Hence, whilst the initial modulus of unfibrillated S9 appears to be a realistic estimate of fibrillated S8 modulus the moduli at higher strains should be treated with some caution.

The secant moduli (Figure 5.2.) also show the expected decrease with strain but at a less rapid rate than for the tangent modulus. No increase in secant modulus at higher strains is exhibited by films S8 and BAR 113.

It was not possible to test the monofilament since a load cell with an adequate range was not available.

5.1.2. Implications of Non-Linearity of Film Stress-Strain Properties upon Composite Performance

As was shown in sections 2.4-2.6 the film modulus affects composite properties in all three zones of composite performance.

In the uncracked zone the strains in both film and matrix are assumed to be equal and are of such a value, less than 0.05%, that the assumption of linear elasticity is reasonably valid, i.e., \( E_f = E(0.1\%) \).
However, as the matrix begins to crack the situation becomes gradually more complex. In calculating the enhanced failure strain of the matrix, $\varepsilon_{\text{mu}}$, the Nottingham model (Korezynskyj et al., 1981) calculates a strain distribution around a Griffith crack (2.4.5.). Using realistic material parameters ($E_f = 7.7 \, \text{GPa}, \, E_m = 31.5 \, \text{GPa}, \, r = 23 \, \mu\text{m}, \, \tau = 0.5 \, \text{MPa}, \, V_f = 5\%, \, \gamma_m = 5 \, \text{J/m}^2$). The maximum fibre strain ($\varepsilon_f$) is calculated to be 0.28% which is the limit of linearity of the film (i.e., BAR 113 - see Figure 5.1.). It should be noted that the value of 23 $\mu$m for fibre radius should strictly be termed the "effective fibre radius" since it was calculated from the measured value of specific surface area, i.e., $E^m = 29.5 \, \text{GPa}, \, r = 6.3 \, \mu\text{m}$ for film E3H the fibre strain is calculated to be 0.29%. At this strain the secant modulus has decreased to ~ 28.5 GPa. Strictly an iterative process should be used in the theoretical study to match fibre strain and modulus, however, the assumption of linear elasticity is close enough before unstable crack propagation considering the other assumptions made.

The ACK model (Aveston et al., 1971) assumes that the crack opens to its full width such that conditions of fixed load are maintained. The maximum strain in the fibre is given by $(1 + \alpha)\varepsilon_{\text{mu}}$, where $\alpha = \frac{E_m V_m}{E_f V_f}$ and $\varepsilon_{\text{mu}}$ is enhanced matrix failure strain.

If film BAR 113 is again considered ($E_f = 7.7 \, \text{GPa}, \, E_m = 31.5 \, \text{GPa}, \, V_f = 5\%, \, \varepsilon_{\text{mu}} = 0.025 \%$) the maximum fibre strain is calculated to be 1.97% which is well into the non-linear region and a lower value of $E_f$ is therefore applicable. Hence, the fibre strain, $\varepsilon_f$, may be re-written thus,

$$\varepsilon_f = [1 + \alpha_f(\varepsilon_f)] \varepsilon_{\text{mu}} \quad \ldots \quad (5.1.)$$
The width of the crack, $b$, and the strain at the end of multiple cracking, $\varepsilon_{mc}$ are also similarly affected, such that,

$$b = \left[ 1 + \alpha f(\varepsilon_f) \right] \varepsilon_{mu} x^-$$  \hspace{1cm} \text{...... (5.2.)}

$$\varepsilon_{mc} = \left[ 1 + 0.659 \alpha f(\varepsilon_f) \right] \varepsilon_{mu}$$  \hspace{1cm} \text{...... (5.3.)}

The term $\alpha f(\varepsilon_f)$ depends upon the fibre-matrix stress transfer since the fibre modulus changes along the length of the fibre according to the stress (strain) in the fibre at any location. Two extremes of stress transfer are shown in Figure 5.3, where the stress is initially transferred rapidly with respect to distance from the crack face (a) and very slowly (b).

![Figure 5.3: Possible Fibre-Matrix Stress Transfer Functions](image_url)
In case (a) the relative length of fibre at high stress (low modulus) is less than that for (b) and, hence, the extension of the fibre would be expected to be less (i.e., lower crack width and strain at the end of multiple cracking). Therefore, to be precise, the fibre modulus should be continually related to the stress (strain) in the fibre along its length. An approximation would be to adopt a secant modulus at a fibre stress, $\sigma_f$, given by,

$$\sigma_f = E_f(0.1\%) \epsilon_{mu} + \frac{\sigma_{mu} V_m}{V_f} \quad \ldots \quad (5.4.)$$

and assume the fibre-matrix stress transfer to be linear as is usually assumed.

In the post crack region the modulus of the composite is given by $E_f V_f$. An appropriate fibre modulus would be a secant modulus which took its origin as the fibre strain across the fully opened crack. However, at such strains (approximately 1.5% depending upon $V_f$) the tangent modulus is sufficiently constant (Figure 5.1) for it to be a reasonable approximation.

5.2. Typical Complete Load/Strain Data for Composites Tested at 28 Days

5.2.1. Results

Figures 5.4. and 5.5. show typical tensile load/strain data for S8 and BAR 21 composites respectively. Figures 5.6.-5.8., 5.9.-5.11. and 5.12.-5.14. show typical load/strain data for BAR 112, BAR 113 and E3H composites respectively containing increasing volume
Fig 5.4  Typical Load - Strain - Acoustic Emission Data

Film - S8, $V_f = 6.4\%$
Fig 5.5 Typical Load - Strain - Acoustic Emission Data
Film - BAR 21, V_f = 6.3%
Fig 5.6  Typical Load - Strain - Acoustic Emission Data

Film - BAR 112, $V_f = 4.4\%$
Fig 5.7  Typical Load - Strain - Acoustic Emission Data

Film - BAR 112, $V_f = 7.8\%$
Fig 5.8  Typical Load - Strain - Acoustic Emission Data

Film - BAR 112, $V_f = 9.9\%$
Fig 5.9  Typical Load - Strain - Acoustic Emission Data
Film - BAR 113, V_f = 4.4%
Fig 5.10  Typical Load - Strain - Acoustic Emission Data
Film - BAR 113, $V_f = 8.8\%$
Fig 5.11  Typical Load - Strain - Acoustic Emission Data

Film - BAR 113, V = 16.3%
Fig 5.12 Typical Load - Strain - Acoustic Emission Data

Film - E3H, $V_f = 3.1\%$
Fig 5.13 Typical Load - Strain - Acoustic Emission Data

Film - E3H; $V_f = 5.5\%$
Fig 5.14  Typical Load - Strain - Acoustic Emission Data

Film - E3H, $V_f = 7.4\%$
Fig 5.15  Typical Load - Strain - Acoustic Emission Data
Film - Monofilament, V = 17.1%
fractions of film. Figure 5.15. shows a typical load/strain curve for the monofilament composite. The composites illustrated in Figure 5.4.-5.14. were manufactured using a 4 times film opening. Also shown on Figures 5.4.-5.15. are the associated AE (Cumulative count) data which will be discussed in section 5.3.

The monofilament composite exceeded the load capacity of the Instron 1122 load cell (5 KN) and the BAR 21 and BAR 113 composites exceeded the strain capacity of the extensometer; hence, the maximum load (strain) shown on the figure is not the ultimate condition.

It can be seen that the three zones of the theoretical ACK tensile stress/strain curve are clearly represented in polyalkene fibre cements. However, there are important variations to which attention will be drawn but which will be discussed in greater detail in Chapters 6 and 7.

5.2.2. Matrix Cracking

Discussion of composite performance at low strain (< 0.1%) may be found in section 5.4. However, it can be seen that multiple cracking did not necessarily occur at constant load as in the idealised case analysed by ACK. Indeed, the majority of multiple cracking could occur at loads lower than, approximately equal to, or continually increasing from the load at which the first few cracks formed (see Figures 5.6., 5.13., and 5.15., respectively).

It is not fully understood why the majority of multiple cracking could occur at loads less than that at which the first crack propagated. This behaviour was particularly noted in composites of low film volume fraction and film modulus. It may be related to the bowing of the specimen and non-uniform distribution of film affecting crack propagation. Also it was noted that at this combination of film volume and modulus the energy released at each
crack formation was sufficient to cause the extensometer to lose contact with the strip (this was overcome by increasing the spring stiffness).

5.2.3. Completion of Multiple Fracture.

Figures 5.4.-5.14. show that, in general, the strain at the end of multiple cracking (taken as the strain at which the approximately linearly increasing portion of the "post crack" curve commences) decreases with an increase in film modulus and volume fraction. However, the multiple cracking behaviour is not as simple as described at ACK, with further cracking occurring on the initial part of the "post crack" curve. Unfortunately, reduction of the original A3 stress-strain record for presentation in an adequate thesis format has resulted in a loss of clarity. However, Figures 5.6. and 5.9. illustrate the effect. It can be seen that more "post crack" cracks have been formed in the BAR 112 composite than have been formed in the BAR 113 composite. Reference to Table 4.1. indicates that the difference between the two films lies in the voiding of BAR 112. This factor appears to be related to the degree of further cracking since it also occurs with S8 and E3H composites to an extent greater than for BAR 113 but less than for BAR 112. BAR 21 exhibits a similar behaviour to BAR 113.

Cracking of a similar type has been observed in GRC by Laws et al. (1973). They attributed it to crack branching rather than to the formation of new cracks. However, visual examination of the NETCEM strips while under test indicated that the further cracking was due to new crack formation rather than solely crack branching. Laws et al., noted their effect at \( \sim 1\% \) volume fraction and similar crack branching appears to be also present in polyalkene cements at close crack spacing (\( \sim 1\mathrm{mm} \)). Such branching makes the assessment of crack spacing a rather arbitrary procedure but results can be consistent if counted by one operator. The crack spacing will be dealt with in Chapter 7.
As discussed in section 5.1.2., the strain at the end of multiple cracking will be affected by the non-linearity of stress-strain properties of the film and also the rate of stress transfer from fibre to matrix. For the purpose of this comparison the film modulus will be taken to be the secant modulus at 3% strain (BAR 112 - 5.1 GPa, BAR 113 - 4.3 GPa, E3H - 13.9 GPa) and a linear stress transfer will be assumed. The experimental value of the strain at the end of multiple cracking is determined as the strain at which the slope of the curve increases to that of the "post crack" curve.

The correlation between the experimental and calculated values of the strain at the end of multiple cracking is shown in Figure 5.15(a) and appears to be reasonable but with high scatter.

5.2.4. "Post Crack" Performance

5.2.4.(a) Effective Film Modulus

Figures 5.4.-5.14. show that the post crack slope increases with both increases in film modulus and volume fraction as expected. Table 5.2. shows the effective film modulus which is calculated by dividing the "post crack" slope by the film volume fraction.

<table>
<thead>
<tr>
<th></th>
<th>BAR 21</th>
<th>BAR 112</th>
<th>BAR 113</th>
<th>E3H</th>
<th>S8</th>
<th>MONO-FILAMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Effective Film Modulus (GPa)</td>
<td>2.3</td>
<td>2.6</td>
<td>2.7</td>
<td>6.9</td>
<td>8.2</td>
<td>3.9</td>
</tr>
<tr>
<td>( ) denote coefficients of variation.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 5.2. Effective Film Modulus from the Post Crack Slope
Figure 5.15(a) Relationship Between Calculated and Measured Strain at the Completion of Multiple Cracking Assuming the Film Modulus to be that of the Secant Modulus at 3% Strain.

Calculated Strain at the Completion of Multiple Cracking (%)

Measured strain at the Completion of Multiple Cracking (%)

- BAR 112
- BAR 113
- E3H
All of the fibrillated films, with the exception of S8, exhibit a lower effective film modulus than the tangent moduli shown in Figure 5.1., the coefficient of variation has increased from a value of less than 2.0% for the film tested on its own.

It must be remembered that the material tested for modulus was not strictly comparable to that incorporated in the composite. In the composite, the fibrils across the crack may not be molecularly oriented in the direction of applied stress as well as in the unfibrillated strip. Also, the fibrillation process produces discontinuous fibrils ("hairs") attached to the main network structure which, whilst included in the film content determination, are not as effective in terms of load carrying capacity as a continuous fibril. Further, the film content determination was made at the end of each strip with the result that the volume fraction in the central 98 mm would be unlikely to be exactly the same as at the ends of the strip. In view of the above it is considered that the agreement between the film moduli determined from the composite and from the film alone is acceptable. The apparent anomaly of S8 has been explained in section 5.1.1.

5.2.4.(b) Ultimate Strain

The ultimate strength and strain of the composite did not form a major part of the investigation. However, attention must be drawn to the difference in behaviour of BAR 112 and BAR 113.

The modulus, strength and failure strain of polyalkenes are broadly related to the draw ratio. Yet, Figures 5.6. and 5.9. indicate that, whilst the moduli are comparable, BAR 112 exhibits a far lower composite failure strain than does BAR 113. Figure 5.6. shows that the composite is adequately ductile at an age of 28 days. However, it is known that strips subject to natural weathering can suffer a loss of ductility with time as a result of continued hydration of the cement (Hannant, 1981). Hence, due regard must be given, in the choice of a film for a commercial composite, to the
balance of film properties. In this case, BAR 112 was specified to be voided in an attempt to achieve a low crack spacing, in the process ductility has been lost.

The most common type of ultimate failure was a diagonal failure, linking many cracks together, the longitudinal length of which ranged between 25-100 mm. More rarely, failures occurred normal to the applied stress with occasional longitudinal splitting.

5.3. Interpretation of AE data for Composites Tested at 28 days

Figures 5.4.-5.8., 5.10.-5.13. and 5.15. also show the AE (cumulative count) as a function of load and strain in addition to the associated load-strain data previously discussed. Figures 5.9., 5.14. and 5.16.-5.25. show similar load-strain data but with the AE (rate count) as a function of strain.

5.3.1. Load/AE (cumulative count)

The necessity of reducing the size of the original A3 plots for thesis presentation has reduced the clarity of the point of first emission. However, emission generally commenced at a lower load than that of the "first crack" (see 2.4.2.). This is clearly shown in Figures 5.7. and 5.11. Although it is not possible to categorically state the exact micromechanical features associated with the initiation of AE it is likely to be related to the stable growth of micro-cracks. AE counting is a function of the test itself, since not only total counts or rates are affected by the amplifier gain but also the onset of counting, unless the micromechanical feature is heralded by a massive, sudden burst of emissions.

Figure 5.11. highlights a number of difficulties of interpreting AE data for brittle matrix composite materials. The load/AE plot approximates to the shape of the load/strain curve. It can be
Fig 5.16  Typical Load - Strain - Acoustic Emission Data

Film - S8, Vf = 6.6%
Fig 5.17  Typical Load - Strain - Acoustic Emission Data
Film - BAR 21, $V_f = 6.7\%$
Fig 5.18  Typical Load - Strain - Acoustic Emission Data

Film - BAR 112. $V_r = 4.1\%$
Fig 5.19  Typical Load - Strain - Acoustic Emission Data
Film - BAR 112, $V_f = 7.7\%$
Fig 5.20  Typical Load - Strain - Acoustic Emission Data

Film - BAR 112, $V_f = 11.5\%$
Fig 5.21  Typical Load - Strain - Acoustic Emission Data
Film - BAR 113, $v_f = 7.8\%$
Fig 5.22  Typical Load - Strain - Acoustic Emission Data

Film - BAR 113, $V_f = 12.0\%$
Fig 5.23  Typical Load - Strain - Acoustic Emission Data

Film - E3H, $V_f = 3.7\%$
Fig 5.24  Typical Load - Strain - Acoustic Emission Data

Film - E3H, $V_f = 6.5\%$
Fig 5.25 Typical Load - Strain - Acoustic Emission Data

Film - Monofilament, \( V_f = 15.5\% \)
seen that a large count (the majority associated with multiple cracking) has been registered at the slightly depressed load after the "BOP". However, at this stage little strain has been registered. Two important factors must be borne in mind (a) much initial cracking is likely to occur near the grips which will not be measured as an increase of strain by the extensometer but will register as AE (particularly since the AE transducer was sited close to the top grip, see Figure 4.12 and (b) cracking transforms the composite into an essentially discontinuous medium with severe attenuation of the AE signals. Signal attenuation is probably the reason why the total count at failure decreases with increases in film volume fraction as shown in Figures 5.6. and 5.8.

5.3.2. Strain/AE (cumulative count)

Consistent interpretation of this data is complicated by the changing form of the curve with film volume fraction (e.g., see Figures 5.6. - 5.8.). At low volume fractions the slope of the curve is, in general, approximately constant to failure, whereas at higher volume fractions there is a distinct change in slope, albeit through a gradual transition. Since this data was only gathered to aid identification of the end of multiple cracking it is not very useful in clarifying cracking mechanisms and will not be considered further.

5.3.3. AE (rate count)/strain

In contrast to the strain/AE (cumulative count) plots the AE (rate count) yields a consistent form of curve. There are strictly three zones to the curve although the first (pre matrix cracking) is indistinct due to its small length, i.e., less than 0.05%. The multiple cracking region is characterised by a high rate count although it shows a decrease with strain, possibly attributable to the previously discussed attenuation. The post crack region is typified by a much smaller rate count which increases towards composite
failure. Even though the relative magnitudes of the second and third zones change the form of the curve is similar. The major change is a decrease in the rate count with an increase in film volume fraction (within each film type).

The applicability of AE (rate count) is admirably demonstrated in Figure 5.19. The strain at the end of multiple cracking is indistinguishable on the load/strain plot. However, the AE (rate count) data indicates a change of form at approximately 2% strain. It can be seen on both plots, that, multiple cracking continues after this strain, the AE (rate count) suggesting that it terminates at approximately 3.4% strain. It is suggested that, in this case, the "post crack" curve started at 2% with further cracking occurring due to a mechanism to be discussed in Chapter 7.

Examination of all the AE (rate count) data (including those not presented) indicates that, at constant film content the magnitude of the rate count may divide the films into two categories. Using Figures 5.16. and 5.17. as examples it can be seen that S8 composite emits signals at a faster rate, in the "post crack" region, than does BAR 21 composite. The major difference between these films lies in the voiding of S8. In fact, this distinction holds for the other films. The voided films exhibit a higher AE (rate count) than the non-voided films when compared at constant film volume fraction.

5.3.4. Kaiser Effect.

The concept of the Kaiser Effect states that no AE is generated if a material is subsequently reloaded to a lower load than had previously been applied. The effect applies within the elastic region but less well when the elastic limit has been exceeded. Further, for some mechanical processes, e.g., frictional sources, the Kaiser Effect is inappropriate (Arrington, 1982).
Examination of Figures 5.4.-5.25. shows for those strips which were unloaded, that immediately upon unloading, AE was halted. In some cases, AE resumed as the load approached zero. The magnitude of this AE (rate count) was always less than that occurring immediately prior to unloading. Possible sources for the emissions may be debris within the cracks or mismatch of opposite faces. The buckling of fibrils crossing cracks has been observed in strips exhibiting a wide residual crack opening. Upon reloading, AE is initiated at a lower stress and strain than that at which the strip was unloaded. Therefore, the material does not obey the Kaiser Effect.

It is perhaps surprising that AE is not registered during the complete unload-reload cycle since frictional sources are said not to obey the Kaiser Effect. It might be imagined that the relative movement of fibre and matrix would generate AE. Since the magnitude of recorded AE signals is gain dependent it may be that a gain of 86 dB did not amplify the frictional signals above the threshold voltage. If this is the case and fibre-matrix interfacial AE is being continuously generated then an alternative source may be responsible for the AE on the post crack curve.

Other sources may include creep and sustained loading effects on the matrix, damage to the fibres or machine noises. Of these, it is suggested that fibre damage is the most likely source. The magnitude of the AE (rate count) increases with strain as fibre and composite fracture is approached although the absolute value will be affected by the fracture location and signal attenuation.

In section 5.3.3, it was stated that the AE (rate count) was higher for the voided films and it is now suggested that this may be related to greater fibre damage occurring in the voided films. In Chapter 7 this will be related to fibre structure and to the mechanisms of fibre-matrix interaction.
5.4. Typical Initial Load/Strain Data for Composites Tested at 28 Days

The form of the initial Load/Strain curve (\(< 0.1\%\)) is not so visibly affected by film modulus and volume fraction as that of the complete curve. Figure 5.26. shows a typical curve and a number of points of detail should be noted.

The strain differential, across a section, mainly due to the bowing of the strip can be clearly seen. The degree of bow is important since it appears to affect the strain (on the most strained face) at which AE is initiated, point A on Figure 5.26. Figure 5.27. shows this strain to be increased with increases in the strain differential. Consequently, the strain at which AE is initiated may not be considered a basic material parameter. It should be remembered that no precise micromechanical significance can be attached to the onset of AE and, also, that the location of the initial AE burst cannot be located with the single transducer system employed.

In 5.2.2. the variability of the load at which matrix cracking occurred was discussed. This is clearly illustrated in Figure 5.26. The first five cracks occurred outside the gauge length, B, probably close to the grips. The first crack within the gauge length subsequently propagated at a load, 1.06 KN, lower than that which the section had previously sustained.

The uncracked composite modulus was found by taking the slope of an average load/strain curve derived from the linear portions of the individual curves. The matrix modulus was calculated using equation 2.3. and assuming the film modulus to be the value measured at 0.1\% strain (see Figure 5.1.).

\[ E_c = E_m V_m + E_f V_f \]
Figure 5.26. Typical Tensile Load/Strain Data Measured on Opposite Faces of a Tensile strip
Figure 5.27. Experimental Relationship Between the Maximum Strain at AE Initiation and the Strain Differential across the Tensile Strip
The average value for $E_m$ was calculated to be 31.5 GPa (standard deviation - 3.5 GPa).

5.5. Typical Load/strain/AE Data for Composites containing Unopened Film and Tested at 28 Days

Two sheets were made with film BAR 113 in which the films were unopened and typical load/strain /AE data are shown in figures 5.28. and 5.29. Both composites exhibit distinctly different regions of post crack linearity. At approximately 2% strain, A, (~15% $V_f$ composite, Figure 5.28.) the effective film modulus shows an increase from 2.62 GPa to 2.92 GPa, the difference being statistically significant. The behaviour of the higher film content composite (~25% $V_f$, Figure 5.29.) is yet more complex, exhibiting 3 post crack slopes. Between approximately 0.4% to 0.8% strain, B, the effective film modulus is 3.36 GPa falling to 2.43 GPa, C, (approximately 1.1% to 1.9% strain). From 1.9% strain, D, the effective film modulus shows an increase to 2.69 GPa. The differences are statistically significant.

If Figure 5.11. is re-examined in the light of these results it can be seen that linearity is not achieved until approximately 2% strain. However, this is not as apparent as the distinctly linear features shown by the unopened film composites.

It is believed that this behaviour is simply a reflection of the film behaviour as shown in Figure 5.1. Sufficient film has been incorporated to ensure that the strain in the film across the cracks is low enough to reveal the subtleties of the film tangent modulus.
Fig 5.28  Typical Load - Strain - Acoustic Emission Data

Film - BAR 113 (Unopened), $V_f = 14.5\%$
Fig 5.29  Typical Load - Strain - Acoustic Emission Data

Film - BAR 113 (Unopened), $V_f = 23.4\%$
Three features of the AE data may be noted. The burst of activity at approximately 6% strain (Figure 5.28.) was caused by the development of a longitudinal split in the strip. Figure 5.29. illustrates severe attenuation of the signal during multiple cracking probably resulting from the higher film volume fraction. Further, the portion of the AE (rate count) data relating to post crack behaviour exhibits a feature which is not believed to be a material parameter. The small rise and subsequent slow fall in signal resemble the charge/discharge of an electrical component. It should be remembered that each count cycle was for only 100 ms before resetting to zero, hence, the response is too slow to be real.

5.6. Typical Load/Strain/AE Data for Composites Tested at 1 Year

Figures 5.30. and 5.31. show typical data for BAR 112 and BAR 113 composites respectively (film opening - 4 times) and the data show similar characteristics to the 28 day results. However, whilst the load/strain curves exhibit the usual, in batch, variations, the general trend is for the multiple cracking region to show an continually increasing load where as 28 day composites cracked at essentially constant load. Further, cracking on the "post crack" curve was more prominent. These features were more apparent for BAR 112 composite than for BAR 113. As a consequence, the comparison of 28 day and 1 year data is rather difficult. For instance, the end of multiple cracking is even more difficult to define than at 28 days, such that the average composite cracking stress, determined at the strain \( e_{\text{mc/2}} \), is less easy to estimate (with a rising multiple cracking load this becomes more significant). As a result, the determination of average matrix failure strain and stress become less certain than at 28 days. The latter parameter, in turn, affects the determination of fibre-matrix stress transfer (see 7.4.2.).
Fig 5.30  Typical Load - Strain - Acoustic Emission Data
Film - BAR 112, $V_f = 6.0\%$, Age - 1 Year
Fig 5.31  Typical Load - Strain - Acoustic Emission Data

Film - Bar 113, $V_f = 7.2\%$, Age - 1 Year
It has been previously stated - section 5.2.2. - that an increasing multiple cracking region could occur in 28 day composites. The difficulty in interpretation for the 1 year composites lies in the fact that the slopes of the multiple cracking and post crack regions are similar, whereas, a distinct discontinuity occurred for those 28 day composites.

Comparison of the BAR 112 composites shows that a slight reduction in failure strain of the composite has occurred with time, falling from approximately 6.1% to 5.7% at 1 year (statistically significant). This is a result of the combined changes in matrix modulus and average matrix failure strain as shown in Table 5.3., rather than a loss in strength of the film (constant at approximately 240 MPa).

<table>
<thead>
<tr>
<th></th>
<th>Age</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>28 Days</td>
</tr>
<tr>
<td>$E_m$</td>
<td>31.6 (1.8)</td>
</tr>
<tr>
<td>$\varepsilon_{mu}$</td>
<td>289 (22)</td>
</tr>
</tbody>
</table>

Table 5.3. Effect of Age on Matrix Properties

Equation 2.23. defines failure strain, $\varepsilon_{cu}$, as,

$$
\varepsilon_{cu} = \varepsilon_{fu} - 0.341 \frac{E_m \varepsilon_m}{E_f V_f} 
$$
Approximating $\varepsilon_{\text{mu}}$ by $\bar{\varepsilon}_{\text{mu}}$, this equation shows that the combined increase of $E_m \varepsilon_{\text{mu}}$ predicts a decrease in composite failure strain.

An unexplained phenomena which occurred in many of the 1 year composites is illustrated in Figure 5.32, which represents the initial load/strain/AE (cumulative count) data until the first crack propagates. It can be seen that the AE has been initiated at a very low load ($\sim 0.5$ KN) in contrast to that for the 28 day composites and was not necessarily accompanied by a departure from linearity of the load/strain curves. The matrix stress at AE initiation ranged between 6.8 MPa and 10.8 MPa at 28 days in contrast to 2.4 MPa and 9.6 MPa at 1 year. As previously discussed, it is not possible to ascribe a micromechanical feature to AE initiation and no such explanation can be offered.

During the tests it was noted that the audibly translated signals from the AE processing unit had a different character to those emitted from the 28 day composites. The emissions sounded "sharper", producing a "pinging" noise rather than the sound of "sand paper on wood". It is possible that these signals suffered less attenuation than within the 28 day composite, and hence, exceeded the threshold voltage at a lower load (see 5.3.1.).

5.7. Pull Out Tests of Narrow Film Strips

Such tests as these are normally performed to evaluate a "bond" strength expressed in MPa. For a simple monofilament the perimeter of the fibre may be relatively easily determined and an "average bond" strength calculated. However, glass fibre strands and voided polyalkene films present difficulties in ascertaining the interfacial contact area. In order to overcome this problem the results will be presented in the form of load/unit length of embedded film assuming a constant
Figure 5.32. Typical Load/Strain/AE Data for 1 Year Old Composite containing film BAR 113
lateral dimension. This parameter will be termed "shear flow", after Bartos (1980).

The results of pull out tests are notoriously variable, with coefficients of variation of up to 50% being commonly reported. The variability of these tests compares favourably with previous work, the maximum coefficients of variation for BAR 112 and BAR 113 being 31% and 15% respectively.

Figure 5.33 shows the "best" and "worst" results for both films whilst the average results are presented in Figure 5.34. The results are presented up to a crosshead displacement of 2 mm (an arbitrary choice), the greater variability of BAR 112 is probably due to shear in the film causing a loss of integrity of the film structure. It will be remembered that in the modulus tests on BAR 112 fibrillation of the film commenced at a lower strain than that encountered in BAR 113. The "best" BAR 112 was obtained as a result of little shear occurring within the area of interest. BAR 112 could never be pulled out at the end of a test whereas BAR 113 could.

Examination of the Instron charts indicates that static friction was overcome at a shear flow of approximately 4N/mm for BAR 113 specimens. Only 2 out of 15 specimens showed a decrease in load immediately slip occurred, and this decrease was only temporary, since the load increased with further relative movement. Hence, unlike many fibres in cement matrices the dynamic frictional fibre-matrix stress transfer is higher than the static case. A shear flow of 5 N/mm is equivalent to an "average bond" strength of approximately 0.8 MPa.

Analysis of the BAR 112 specimens is complicated by the structure of the voided film. Unlike the glass strands which present a constant, if unknown, contact area to the matrix, a voided film presents an unknown and changing area along its length. This results from the random distribution
Figure 5.33. Shear flow Data from Narrow Strip Pull Out Tests showing the Range of Results Obtained.
Figure 5.34. Average Shear Flow Data for films BAR 112 and BAR 113 (average of 15 strips each)
of voids along the film length. Hence, in some specimens the film area at the entry point is low (i.e., same as BAR 113) and in others it will be much higher. Consequently, the shear flow at first slip is likely to vary considerably. Subsequent to slip, the behaviour depends upon the structural integrity of the film. However, possibly the most important result is that, BAR 112 exhibits the potential for a more rapid increase in shear flow with respect to relative movement of fibre and matrix.

5.8. Matrix Work of Fracture

These tests were carried out on a test rig developed in the Metallurgy and Materials Science Department for similar work of fracture tests on cement pastes. Figure 5.35. shows a typical load/deflection curve obtained. It will be noted that immediately after the maximum load the rate of decrease in load is gradual, implying stable crack growth.

The load/deflection data for all specimens was input manually to a PET microcomputer and the work of fracture calculated, after allowance had been made for kinetic effects.

The matrix work of fracture, \( \gamma_m \), was determined to be 5 J/m\(^2\), coefficient of variation - 13%.

5.9. Matrix Control specimens

The average flexural strength was 13.3 MPa with a coefficient of variation of 10%.
Figure 5.35. Typical Load/Deflection Data for a Notched beam
5.10. Summary

The typical results presented in this chapter show that the experimental data broadly reflect the predictions of Aveston et. al. (1971). In the two subsequent chapters the more contentious question of predicting enhanced matrix failure strain and the source of fibre-matrix stress transfer will be discussed.
6.1. Experimental Determination of Matrix Failure Strain

In developing failure models to predict matrix failure strain, $\varepsilon_{mu}$, it is normally assumed that the stress-strain behaviour is linear until the first crack propagates catastrophically. It is also assumed that there is a uniform strain distribution across the composite. Figure 5.26 shows that these assumptions are inapplicable to the composites under discussion and therefore some judgement has to be used in the choice of strain parameter to be obtained from the experimental stress-strain curve for comparison with theoretical predictions. A similar judgement was made by Aveston et. al. (1974) who examined the acoustic emission, AE, behaviour of steel fibre cements undergoing multiple fracture. They associated maximum cracking with maximum acoustic emission and determined an average matrix failure strain, $\bar{\varepsilon}_{mu}$, as follows

$$\bar{\varepsilon}_{mu} = \frac{\bar{\sigma}}{E_c}$$

(6.1.)

where $\bar{\sigma}$ is the average composite cracking stress, coinciding with maximum acoustic emission.

Aveston et. al. (1974) stated that the maximum acoustic emission, for steel fibre cement, occurred at the minimum slope of the stress-strain curve. Examination of the published stress-strain curve suggests that the minimum slope occurs approximately in the middle of the multiple cracking region. Hence, it was decided in this work to calculate the average composite cracking
stress at a strain equal to a half of the strain at the completion of multiple cracking, i.e., at $\varepsilon_{mc/2}$. In comparison with glass reinforced cement and steel fibre cements, the slope of the multiple cracking region of polyalkene cements is relatively shallow. Thus, the range of stress over which cracking occurs is more restricted and errors so reduced.

Whilst it is realised that the average matrix failure strain calculated in this way is not ideal for comparison with theoretical predictions, it is the most representative parameter available from a tensile strip specimen.

In order to ensure that results were only obtained from samples with a relatively uniform fibre distribution, those composites with different crack spacing on each face were discarded.

6.2. Experimental Values of Average Matrix Cracking Strain and Existing Theoretical Predictions of Enhanced Matrix Cracking Strain

6.2.1. Experimental Results

Figures 6.1.-6.5. show both the experimental values of the average matrix failure strain and the theoretical values of enhanced matrix failure strain as predicted by the ACK (Aveston et. al., 1971) and Nottingham (Korczynskyj et. al., 1981) models for films BAR 112, BAR 113, E3H, S8 and BAR 21 respectively. It is apparent that the incorporation of films of differing physical properties has affected the average matrix failure strain. As is common with brittle materials, the scatter of results is substantial and tends to mask small differences, which may possibly exist,
Figure 6.1. Relationship between the Experimentally Determined Average Matrix Failure Strain and the Theoretically Predicted Enhanced Matrix Failure Strain with Film volume Fraction for BAR 112 composites
Figure 6.2. Relationship between the experimentally determined average matrix failure strain and the theoretically predicted enhanced matrix failure strain with film volume fraction of BAR113 Comp.
Figure 6.3. Relationship between the Experimentally Determined Average Matrix Failure Strain and the Theoretically Predicted Enhanced Matrix Failure Strain with film Volume Fraction for E3H Composites.
Figure 6.4. Relationship between the Experimentally Determined Average Matrix Failure Strain and the Theoretically Predicted Enhanced Matrix Failure Strain with Film volume Fraction for film S8 composites.
Figure 6.5. Relationship between the Experimentally Determined Average Matrix Failure Strain and the Theoretically Predicted Enhanced Matrix Failure Strain with Film Volume Fraction for BAR 21 Composites
between BAR 112, BAR 113 and BAR 21. Nevertheless, it can be seen that the average matrix failure strain is increased by increases in film modulus and volume fraction. For example, BAR 112 (initial modulus, 8.7 GPa) yields an average matrix failure strain of approximately $275 \times 10^{-6}$, $310 \times 10^{-6}$ and $360 \times 10^{-6}$ at 5%, 8% and 11% film volume fractions, respectively. In contrast, E3H (initial modulus, 29.5 GPa) yields an average matrix failure strain of $275 \times 10^{-6}$, $325 \times 10^{-6}$ and $400 \times 10^{-6}$ at 3%, 5% and 8% film volume fractions respectively. Both films possess a similar specific fibre surface area, a major difference lies in the initial value of modulus.

This is a very important result for the development of a commercial composite, since its effect would be to increase the working stress range of a cladding element before cracking was apparent. However, to be able to design suitable composites, it is necessary to possess a model which is capable of predicting the enhanced matrix failure strain. The ACK and Nottingham models will now be examined in the light of experimental results.

6.2.2. Theoretical Predictions

The film and matrix parameters required by both models are described below:

(a) Matrix modulus, $E_m = 31.5$ GPa

(b) Film modulus, $E_f$ - tangent modulus at 0.1% strain.

BAR 112, $E_f = 8.7$ GPa
BAR 113, $E_f = 7.7$ GPa
BAR 21, $E_f = 7.9$ GPa
E3H, $E_f = 29.5$ GPa
S8, $E_f = 12.0$ GPa.
(c) Bond strength, \( \tau = 0.5 \text{ MPa} \) (see Chapter 7 for full discussion).

(d) Effective fibre radius, \( r \), given by \( r = \sqrt{\frac{2}{SFS}} \).

\[
\begin{align*}
\text{BAR 112, } r &= 6.4 \mu\text{m} \\
\text{BAR 113, } r &= 23 \mu\text{m} \\
\text{BAR 21, } r &= 12 \mu\text{m} \\
\text{E3H, } r &= 6.3 \mu\text{m} \\
\text{S8, } r &= 8.7 \mu\text{m}
\end{align*}
\]

(e) Matrix work of fracture, \( \gamma_m = 5 \text{ J/m}^2 \).

(f) Matrix failure strain, \( \varepsilon_{\text{mu}} \), i.e., at film volume fraction = 0%. The values of average matrix cracking strain for BAR 112, BAR 113 and BAR 21 are very similar within the experimental scatter. Using a linear regression analysis of all these results (\( \sim 150 \)) a strain value of \( 222 \times 10^{-6} \) (0.0222\%) was obtained.

(g) Griffith half crack length, \( c = 2.05 \text{ mm} \).

The experimental results (Figures 6.1.-6.3.) indicate that the average matrix failure strain is enhanced even at relatively low film volume fractions of 3\%. This is in contrast to the ACK model which predicts no enhancement of failure strain at film volume fractions of less than 15\% for BAR 113. Clearly, the Nottingham model is a better fit to the experimental data, even though, in general, the prediction is lower than the experimental data.

We shall now examine each model in further detail to determine the precise differences between the two approaches and to investigate the ways in which the predictions can be improved.
6.2.3. Examination of ACK and Nottingham Models

6.2.3. (a) ACK Model (Section 2.4.4.)

In Section 2.4.4. it was stated that the ACK model considered the energy balance before and after the complete, instantaneous propagation of a crack. The crack width, \( b \), is given by,

\[
b = (1 + \alpha)e_m \mu x'
\]

for crack spacing of \( 2x' \), where \( x' \) is the minimum crack spacing.

Assuming typical values, \( E_m = 31.5 \text{ GPa}, E_f = 3 \text{ GPa}, \)
\( V_f = 7\% , \epsilon_m = 0.0222\% \) (i.e., as predicted by ACK),
\( x' = 1 \text{ mm} \) the crack width is approximately \( 30 \mu\text{m} \). The width of the Griffith crack in the unreinforced matrix, \( B \), is given by,

\[
B = \epsilon \pi c
\]

for plane stress.

Assuming the same value of matrix failure strain, the crack width is approximately \( 1.4 \mu\text{m} \) at the centre of the crack.

It is likely that the critical crack in the composite will be of a similar size to the Griffith crack. This crack will only attain its ACK width after the critical flaw has propagated and the faces of the crack moved apart, such that the fibres are strained to carry the load which the composite carried prior to crack propagation. Consequently, ACK over estimates the work done against friction, the increase in strain energy of the fibres and the work done separating the crack faces.
Aveston, Mercer and Sillwood (1975) recognised the link between the Griffith crack width and the ACK crack width but suggested that the matrix would crack at the same strain as the unreinforced matrix until \((1 + \alpha)\varepsilon_{\text{mu}}^{\text{X}} < B\). This would further increase the film volume fraction lower than which no enhancement would be expected for the polyalkene cements examined herein. It is considered that the ACK and AMS approaches have not adequately considered the energy balance for a crack at the instance of propagation. Consequently, the ACK model will not be considered further. However, it should be noted that the basic mechanisms of energy absorption are still valid.

6.2.3. (b) Nottingham Model (Section 2.4.5.)

The assumption of an elliptical strain relief zone around the Griffith crack is reasonable for an unreinforced brittle material. However, the assumption that the size of the relief zone remains unaltered by the inclusion of fibres is probably unrealistic. It is this assumption that governs the strain distributions within the zone and it is particularly difficult to imagine the physical reason for the distance, \(L_1\) to \(L_2\) (see Figure 2.6.) where the strain in the fibre and matrix is assumed to remain constant. It is more logical to consider that the zone would become smaller, as the film volume fraction is increased, as a result of the more rapid transfer of load back into the matrix. However, the Nottingham approach appears to simulate the propagation of a crack more satisfactorily than does ACK.

6.3. General Examination of Cracking in Brittle Matrix Composites

An attempt to rationalise the failure of fibre reinforced cements has been made by Hannant, Hughes and Kelly (1983). Their approach was to examine the surface to surface fibre spacing in relation to the Griffith crack length in order to establish whether one or more fibres would always be present within a Griffith crack.
In the unreinforced matrix the failure strain, $\varepsilon_{\text{mu}}$, is given by,

$$
\varepsilon_{\text{mu}} = \left[ \frac{G}{\pi E_m c} \right]^{\frac{1}{2}}
$$

for plane stress

and the crack width is as equation (6.3.)

Figure 6.6. Showing the Fibre Spacing and the Griffith Crack

If fibres are introduced with a surface to surface spacing, $S$, much greater than the Griffith crack length, $2c$ (Figure 6.6.) then the crack is likely to advance unstably before it encounters fibres. When the crack passes the fibres, the usual energy
absorbing mechanisms will operate and, in principle, the fracture
toughness, to complete failure, will be slightly increased. It
is unlikely that this will substantially affect the matrix cracking
strain. Consequently, it is important to ascertain when this
state of affairs is altered, i.e., when $S \leq 2c$.

The surface to surface fibre spacing is given by

$$S = 2r \left\{ \left[ \frac{\alpha'}{V_f} \right]^{\frac{1}{2}} - 1 \right\}$$

..... (6.5.)

where $\alpha' = 0.912$ for a hexagonal fibre array and
$\alpha' = 0.785$ for a square fibre array.

To obtain the condition, $S \leq 2c$, we can equate equations
(6.4.) and (6.5.), i.e.,

$$\frac{2G}{E_m \pi \varepsilon_{mu}^2} \geq 2r \left\{ \left[ \frac{\alpha'}{V_f} \right]^{\frac{1}{2}} - 1 \right\}$$

..... (6.6.)

Thus, using typical values for BAR 113 $E_m = 31.5$ GPa,
$\varepsilon_{mu} = 222 \mu e$, $r = 23 \mu m$ and $G = 10$ J/m$^2$,
equation (6.6.) is satisfied by film volume fractions in excess
of 0.01%. In a similar matrix, a steel fibre cement ($r = 66 \mu m$)
requires a fibre volume fraction of at least 0.09%. In contrast,
for a carbon fibre reinforced pyrex glass of $E_m = 70$ GPa,
$\varepsilon_{mu} = 0.14\%$, $r = 4 \mu m$ and $G = 8$ J/m$^2$ (Aveston et. al., 1971)
the minimum fibre volume fraction is approximately 3%. The latter result is basically because the Griffith crack is so short for the pyrex glass.

Thus, polyalkene cements would be expected to show an enhanced matrix failure strain for all practical film volume fractions.

6.4. New Model for Estimating the Enhanced Matrix Failure Strain of Brittle Matrix Composites

6.4.1. Development of the Model

The model is essentially a development of the Nottingham analysis. The same assumption is made concerning the shape and size of the relief zone around the Griffith crack in the unreinforced matrix, i.e., elliptical with a major axis of length 3 times that of the Griffith crack. The rate of increase of strain in the matrix with distance from the crack face is given by \( \frac{\varepsilon_m u}{L_3} \)

where \( L_3 \) is the distance from the crack face to the edge of the elliptical zone.
It is now assumed (incorrectly) that the presence of fibres does not alter the length of the critical crack. The assumption is incorrect since it is likely that the fibres will divert the crack or arrest it (Cook and Gordon, 1964), but not restrict its length to the interfibre spacing (Hannant et al., 1983). However, upon straining the composite, the crack width is related to the requirement that plane sections remain plane. Thus, only one crack width is permissible for a given value of matrix strain and is a function of the rate of increase of strain in the matrix with distance from the crack which is itself controlled by fibre parameters. It is also assumed that the fibres are aligned and linear elastic and the fibre-matrix bond strength is constant.

The method of analysis is similar to that of the Nottingham model, the crack being divided into a number of segments of width, $\delta y$, distance $y$ from the centre of the crack. Any interactions between segments are ignored. Figure 6.8 shows the longitudinal strain distributions in the fibres and matrix perpendicular to the plane of the crack.
Figure 6.8. **Longitudinal Strain Distributions Along a Section Perpendicular to the Crack Face**

The maximum strain in the fibre, \( \varepsilon_f' \), occurs across the crack. Load in the fibre is transferred back into the matrix by relative displacement of the fibre and matrix between 0 and \( L_1 \). The rate of change of strain in the fibre and matrix are shown by AB and OA respectively and are given by

\[
\frac{d\varepsilon_f}{dx} = \frac{-2\tau}{E_f r} 
\]  

..... (6.7.)
The maximum fibre strain, $\varepsilon'_f$, is given by,

$$\varepsilon'_f = L_1 \left( \frac{d\varepsilon_f}{dx} + \frac{d\varepsilon_m}{dx} \right) \quad \cdots \quad (6.9.)$$

and also,

$$\varepsilon'_f = \frac{B^{'}}{L_1} \quad \cdots \quad (6.10.)$$

where $B'$ is the crack width at distance $y$ from the centre of the crack.

At the position $L_1$, the strains in the fibre and matrix, $\varepsilon_r$, are equal and $\varepsilon_r$ is given by,

$$\varepsilon_r = \frac{d\varepsilon_m}{dx} \quad \cdots \quad (6.11.)$$

Between $L_1$ and $L_2$ the strain in the fibre and matrix increases together at a rate, $AC$, corresponding to the unreinforced matrix, $\varepsilon'_{mu}/L_3$. 
The distances \( L_1 \), \( L_2 \), and \( L_3 \) are given by,

\[
L_1 = \left[ \frac{B^*}{\frac{d\varepsilon_f}{dx} + \frac{d\varepsilon_m}{dx}} \right]^{\frac{1}{2}}
\]

\[ \cdots \quad (6.12.) \]

\[
L_2 = L_1 + (\varepsilon_{\text{mu}} - \varepsilon_r) \frac{\varepsilon_{\text{mu}}}{L_3}
\]

\[ \cdots \quad (6.13.) \]

\[
L_3 = 3(c^2 - y^2)^{\frac{1}{2}}
\]

\[ \cdots \quad (6.14.) \]

The first stage of the analysis is an iterative process to calculate the crack width, \( B^* \), for which the total extension of position \( L_2 \) inside and outside the relief zone is the same.

\[
i.e., \ v_{\text{mu}} L_2 = \frac{B^*}{2} + \frac{\varepsilon_r L_1}{2} + \left( \frac{\varepsilon_{\text{mu}} + \varepsilon_r}{2} \right) \left( L_2 - L_1 \right)
\]

\[ \cdots \quad (6.15.) \]

A crack width is deemed acceptable when the two extensions are calculated to be within \( \pm 1\% \) of each other. Thus, the solution is not exact, since it will depend upon the precision of the comparison of the extensions within the preset limits.
Once the shape of the crack has been determined, the strain energy released, $U_r$, by a segment of width, $\delta y$, and unit thickness at a distance, $y$, from the centre of the crack may be calculated.

$$U_r = U_i - U(0 - L_1) - U(L_1 - L_2)$$

..... (6.16.)

where,

$$U_i = \frac{(\varepsilon_{mu})^2}{2} E_c \frac{L_2 \delta y}{\delta y}$$

..... (6.17.)

$$U(0 - L_1) = \frac{E_m V_m \varepsilon_f^2 L_1}{6} \delta y + \frac{E_f V_f (\varepsilon_f^2 + \varepsilon_r^2 + \varepsilon_r^2)L_1 \delta y}{6}$$

..... (6.18.)

$$U(L_1 - L_2) + \frac{E_c (\varepsilon_{mu})^2}{2} \left[ \frac{L}{3} + \frac{L_3^2}{3} - \frac{L_2^2}{L_3} \right] \delta y$$

..... (6.19.)

where,

$$L = L_2 - L_1$$

Between 0 and $L_1$, work is also done as a result of friction between the fibres and matrix, $U_f$, and is given by,

$$U_f = \frac{V_f \varepsilon_f^2 L_1^2}{3} \delta y$$

..... (6.20.)
Energy is also absorbed at a rate, $G V_m$, by the crack surfaces.

The crack will propagate when the rate of release of strain energy is infinitesimally greater than the rate of absorption. The total decrease in elastic strain energy is obtained by numerically integrating equation (6.16) along the crack. This is repeated for a slightly larger value of $c$ and then the rate of release of energy is obtained by dividing the difference between the two energies by the crack extension. The rate of absorption is similarly obtained from equation (6.20) to which is added the contribution from the crack surfaces.

6.4.2. Theoretical Predictions of Enhanced Matrix Failure Strain Using the New Model

The crack length was divided into 20 segments, i.e., $\delta y = 0.1c$, and was incremented by 10% of its original length. Using the same parameters as defined in section 6.2.2., values of enhanced matrix failure strain were calculated and are shown in Figures 6.9.-6.13. for films BAR 112, BAR 113, E3H, S8 and BAR 21 respectively. The theoretical predictions are superimposed upon the experimentally determined average matrix failure strain.

It can be seen that, in general, the fit to the experimental data is very good and is better than the predictions of the ACK and Nottingham models (Figures 6.1.-6.5.). In the case of E3H (Figures 6.3. and 6.11.) the prediction would appear to be rather high whereas the Nottingham model yields a rather low value. Only the prediction for BAR 112 (Figure 6.9.) would appear to have become less certain. However, this may be related to the value of bond strength, $\tau = 0.5$ MPa, assumed to be relevant to the voided film.
Figure 6.9. Predicted Values of Enhanced Matrix Failure Strain of BAR 112 Composites Using the New Model
Figure 6.10. Predicted Values of Enhanced Matrix Failure Strain for BAR 113 Composites Using the New Model.
Figure 6.11. Predicted Values of Enhanced Matrix Failure Strain for E3H Composites Using the New Model
Figure 6.12. Predicted Value of Enhanced Matrix Failure Strain for s8 composite using the New Model
Figure 6.13. Predicted value of Enhanced Matrix Failure Strain for BAR 21 Composite using the New Model
In Chapter 5 it was shown that, for both voided and non-voided films, the dynamic shear flow (load/unit length) increases after the first slip of the film, with the voided film, BAR 112, showing the greater rate of increase. Although there is no direct relationship between results from pullout and multiple cracking experiments it will be suggested in Chapter 7 that the "average bond" strength, $\tau$, also increases with relative displacement, and at a greater rate for the voided film. Since the "average bond" strength is similar for both films at large relative displacements, i.e., the completion of multiple cracking, it is suggested that the "average bond" strength relevant to the first cracking strain may be lower for BAR 112 than for BAR 113 due to the low relative displacement and therefore two lines are shown on Figure 6.9. to clarify this effect.

This feature may apply to the other voided films and will be discussed in greater detail in Chapter 7.

Figure 6.14. shows the predicted values of enhanced matrix failure strain for both the new model and the Nottingham model assuming an "average bond" strength of 0.25 MPa. The fit for the new model has greatly improved and the Nottingham model yields a lower result as displayed by the other films.

The results of the new model suggest that it is applicable to polyalkene cements. A comparison will be made of the strain distributions and crack widths predicted for polyalkene cements by the new model and the Nottingham model before the more general applicability to cement matrix composites is tested.
Figure 6.14. Predicted Values of Matrix Failure Strain for BAR 112 Composites assuming "Average Bond" Strength = 0.25 MPa
6.5. Comparison of the New Model and the Nottingham Model

For the purpose of this comparison only composites containing BAR 113 will be considered at film volume fractions of 5% and 25%.

The Nottingham model predicts enhanced matrix failure strains of $237 \times 10^{-6}$ and $371 \times 10^{-6}$ for film volume fractions of 5% and 25% respectively. For identical quantities of film the new model predicts enhanced matrix failure strains of $262 \times 10^{-6}$ and $452 \times 10^{-6}$ respectively. The predicted longitudinal strains on a plane perpendicular to the crack face are shown in Figures 6.15. and 6.16. for the Nottingham model and the new model respectively. The plane is located at a distance 0.05 c from the centre of the crack, i.e., at the centre of the segment adjacent to the crack centre.

Whilst both models assume that plane sections remain plane, the Nottingham model assumes a constant distance from the crack face at which the equality is maintained, i.e., at the edge of the fixed elliptical zone, $L_3$. This is shown in Figure 6.15. where $L_3 = 6.14$ mm for both film volume fractions. However, the new model establishes the equality by an iterative process and the size of the relaxation zone is shown to decrease with increases in film volume fraction from $L_2 = 5.34$ mm at 5% to $L_2 = 3.58$ mm at 25% volume fraction. This distance over which relative slip occurs is similar for both models and film volume fractions at approximately 0.5 mm.

The major objection raised against the Nottingham model is the portion $(L_1 - L_2)$ of constant strain at a value less than the strain in the bulk of the composite. It can be seen in Figure 6.15. that the distance at which strain commences to rise again increases from 1.42 mm at 5% to 4.09 mm at 25% film volume fraction.
Figure 6.15. Strain Distribution along a Section 0.05C from the Centre of the Crack as Predicted by the Nottingham Model.
Figure 6.16. Strain distribution along a section 0.05C from the centre of the crack as predicted by the new model.
The fundamental parameter in the new model is that of crack width. The width of the Griffith crack in the unreinforced matrix is 1.43 μm and the Nottingham model predicts a very similar value for both film volume fractions. However, the new model indicates that the critical crack width at unstable propagation may be expected to decrease from 1.26 μm at 5% to 1.04 μm at 25% film volume fraction.

The strain in the fibre at the crack face is predicted to be approximately 0.27% justifying the assumption that the film tangent modulus at 0.1% strain is applicable.

6.6. The General Applicability of the New Model to Brittle Matrix Composites

6.6.1. Limitations on Comparison with Other Materials

It is not possible to accurately compare the new model with other fibre cements since such terms as unreinforced matrix failure strain, matrix modulus, matrix work of fracture, fibre radius are either not precisely known or are not precisely calculable. Also, the accurate experimental determination of average matrix failure strain, made by other researchers has to be assumed. However, results published by authors well recognised in the field are available in the literature and will be compared with the theoretical prediction.

6.6.2. Glass Reinforced Cement

Experimental results have been published by Ali, Majumdar and Singh (1975) and using their parameters, values of enhanced matrix cracking strain have been calculated.
Figure 6.17. Theoretical and Experimental Values of Enhanced Matrix Failure Strain for GRC

\[
\begin{align*}
E_m &= 26 \text{ GPa}, E_f = 76 \text{ GPa}, r = 38 \mu m, \gamma_m = 5 \text{ J/m}^2 \\
\tau &= 3 \text{ MPa}, \epsilon_{mu} = 200 \mu \epsilon \text{ (assumed) and an efficiency factor of 0.27 for the fibre volume fractions (Hannant, 1978).}
\end{align*}
\]

The fit to the water stored GRC is very good.

6.6.3. Steel Fibre Reinforced Cement

Aveston, et. al., (1974) have published values of average matrix failure strain for steel fibre reinforced cement and the results compared favourably with the ACK theory. The prediction from the new model is shown in Figure 6.18.
Figure 6.18. Theoretical and Experimental Values of Enhanced Matrix Failure Strain for Steel Fibre Reinforced Cement

\[ (E_m = 19 \text{ GPa}, \ E_F = 192 \text{ GPa}, \ r = 66 \mu m, \ \gamma_m = 4 \text{ J/m}^2, \]

\[ \tau = 6.8 \text{ MPa and } \varepsilon_{mu} = 500 \mu \varepsilon. \]

The fit of the new model to the experimental data is reasonably good especially since Aveston et al. (1974) stated that the failure strain of the matrix alone lay between 0.02%-0.06%. A strict comparison is further complicated since the value of "average bond" strength was calculated from a multiple cracking experiment. Pinchin and Tabor (1978) have shown that compaction of the fibre-matrix interface upon relative displacement results in a reduction of "bond". Thus, a value of bond strength determined from a multiple cracking experiment may not be strictly applicable to the growth of a Griffith type flaw.
6.6.4. Asbestos Cement

The literature contains very little information on the tensile stress-strain behaviour of asbestos cement. The failure mechanism is still a question for debate, and it is not known whether the material exhibits true multiple cracking or whether cracks are suppressed until the propagation of one crack causes complete failure. Allen (1971) has stated that the ultimate failure strain increases with increases in fibre volume fraction. However, asbestos cement is a difficult system to analyse since increases in fibre volume are accompanied by substantial increases in void content (up to 60%).

Assuming the following parameters, \( E_m = 15 \text{ GPa}, \)
\( E_f = 160 \text{ GPa}, \)
\( r = 4 \mu m, \gamma_m = 4 \text{ J/m}^2, \tau = 2 \text{ MPa} \)
and \( \varepsilon_{mu} = 200 \mu \varepsilon \) and also assuming that the fibres are aligned and linear elastic (failure strength = 320 MPa) the new model predicts fibre failure at \( 855 \times 10^{-6} \) for 5\% fibre volume fraction and \( 1200 \times 10^{-6} \) for 10\% fibre volume fraction. The prediction is actually for ultimate strain since the fibre strength was exceeded across the crack faces, the crack being energetically stable at that strain. Hence, the model suggests that cracking is suppressed in asbestos cement until complete failure.

6.6.5. Carbon Fibre Reinforced Pyrex Glass

The general applicability of the new model is shown by considering carbon fibre reinforced glass. With \( E_m = 70 \text{ GPa}, \)
\( E_f = 380 \text{ GPa}, \)
\( r = 4 \mu m, \gamma_m = 4 \text{ J/m}^2, \tau = 55 \text{ MPa} \)
and \( \varepsilon_{mu} = 0.14\% \) the predicted values of enhanced matrix failure strain are 0.3\% at 23\% fibre volume fraction and 0.35\% at 30\% fibre volume fraction. Aveston et. al., (1971) quote experimental
values of enhanced matrix failure strain of 0.2% and 0.37% at 23% and 30% fibre volume fraction respectively. These results were used to justify the ACK approach. It is interesting to note the similarity between the current predictions and those of ACK which predicts 0.29% and 0.34% respectively for the appropriate fibre volumes. When a crack is heavily constrained the two models approach the same prediction. Korczynskj et. al., (1981) also noted that their model approached ACK as the energy release rate became insensitive to crack length at high degrees of constraint.

6.7. Summary

This chapter has compared two existing models and presented a new model for predicting the enhanced matrix failure strain of brittle matrix composites with experimental results of composites containing five different polyalkene films. The new model appears to yield a satisfactory correlation to the experimental data and should assist the design of commercial cladding elements. However, the calculated length of the strain relief zone around the Griffith flaw (~3.5 mm for 25% BAR 113) is greater than the observed final crack spacing of approximately 0.5 mm. Thus, further work is required to refine the model to increase its compatibility with the basic ACK theory.
CHAPTER 7

MULTIPLE FRACTURE AND FIBRE-MATRIX STRESS TRANSFER

7.1. Definition of Terms

In previous chapters fibre-matrix stress transfer has been discussed. To simplify the terminology, reference will, henceforth, be made to "bond" strength and "average bond" strength, defined as follows -

"Bond" strength - dynamic shear stress across the interface at a contact point.

"Average Bond" strength - average dynamic shear stress along a fibre, assuming 100% interfacial contact and linear stress transfer, i.e., calculated from the ACK analysis.

Other terms which are used include -

Matrix channel - a hole in the matrix which would be present if the fibre was removed.

Fibril - see Chapter 3 for precise definition of primary and secondary fibrils. In this Chapter the differentiation will be ignored.

7.2. Multiple Fracture

7.2.1. Average Crack Spacing and Film Volume Fraction

A substantial transfer of stress from polyalkene fibres to cement matrices has been confirmed by the phenomenon of multiple fracture which, consequently, requires an examination of the previous assumptions
leading to the prediction of single fracture for aligned poly-
alkene cements (Kelly and Zweben, 1976; Pinchin, 1976; Baggott
and Gandhi, 1981). The source of the bond is the question to
which this chapter is addressed.

Figures 7.1.-7.5. show the relationships between the final
average crack spacing ($\bar{x}$) and the film volume fraction ($V_f$) for
BAR 112, BAR 113, E3H, BAR 21 and S8 respectively. At low film
volume fractions the scatter of the average crack spacing can be
large as in Figure 7.2. and 7.3. In this range, the ACK theory
suggests that crack spacing is highly sensitive to small changes
in film volume fraction, and hence, any variability in film volume
fraction along the specimen will affect the average crack spacing.
The effect of locally variable matrix strength can also have a
marked effect on crack spacing when the number of cracks within
the gauge length is small. Multiple fracture of BAR 112 composites
below approximately 4% film volume fraction was not obtained due to
the low film strength.

A comparison of the effect of film volume fraction on crack
spacing is only of practical use in ranking films in order of
performance for a particular specimen preparation. It does
not describe fundamental material interactions.

Additional composite sheets were manufactured with unopened
film so that the alignment of the fibrils was more axial. The
average crack spacing against film volume fraction data is presented
in Figure 7.6. The average crack spacing of the composite with
25% film by volume could not be measured with any accuracy since
it was less than 0.5 mm. Comparison of Figures 7.2. and 7.6. indicates
that at a film volume fraction of 15% the BAR 113 composite with
unopened film exhibits a higher average crack spacing than that
with the four times opened film.
Figure 7.1. Crack Spacing Data for BAR 112 Composites

Average Crack Spacing (mm)

Film Volume Fraction (%)
Figure 7.2. Crack Spacing Data for BAR 113 Composites
Figure 7.3. Crack Spacing Data for E3H Composites
Figure 7.4. Crack Spacing Data for BAR 21 Composites
Figure 7.5. Crack Spacing Data for S8 Composites
Figure 7.6. Crack Spacing Data for Composites containing Aligned Fibres
7.2.2. Average Crack Spacing and Specific Film Surface Area

Krenchel (1975) suggested that crack spacing would be related to the surface area of film per unit volume of composite. This value is simply the product of the specific surface area of the film alone and the film volume fraction. Hannant et. al. (1978) linked Krenchel's approach to that of ACK to show that for the relationship to hold for all fibre cements, $\sigma_{m'V_m/\tau}$ must be a constant. While this may be approximately true for individual fibre types in the same matrix, it is unlikely to be a universal constant.

At this stage it is worth looking at the theoretical relationship between crack spacing and specific fibre surface within the composite ($SFS_c$) in order to examine its implications.

Equation (2.13.) shows that minimum crack spacing, $x'$, is given by,

$$x' = \frac{\sigma_{m'V_m r}}{2\tau V_f}$$

which may be re-written as,

$$x' = \frac{E_m \varepsilon_{m'V_m}}{\tau SFS_c} \quad \ldots \quad (7.1.)$$

In the previous chapter it was shown that the matrix failure strain is not a material constant but is a variable related to certain film parameters. Whilst it was shown that the ACK analysis is not the best fit to polyalkene cements at realistic film volume fractions, the parameters involved certainly provide an illustrative basis for discussion. Thus,
It will be noted that the specific fibre surface is independant of fibre modulus, yet the matrix failure strain, \( \varepsilon_{\text{mu}} \), will be increased by increases in fibre modulus. In a more complex manner, a decrease in fibre radius increases the specific fibre surface which from equation (7.1.) would suggest a decrease in crack spacing. However, the matrix failure strain will also be increased although not sufficiently to alter the trend in crack spacing.

Thus it may be seen that there can be no unique relationship between crack spacing and specific fibre surface for all film types.

The relationship between the average crack spacing and specific fibre surface per unit volume of composite for films at four times opening is shown in Figure 7.7. It is apparent that Krenchel's unique relationship does not hold for polyalkene cements in general. It is interesting to note that two particular relationships are apparent on Figure 7.7. and are distinguished by the microstructure of the films, i.e., voided or non-voided. The voided films require a higher specific fibre (\( SFS_c \)) surface than the non-voided films to achieve a given crack spacing. It may be thought that the difference is caused by a major change in "average bond" strength, being lower for the voided films. However, reality is not quite so simple.

The scatter of results between 2 mm and 4 mm average crack spacing is larger for the voided films. This is probably a consequence of the differences in film modulus. Although E3H and BAR 112 possess similar specific surface areas for the film alone,
Figure 7.7. Relationship Between Average Crack Spacing and the Surface Area of Film per Unit Volume of Composite, SFS\textsubscript{C}
E3H yields an apparently higher average crack spacing (admittedly small). It should be remembered that E3H yields a significantly higher average matrix failure strain due to its higher film modulus (E3H - 29.5 GPa, BAR 112 - 8.7 GPa) which would increase the crack spacing.

7.2.3. "Average Bond" Strength and Film Volume Fraction

"Average bond" strength, \( \tau \), may be calculated from equation (2.13.), assuming that the average crack spacing is larger than the minimum by the factor of 1.364. The results are shown in Figure 7.8. and a general trend is displayed whereby "average bond" strength increases with increasing film volume fraction, e.g., the "average bond" strength of BAR 112 rises from 0.23 MPa to 0.5 MPa as the film volume fraction is increased from 4% to 10%. The scatter of the results makes it difficult to detect a consistent difference in "average bond" strength between films. However, it would appear that BAR 21 exhibits a higher "average bond" strength (0.6 MPa) than the other films (0.4 MPa). This would explain why BAR 112 and BAR 21 produced similar average crack spacings despite BAR 21 possessing a lower specific surface area. The significance of this observation is weakened since the range of film volume fraction is very limited.

A comparison may be made of the "average bond" strength for the unopened and four times opened films. At approximately 15% film by volume the "average bond" strength is 0.79 MPa (cv 7.6%) for the opened BAR 113 whereas the equivalent composite with unopened film yields 0.63 MPa (cv 8.7%). The difference is statistically significant. This suggests that a certain amount of the "average bond" strength may be attributable to the interaction of fibril and matrix as the fibril is aligned across each crack. This mechanism may be more apparent in polyalkene cements since the low stiffness fibrils are likely to cause less damage to the matrix, local to the crack, than a high stiffness fibre (e.g. 0.5 mm diameter steel fibre). Alternatively, as the fibrils
Figure 7.8. Showing the Dependence of "Average Bond" upon Film Volume Fraction
are drawn through a curved matrix channel interfacial contact pressure on the inside of the curve may increase the frictional restraint.

The average crack spacing of the monofilament composite exhibits a very high scatter (see Figure 7.6.) and consequently, the calculated "average bond" strength is also highly variable (0.71 MPa, cv 35.6%) making comparison with the fibrillated films difficult. However, despite the variation, the relationship between the average crack spacing and specific fibre surface (SFS\textsubscript{c}) is similar to that of the non-voided films, falling at the extreme left side of the curve on Figure 7.7. It is likely, therefore, that the physical mechanisms of stress transfer are similar for both monofilaments and fibrillated film.

The "average bond" strength of the composite containing 25\% by volume of unopened BAR 113 is in excess of 1.3 MPa, but no precise value could be calculated because the cracks were too close together to be counted accurately.

7.2.4. Discussion

It is apparent that the topic of fibre-matrix stress transfer is complex and cannot be understood without reference to the actual behaviour of the composite.

The two relationships of average crack spacing and specific film surface for voided and non-voided films (Figure 7.7.) may be explained by the variations in "average bond" strength. The voided films generally have a much higher value of specific film surface area for the film alone (see Table 4.1.) than the non-voided films. Consequently, at a given value of specific film surface within a composite, a non-voided film will be present with a much higher volume fraction. Since the "average bond" strength increases with film volume fraction (Figure 7.8) the non-voided film composite would exhibit the smaller crack spacing. This assumes a unique relationship between "average bond" strength
and film volume fraction and ignores any film modulus effects upon the matrix failure strain.

It has so far been assumed that the "average bond" strength has a physical significance. The ACK theory is based upon the following assumptions:

(a) bond strength is constant regardless of the relative displacement of fibre and matrix.

(b) matrix strength is constant for the specimen under consideration.

(c) fibre and matrix are in complete, intimate contact.

These assumptions will be briefly discussed before a model for the mechanism of stress transfer is proposed.

It will be remembered that cracking was more apparent on the "post crack" curve for composites containing voided film. Also, in section 5.7. it was shown that a voided film exhibited a higher rate of change of shear flow with relative displacement than a non-voided film. Although it is impossible to directly relate pull out results to the composite, it is believed that the further matrix cracking on the "post crack" curve is a reflection of the rate of change of shear flow. Consequently, assumption (a) is invalid and, strictly, crack spacing and "average bond" strength should be related to relative displacement (where the maximum relative displacement is half the width of the crack).

The accuracy of assumption (b) is always uncertain for cement matrices because of their inherent variability in strength. It is therefore possible that local regions of high matrix strength could be stressed to failure with one film type, whereas for a film with a lower shear flow characteristic, the failure stress could not be attained at the same distance from the adjacent crack. Further, the relationship, \( \bar{x} = 1.364 x' \), may not hold for a matrix of variable strength.
Previous research (see section 2.8.3.) has shown that even with relatively hydrophilic fibres the fibre-matrix interfacial contact is discontinuous. The interfacial zone of BAR 112 and a cement paste (same mix as for the pull out strips) hydrated under water for 28 days is shown in Figure 7.9. No matrix material was displaced when the film was removed, and hence, the interfacial zone, as seen, was that presented to the film. Whilst some regions conform to the film surface others maintain only point contact. The morphology of the latter regions (Figure 7.10.) is similar to that observed in voids of hydrated cement pastes. It is possible that these less dense regions may be reduced if the polymer were to be treated to make the surface more wettable. If this occurred, it would be anticipated that the "average bond" strength (as calculated) would be increased and the crack spacing reduced. However, this remains to be confirmed by experiment. Given the nature of the interface the "bond" strength at the actual contact points is likely to be much higher than the "average bond" strength.

However, the concept of "average bond" cannot be improved upon until the fibre-matrix stress transfer mechanisms are fully understood and quantified. The following discussion does not make any claims to the complete answer, but suggests a new line of approach for further research to follow.

7.3. Fibre-Matrix Misfit and Multiple Fracture

7.3.1. Validity of Previous Assumptions

Three theories have been proposed for material parameters necessary to achieve multiple fracture of brittle matrix composites (Kelly and Zweben, 1976; Pinchin, 1976; Baggott and Gandhi, 1981). Although there are variations of approach, as a consequence of the low fibre modulus and high Poisson's ratio none predict multiple fracture in polypropylene cements. However, one of the common assumptions is that the fibres are rigorously parallel
Figure 7.9. Typical View of Interface Between BAR 112 and Cement Paste
sided and perfectly aligned in the direction of stress. Obviously, the latter assumption is not strictly applicable to the current work, but nevertheless a substantial "average bond" strength was recorded for the unopened film composites which at the highest film volume fraction were the nearest to aligned films which could be achieved. Whilst non-alignment is an important factor in increasing "average bond" strengths, it is not felt to be the fundamental transfer mechanism and cannot, for instance, satisfactorily explain the increase in "average bond" strength with film volume fraction (Figure 7.8.)

It is known that the edge of a drawn polyalkene film is thicker than the central portion, e.g., BAR 113 measured 73µm reducing to 56µm at one section. Further, measurements in this laboratory have shown that the weight of unit lengths (1 m) of fibrillated film vary along the length of film roll indicating a change in cross sectional area. For instance, further along the same length of BAR 113 the edge thickness had increased from 73µm to 95µm. Such a large thickness variation may be due to "draw resonance" which is observed in films with draw ratios exceeding approximately 4:1 and may show a wavelength of at least one metre. However, "draw resonance" is not associated with fibre roughness which may result from inhomogeneous materials, fluctuations in the feeding rate of the extruder or in the melt temperature, surging of the extruder or lack of mechanical stability and stiffness of the equipment. In the extreme, melt fracture causes large surface irregularities as a certain critical extrusion rate is exceeded (Schuur and Van der Vegt, 1975). Hence, although measures are taken to negate these factors the assumption of a rigorously parallel sided fibre is open to question.

Obviously, if significant effects are to be observed at the fibre-matrix interface, a non-parallelism must occur on a scale, orders of magnitude smaller than that mentioned above, with, maybe, changes in thickness or in fibril width being measured within individual micron lengths of film. In an attempt to reveal such changes in film thickness, short lengths of BAR 113
were embedded in wax and thin longitudinal sections, exposing the thickness, were cut using a microtome. Each section was mounted on a glass slide and examined in a optical microscope. However, the shallow depth of field and problems with the wax separating from and apparently overlapping the film, resulted in the observations being inconclusive.

Identically prepared thin sections were mounted on stubs for examination by scanning electron microscopy, the wax being chemically removed. Unfortunately, charging of the edges of the film in the electron beam, which is always a problem with polyalkenes, produced images without a precisely definable edge from which to take measurements. Crude measurements from the micrographs suggested that the approach was worth pursuing, although the objectives would have to be curtailed due to pressure of time.

It was therefore decided to measure the change in thickness rather than the absolute thickness at points approximately 0.5 mm apart, along sections of the film strips used for the pull out tests. The apparatus used was a Solex comparator Unit 1-HH51, with the film supported on a fine anvil. The comparator measurement is based upon changes in air pressure and was calibrated to 0.25 µm/division with a full scale deflection of ±10µm. Typical results are shown in Table 7.1. below, being reported as the change in thickness related to the thickness at the previous measurement position.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Change in Film thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specimen 1</td>
<td>-0.5, -8.5, +11.5, +1, +0.5, +0.75, -1, -1, +2.5, 0</td>
</tr>
<tr>
<td>Specimen 2</td>
<td>+1.75, +0.25, 0, -2.5, -1.25, +3.5, +2.25, +1, +0.5, +0.25</td>
</tr>
</tbody>
</table>

Table 7.1. Changes in Thickness of BAR 113 along its Length
Similar measurements were attempted on BAR 112 but without success since although the variation was much greater than for BAR 113, the reading drifted rapidly. SEM examination of the surfaces on BAR 112 and BAR 113 (Figures 7.11. and 7.12. respectively) illustrates that the surface of BAR 112 changes so rapidly that this could explain why a stable signal was unobtainable. In contrast, the surface of BAR 113 is relatively flat. The difference in surface topography is probably a consequence of the expanding agent included in the polymer. A roughly cut section through BAR 112 is shown in Figure 7.13. In the centre of the micrograph three large internal voids may be seen. The area shown in Figure 7.13. may be seen to a smaller scale in the centre of Figure 7.14. Despite the poor quality of the micrograph it is apparent that the overall film thickness, in the region of the internal voids is greater than adjacent areas.

Even though these measurements do not yield as much data as had been originally hoped, they do indicate that the assumption of a rigorously parallel sided fibre is inappropriate for normally produced drawn polypropylene films.

7.3.2. Criteria for Achieving Multiple Fracture

In section 2.8.4. it was suggested that a varying film thickness would result in fibre-matrix misfit accompanied by a variable interfacial contact pressure as the film moves through the matrix channel. The misfit can be defined as the difference in dimension between the matrix channel and the strained fibre, the dimension being measured in any direction in a plane at right angles to the length of the fibre. However, no misfit will occur if the Poisson contraction is larger than twice the maximum profile height (see Figure 7.15.).

Let us consider the simplified fibre shown in Figure 7.15.
Figure 7.14. Micrograph of Section Through Film BAR 112
If it is assumed that, upon propagation of a crack, the matrix channel at the crack surface relaxes to its initial dimensions, then the separation, between B and C (Figure 7.15.), of the fibre and matrix is given by the Poisson contraction of the fibre, $\delta t -$

$$\delta t = tv_f \varepsilon_{\text{mat}}(1 + \alpha) \quad \ldots \quad (7.3.)$$

where $t = \text{film thickness, } t \gg \delta$

$v_f = \text{Poisson's ratio of film (0.3.)}$

It is apparent from equation (7.3.) that the Poisson contraction is a function of film thickness, film modulus and film volume fraction, whilst it must also be remembered that the enhanced matrix failure strain is complex function. Figures 7.16. and 7.17. show the relationship between Poisson contraction and film modulus and volume fraction for films of 70\mu m and 15\mu m.
Figure 7.16. Relationship Between Poisson contraction and Film Modulus and Volume Fraction for 70 μm Film
Figure 7.17. Relationship Between Poisson Contraction and film Modulus and Volume Fraction for 15 μm Film
thickness. These values were chosen to simulate BAR 113 and BAR 21 respectively. The values of enhanced matrix failure strain, $e^m_{yu}$, for each film volume were taken from the combined experimental average matrix failure strain results of BAR 21, BAR 112 and BAR 113. Hence, although the Poisson contraction at low film moduli may be overestimated and likewise underestimated at high moduli, the general trend indicates Poisson contraction decreasing with both increases in film modulus and volume fraction and a decrease in thickness.

A minimum criterion for multiple fracture is that the Poisson contraction should less than twice the profile height, $\delta$. If $\delta$ is too small, the fibre would simply pull out. On the other hand, where $2\delta$ is greater than the Poisson contraction, relative displacement of the fibre will result in misfit of differing magnitude along the length of the displaced fibre. Some regions of the fibre, e.g., A, will exhibit relatively large misfit, whereas others, e.g., B, will be completely debonded. The case of intermediate regions will depend upon the direction of relative displacement, i.e., of increasing or decreasing film thickness. Of course, should a length of fibre be displaced by one profile pitch, then no misfit will occur. As load is transferred back into the matrix, the size of the matrix channel will reduce due to Poisson contraction and the Poisson contraction (fibre) will be less, increasing the potential for misfit. To counteract this effect the relative displacement is reduced.

It should also be remembered that the rectangular shaped fibrils will also contract laterally across their width, and since the profile height is a two dimensional phenomenon (i.e., along the fibre length and width) misfit would be anticipated as lateral relative displacement is induced.

The minimum criteria for a real film cannot be precisely defined since, at the micron level, the distributions of profile height and pitch are likely to be random. Thus, a statistical approach is required. Nevertheless, from Table 7.1. and Figure 7.16.
it would appear that $2\delta$ is greater than the Poisson contraction for BAR 113, such that multiple fracture would be anticipated.

7.3.3. Fibre-Matrix Misfit and Film Structure

The previous discussion showed that misfit would be expected with BAR 113 simply through changes in film thickness. Visual examination of the fibrillated film (Figure 4.3.) shows that the individual fibrils have discontinuous "hairs" attached to them such that a fibril does not possess a uniform width along its length (Figure 7.18.).

Figure 7.18. Simplification of Discontinuous Hairs on BAR 113

As the fibril is pulled relative to the matrix the hair may be pulled out of its channel and into the channel of the fibril. The subsequent behaviour is likely to depend on the direction of fibril displacement. Considering Figure 7.18., if the displacement is to the right, the hair could have sufficient space in the fibril channel without causing matrix-hair-fibril interaction. However, if displacement is to the left the situation is more complicated. Obviously, the unstrained width of hair and fibril is greater than the width of the fibril channel. Thus, initially, lateral misfit may occur.
However, as relative displacement increases, it is likely that the shear strength (or peel strength) of the fibril is exceeded in the vicinity of the junction of the hair and the fibril. The effect of such damage is unquantifiable but it may reduce the apparent strength of the film in the composite while possibly causing some jamming in the matrix channel.

If we now consider the fibrillation of a voided film, another source of misfit is revealed. As the film is passed over the pin roller, the overall width of the fibrils so formed will be approximately constant. However, within the fibrils, part of the width will be taken up by the voids. Consequently, the width of polymeric material changes along the length of the fibril as shown in Figure 7.19, i.e., \( a > b + c \).

![Figure 7.19. Simplification of the Structure of a Film showing a Void Completely Penetrating a Fibril](image)

During hydration of the cement, the voids may be filled with cementitious material as shown in Figure 7.20. As such a fibril is displaced it will fibrillate further as it is drawn past the matrix filled void. Lateral misfit may then occur due to the greater width of fibril being drawn through the channel.
Figure 7.20. Shows Void in Film Penetration by Cement Hydration Products. Width of Micrograph is approximately 100 μm.
on either side of the filled void. Should the difference in width \[ a - (b + c) \] be less than the Poisson contraction, then stress transfer will still occur, since the strained fibril will "bite" onto the filled void.

If the void does not completely penetrate the film a "trough" may still be formed (Figure 7.21.) and the resulting fibre-matrix interaction may be a "ploughing" function. Fibril damage would again occur as portions of the fibril were "chiselled" off.

It would appear that these mechanisms may lead to greater film damage in voided films in comparison to non-voided films. This was certainly noted during the narrow strip pull out tests. Whereas BAR 113 was pulled out apparently undamaged, BAR 112 always appeared to be shredded. Further, during the composite tests, it was noted that voided film composites produced the greater acoustic emission which is believed to be a reflection of film damage.

7.4. Shear Flow and Fibre-Matrix Misfit

In section 5.7. it was shown that the dynamic shear flow increased with fibre-matrix relative displacement, BAR 112 exhibiting the greater rate of increase. It is apparent that misfit is a dynamic mechanism and is compatible with the experimental data. The additional sources of misfit possessed by a voided film could explain the more rapid increase in dynamic shear flow. The load at which static resistance is overcome is probably dictated by the distribution of voids in the vicinity of the entry point of the film strip into the matrix. A heavily voided region would show a higher surface area than a region with few voids and in the latter case the maximum static load may be similar to that for a similar non-voided film.
7.5. "Average Bond" Strength and Fibre-Matrix Misfit

7.5.1. Comments on 28 Day Test Results

Although "average bond" strength has no physical significance it may be instructive to examine the results and see how they support the concept of misfit. To summarise, fibril Poisson contraction, equation (7.3.), decreases with increases in film modulus and volume fraction and a decrease in thickness. If the profile height is constant, then "average bond" strength may be expected to increase as Poisson contraction decreases.

For each film type with an adequate spread of film volume fraction (i.e., BAR 112, BAR 113 and E3H) it can indeed be seen (Figure 7.8.) that "average bond" strength increases with an increase in film volume fraction. However, the effect of different film moduli is difficult to isolate; the higher moduli of E3H and S8 have not made very great improvements upon the performance of BAR 112 and BAR 113 implying that the modulus effect is of secondary importance.

A reason may lie in the manufacturing procedure of each film. Whereas BAR 112 and BAR 113 had draw ratios of approximately 8:1, S8 was drawn 18:1 and E3H, 25:1. It is possible that the drawing procedure not only makes the films narrower and thinner but also reduces the profile height. Thus, S8 and E3H may exhibit a smaller "average" profile height than BAR 113. Indeed, when measured as for BAR 113, the unfibrillated S8 replacement (as used for modulus determination) exhibited a change in thickness of only ~0.5µm compared with up to 11µm for BAR 113. Another factor, which cannot be quantified, is that although the fibrillated films were voided, this was not apparent in the unfibrillated film. It is not known whether the mechanism of transfer is the same for voiding with different origins.
Figure 7.17. suggested that a thinner film may exhibit a greater misfit for a given profile height. The "average bond" strength of BAR 21 (15μm thickness in contrast to 70μm of BAR 113) indeed appears to be higher than that of the other films (Figure 7.8) but no measurements were made of profile height. Baggott and Gandhi (1981) reported an "average bond" strength of 0.05 MPa for 340 μm diameter monofilaments. It is possible that the reasons for the low bond include the large fibre diameter and high degree of fibre alignment.

7.5.2. Comments on 1 Year Test Results

In discussing the 1 year test results in section 5.6., the "average bond" strengths were not quoted. The "average bond" strengths are shown in Table 7.2.

<table>
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<tr>
<th>Age of Test</th>
<th>28 Days</th>
<th>1 Year</th>
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<tbody>
<tr>
<td>&quot;Average Bond&quot; Strength (MPa)</td>
<td>BAR 112</td>
<td>0.36 (0.04)</td>
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<tr>
<td></td>
<td>BAR 113</td>
<td>0.45 (0.06)</td>
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<tr>
<td>Matrix Modulus (GPa)</td>
<td>BAR 112</td>
<td>31.5 (2.0)</td>
</tr>
<tr>
<td></td>
<td>BAR 113</td>
<td>31.7 (1.5)</td>
</tr>
<tr>
<td>Average Matrix Failure Strain (x10^-6)</td>
<td>BAR 112</td>
<td>296 (22)</td>
</tr>
<tr>
<td></td>
<td>BAR 113</td>
<td>282 (21)</td>
</tr>
</tbody>
</table>

( ) denotes standard deviation.

Table 7.2. "Average Bond" Strength and Relevant Parameters at 28 Days and 1 Year
The "average bond" strengths appear to show a decrease at 1 year, although only the BAR 113 difference is statistically significant. It might have been expected that "average bond" would remain constant or increase due to a possible increase in contact area.

It is proposed that a decrease in "average bond" strength would result from a decrease in misfit. Assuming that the morphology of the interfacial zone remains essentially constant, misfit will decrease if the Poisson contraction of the fibrils across a crack increases. Equation (7.3.) indicates that the only two variable parameters (with age) affecting Poisson contraction are the matrix modulus and failure strain. Whilst the matrix modulus increases and the average matrix failure strain decreases with age, the product \((E_m \varepsilon_u)\) increases by approximately 12%. Hence, the Poisson contraction of the fibrils across a crack will increase, reducing misfit and reducing the "average bond" strength.

A decrease in the "average bond" strength may be seen in BAR 113. It is felt that the proposed damage, occurring to BAR 112, probably masks any changes in behaviour.

7.5.3. Comments on "Average Bond" Strength for Composites with Unopened and Four Times Opened Film.

In section 7.2.3. it was reported that the "average bond" strength for the four times opened film is 0.79 MPa and 0.63 MPa for the unopened film (BAR 113, \(V_f \sim 15\%\)). Hence, the effect of departure from axial alignment can be directly obtained by comparison of the two results. The difference in crack spacing, 1.33 mm for the four times opened film and 1.70 mm for the unopened film is statistically significant and this is in contradiction to the prediction of Aveston and Kelly (1973) who stated that \(x'' = kx'\).
where $x''$ = minimum crack spacing for non-aligned fibre composite.

$x'$ = minimum crack spacing for aligned fibre composite.

$k$ = factor depending upon degree of non-alignment
(k > 1).

$k$, in fact, is related to the number of fibres crossing each crack which decreases with non-alignment (constant film volume fraction) such that $k = \pi/2$ for random planar fibres and $k = 2$ for 3D random fibres.

For non-aligned fibres, Aveston and Kelly suggested that the stress transfer comprises the previously discussed frictional transfer and a "pulley" force due to the fibres being realigned across a crack. It is possible that, for polyalkene cements, the "pulley" force is larger than predicted. Polyalkenes are relatively flexible and little damage would be anticipated in the matrix close to the crack face from the "pulley" action. Further, the increased normal force at the interface would lead to an additional local stress transfer. If the "pulley" force is the source of the increased stress transfer it may have important consequences for the specification of the fibrillation pattern of future films.

The angle through which the fibril is realigned is controlled by the slit length, all other factors being constant. The shorter the slit length, the greater is the angle of realignment. Hence, whilst it is not currently possible to predict the optimum angle, it may be fruitful to examine the average crack spacing produced by films of various slit lengths, as they are opened to increasing amounts at constant film volume fraction. Fibril width may also be an inter-related parameter.

It is not possible to quantitatively separate the mechanisms of misfit and fibril realignment, however, the latter mechanism may not be as sensitive to Poisson contraction of the fibrils such that its relative contribution may be larger at low film volume fractions.
7.5.4. Comments on Stress Transfer and First Cracking of the Matrix

Chapter 6 was concerned with predicting the first cracking strain of the matrix. One parameter common to all of the theoretical predictions is a bond strength. It is apparent that stress transfer is highly complex and that this chapter has dealt with stress transfer after considerable relative displacement, i.e., during and after completion of multiple fracture. The bond strength required to predict matrix failure strain is a different matter.

The growth of the Griffith crack not only involves smaller relative displacement but also smaller strain in the fibre. The relative displacement may lead to a smaller misfit, however, the reduced fibre strain is accompanied by a smaller Poisson contraction. As a compromise and until the stress transfer can be mathematically described, a value for bond has to be assumed in order to carry out numerical predictions. The most realistic value for the purposes of this thesis is to assume that the "average bond" strength equals 0.5 MPa.

7.6. A Tentative Film Specification

Two composite properties, above all others, are of paramount interest for successful commercial exploitation of a pseudo-ductile material such as NETCEM. The composite should exhibit a "first crack" stress and strain which are high in relation to the working stresses and the crack widths should be small. Such a composite would be achieved with a film of high elastic modulus and good bonding characteristics. A tentative specification will be outlined based upon the films so far supplied and then potentially beneficial improvements will be suggested. It is known that the films examined herein do not represent the best available polypropylene films. However, commercial secrecy has precluded both microstructural examination and a discussion of the results from such films.
From Figures 6.1.-6.5. it can be seen that the primary factor affecting the average matrix failure strain at constant film volume fraction is the film modulus. Hence, a high modulus and draw ratio is the first requirement. A secondary factor is the specific surface area of the film, i.e., higher specific surface area yields a higher average matrix failure strain. This may be achieved in one of two ways:

(a) the film may be very thin, \( \leq 15 \mu m \) with a small fibril width. The minimum fibril width is governed by the fibrillation techniques but may be approximately 0.2 mm. Obviously, to achieve a given film volume a large number of film layers is required as the film thickness is reduced. Hence, there may be a limit to film thickness governed by the ease of incorporation of a number of film layers into the matrix.

(b) the film may be thicker (\( \sim 50 \mu m \)) but also voided such that the specific surface area is increased. This would solve the problem of film incorporation but attention should be directed to the ultimate strength and strain of the film. If these properties are reduced drastically by the voiding then durability problems may be encountered as a result of changing matrix properties. This could result in the critical fibre volume fraction increasing with time as the matrix strength increases, possibly leading to brittle failures if the product of fibre strength and volume fraction is too low.

A good commercial film would be a voided 50\( \mu m \) film which would give a composite with close crack spacing whilst making commercial production relatively easy. The combination of high film modulus and low crack spacing produces a small crack width.

Thus, a film specification, but not to be considered a minimum commercial specification, may be:
<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strength</td>
<td>≥ 500 MPa</td>
</tr>
<tr>
<td>Initial Modulus</td>
<td>≥ 15 GPa</td>
</tr>
<tr>
<td>Specific surface Area</td>
<td>≥ 350 mm²/mm³</td>
</tr>
<tr>
<td>Slit Length</td>
<td>15 - 20 mm</td>
</tr>
<tr>
<td>Fibril Width</td>
<td>0.2 mm</td>
</tr>
<tr>
<td>Film Thickness</td>
<td>50 μm</td>
</tr>
<tr>
<td>Structure</td>
<td>Voided</td>
</tr>
</tbody>
</table>

Appropriate anti-oxidants to be included to give a service life of greater than 30 years.

We may now consider modifying the film to enhance certain properties, notably "average bond" strength.

It has been suggested that the fundamental mechanism of stress transfer is that of misfit. Hence, the production process should be arranged to produce an adequate but so far undefined profile thickness distribution. This may possibly be achieved during the passage of the film though the various rollers, surface treatment of which may produce the desired changes in film thickness. However, it is suggested that the misfit is not so large as to cause film damage within the composite.

The angular alignment of the fibrils across a crack could be important to crack spacing. If this is confirmed experimentally then the slit length could be adjusted accordingly. However, care must be taken to ensure that the combination of fibril width and slit length does not result in buckled fibrils during the film opening process. Such buckling will affect ease of composite manufacture and reduce the lateral strength of the composite.

If it is confirmed that the morphology of the interfacial zone is beneficially altered by increasing the wettability of the film, treatments such as corona discharge may be included. Benefits may be gained by including chalk or cement particles to produce an inhomogenous material which may increase the surface roughness.
8.1. General Tensile Behaviour

Polyalkene reinforced cements containing more than the critical volume of film are shown to comply with the form of the generally accepted theoretical tensile stress-strain curve. However, the accurate prediction of the curve is complicated by the non linear stress-strain behaviour of polyalkene films.

Cracking is shown to continue to occur on the "post crack" slope, predominantly with films possessing a voided structure. This type of behaviour implies that increasing slip between fibril and matrix results in an increase of stress transferred to the matrix, which is contrary to the assumptions of the simple theory.

8.2. Enhanced Matrix Failure Strain

Due to bowing of the tensile specimens it was not possible to measure the actual matrix failure strain. An additional complication is that the matrix failure strain was not a single valued parameter and, thus, an average matrix failure strain was calculated from the stress-strain curve.

The average matrix failure strain is shown to increase with increases in film volume fraction and modulus. The model initially described by Aveston, Cooper and Kelly (1971) always gives a lower limit to the matrix failure strain since it does not consider the growth of a crack or flaw. The Nottingham model (McColl and Morley, 1977), whilst yielding an improved fit to the experimental data, also
provides an underestimate. The assumption of a constant size for the strain relaxation zone around an internal crack or flaw produces an unrealistic strain distribution normal to the crack face.

An improved prediction of matrix failure strain is obtained using a new model developed herein which predicts that the size of the relaxation zone and critical crack width decrease with increasing film volume fractions. The model also shows that an increased matrix failure strain would be expected if the "diameter" of the fibrils were to be reduced or if the "bond" strength is increased. Due to problems of accurately determining these parameters for a polyalkene film it is not possible to separate the effects of surface area and bond strength within the scatter of the experimental results.

8.3. Fibre-Matrix Stress Transfer

Typical drawn polyalkene films are shown not to possess a constant thickness. Thus, upon relative displacement of fibril and matrix an interfacial contact pressure will occur, due to the resulting misfit, for as long as the Poisson contraction is less than the local difference in film thickness. The magnitude of the interfacial contact pressure depends upon the degree of misfit and, hence, will vary along the fibril, such that the concept of a unique "bond" strength is inapplicable. Further, the interfacial contact area is shown to be discontinuous and indeterminate and hence the "average bond" strength derived from a multiple fracture experiment has no physical significance, even though it may be convenient to use such a value in certain calculations.

The simple equations developed suggest that, for a given surface profile, the "average bond" strength would increase for a decrease in fibre strain across a crack or for a decrease in film thickness. The decrease in fibre strain across a crack could result from either an increased film volume fraction or an increased film modulus. The experimental data follows this prediction
in respect of film content and thickness whilst not showing a dependance on film modulus. This is possibly due to the surface irregularities on such films being reduced as a function of the further drawing necessary to achieve higher moduli. Unfortunately, the experimental techniques necessary to check this possibility were not available to the author.

Pull out tests show the shear flow (load/unit length of embedment) to increase with increasing relative displacement and this effect increases more rapidly for voided films. Thus, the "average bond" strength derived from a multiple fracture experiment, which involves large relative displacement, is not necessarily the precise value applicable to the growth of a Griffith type flow in which relative displacements are small. The situation is further complicated since the Poisson contraction of the fibre is less in the case of growth of a Griffith crack than at the completion of multiple cracking, consequently, the potential misfit may be larger.

A supplementary source of fibre-matrix stress transfer results from realignment of fibres across a crack. At film volume fractions of approximately 15%, composites with four times opened film yield closer crack spacking than those with aligned films. It is not possible to quantitively separate the relative contribution of either mechanism which may vary with film opening and film volume fraction.

8.4. Closure

As a result of the research described in this thesis it is possible to design films with improved characteristics which will enable the production of composites with enhanced serviceability in the sense of higher potential design stresses and closer crack spacking and reduced crack width.
CHAPTER 9

RECOMMENDATIONS FOR FURTHER WORK

Two specific properties are of particular importance and require further investigation, namely enhanced matrix failure strain and fibre matrix stress transfer.

The growth of a crack through a fibre cement is not fully understood and the tensile strip used in this investigation is not the ideal specimen configuration to study this parameter. A specimen is required in which parallel sides and axial loading is guaranteed with precise axial alignment of the fibres. The incorporation of a notch would accurately locate the failure plane. Development of a straining stage allowing for examination of the notch in a Scanning Electron Microscope may reveal some details of the crack path.

A major problem with fibre cements is the low work of fracture of the matrix. Means of increasing this parameter should be examined, possibly by including small quantities of a very fine chopped polyalkene flock or other finely distributed fibre which would increase the frictional work done during crack propagation.

Also, having accurately determined the enhanced matrix failure strain, its relationship to the flexural load-deflection behaviour of flat and corrugated sheeting requires examination.

Additionally, a continuous description of the width and thickness profile of fibrillated films is required so that a mathematical model of misfit and fibre matrix stress transfer can be derived. A suitable pull out test which allows for various pull out loads and an accurate determination of the relative displacement between fibre and matrix may be an advantage. The effects of fibril alignment, film wettability and film inclusions requires quantifying.

With a description of the stress transfer mechanism and of the film modulus with increasing strain it may be possible to improve the prediction of the strain at the completion of multiple cracking.
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CHAPTER 2


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CHAPTER 3


CHAPTER 4


CHAPTER 5


CHAPTER 6


CHAPTER 7


CHAPTER 8


APPENDIX

ANALYSIS OF THE CEMENT

Ordinary Typical Cement - Batch No. 103

B.S. 12 Physical Tests

Setting Time  Initial (mins) = 125  Final (mins) = 170
Standard Consistency Water  (%)  = 25.8
Soundness  Le Chatelier Expansion (mm)  = 0.3
Fineness  Specific Surface (m^2/kg)  = 350
Compressive Strength  N/mm^2  3 days  7 days  38 days
(1:6/0.6 Concrete)  21.5  30.0  41.5

Chemical Analysis

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