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THE MECHANICAL PROPERTIES OF UNIAXIAL CARBON FIBRE REINFORCED EPOXY RESIN COMPOSITE SYSTEMS

By

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Submitted as a thesis for the degree of
MASTER OF PHILOSOPHY
In the Faculty of Mathematical & Physical Sciences of the
UNIVERSITY OF SURREY

The Department of Metallurgy & Materials Technology, The University of Surrey.

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The aim of this work is to investigate the mechanical properties of carbon fibre reinforced epoxy resins and some of the factors which affect these properties. Different fibre/resin systems are examined and the properties investigated are the flexural strength, interlaminar shear strength, fatigue behaviour and fracture toughness. In order to use the composites safely and efficiently it is important to understand the variables which affect the mechanical properties of the material and the failure mechanisms which are likely to occur under a given set of circumstances. In some cases the results of the experiments must be treated with caution, e.g. short beam interlaminar shear strength ILSS tests, because the results are difficult to interpret in terms of the fracture mode observed. The interpretation of the results of the tests and their significance in terms of the improvement of composite properties are discussed.
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1.1 INTRODUCTION

The development of high strength, high stiffness carbon fibres from a polyacrylonitrile precursor P.A.N. (1), has caused a great deal of interest in the field of continuous fibre reinforced composite materials.

Carbon fibre is important as a reinforcing material because of its high specific strength and high specific stiffness. Figure 1 (2) compares these properties for several different materials. The high specific stiffness makes carbon fibre a more attractive material than glass, especially in the aircraft industry. Unfortunately, the high production costs of large scale carbon fibre manufacture have limited its use to specialized applications where cost is not the most important consideration. The fibre should therefore be used as efficiently as possible and in order to do this an understanding of the composite properties and the factors which affect them is essential.

The aim of this chapter is to review the work carried out to investigate the mechanical properties of carbon fibre - epoxy resin composite materials. The properties of both fibre and resin, the factors which affect these properties, the design requirements of composite materials and the testing methods used to obtain mechanical property data are all investigated.

1.2 CHEMISTRY AND MORPHOLOGY OF CARBON FIBRES

The two main sources of commercial carbon fibres are synthetic fibres and pitch. The mechanical properties of the carbon fibre are dictated by its structure, synthetic fibres are much more ordered than pitch and they present a more attractive starting point providing this structure can be retained. The process of converting synthetic fibres into carbon fibres usually involves heating the fibre to at least 1500°C and most synthetic fibres melt before decomposition and thus lose their oriented structure. The high melting temperatures of P.A.N. and rayon, attributable to the strong intermolecular forces inhibiting molecular motion, make them attractive precursor materials. The conversion of P.A.N. to carbon fibre is cheaper and easier to carry out than that of rayon which requires an additional expensive hot stretching process.
FIG. 1 COMPARISON OF THE MECHANICAL PROPERTIES OF CARBON FIBRES WITH THOSE OF OTHER MATERIALS
Many other potential precursor materials have been considered (3, 4, 5, 6, 7, 8, 9) but these have not been widely commercially exploited.

1.2.1 PRODUCTION OF CARBON FIBRES FROM A P.A.N. PRECURSOR

There are three main stages in the conversion of P.A.N. to carbon fibre, oxidation, carbonization and graphitization.

1.2.1.1 Oxidation

P.A.N. fibres which have been oriented by spinning and hot stretching are heated on a wooden frame in air at 220°C for five hours. The frame prevents shrinkage of the precursor which would cause relaxation and misorientation of the molecular chains. The importance of oxidation is to convert the P.A.N. into thermally stable ladder polymer which does not undergo chain scission when strongly heated (1 and 10).

1.2.1.2 Carbonization

The oxidized fibres are heated to a temperature of 1000 – 1500°C and the side chain elements are removed in the form of ammonia, hydrogen cyanide and water. In this stage there is a tendency to produce six membered rings in reactions which occur during carbonization and these presumably lead to the hexagonal arrangement of atoms in the carbon fibre (11).

1.2.1.3 Graphitization

This is a high temperature treatment - 3000°C which involves changing the structure of the carbonized fibre and is described more fully in the next section.

1.2.2 STRUCTURE OF CARBON FIBRES

At heat treatment temperatures of about 1000 - 1500°C electron microscope (12) and X-ray investigations (11 and 12) reveal that the carbon atoms form an hexagonal configuration held together by covalent bonding between the atoms and the hexagons are linked to form planar structures (11). The hexagonal layers form long narrow ribbon like structures which pack together to form microfibrils of turbostratic graphite. The ribbons become twisted together to form a larger structural unit known as a fibril and bundles of fibrils constitute the fibre (11 and 13).
Many workers have characterized carbon fibre structure in terms of the parameters $La$ and $Lc$ determined from electron microscopy and high angle X-ray diffraction (14, 15 and 16), these parameters represent the dimensions of crystallites in the $a$ and $c$ directions of the graphite lattice. If a continuous bending of the lattice planes occurs, $ha$ more accurately represents the mean length of the straight basal plane (17 and 18). It is generally agreed that $La$ and $Lc$ increase with increase in heat treatment temperature, this may be interpreted in terms of an increase in preferred orientation.

The carbonization and graphitization processes involve a realignment of crystallites towards the axial direction (19 and 20) and at temperatures of about $2800 - 3000^\circ C$ there is a high degree of preferred orientation. Wicks, Coyle and Gillin (21) suggested that the morphology of the fibre formed at high temperature involves sheet like elements arranged concentric to the fibre's axis, this was confirmed by Stewart and Feughelman (22) using Stereoscan Electron Microscopy techniques. Stewart and Feughelman also found that the cohesive forces within the sheet like units are greater than those between them. The core-sheath structure demonstrates that the fibre structure is not homogeneous throughout its cross-section and it implies that a memory of the original textile fibre orientation is preserved only in a local part of the fibre while elsewhere the texture may be severely degraded.

### 1.2.3 MECHANICAL PROPERTIES OF CARBON FIBRE

There are four types of carbon fibre available for use in composites:-

**Type 1** - A high modulus carbon fibre which is produced at $2800 - 3000^\circ C$. It is expensive because of the high heat treatment temperatures used. It has a low strain to failure and relatively low energy to fracture.

**Type 2** - This has a high strength and high strain to failure which make it a more attractive proposition for most applications where high modulus is not essential. It is also cheaper because of the lower heat treatment temperatures.

**Type 3** - This has a slightly lower strength than type 2, it is heat treated at a lower temperature and is therefore cheaper.

**Type 4** - This is processed at $2000 - 2500^\circ C$ and has properties intermediate between types 1 and 2.
During the oxidation of P.A.N. based fibres, Youngs modulus remains almost constant while the tensile strength falls (23 and 24). At a carbonization temperature of only 400°C both tensile strength and modulus start to rise (23, 24 and 25) and continue to do so until heat treatment temperatures above 1200°C are reached. Between 1000 and 3000°C the modulus rises continuously with temperature while the tensile strength shows a maximum between 1200 and 1500°C (2 and 26). The rise in modulus may be explained in terms of the orientation of the graphitic layers, however the decrease in strength at high temperatures is more difficult to explain (17).

Flaws which include voids from dissolved gases, inclusions, and surface irregularities obtained from the spinning process affect the values of fibre strength (27, 28, 29 and 30). Murphy and Jones have demonstrated the effect of surface flaws by etching the fibre surface which raised the tensile strength of the fibres without affecting the maximum obtained at 1500°C (17). Cooper and Mayer explained the loss in strength above 1500°C in terms of the growth of crystallites and that the strength at low HTT's strength is dictated by the removal of H and N atoms which produces a densification of the fibre and voids are trapped within the fibre structure. At higher temperatures the increase in crystallite size leads to a reduction in strength as indicated by the Hall-Petch equation:

\[ \sigma_t = \sigma_0 + Kd^{-1} \]

- \( \sigma_t \) tensile flow stress
- \( \sigma_0 \) resistance to "dislocation" motion
- \( K \) constant
- \( d \) crystallite diameter

The mechanical properties of the fibre may be modified by doping it with boron atoms or irradiation. Both of these treatments increase the fibre modulus and this is attributed to enhanced recrystallization and to an increase in the shear stiffness of the graphite by boron or by displacement of atoms which pin dislocations.

Bullock (31) explained the strength properties in terms of the core-sheath structure of the fibres in which the core is more heavily flawed, controlling the strength, while the more perfect sheath controls the modulus and is readily affected both/irradiation and oxidation when the fibre is heated in air.

1.2.4 SURFACE TREATMENT OF CARBON FIBRES

The surface of carbon fibres is particularly unreactive chemically and poor bonds are formed with polymers because of the nature of carbon and
the fact that the fibres have been formed in an inert atmosphere. This poor bond can lead to poor mechanical behaviour of the composite and surface treatment is used to overcome this. The treatment is similar to that used for the activation of charcoal, where atoms which will take part in the electron delocalization phenomena are chemically bonded to the fibre surface. These atoms can include oxygen, sulphur and nitrogen, in practice oxygen is used because oxidation is a simple process and the fibres are heated for short periods in air or ozone, or immersed in a liquid oxidizing agent e.g. nitric acid or a hypochlorite solution. There is a major disadvantage in surface treatment however, the surface of the fibres becomes more hydrophilic and water is absorbed at the fibre surface. Water bonds to the surface preferentially causing debonding and fibre pull-out especially in hostile environments (32). Nevertheless surface treated fibres are most frequently used in composites because of the improvement in the mechanical properties.

1.3 CHEMISTRY AND PROPERTIES OF EPOXY RESINS

Epoxy resins are used in a wide variety of applications, one of the most important of which is as a matrix for fibre reinforced materials. The function of a matrix is as follows (33):-

(i) It provides a means by which the bad can be applied to the strong fibres.
(ii) It separates the fibres and prevents cracks from passing catastrophically from one fibre to another.
(iii) It protects the fibres from abrasive damage and also from environmental damage.
(iv) A relatively ductile matrix can provide a mechanism for slowing down cracks which might pass rapidly through brittle fibres.
(v) Through its interfacial strength the matrix can be an important mechanism in controlling the toughness of the composite.

The uncured resins are characterized by the three membered epoxide ring group:-

\[ \text{C} - \text{O} - \text{C} \]

The reactions of this group resulting in ring opening are responsible for cross linking (i.e. curing). The epoxide resin is made
up of at least two constituents, the basic resin and the curing agent. It is the versatility of the epoxy group that makes the resins so attractive for use in composite materials as many different curing systems can be used to modify the properties or facilitate fabrication. Other constituents may also be added, these include: fillers, dyes, solvents, plasticizers and accelerators and these aid the production of systems with particular properties for specific applications. The choice of resin system is therefore governed by the requirements of the particular application.

The parent resins may be classified broadly into the following groups:

(i) Glycidyl ethers - \[ R-O-CH_2-CH-CH_2 \]

(ii) Glycidyl esters - \[ R-CO_2-CH_2-CH-CH_2 \]

(iii) Glycidyl amines - \[ RRN-CH_2-CH-CH_2 \]

(iv) Linear aliphatic - \[ R-CH-CH-R-CH-CH-R \]

(v) Cycloaliphatic -

By far the most important commercial resin group are the glycidyl ethers of dihydroxy compounds. The diglycidyl ether of bisphenol A accounts for 9% of epoxide resin production.

1.3.1 RESINS FROM DIGLYCIDYL ETHER OF BISPHENOL A

The diglycidyl ether of bisphenol A (DGEBA) is produced by the reaction of bisphenol A and epichlorohydrin at elevated temperatures in the presence of aqueous caustic soda. The DGEBA produced has the general formula:-

\[
\begin{align*}
\text{CH}_2\text{CH}-\text{CH}_2 & \quad \text{O} & \quad \text{CH}_3 \\
\text{CH}_3 & \quad \text{O} & \quad \text{CH}_2\text{CH}-\text{CH}_2 \\
\text{CH}_3 & \quad \text{O} & \quad \text{CH}_2\text{CH}-\text{CH}_2 \\
\text{CH}_3 & \quad \text{O} & \quad \text{CH}_2\text{CH}-\text{CH}_2 \\
\text{n} & \quad & 
\end{align*}
\]
In practice \( n \) usually varies between \( n = 1 \) and \( n = 12 \). The DGEBA is characterized by an "epoxy equivalent" and the weight of resin which contains 1 gram equivalent of epoxide (when \( n = 0 \) epoxy equivalent is 170). A series of resins may be obtained, each grade consisting of a mixture of molecules differing in chain length and molecular weight. Resins of short chain length (low value of \( n \)) are syrupy viscous liquids, whereas resins of longer chain length are hard, brittle amber coloured solids at room temperature. The epoxide resin may be characterized by a number of parameters including: epoxide equivalent, resin viscosity, average molecular weight and distribution, melting point of the solid resin, heat distortion temperature or hydroxyl equivalent (the weight of resin in grams containing one gram equivalent of hydroxyl (34 and 35)).

The chief reasons for the development of resins based on DGEBA are (a) the availability of bisphenol A from the petroleum raw materials and (b) the good mechanical and chemical properties exhibited by these resins when hardened by a large variety of crosslinking agents.

Other hydrogen containing molecules used instead of bisphenol A are monohydric and polyhydric phenols which produce a high degree of cross-linking and tetrabromobisphenol A which is used to produce flame retardant epoxide resins (35).

1.3.2 OTHER RESIN SYSTEMS

1.3.2.1 Epoxy-novolak resins

Since the beginning of resin technology attempts have been made to obtain resin systems which retain their properties at higher temperatures. One approach was to increase the cross-link density in the cured polymer network by using polyfunctional resins or polyfunctional curing agents. This was the case for the epoxidised novolak resin which may be represented generally as follows:
Novolaks based on alkyl substitutes or other phenols can also be used to prepare epoxide resins.

1.3.2.2 Cycloaliphatic resins

In these resin systems the aromatic ring of the glycidyl ether resins is replaced and the compact molecules which increase cross-link density and impart rigidity to the thermoset resin are obtained from cycloaliphatic ring structures. These structures are incorporated to improve the high temperature mechanical strength for long periods and to improve the electrical properties. Examples include (36):

- Cyclohexene oxide
- Tricyclodecene oxide
- Cyclopentene oxide

Where X is an ester, ether, imide or amide and Y is a hydrogen or the cyclic group attached to X.

The cycloaliphatics and the glycidyl ethers react at different rates with the various types of curing agents. The cycloaliphatics react more slowly with aliphatic polyamines but there is much less difference with acid-anhydride curing agents. (35).

The cycloaliphatics form brittle solids with high HDT and may have higher tensile and compressive strengths, lower flexural and impact properties, lower shrinkage and better long term temperature characteristics than the DGEBA resins.

1.3.3 Curing Agents

These are used to convert the epoxide resin into a three-dimensional infusible network held together by covalent bonds. There are many chemical compounds which can be used to cure epoxide resin and the correct choice of a curing agent can be as important as the choice of resin itself, both playing a part in determining the extent and nature of the intermolecular crosslinking. The choice of curing agent to be used depends on (35):

(a) the handling characteristics required or tolerable in the uncured system, such as viscosity at working temperature, pot life, exotherm, and toxicity
(b) the cure and post cure time and temperature requirements
(c) the properties (physical, mechanical, electrical and chemical) required of the cured system
(d) the cost of the curing agent.

Curing agents have been considered in the following broad categories:

1.3.3 Acid and acid anhydride curing agents

Anhydrides are usually preferred because acids produce water which may lead to foaming. The curing mechanism is complex but the first stage is probably the opening of the anhydride ring by an alcoholic hydroxyl group.

Other possible reactions are:
(i) Reaction of the epoxy group with the carboxylic group formed in the above
(ii) Etherification of the epoxy groups
(iii) Reaction of the monoester with an hydroxyl group to form a diester
(iv) Hydrolysis of the anhydride to the acid
(v) Hydrolysis of the monoester to produce an acid and an alcohol.

Reactions (i) and (ii) are most important as they produce crosslinking. Examples of common acid anhydride curing agents are phthalic anhydride (PA), hexahydrophthalic anhydride (HPA) maleic anhydride (MA) and methyl (MA) and methyl nadic anhydride (NMA).

When used with glycidyl ether resins, anhydrides provide cured systems which have good mechanical properties and better high temperature stability than the amine-cured systems. The resin anhydride mixture has a low viscosity, long pot life and low volatility. During the hot cure there is only a little shrinkage of the system and generally low exothermic heat evolution. The limitation to the use of anhydrides is the long and high temperature cure schedules to obtain optimum properties, although the use of accelerators assists in overcoming this drawback (35 and 37).

1.3.3.2 Amino curing agents

Resins may be crosslinked by agents which link epoxy molecules. The reaction with a primary amine leads to the formation of a secondary
hydroxyl group and a secondary amine. This secondary amine can then undergo reaction with another epoxide group to form another secondary hydroxyl group and a tertiary amine.

\[
\text{RNH}_2 + \text{CH}_2\text{OCH} \rightarrow \text{RNH} - \text{CH} - \text{CH} -
\]

\[
\text{RNH} - \text{CH} - \text{CH} - + \text{CH} - \text{OCH} \rightarrow \text{RNH} - \text{CH} - \text{CH} -
\]

Generally, linear and branched primary and secondary aliphatic amines give good fast room temperature cures with DGEBA, resulting in heat deflection temperatures in the range 70 - 160°C depending on the resin, the concentration of the amine and the cure cycle (38). Cyclic aliphatic amines require a high temperature cure and give properties in the cured casting similar to those obtained from aromatic amines. Examples of common aliphatic amines include diethylene triamine (DETA) and triethylenetetramine (TETA).

Modifications have been made to aliphatic amines in order to achieve:

(i) Improved characteristics such as longer pot life, faster or slower cures and resin compatibility.

(ii) Easier handling, lower viscosity and easier mixing.

The most commonly used modifications are those based on adducts of the polyamine with (a) ethylene oxide (b) acrylonitrile (c) diglycidyl ethers (d) compounds containing groups reactive towards the amine group and ketones. The properties of the cured resin are usually similar to those obtained using the unmodified amine.

Aromatic amines are also used because they incorporate a rigid benzene ring into the network and this produces cured resins with a much higher heat distortion temperature. The aromatic amine cured systems also have the advantage of forming dry brittle soluble solids when partially cured (B stage) which flow when heated before finally forming a cured infusible network. This property is very useful in the preparation of dry lay-up laminates.

Unfortunately amines react sluggishly with DGEBA because of steric factors between the amine and epoxide groups, lower basicity than aliphatic amines, and the relative lack of mobility of the polymer network and therefore cures must be performed at elevated temperatures sometimes in conjunction with an accelerator. Examples of common aromatic amines used as curing agents are (39 and 40):

\[
\begin{align*}
\text{M.P.D.A} & \\
\text{D.D.M.} & \\
\text{D.D.S.} &
\end{align*}
\]
To avoid difficulty in mixing eutectic blends have been developed which are liquid at room temperature, alternatively the amine may be dissolved in a solvent (dimethylformamide or butyrolactone (35)) and the resin may be cured at room temperature.

DDS generally gives the highest heat distortion temperature but it is slow to react even at elevated temperature and the addition of an accelerator is desirable (BF$_3$ - MEA) (35).

Polysiloxanes may also be used as curing agents to produce room temperature curing without unpleasant odours to form a tough, flexible cured product.

1.3.3.3. Catalytic Curing Agents

These curing agents achieve cross-linking by initially opening the epoxide ring and causing homopolymerization of the resin. The resin molecules react directly with each other and the cured polymer has essentially a polyether structure. Catalytic curing agents can be used either as a sole curing agent, as a co-curing agent or as an accelerator. The most common catalytic curing agents are Lewis acids and bases, which are substances having either unsheared pairs of electrons in an outer orbital (bases) for bond formation, or empty electron orbitals (acids) which can be used to form a bond with an atom which donates both electrons.

The most important of the Lewis acids is boron trifluoride BF$_3$. BF$_3$ is a highly corrosive gas capable of polymerizing epoxide resins extremely rapidly. To obtain a material suitable for handling, complexes formed between BF$_3$ and amines or ethers are used. The most important of these complexes is between BF$_3$ and monoethylamine known as BF$_3$MEA. The rate of cure using BF$_3$MEA is very sensitive; below 100°C the rate is negligible but at 120°C a very rapid reaction occurs accompanied by considerable evolution of heat and a large rise in the temperature of the casting. Care must be taken when producing castings over 100 g wt. and large castings should not be attempted. The advantage of this system is the long pot life at room temperature.

One of the most important Lewis bases used as a catalytic curing agent is the tertiary amine benzylidimethylamine (BDMA).

\[
\begin{align*}
\text{CH}_2\text{N(CH}_3\text{)}_{2}
\end{align*}
\]
Tertiary amines are able to act as catalysts in the anhydride epoxy reactions by opening anhydride rings.

The above curing agents influence the rate of reaction and the reaction products when used in conjunction with other curing agents and they can therefore be used to help control the properties of the cured product.

1.3.4 ADDITIVES

Other materials may be incorporated to modify the properties of the cured resin.

(i) Diluents - reduce the viscosity and acid impregnation. They may affect pot-life, exotherm, HDT and physical properties. Diluents may be reactive or non-reactive, the former join in the polymerization reactions while the latter merely reduce the resin concentration. Reactive diluents include mono and diepoxide compounds, triphenyl phosphite, toluene and xylene are examples of non-reactive diluents.

(ii) Fillers - generally used to lower the cost of the product, reduce curing shrinkage, decrease the exotherm, increase HDTs, reduce water absorption and to improve the abrasion resistance and compressive strength of the moulding. However fillers may also increase wt and viscosity and reduce the impact and tensile strengths. Common fillers include silica powder, zircon flour and sand.

(iii) Flexibilisers - are used to improve toughness and flexibility of hard and brittle resin systems. This may be achieved by introducing long flexible molecular chains which improve the impact resistance but reduce the tensile strength.

1.3.5 MECHANICAL PROPERTIES OF CURED EPOXY RESINS

The characteristics of the cured resin system are derived from the basic molecular structure of which the following are important:

(i) The extent of cross-linking
(ii) The nature of the resin molecule between cross-links
(iii) The nature of the curing agent molecule.

It is important to try to achieve a high degree of crosslinking for most uses. Crosslinking may be hindered by the trapping of reactive molecules in the polymer network at the gel stage. Post curing at a
temperature above the original cure temperature and above the Tg of the system often alleviates this problem by increasing molecular mobility and opportunity for bond formation (37).

The cross-link density in different systems depends on the functionality of the reacting species and the distance between reactive groups. The nature of the resin or curing agent molecule between reactive groups also has an influence on physical characteristics. High cross-link density is usually associated with high HDT's and glass transition temperatures while chain stoppers and diluents are used to produce resins with low curing shrinkage and increased toughness and elongation.

As the temperature of the polymer is raised through Tg the nature of the polymer changes from a hard, glassy and brittle polymer to one which is flexible and rubbery because relatively large sections of molecular segments can move under the influence of the increased thermal energy which produces rotation of the C-C bonds (42). This change is generally accompanied by changes in other properties such as expansion coefficient, mechanical properties and heat capacity. In some highly crosslinked systems polymers pyrolyse before the change from the glassy to the rubbery state. This is generally the case with epoxides and the Tg of a cured epoxide resin reflects the extent and nature of its cross-linking and is used as a measure of its thermal stability. The method used to investigate Tg include differential thermal analysis and measurement of the dynamic mechanical properties using a torsion pendulum.

The large number of resins and curing agents etc. available may be used to produce epoxy systems with a wide variety of properties. The properties have been assessed generally for unfilled epoxy castings in Table. It should be noted that the cured resin has a low fracture energy (35) and is very notch sensitive.

Lee and Neville (37) have studied initiation and propagation of cracks in this material and they concluded that the fracture is generally initiated at flaws and the crack spreads out in a radial direction until it reaches a critical size corresponding to the applied stress at which it becomes unstable and propogates rapidly. Low strain rates favour crack propagation by decohesion prior to the critical stress whereas at high strain rates the stresses are built up too quickly for decohesion to occur. Experimental observation on the effect of temperature and strain rate on the compressive properties of epoxide resins have been made by Ishai (43), who observed a change in failure mode at various temperatures using a plasticized
The epoxide resin system and the various stress strain curves are shown in Fig. 2. The failure mode changes from brittle at low temperature and high strain rates, to ductile at intermediate temperatures and rubbery at high temperatures. The fracture surfaces which he observed were similar to those described by Andrews (44) in which slow fracture is accompanied by a smooth failure and as the crack velocity increases more energy is available for crack propagation on different planes.

The shear strength of epoxide resins has also been measured (45). The recommended shear strength test involves punching out a circular disc from a resin sheet and the shear strength is given by:

\[ \tau = \frac{P}{2\pi rd} \]

\( \tau \) = shear stress
\( P \) = force in newtons
\( r \) = radius of the disc
\( d \) = thickness of the sheet

The disadvantage of this test is the stress concentration which exists at the edge of the punch. An alternative test which is also used is the plane strain compression test in which the sheet is pressed between two flat discs (46, 47 and 48).

In order to measure the adhesive properties of epoxy resin the lap shear test is used, this will be described in a later section. The adhesive properties of the resin are attributed to the pendant secondary hydroxyl groups which occur along the molecular chain which are strongly adsorbed onto oxide and hydroxyl surfaces (49 and 50). For good adhesive properties it is important to achieve effective wetting of the adherend because incomplete contact results in the formation of stress concentrations. Rigid structures enhance this problem as they are unable to accommodate and dissipate the stresses built up in the system. In the case of composites this problem is offset somewhat by the tendency of the resin to shrink onto the fibre during cure to provide an effective mechanical bond as well as a chemical bond although differences in thermal expansion coefficients of resin and fibre may counteract this advantage.

The epoxide resin properties are extremely versatile and depend on the curing system and conditions which determine the resin structure. The development of good adhesive properties and the avoidance of large voids which produce stress concentrations are important considerations when choosing a resin system for composite fabrication.

1.4 FABRICATION TECHNIQUES OF CFRP

All of the fabrication processes are designed to produce a composite in which the fibre is thoroughly impregnated with resin. The techniques fall
<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elongation at Break</td>
<td>3-5%</td>
</tr>
<tr>
<td>Ultimate Compressive Strength</td>
<td>100-200 MN/m²</td>
</tr>
<tr>
<td>Ultimate Tensile Strength</td>
<td>30-90 MN/m²</td>
</tr>
<tr>
<td>Impact Strength (IZOD)</td>
<td></td>
</tr>
<tr>
<td>Young's Modulus</td>
<td>1.5-3.5 GN/m²</td>
</tr>
</tbody>
</table>

**Table 1** General Properties of Epoxy Resins

**Fig. 2** Compressive Stress-Strain Curves at Constant Strain Rate.

% Strain

0  5  10

25°C, 35°C, 40°C, 50.5°C, 55°C, 61.2°C, 74.8°C
into two main types, wet lay-up and dry lay-up.

1.4.1 WET LAY-UP METHODS

These involve the use of a liquid resin system to impregnate the reinforcing material.

1.4.1.1 Hand lay-up

This is the simplest of the wet lay up methods using curing at contact pressure only. The process is manual and the quality of the laminate depends on the moulder. Either male or female moulds may be used and the mould is first treated with release agent to facilitate component removal. A layer of resin is poured into the mould and successive layers of fibre and resin are added, each layer being thoroughly impregnated before the next is added. Entrapped air may be removed by rolling and the laminate is then allowed to harden usually at room temperature although heat may be applied if possible to increase the rate of cure (35). This method is especially applicable for articles required in small quantities where a large investment in tooling costs is not justified or for complicated articles which would be difficult to mould by other techniques.

1.4.1.2 Vacuum bag technique

This is for better quality laminates and consists of laying up as described in (i) and then placing a flexible bag over the mould which is clamped down round its circumference. A vacuum is then drawn under the sheet and atmospheric pressure forces air and excess resin out of the laminate (51). Laminates produced by this method usually have a lower void content than those produced by simple hand lay up.

1.4.1.3 Resin injection method

The required amount of reinforcement is placed between matched moulds treated with release agent. Resin and curing agent are then mixed and pumped through the mould under pressure removing all entrapped air and impregnating the reinforcement. The laminate is cured with the aid of heaters built into the surface of the mould. This is a useful method for the production of sizeable mouldings (35 and 51).

1.4.1.4 Filament winding

A continuous roving or tape is impregnated with the binder resin and
it is then wound onto a mandrel with the shape of the final product. The rovings are kept in tension throughout the winding process and the pattern is calculated to produce the required hoop and longitudinal strengths. After winding the laminate is usually hot-cured in an oven and then removed from the mandrel, though in some cases the mandrel may be part of the finished article.

Filament winding can be used to produce cylinders and any shape can be made if it can be wound and if it has an axis of rotation (52). Fully automatic machinery has been used to produce pipes, storage tanks, sporting goods etc. and filament winding is one of the most important composite fabricating techniques (53, 54 and 55) because:

a) It produces very high strength to weight ratio composites
b) The composites are highly reproducible with good dimensional tolerance and mechanical performance
c) The process is fully automatic.

1.4.2 DRY LAY-UP METHODS

The major limitation of wet lay-up techniques is that they are restricted to resin systems which have low viscosities at working temperatures and reactive diluents used to overcome this problem often degrade composite properties. The dry-lay-up technique overcomes this problem by impregnating the fibre with either the resin system which is dissolved in a solvent (acetone or MEK) or a molten ‘B’ staged resin. In the case of the solvent method, the solvent is removed in a dry oven and the impregnated fibre is ready for laminating (35).

Liquid and solid diglycidyl ether resins and epoxidized novolaks may be used with a variety of curing agents. Aromatic amines e.g. MPD, DDM and DDS are particularly suitable since they can be partially advanced to the B stage where cure proceeds only slowly at room temperature. The resulting pre-preg material usually has a limited shelf life at this intermediate stage (37). Final curing is carried out in a press at elevated temperatures with possibly a subsequent post-cure.

Pre-pregs with longer shelf lives are obtained by using BF3-MEA as a curing agent since it does not react at room temperature. Acid anhydrides are also used to prepare dry lay-up systems but the cure accelerator usually prevents the possibility of 'B' staging and the
Impregnated material must be stored under refrigerated conditions. Dry filament winding has also been used (51) final cure being performed in an oven. Laminating with prepreg enables close control of $V_f$ of the reinforcement and therefore the composites produced usually have consistent properties.

1.4.3 MATCHED DIE MOULDING

This technique may be used for both wet lay up and dry lay up methods. The cure is carried out while the material is restrained between two mould surfaces. (51). It is usually an automated, high production process in which the resin is added to a pre-cut, shaped sheet or a preform positioned in a heated mould and pressure is usually applied hydraulically.

The lay up is placed on one of the matched moulds and the other mould is pressed against it. The moulds are usually heated, liquifying or reducing the viscosity of the resin and enabling it to wet the fibre. Excess air may be removed and since air is more readily soluble in liquids under pressure, some of the air is dissolved permanently in the resin producing a non-porous product.

Articles made by matched die moulding are usually stronger, less porous and have a better finish than parts moulded by vacuum bag methods. The method is suitable for large scale production of parts requiring high dimensional accuracy. The leaky mould technique is a variation of the matched die moulding which is used in the laboratory.

1.5 DESIGN CONSIDERATIONS FOR CFRP

To designers familiar with approximately isotropic, homogeneous and ductile metals, composites present many unfamiliar characteristics some of which are favourable and some detrimental. Several workers have given excellent reviews of the material characteristics which affect the designer and the design philosophy (61 - 67). Among the most important properties which affect design considerations are:

1.5.1 Strength and Stiffness

The high specific strength and stiffness of CFRP have already been noted, but the anisotropic nature of these properties means that care must be taken in designing CFRP components and the orientation of the fibres must match the load/stiffness requirements. Carbon fibre composites do not possess a plastic
range in the fibre direction and although Petit and Waddoups (68) have shown that transverse stress-strain behaviour is non-linear, this is small however compared to normal engineering materials such as mild steel or aluminium.

1.5.2 Fracture Characteristics

The anisotropic nature of the composite also affects the fracture behaviour and the designer must be able to predict the static and fatigue strengths of a composite and its susceptibility to delamination. An understanding of fracture initiation and propagation mechanisms as well as the effects of local and concentrated loads and other stress raisers is essential to avoid in-service failures.

1.5.3 Thermal Expansion Characteristics

Generally the thermal expansion characteristics of a metal are approximately uniform and positive. This is not the case in an anisotropic composite, which has a zero or negative thermal expansion coefficient longitudinally and a positive thermal expansion coefficient transversely. The designer must take this into account when designing components especially when compatibility with metals is required.

1.5.4 Built-in stresses

Built-in stresses from fabrication may be reduced or relieved in a metal by appropriate annealing treatments. In a composite however there is generally a shrinkage during cure which may produce stresses which cannot be relieved. The effect of these stresses on the mechanical properties should be assessed in order to allow safe utilization of components.

1.5.5 Environmental characteristics

The effect of acids, salts, water and solvents must be studied with special reference to the environmental requirements of a particular component. One of the disadvantages of CFRP is its susceptibility to erosion damage and for some applications this may be important and a protective surface coating must then be applied.

1.5.6 Fabrication characteristics

Conventional metal fabrication techniques are very well established and standard treatments are available to produce desired properties. This
enables the metallurgist to produce high grade metals in a final form with a typical coefficient of variation of about 3%. In the case of composite technology the finished material is produced as required and a wide range of lay up techniques can give the designer the opportunity to control the properties of the composite in each direction. Unfortunately the coefficient of variation in the mechanical properties of nominally identical materials is about 10 - 15% which makes designing for safety more difficult than for metals.

1.5.7 Economic considerations

It is well known that carbon fibre is very expensive to produce. This has meant that the initial use of CFRP was limited mainly to the aerospace industry and components which normally require complicated machinery.

1.6 TESTING OF C.F.R.P.

The first consideration when testing material is with theoretical aspects based on the nature of the composite material in order to get some idea of the likely properties and failure modes which may occur in practice.

Kelly (50) has calculated the strength of a composite material based on a law of mixtures prediction. If a composite containing more than a certain volume Vmin of continuous fibres is loaded in the direction of the fibres the UTS of the composite is reached when the strain in the composite is equal to the breaking strain of the fibres.

\[
\sigma_c = \sigma_f V_f + \sigma_m (1 - V_f) \quad V_f > V_{\text{min}}
\]

\(\sigma_c\) is the UTS in the composite \(\sigma_f\) is the UTS in the fibre \(\sigma_m\) is the tensile stress in the matrix when the composite is strained to its UTS and \(V_f\) is the volume fracture of fibres.

In the case of brittle carbon fibres, the failure of one fibre will cause transference of the load carried by that fibre back into the matrix. Aveston (57) pointed out that if the breaking strain of the fibre is less than that of the matrix then single fracture occurs when:

\[
\sigma_f V_f > \sigma_u (1 - V_f) - \sigma_m (1 - V_f)
\]

where \(\sigma_u\) is the UTS of the matrix. The equation means that when the fibres break, the matrix is unable to withstand the additional load transferred to it. If however:

\[
\sigma_f V_f < \sigma_u (1 - V_f) - \sigma_m (1 - V_f)
\]
the fibres will be successively fractured into shorter lengths by multiple fracture until the matrix reaches its failure strain. The change from multiple fracture to single fracture defines $V_f^{\text{min}}$. Figure 3 illustrates the conditions under which multiple fracture should occur.

1.6.1 STRESS TRANSFER

If multiple fracture occurs, the stress carried by the fibre which fails must be transferred to other fibres via the matrix and the stress distribution changes in the region close to the break. Kelly (56) has studied the stress transfer between the matrix and the interface in a composite containing fibres each of constant length and stressed parallel to the fibre axis. The axial displacements in the fibre and matrix are different because of the difference in elastic module of the two components. Shear strains are produced on planes parallel to the axis of the fibre in the direction of the axis. The strain and resulting shear stress are the means by which load is transferred from the broken fibre, and Kelly (56) has used equations derived by Cox and Rosen (58, 59) to relate the maximum shear stress $\tau_m$ in the matrix at the fibre matrix interface to the maximum tensile stress $\sigma_{\text{max}}$ in the fibre which for a long thin fibre is given by

$$\tau_m = \frac{G_m}{\sigma_{\text{max}}} = \sqrt{\frac{G_m}{2 E \log R r_o}}$$

where $G_m$ = shear modulus of the matrix
$E_f$ = Young's mod. of the fibre
$r_o$ = radius of the fibre
$R$ = half of the centre-centre separation of the fibres normal to their length

This equation represents the upper limit to the stress which may be transferred into the fibre since if $\tau_m$ is exceeded shear failure occurs in the resin. The stress distribution is shown schematically in (4) where $l_c$ is the critical length beyond which the fibre may be loaded to its breaking stress and

$$l_c = \frac{\sigma_f}{\tau_y}$$

$\tau_y$ = yielded stress of the matrix
$\sigma_f$ = breaking stress of the fibre
$r_o$ = radius of the fibre

For polymer matrices Outwater replaced $\tau_y$ by $\mu n$ where $\mu$ is the coefficient of sliding friction between fibre and matrix and $n$ is the radial pressure due to shrinkage of the fibre and represented by the
FIG. 3 CONDITIONS FOR MULTIPLE FRACTURE
pressure exerted by a thin walled tube of thickness \( t/2 \), internal diameter \( d \) and hoop stress \( F \)

\[ \tau = \frac{Ft}{d} \]

in this case \( t \) is the surface-surface separation of the fibres, \( d \) their diameter and \( F \) the yield point of the resin. The fibres may be broken down into shorter lengths as the strain is increased provided \( l_c \) is exceeded and \( l_c \) is an important parameter in the control of composite properties.

1.6.2 TRANSVERSE PROPERTIES

If the composite undergoes an extension in the transverse direction, the stress is the same in each component and the total strain in the transverse direction is given by:

\[ \varepsilon = \left( \frac{V_f}{E_f} + \frac{(1-V_f)}{E_m} \right) \varepsilon \]

and the approximate transverse elastic modulus \( E_T \) is approximately:

\[ E_T \sim \frac{E_m}{1-V_f} \]

The transverse shear modulus \( G_T \) may be calculated similarly

\[ G_T = \left( \frac{V_f}{G_f} + \frac{(1-V_f)}{G_m} \right) \tau \]

\( \gamma = \) shear strain

\( G_f = \) shear modulus of the fibre

\( G_m = \) shear modulus of the matrix

and since \( G_f \gg G_m \)

\[ G_T \sim \frac{G_m}{1-V_f} \]

This situation may be represented by a model consisting of elastic spring and viscous dashpots (5). In this case the fibre is represented by the stiff spring. The behaviour in transverse tension and axial shear is mainly governed by the properties of the matrix or the fibre matrix interface whichever is the lesser.

The composite therefore behaves anisotropically and this must be taken into account when designing components of CFRP. In order to obtain useful information from mechanical tests it is important to understand the design for CFRP and the parameters which control composite properties.
FIG. 4  STRESS VARIATION IN A FIBRE
OF LENGTH L
The bend test is one of the simplest tests used for the determination of the mechanical properties of composite materials. It is generally performed in either the three or four point mode. If a length of beam is acted upon by a constant bending moment, the stresses set up on any cross-section must constitute a pure couple equal in magnitude to the bending moment. One part of the cross-section is in compression and the other part is in tension. If the following assumptions are made (69):—

a) The material is homogeneous, isotropic and has the same value of Young's modulus in tension and compression.

b) The beam is initially straight and all longitudinal filaments bend into circular arcs with a common centre of curvature.

c) Transverse cross-sections remain plane and perpendicular to the neutral surface after bending.

d) The radius of curvature is large compared to the dimensions of the specimen.

e) The stress is purely longitudinal and local effects near concentrated loads are neglected.

f) The lateral stresses are neglected and the problem is treated as one of one dimensional stress.

The magnitude of the maximum tensile or compressive stress may be calculated by the use of elementary elastic theory using:—

\[ M = \frac{E}{I} = \frac{\sigma}{R} \]

- \( M \) is the bending moment.
- \( I \) is the moment of inertia.
- \( E \) is the Young's modulus of the material.
- \( R \) is the radius of curvature of the neutral axis.
- \( \sigma \) is the longitudinal stress.
- \( y \) is the distance from the neutral surface.

1.6.3.1 Application of bending theory to the 3 and 4 point bend test

By applying equation to the three and four point bending situations it is possible to obtain expressions for the maximum bending (flexural) stress \( \sigma_{\text{max}} \) & the maximum bending modulus \( E_b \). The variations of the bending moment in three and four point bending are shown in Fig. 5 and 6.

(i) Three point bending.

The flexural stress is a maximum at the surface of the beam (Fig. 5)
\[ \sigma_{\text{max}} = \frac{3PL}{2bd^2} \]

\( b = \text{beam width} \)
\( d = \text{beam thickness} \)
\( L = \text{span between outer rollers} \)
\( P = \text{force} \)

The bend modulus \( E_b \) is given by
\[ M = \frac{Eb}{R} \]

where \( R \) is calculated from the equation (70).
\[ \frac{1}{R} = \frac{d^2}{dN^2} \]

Thus the bend modulus may be obtained by integrating twice and
\[ E_b = \frac{PL^3}{4bd\gamma} \]

where \( \gamma \) is the mid-span deflection of the beam.

(iii) Four point bending.

From Fig 6 the maximum bending moment is given by
\[ M = \frac{PL}{2} \]

The maximum flexural stress at A is given by
\[ \sigma_{\text{max}} = \frac{3PL}{bd^2} \]

Calculating the bend modulus is more difficult than in the case of three point bending. The deflection must be split up into three parts (Fig. 6). \( Y_1 \) is calculated as in three point bending, \( Y_2 \) is given by the slope of the beam at point A and \( Y_3 \) is the deflection of a cantilever beam with a load \( P/2 \) at the end from the tangent at 0. Combining these contributions, \( E_b \) is given by (71):
\[ E_b = \frac{-P|l|}{2I(\gamma_1 + \gamma_2 + \gamma_3)} \left[ \frac{l_1^2}{3} + \frac{l_2^2}{2} + \frac{l_3^2}{8} \right] \]

1.6.3.2 Shear stress in beams.

The shearing force at any cross-section of a beam sets up a shear stress on transverse sections which in general varies across the section (69). The shear stress on transverse planes causes an equal complementary shear stress on longitudinal planes parallel to the neutral axis. The shearing force diagrams are shown in Fig. 7.

Following assumptions are made:

a) The stress is uniform across the width.

b) The shear stress does not affect the bending stress.

Elastic theory can be used to show that the shear stress \( \tau \) is given by:
\[ \tau = \frac{3P}{bd^3} \left[ \frac{d^2}{4} - \gamma \right] \]
FIG. 5  THREE POINT BEND TEST

\[ \frac{y_3}{y_2} = \frac{l_1}{l_2} \]

FIG. 6  FOUR POINT BEND TEST
FIG. 7 SHEARING FORCE DIAGRAMS
Figure 7 shows the shearing force diagrams for three and four point bending. It is evident in the four point bend test there are no shearing forces in the region between the two inner rollers. Equation represents a parabolic variation of shear stress with $y$ and the maximum shear stress occurs when $y = 0$ and at the neutral axis.

$$\tau_{\text{max}} = \frac{3P}{4bd}$$

### 1.6.3.3 Comparison of the three and four point bend test.

The four point bend test is generally favoured for the determination of $\tau_{\text{max}}$ for the following reasons (72):

(i) The specimen undergoes pure bending between the inner pair of loading points and there is no shear component.

(ii) Between the inner loading points there is a uniform stress and strain along the specimen surface, whereas in the three point bend test the peak stress is concentrated near the loading points. A much larger area of the specimen is therefore tested in four point bending and the results are therefore more representative.

(iii) Local damage at the loading points is much less likely in the four point bend test because the load required to produce an equivalent stress is divided between two loading points.

### 1.6.3.4 The effect of local stress concentration in the three point bend test

The large local stress concentration beneath the central loading point is ignored in the elastic theory equations. In the case of a narrow rectangular beam the irregularities due to the local stress concentration at the loading points may be studied by using the solution for stress distribution in an infinitely large plate subjected to a concentrated force (70 and 73).

The Principle of Superposition is used to determine the local stresses. This Principle states that if several transverse forces all having the same direction are applied to the beam simultaneously, the bending moment produced at any cross-section is equal to the sum of the bending moments produced at the same cross-section by the individual loads acting separately. It follows that the deflection produced at any point of a beam by a system of two simultaneously acting transverse forces can be obtained by summing the deflections produced at that point by the individual loads. The Principle is valid only if small displacements in deformation do not affect the action of external forces, if this is not the case (and beyond the elastic limit) then the Principle of Superposition is invalid.
Another approach to the investigation of the stress distribution near the point of application of a concentrated load was made by Karman and Seewald (74) who represented the stresses in terms of a Fourier integral. Seewald showed that the stress can be split into two parts, one of which can be calculated by the elementary beam theory and the other which represents the local effect near the point of application. The local stresses decrease very rapidly with increase in distance from the point of application. There is good agreement between the above methods except at the surface of the beam and when $y = d/2$.

Practical evidence for the effect of local stress concentrations is given by Kedward and Hindle (75) who used Moire birefringent coatings and brittle lacquers. The importance of local stress concentrations means that great care must be taken to choose a supporting system which minimizes the large variation in local stresses.

Ogorciewicz and Mucci (76) examined several methods of supporting specimens in a bending test, these include: knife edges, large radius rollers, rotating roller supports and rotating rollers on swinging links. Line contact between specimen and supports is assumed when calculating the stress derived in the beam from elastic theory. A line contact may be achieved by using knife edges, but this presents the problem of severe indentation. The use of large radius rollers on the other hand eliminates this problem, but the point of contact between the supports and the specimen changes and this is not accounted for in the simple bending theory. Ogorciewicz and Mucci concluded that rotating rollers offer no advantage over fixed rollers.

1.6.3.5 Application of bending to CFRP.

In comparison with other test methods such as unidirectional tension or torsion, bending represents the most inexpensive and widely used test configuration. Beam specimens may be used to measure both flexural and shear properties with the span-to-depth ratio governing the mode of failure.

Consider a three point bend test of a composite in which the reinforcing fibres are aligned parallel to the span length so that the principal axes of orthotropy coincide with the axes of symmetry of the beam. From equations

\[ \tau_{\text{max}} = \frac{3P}{4bd} \]
\[ \sigma_{\text{max}} = \frac{3PL}{2bd^2} \]
The assumptions used to derive these expressions are given in section (163). Most composites with parallel fibre arrays are considered macroscopically homogeneous and because their strength is governed by the very strong and stiff fibres, they exhibit very nearly linear elastic response to failure. For very short beams where the stress field is influenced by local stress concentrations at the contact points, the plane sections of the beam do not remain plane after bending.

The above equations may be combined to obtain a relationship between maximum shear stress and maximum flexural stress as follows:

$$\tau_{\text{max}} = \frac{C_{\text{max}}}{2 \left(\frac{L}{d}\right)}$$

Figure 8 shows $\tau_{\text{max}}$ plotted against span-to-depth ratio. Point A represents the transition between flexural failure and shear failure and at values of $L/d$ below $(L/d)^{\text{flex}}_{\text{ratio}}$, shear is the primary mode of failure. In practice this sharp transition is not observed (77, 78 and 79) and mixed mode failures are common around the transition point. Mullin and Kn oell and Sattar and Kellog (79) have done beam experiments on type II carbon fibre/epoxy resin composites at various $L/d$ ratios. They found that although shear failures occur around the transition point the value of $\tau_{\text{max}}$ increases at lower values of $L/d$ (Fig 8) indicating a mixed mode type failure. The flexural strength properties exhibit a dependence on the span-to-depth ratio and a mixed mode of failure makes meaningful calculations of shear strength very difficult, Mullins and Knoell (78) also found that void content and variations in fibre distribution have a significant effect on the calculated shear strength of the beam. In order to reach the value of $\tau_{\text{max}}$ shown in Fig 8 it is necessary to use an $L/d$ ratio of approximately 3:1. Unfortunately the stress distribution at such low $L/d$ ratios is very difficult to calculate because of the large local stresses beneath the central loading roller, and the non elastic behaviour of the beam. Another disadvantage of this method of shear testing is the small volume of material subjected to the maximum shearing stress (80).

Several workers have commented on the use of short beam tests for the determination of ILSS (33, 77, 81, 82, 83, 84). The values obtained are extremely sensitive to fibre $V_F$ and span-to-depth ratio and failure rarely occurs in the classical manner predicted by the elastic theory equations (and shear failure on the neutral plane). Characterization of fracture surfaces and proposed mechanisms for failure have been investigated by Daniels, Harakas and Jackson (84), who carried out short beam tests on treated and untreated fibre composites. They classified shear failures into various failure modes (Fig 9) including:
FIG. 8 EFFECT OF \( \frac{L}{d} \) ON THE CALCULATED SHEAR STRENGTH
FIG. 9  SHORT BEAM FAILURE MODES
a) Discrete shear failures characterized by a single crack which may be flat, irregular or only at the side of the specimen. These correspond to brittle behaviour and show a load drop.

b) Homogeneous shear failures in which there is no visible sign of failure after the load has passed through a maximum. These failures are assumed to have occurred by a shearing mechanism which is homogeneously distributed throughout the sample. In this case there is no sharp load drop.

c) Permanent deformation and compression jamming may occur especially at high temperature. The load-deflection curves exhibits a point of inflection in the case of compression jamming because a point is reached where the forces are transmitted to the sample in compression rather than in shear.

d) Tensile failures may be either through the specimen or confined to the outermost fibres.

e) The compressive stress beneath the central roller can also cause failure of the beam because of the local stress concentration.

The mode of failure in (e) was not observed by Daniels et al, but it has been reported by other workers (77 & 81) in the short beam tests and by Hancov in impact tests. Harris, Novak and Bader et al have also commented on the effect of fibre treatment on the value of ILSS obtained using the short beam test and a change in fracture mode has also been observed. The ILSS of CFRP falls slightly with increasing Vf if the fibre is untreated and increases with Vf if the fibre resin bond has been improved by fibre surface oxidation. Bader et al noted that shear failures are generally observed in untreated type I composites and at high values of Vf in other materials. Most treated fibre composite fracture surfaces have two distinct failure zones, a relatively smooth compression region beneath the central loading roller and a tensile region which exhibits fibre pull out. Where interlaminar failure occurs it is often associated with flexural failure and it is frequently away from the neutral plane at which the maximum shear stress should be exerted.

The results of flexural tests also require careful interpretation. It is important to avoid premature compressive buckling (82) which lowers the value of γmax, obtained. A careful analysis of the failure mode during beam testing is necessary in order to understand the fracture process and the applicability of the elastic theory equations.

1.6.4 DIRECT SINGLE SHEAR TEST

This is an alternative method to determine the ILSS in which notches are made in the specimen to produce a shear failure when the specimen is loaded in
tension or compression. This type of test is used for the assessment of adhesive joints and in a suitable form it can be used on reinforced plastic joints. In its simplest form it consists of a plane parallel-sided specimen notched on opposite sides and gripped in a tensile testing machine. The disadvantage of this test is the stress concentrating effect of the notch. Volkersen (86) and Goland and Reissner (87) have examined this effect and conclude that the ideal conditions to avoid large stress concentration are:

(i) A flexible resin,
(ii) A shaped or rotated joint,
(iii) A small overlap region,
(iv) High modulus adherend,
(v) Thick adherend and adhesive layers.

There has been very little work relating DSS shear results for CFRP to the above factors although several workers have obtained results for shear strength from DSS tests. Ball and Raymond (88) compared the DSS test with the short beam test and found that the latter gave higher results probably because of the stress concentrating effect of the notch and they concluded that in the DSS test failure always occurs on a predetermined plane whereas the short beam often failed in a mixed mode. The DSS test is more searching in its exposure of interlaminar weakness although more work is required to ascertain which test is most suitable for ILSS determination.
Fracture toughness is a measure of the ability of the material to resist fracture. Materials with low fracture toughness are susceptible to catastrophic failure. There are three parameters which may be used to describe the crack resistance: \( K_{IC} \) characterizes the intensity of the stresses at the crack tip in the cleavage mode of the fracture; \( Y_F \) is the local work required to form unit area of crack \( (33) \); \( G_{IC} \) is the rate of release of elastic strain energy as the crack grows \( (i.e. 2Y_F) \). There are several different methods of calculating these parameters, \((114)\).

Impact Tests

Many materials behave in a brittle manner under impact loading conditions and the energy absorbed by a specimen from the pendulum in a rapid test such as the Charpy or Izod test is one of the easiest properties to measure. Unfortunately these tests produce results which are difficult to relate to service conditions and also sensitive to specimen geometry. A very sharp notch is often used to prevent the energy of crack initiation from contributing to and obscuring the result, it also ensures that failure occurs at the largest flow which is of known dimensions. The results of pendulum tests are quite often comparable with those of more sophisticated controlled crack propagation tests \((33)\) and fracture energy \( Y_F \) is given by:

\[
\begin{align*}
\gamma_F &= \frac{U_F}{2bd} = \frac{U}{2} \\
\gamma_{NF} &= \frac{U}{2b(d-n)} = \frac{U_{NF}}{2}
\end{align*}
\]

\( U_F = \) Impact fracture energy
\( U = \) energy absorbed
\( d = \) thickness of specimen
\( b = \) width of specimen
\( U_{NF} = NI \) fracture energy
\( n = \) notch depth

There are several contributions to the energy absorbed by the specimen:

(i) The work of fracture of the matrix
(ii) The work of fracture of the fibres
(iii) The energy required to debond the fibres from the matrix
(iv) The elastic energy release when the fibre and matrix debond
(v) The frictional work required to pull the broken fibre from the cracked matrix
(vi) The plastic work done in shearing fibres that are not normal to the crack face
(vii) Parallel splitting
It is important to understand how different parameters affect the energy absorbed and the fracture mode.

(I) Strain energy release rate

Harrison (89) observed that cracks propagate in their own plane if the strain energy release rate $G$ for propagation in that direction equals the corresponding energy to create unit area of crack. A necessary condition for split formation at the end of a crack whose plane is perpendicular to the fibres is:

$$\frac{G_X}{G_Y} < \frac{R_X}{R_Y}$$

$R_X$ and $R_Y$ are the energies to create unit surface area $G_X$ and $G_Y$ are the strain energy release rates for propagation perpendicular and parallel to the fibres respectively. The load at which a split forms is such as to make $G_Y = R_Y$. Anything which affects $G_X / G_Y$ and $R_X / R_Y$ may influence the failure mode of the composite.

(II) Geometric factors

The most important geometric factor influencing the impact behaviour of unidirectional CFRP is $1/d$ ratio. The tendency for parallel splitting increases at lower $1/d$ and Hancox (90) found that for constant width of specimen impact energy increases non-linearly with depth, approximately as its square. Ellis and Harris (91) on the other hand obtained a linear relationship between impact energy and depth. Increase in impact energy with depth may be attributed to the contribution of shear strain energy in thicker specimens (90) and therefore properties must be related to the geometry and care must be taken when scaling results up or down.

(iii) Fibre type

Type II fibre composites exhibit a greater impact resistance than those of Type I with corresponding surface treatments (92) and this is attributed to the strain energy stored in the specimen which is proportional to $\sigma^2$. Et al (92) ranked composites according to Charpy impact performance using a model based on the strain energy in the specimen and independent of fibre/matrix bond strength. Bader et al (92) proposed a model which accounts for other contributions which vary according to the composite failure mode. Fractures are classified as:

(a) Brittle - The strain energy requirements for crack propagation are derived from the strain energy stored in the test piece and the impact energy is given by:

$$U_B = \frac{d}{18} \frac{\sigma V_t}{E_f}$$
(b) Progressive:- The crack moves forward in a series of small steps at a slower rate than the pendulum. The cross section and the stiffness of the test piece is progressively reduced, but the pendulum continues to transfer energy to the specimen and the impact energy $U_p$ is given by:

$$\frac{U_p}{U_B} = 2.33$$

(c) Multiple shear:- Failures occur parallel to the fibre axis and the stiffness of the beam is reduced and there is a decrease in the reaction of the pendulum. The energy absorption is more difficult to estimate in this case since there is no way of predicting the number or position of interlaminar failures. Bader et al did a specimen calculation for a beam which splits into four laminates and found that:

$$\frac{U_m}{U_B} = 3.09$$

These values correspond well with those observed experimentally and a general assessment of impact behaviour may be obtained from the fibre strength and fibre modulus. The failure mode may be predicted from a consideration of interface properties and the relative values of $G_\chi$, $G_\gamma$, $R_\chi$ and $R_\gamma$.

(iv) Fibre surface treatment

Surface treatments are used to increase the adhesion between fibre and matrix, it also increases the value of $R_\gamma$ two or three times and this may affect the failure mode (33). Treated fibre composites generally fail by transverse cracking while the untreated fibre specimens exhibit multiple shear cracking with higher energy absorption.

Models based on pull out (94), the work done in extracting fibres from a broken matrix, and debonding (95), the work done in breaking the fibre matrix bond, have been proposed. Experimental work (96, 97 and 98) indicates that in a ductile matrix fibres pull out of the matrix while in a brittle matrix debonding occurs before pull out. The relative contributions of pull out and debonding have been assessed (95 and 99). The work done in debonding appears as strain energy in the fibre and if debonding occurs over a length $\chi$ the strain energy in the fibre is given by:

$$W_D = \frac{\pi d^2}{8 E} \int_0^\chi \left( \sigma - \frac{4 \tau}{d} \right)^2 d\chi = W_d d\Pi_\chi$$

$W_d$ = energy/unit area to debond the fibre and matrix.

$W_D$ = Total work done debonding the fibre and matrix.

The maximum value of this work is when $\sigma$ reaches the breaking strength of the fibre at $d$

$$\chi = \frac{d \sigma_f}{4 \tau}$$

Integrating $W_D = \frac{\pi d^2}{24} \left( \frac{\sigma_f}{E} \right) \sigma_f \chi$
The work required to pull a fibre from the matrix over a distance is given by (99): 
\[ W_{PO} = \frac{1}{2} \pi d \tau x^2 = \frac{1}{8} \pi d^2 \sigma f x \]
and the ratio of the maximum work of pull out to the work done in debonding is:
\[ \frac{W_{PO}}{W_D} = \frac{3E}{\sigma f} \]
For carbon fibres \( \frac{W_{PO}}{W_D} \) is usually large. If the work of fracture is large \( \tau \) must be small otherwise the fibre breaks rather than debonds. If \( \tau \) is small \( \frac{W_{PO}}{W_D} \) is large, but if 
\[ \frac{W_{PO}}{W_D} > \frac{\sigma f d^2}{8E} \]
debonding is impossible and the fibre breaks without debonding or pulling out.

Bradshaw, Dorey and Sidey (100) studied the impact behaviour of boron composites and conclude that there is no increase in impact energy with fibre diameter which contradicts the proposed model. They point out however that increased fracture energy with larger fibre diameter may be confirmed in controlled fracture experiments.

(v) Volume Fraction
The energy absorbed during impact is related to fibre \( V_f \) by the rule of mixtures fairly closely up to 0.6 \( V_f \) (92). At about 0.7 \( V_f \) however a maximum is reached probably because of the difficulty in producing sound composites at high \( V_f \)'s.

(vi) Strain Rate
At sufficiently high strain rates increasing shock wave inertia effects and decreases in relaxation processes produce changes in impact performance. Compression shock waves reflected from the back of the specimen may produce a greater tendency to failure interlaminarly (101). Bradshaw, Dorey and Sidey found that the threshold energy for initial damage is insensitive to strain rate up to impact velocities of 300 m/s.

(vii) Resin modifications
If the fracture strain of the resin is below that of the fibres the resin fails first and the impact performance is poor. This is therefore generally avoided in practice. It is important to understand whether further
increases in matrix strain, obtained by chemical modification or the use of plasticizers will improve the impact properties of the composite. Impact tests on GRP (102) indicate that an increase in matrix strain allows the development of greater shear, provided that the matrix bond strength is not significantly reduced. This increases the threshold for initial damage and where further damage involves propagation of the delamination there may be an increase in impact energy, but where failure occurs transverse to the fibres little benefit is derived from a plasticized resin (103).

(viii) Temperature

Barker et al (104) and Bader et al found that in broken impact specimens the proportion of tensile (fibrous) fracture surface in a composite decreases as the temperature is raised. Barker suggested that more energy is required to initiate and propagate a tensile failure than a compressive failure because of the increase in fibre pull out and the impact energy should decrease at higher temperatures. This may be modified near the resin Tg because of the increase in resin toughness and ductility. Barker also studied various fibre resin combinations and found that the impact energy was much more sensitive with temperature in the untreated fibre composites, this may be linked with the different mode of failure.

OTHER FRACTURE TOUGHNESS TESTS

Double Cantilever test

This test consists of a cantilever bar with side grooves which guide the crack down the specimen when the cantilever arms are pulled apart (105 and 106). Elastic beam theory is then used to calculate \( Y \) the fracture surface energy.

The test is useful for transparent polymers but with composites it is very difficult to follow a crack which is bridged by fibres along its length. The mechanism of failure agrees well qualitatively with the models predicted by Kelly and Outwater but quantitative measurements are difficult and unreliable. In the case of fibres aligned parallel to the fracture plane, the crack can be followed more easily, but the results are again difficult to interpret because of fibres crossing the fracture plane and it is therefore of limited use.

Slow bend test

The slow bend test (107) may also be used to obtain a value for fracture energy of filled and unfilled resin systems. It consists of propagating a crack into an increasing area thereby controlling its rate of propagation. Bader et al found that the results obtained from this test correspond well with impact tests except at high \( V_f \) s.
At low $V_f$'s failure is generally progressive and occurs in a number of steps from the apex to the base of the triangular cross-section, but at high $V_f$'s interlaminar splitting occurs as the main crack propagates parallel to the fibres. The factors affecting the energy and type of failure obtained should also affect the results of the slow bend test.

1.6.6 FATIGUE

Fatigue failures is one of the most common service failures in metals and it is helpful if the test methods used in the fatigue testing of CFRP are related as closely as possible to those of metals. There are various possible testing modes; rotating, plane bending, axial loading, torsion and special tests including component tests etc. (108). Bonding is one of the most important testing methods of CFRP.

Cyclic fatigue occurs in homogeneous materials as a result of localized plastic deformation which first forms surface defects and then facilitates the spread of these defects under the fluctuating stress (Harris 33), and in polymers this deformation results from the viscous motion of polymer chains. In fibre reinforced materials there are no cyclic fatigue effects on composites tested in tension (33, 109 and 110), the fibres support the load completely and the total strain range in the composite is small because of the high modulus of the fibres, there is therefore little scope for either matrix heat build up (33) or matrix crazing and cracking. Owen and Morris (110) performed experiments on cross-plied CFRP and concluded that the behaviour is similar to that of the unidirectional equivalent.

Owen and Morris and Harris have shown that repeated bending has a significant effect on the S-N curve producing a definite negative slope on a log-log plot, this is more pronounced in reversed bending. Owen (108) also showed that the failure in interlaminar shear fatigue was similar to that observed in the static tests and the initial failure occurred on the neutral plane in the untreated fibre composites, while a mixed mode failure generally occurred in treated composites. Damage due to roller indentation observed in static bending was enhanced during fatigue (81 and 82).

One of the problems of using CFRP is the difficulty in predicting a satisfactory failure criterion because of the various possible failure modes. For certain applications where the avoidance of leakage is important, debonding must be prevented. Smith and Owen have established a failure criterion for GRP based on the strain at the onset of debonding. In both GRP and CFRP the anisotropic nature of the composite controls the fatigue behaviour and ILSS, fibre buckling strength and transverse shear strength may be the controlling
parameters in the fatigue of CFRP.

There have been several attempts to use predictive rules for CFRP (108), these criteria are of limited importance however because the predictions tend to be too optimistic. In metallic structures it was realized that the prediction of safe life is concerned with the knowledge of the initial size of flaws, the crack propagation rates and critical crack sizes (108). In small specimens resin cracking occurs uniformly throughout during fatigue until just before final failure when a localized region of intense damage occurs (108). It is possible in small specimens for a large damage zone to precede a running crack under a single application of load and Owen and Rose (111) consider that this casts doubt on the validity of critical stress intensity measurements. They found that as the specimen size is increased the micro-damage zone becomes relatively smaller and in some cases in GRP, Paris' crack propagation relationship is applicable (112):

\[
\frac{da}{dn} = C \Delta K^m
\]

\( \frac{da}{dn} \) is the rate of crack growth
\( C \) is a material constant
\( \Delta K \) is the alternating stress intensity factor
\( m \) is the number of cycles required for a crack of length \( a \) to grow to the critical size

The objectives of fatigue testing are as follows (108):

1. To understand the fundamental failure mechanisms
2. To determine the comparative fatigue resistance of materials
3. To determine the effect of material composition, processing and environment on the fatigue properties
4. The formulation of predictive design rules
5. Confirmation of the design predictions by testing the components.
In this section the various experimental techniques used to prepare the composites and to determine their properties are described.

2.1 PREPARATION OF COMPOSITES

The composites were made in one of two ways.

2.1.1 Pre-impregnated material

Type II (HTS) carbon fibre manufactured in continuous lengths by Courtaulds Limited was made into 1" wide prepreg tape 0.01" thick by CIBA-ARL using their resin system DLS 60. The tape was laminated by British Aircraft Corporation into bars each bar being 25 x 25 mm section, with 10 laminates in each section, and 1.2 m long. The fibre content of the finished moulding was nominally 0.60 Vf and the material was supplied in the fully cured condition.

2.1.2 Wet lay-up material

The leaky mould technique was used to produce carbon fibre composites using the following fibres:-

(a) MORGANITE MODMOR Types I and IS high modulus fibres
(b) MORGANITE MODMOR Types II and II S high strength fibres

In each case S denotes surface treated fibre.

The fibres were moulded in a SHELL epoxy resin system, EPIKOTE 828 with hydrophthalic anhydride HPA as a curing agent and benzylidimethyl amine BDMA as a catalyst in the proportion 100:80:1. This resin system was used because its low viscosity and long gel time facilitated good wetting of the fibre. The moulding procedure was carried out as described below.

A quantity of resin in excess of moulding requirements was weighed out in a beaker and the calculated weights of curing agents and catalyst were added. HPA is a white solid at room temperature (melting point = 30° C), it was therefore preheated at 80° C for about half an hour to produce a clear liquid which mixed readily with the resin, and the BDMA was added dropwise. The contents of the beaker were stirred thoroughly and placed in an air circulating furnace at about 80° C for five minutes, during which time the viscosity of the mixture decreased. Part of the resin mixture was poured into a preheated mould coated with mould release agent. The calculated weight of precut fibres was then placed in the mould and thoroughly wetted before the
remainder of the resin was added. The lid was placed on the mould which was inserted between the platens of a preheated press (80° C) on an aluminium foil tray. The excess resin was squeezed out over a period of twenty minutes, the lid being closed slowly to allow the air bubbles to rise to the surface and escape along with excess resin. The resin was cured at 80° C for three hours and allowed to cool slowly to reduce any shrinkage stresses. An optional post curing treatment of four hours at 120° C was also carried out to modify the resin mechanical properties. The composites produced by this technique were reasonably well aligned with low void contents.

2.2 DETERMINATION OF FIBRE VOLUME FRACTION

The fibre volume fraction $V_f$ has a significant effect on the mechanical properties of the composite and therefore an accurate assessment of $V_f$ is important in the analysis of composite properties. An investigation of the various methods of $V_f$ determination was carried out on the HTS/DLS 60 prepreg material.

2.2.1 PHOTOMICROSCOPIC METHODS

For continuous unidirectional fibre composites the area occupied by the fibre on a plane perpendicular to the fibre direction is a measure of the fibre $V_f$. Various methods were used to analyse the $V_f$ of a polished section. The sections were polished on diamond impregnated pads, the final polishing being performed on 1 micron diamond compound fitted on a vibratory polisher. A polishing time of up to four days was necessary to obtain a satisfactory surface finish. The $V_f$ was then estimated by the following techniques.

2.2.1.1 Quantimet Television Microscope analysis

The polished specimen was investigated on a Quantimet Television Microscope (QTM). With the QTM it is possible to measure the volume fraction of different phases in the microstructure as a function of their relative brightness. The success of this technique therefore depends on the contrast between the phases present. In the carbon fibre composites investigated the fibres were much lighter than the resin matrix and reasonable results could therefore be obtained. Unfortunately the value for fibre $V_f$ is very sensitive
to the standard of polish since any stains especially around the edges of the fibres were usually counted as resin. Measurements were made for twenty different areas of each specimen. A value for the mean $V_f$ and standard deviation are given in Table 2.

2.2.1.2 Point count

A transparent photographic plate with 100 points arranged 1 cm apart in a 10 cm square was placed on a photomicrograph of the specimen, obtained by a Zeiss Ultraphot photomicroscope. The $V_f$ was obtained by counting the number of points which covered the fibres, a half being counted for those points coinciding with the interface. The process was repeated twenty times for each of five separate photomicrographs. The results are given in Table 2.

2.2.1.3 Area count

This method is similar to the previous one. The diameters of 100 fibres were measured on a photomicrograph and the mean diameter and its standard deviation were calculated. The number of fibres were counted and the total area occupied by these fibres was estimated. The process was repeated for five different photomicrographs and the results are shown in Table 2.

The photographic techniques produced fairly similar values for $V_f$ although those of the Quantimet were slightly lower due to the difficulty in defining the edges of the fibres accurately. The area count method gave the largest standard deviation because of the squared radius term used to calculate the fibre area. The point count method was the most satisfactory of the photographic techniques, but it was very time consuming since a large number of regions had to be considered in order to obtain a reliable value of $V_f$.

2.2.2. Burn-off method

The method consists of burning-off the resin and estimating the weight and hence the $V_f$ of the remaining fibre. A composite specimen weighing about 200 mg was placed in a crucible and heated to 500° C on a Stanton thermobalance. The weight of the specimen was recorded continuously as the resin was burnt off (Fig. 10). The rate at which the composite lost weight gradually decreased until after about forty minutes it became constant. The specimen was removed from the thermobalance after 2$\frac{1}{2}$ hours and since there was no trace of resin in the
<table>
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<th>BATCH</th>
<th>METHOD OF DETERMINATION</th>
<th>$V_f$</th>
<th>STANDARD DEVIATION</th>
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</thead>
<tbody>
<tr>
<td>2</td>
<td>QUANTIMET</td>
<td>0.555</td>
<td>0.068</td>
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<tr>
<td></td>
<td>POINT COUNT</td>
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<td>0.055</td>
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<td>AREA COUNT</td>
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<tr>
<td>3</td>
<td>AREA COUNT</td>
<td>0.462</td>
<td>0.106</td>
</tr>
<tr>
<td></td>
<td>ACID DIGESTION</td>
<td>0.467</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>BURN OFF</td>
<td>0.404-0.476</td>
<td>-</td>
</tr>
</tbody>
</table>

**TABLE 2**

Determination of Fibre $V_f$ of prepreg material by the various methods
crucible it may be concluded that the constant rate of weight loss is caused by oxidation of the fibres and not by burning-off of the resin. If the curve in Fig. 10 is extrapolated back to the y-axis, the value obtained represents the original weight of the fibres in the composite assuming that oxidation of the fibres occurred immediately. This is however unlikely since the fibres are embedded in the resin matrix, some of which must be removed before oxidation can occur. The fibre $V_f$ was calculated from Fig. 10, the outer limits of the original weight of fibre being given by points A and B. This method therefore only provides an approximate value of $V_f$ but it is a useful check for comparing the results obtained by other methods.

2.2.3 ACID DIGESTION TECHNIQUE

This method of fibre $V_f$ determination consists of dissolving the matrix and estimating $V_f$ from the weight of the fibre remaining. A sample of the composite weighing 0.2 - 0.5 grams was placed in the reaction vessel and 20 mls of concentrated sulphuric acid were added. This mixture was heated at 150°C, the acid turned brown as it attacked the resin. 30 mls of hydrogen peroxide $H_2O_2$ were added, this was done very carefully since the reaction is very exothermic. The vessel was heated until the mixture fumed and then the vessel was allowed to cool down. The contents of the vessel were then poured into a beaker of water ensuring that all of the fibre was removed from the reaction vessel. A dry filter of known weight was fitted on a Buchner flask and the contents of the beaker were filtered. The fibres were then thoroughly washed and dried before being weighed. The weight of fibre was then used to calculate the fibre $V_f$ in the original sample and the results are given in Table 2.

The results obtained from this method were usually slightly higher than those of the photographic methods this may be because of voids in the sample or retained water which was not completely removed. The samples used had to be small because of the exothermic nature of the reaction and this limited the accuracy of the experiment.

2.3 MICROSTRUCTURAL EXAMINATION

Microstructural examination is extremely important because studies of the fracture surfaces of broken specimens often give some indication of the failure mechanism of the material. Specimens were examined
Fig. 10  Weight Loss during burn-off of HTS/DLS 60 prepreg material
microscopically using the Scanning Electron Microscope (S.E.M.) or by the optical technique described in section 2.2.1. In the S.E.M. investigation the sample was vacuum coated with a gold/palladium alloy to render the surface conductive, it was then examined in the S.E.M. Mk. II at magnifications of up to 10,000. In some cases montages were made by taking several overlapping photomicrographs which were then joined to produce a composite picture of a particular region at fairly high magnification.

2.4 DETERMINATION OF FLEXURAL STRENGTH

The bend test may be used to determine the flexural strength of the fibre reinforced composite if the tensile (or compressive) strength of the material is exceeded before the interlaminar shear strength is reached. Three point bend tests were carried out on an Instron testing machine at a crosshead speed of 2 millimetres (mm)/minute (min) and the flexural strength was calculated from the equation:

\[ \sigma = \frac{3PL}{2bd^2} \]

where \( P \) is the load in newtons
\( L \) is the beam span
\( b \) is the test piece width
\( d \) is the test piece depth

The variation of flexural strength with fibre \( V_f \) at an \( l/d \) of approximately 20 is shown in Fig. 11. Results are given for both the MODMOR - 828/HFA/BDMA wet lay up composites and the HTS/DLS 60 prepreg material, although the latter was only available in two fibre \( V_f \)'s. In each of the composite systems examined the flexural strength increased up to 0.6 \( V_f \), above this value however only type II treated fibre composite showed any significant increase.

The effect of other parameters on the flexural strength of the composite was also investigated.

2.4.1 THE EFFECT OF \( l/d \)

The flexural strength of the HTS/DLS 60 prepreg material was calculated from the results of three point bend tests carried out at \( l/d \) ratios ranging from 5 : 1 - 30 : 1. The values obtained are shown in
Fig. 11 The Variation of Flexural Strength with Fibre Volume Fraction

Volume Fraction $V_f$

Flexural Strength $CN/A^2$
Fig. 12. Even at low values of l/d failure occurred by flexure rather than by interlaminar shear on the neutral plane.

The failed specimens were examined by S.E.M. and optical microscopy and the general features of the fracture surface of specimens tested at low l/d (5 : 1) are shown in Fig. 12. The fracture surface consists of two regions, the compressive failure region which is characterized by very short fibres and fragments of resin and fibre debris (Figs. 13 and 14), and the tensile failure region in which the broken fibres are much longer (Figs. 15, 16 and 17). The fibres in the tensile region appear to have been pulled out in clumps rather than as single fibres. Montages of the region beneath the central loading roller (Fig. 18) show that the compression failure was caused by fibre buckling at the contact edge of the loading roller, the angle between the plane of the buckle and the fibre axis in Fig. 18 is approximately 60°. The failure mode of the MODMOR - 828/HPA/BDMA composites was similar to that described above except for the untreated type I composites at high fibre Vf which failed by interlaminar shear.

Experiments were also carried out to determine the sequence of failure events in the above tests. Several specimens were loaded to different points on the load-deflection curve and then examined using S.E.M. and optical microscopy. The load deflection curve is shown in Fig. 19 and it consists of four sections:-

(i) Section a-b is the take up region in which the load is applied to the specimen.
(ii) In section b-c the load is proportional to the deflection.
(iii) In section c-d the slope of the curve decreases due to roller indentation.
(iv) At d the first load drop occurs and this is followed by a series of load drops until at (e) the failure process is complete.

Microscopic investigation of the off-loaded specimens indicates that fibre buckling occurred at point d or soon afterwards. Damage was also observed parallel to the fibres at the base of the compression region (Figs. 20 and 21) this should not be confused with interlaminar shear failure which occurs on the neutral plane.

The general features of the fracture surfaces of specimens tested at high l/d (20 : 1) were similar to those described above although in this case the compression buckle was more complex (Fig. 22). In Fig. 22 two compression buckles merge to produce the failure at 90° to the fibre axis.
Fig. 12 L/d Ratio on Flexural Strength
Fig 18  Montage of the failure in the compressive region.
Centre Span Deflection

Fig. 19 Short Beam Load-Deflection Curve
FIG. 20

COMPRESSIVE BUCKLE DUE TO ROLLER INDENTATION

FIG. 21
Fig 2.2  Compressive buckle in the long beam specimen.
The specimen was prepared for the S.E.M. examination by cleaving the failed beam parallel to the fibres therefore the resinous nature of the surface has no significance.

2.4.2 THE EFFECT OF TEMPERATURE

Three point bend tests were performed on the HTS/DLS 60 prepreg material over the temperature range -50 to +70°C in a Sondes Place Research Institute temperature chamber adapted for use on the Instron testing machine. The results of the tests carried out at \( \frac{1}{d} = 20 \) are shown in Fig. 23. Examination of the broken test pieces at low magnification (Figs. 24 - 30) shows that:

(a) At low temperatures (-50°C to -30°C) failure occurred on the tension side of the beam and it was accompanied by extensive delamination. There was however no sign of compressive damage (Figs. 24 and 25).

(b) At intermediate temperatures (-10°C to +20°C and +30°C) delamination decreased progressively with increase in temperature. Compressive buckling damage was observed beneath the central loading roller at each of these temperatures (Figs. 26, 27 and 28).

(c) At high temperatures (+50°C and +70°C) there was no delamination but the compressive buckle became more severe (Figs. 29 and 30). In Fig. 31 there are two compressive buckles, one on each side of the roller.

From the above results it may be concluded that a high flexural strength is associated with a suppression of the fibre buckling on the compression side of the beam. The mode of failure was similar in the short beam test on HTS/DLS 60 prepreg material however compressive buckling was not completely suppressed at low temperatures. There was no evidence of interlaminar shear failure on the neutral plane. At higher temperatures the effect of roller indentation became more significant and there was some delamination in the resin rich regions between the prepreg laminates. In both long and short beam tests the flexural strength of the composite decreased rapidly at temperatures above -10°C.

2.4.3 THE EFFECT OF FOUR POINT LOADING

In order to reduce the effect of the stress concentration beneath the central loading roller in the three point bend test, a four point
Fig. 23  Effect of temperature on the flexural strength of C.F.R.P.
EFFECT OF TEMPERATURE ON THE FAILURE MODE OF THE COMPOSITE

FIG. 24  $T = -50^\circ C$

FIG. 25  $T = -30^\circ C$

FIG. 26  $T = -10^\circ C$

FIG. 27  $T = 10^\circ C$

FIG. 28  $T = 30^\circ C$

FIG. 29  $T = 50^\circ C$

FIG. 30  $T = 70^\circ C$
Fig. 31  A montage of the long beam specimen tested at +70°C
test was carried out on the HTS/DLS 60 prepreg material. The flexural strength is given by the equation:

\[ \sigma_{\text{max}} = \frac{3PL}{bL^2} \]

Where \( L \) is the distance between the inner and outer rollers.

Visual observation of the broken test pieces revealed that despite the reduction of load beneath the inner rollers there was still some damage in this region. The problem was overcome by using aluminium alloy backing plates to allow gradual diffusion of load onto the specimen. The results of the four point bend tests on the composites protected by backing plates are shown in Fig. 11. The values for \( \sigma_{\text{max}} \) at room temperature are approximately equivalent to those obtained at \(-50^\circ C\) in the three point bend tests and in specimens which did not undergo roller indentation.

2.4.4 EFFECT OF REPEATED LOADING

Fatigue tests were performed on the HTS/DLS 60 prepreg material at stresses below the static failure stress. Short beam specimens were tested in 3-point bending on an Instron testing machine using a crosshead speed of 10 mm/min. The load was cycled from a small positive load to a selected maximum in order to avoid the zero load situation and the need for elaborate location fixtures. Experiments were carried out at temperatures ranging from \(-50^\circ C\) to \(+70^\circ C\) and the results are shown in Fig. 32. The short beam results lie on a shallow curve of convex form which contrasts with the more usually observed concave shape. The stress axis refers to the calculated shear stress at the neutral plane (ILSS).

A diagrammatic representation of the fracture surfaces is shown in Fig. 33. S.E.M. examination revealed that at low temperatures flexural failure was accompanied by delamination (Fig. 34), while at high temperatures (\(750^\circ C\)) there was significant plastic deformation of the specimen and Figs. 35 - 37 show the tendency towards separation into the original prepreg layers during fatigue. This plastic deformation at high temperatures makes the establishment of a satisfactory failure criterion very difficult.

Long beam fatigue tests were also carried out in both 3 and 4-point bending. Three-point tests (\(L/d = 16 - 20\)) were performed on an Instron
Fig. 32. Effect of temperature on short beam fatigue behaviour

<table>
<thead>
<tr>
<th>TEMP °C</th>
<th>Cycles to failure</th>
<th>Type of Fracture</th>
</tr>
</thead>
<tbody>
<tr>
<td>-50</td>
<td>300</td>
<td></td>
</tr>
<tr>
<td>-30</td>
<td>120</td>
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</tr>
<tr>
<td>-10</td>
<td>24</td>
<td></td>
</tr>
<tr>
<td>+10</td>
<td>1</td>
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<table>
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<tr>
<th>TEMP °C</th>
<th>Cycles to failure</th>
<th>Type of Fracture</th>
</tr>
</thead>
<tbody>
<tr>
<td>+20</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>+30</td>
<td>200</td>
<td></td>
</tr>
<tr>
<td>+50</td>
<td>650</td>
<td></td>
</tr>
<tr>
<td>+70</td>
<td>4000</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 33 Diagrammatic representation of typical short beam fatigue failures
Fig. 34  Delamination during fatigue at -50°C

Fig. 35  Delamination during fatigue at +30°C

Fig. 36  Montage of a fatigue failure at +30°C

Fig. 37  Delamination of all prepreg layers at +50°C
testing machine using the load cycling mode at a frequency of about 4 cycles/minute and on an Avery midget pulsator machine using a constant displacement mode at up to 50 Hz. The 3-point bending rig used on the Avery machine is shown in Fig. 38 in this case for reversed bending. By removing three of the loading rollers it could also be used for unidirectional fatigue testing. Unfortunately local damage beneath the central loading rollers made it impossible to carry out satisfactory 3-point tests in reversed bending. The fatigue curve obtained in unidirectional 3-point bending is shallow (Fig. 39) and the results were widely scattered. The minimum value of fatigue strength at 10 cycles was approximately 75% of the static flexural strength of the composite.

Results obtained at different frequencies fell into a single scatter band and there was therefore a negligible frequency effect over the frequency range examined. The failure mode was similar to that observed in the static tests and initiated by compressive buckling. Failure appears to have occurred suddenly rather than in a progressive manner.

The effect of temperature on low frequency fatigue was also examined and found to be similar to that observed in the short beam test. An increase in temperature shifted the S-N curve down the y axis. Visual and S.E.M. examination revealed delamination and compressive damage in the beam tested at -10°C while at higher temperatures plastic deformation occurred beneath the central loading roller. (Figs. 40 - 44). The complicated nature of the failure in the compression region of the specimen tested at 50°C is shown in Fig. 42, and the drastic effect of roller damage during fatigue at 70°C is shown in Fig. 44. The specimen did not separate into two halves and the photomicrograph shows how the compressive buckles occur at the contact edges of the roller.

Fatigue tests were also carried out in 4-point bending using aluminium alloy backing plates on the test pieces to prevent the roller indentation problems. Although the backing plates were satisfactory for the static tests it was found that during fatigue the backing plates were indented and they eventually peeled away from the test piece. To overcome this a 4-point pure bending rig was designed in which a strip test piece cemented into two aluminium alloy end pieces was used (Fig. 45). The load was applied through trunnions set on the end pieces, this eliminated the difficulties of the 3-point test. Two test piece configurations were used, a plain test strip and a waisted test piece in which the central section was reduced to 1.5 mm thickness by grinding from each side with a
Fig. 38 Avery fatigue rig
TYPICAL FRACTURE SURFACES OBTAINED IN THE FLEXURAL FATIGUE TESTS

Fig. 40 Fracture surface in the tensile region at -10°C

Fig. 41 Delaminated surface in the tensile region at -10°C

Fig. 42 Compressive failure region at +50°C

Fig. 43 Compressive failure region at +50°C

Fig. Effect of roller damage at +70°C
Aluminium alloy block

Arrows indicate bending mode

FIG. 45 Test piece used in 4-point pure bending experiments.
250 mm diameter wheel. The results of these experiments are also shown in Fig. 39. It should be noted that since the 4-point bending tests were carried out in reversed bending, the stress scale represents the semi-range of stress for 4-point tests and the whole stress range for unidirectional 3-point tests.

The effect of fatigue on reversed 4-point bending was fairly drastic and the strength after $10^4$ cycles was only 30% of the static strength. The plain test piece failed by delamination close to the neutral plane, but there was no evidence of any other damage to the specimen and the crack did not appear to penetrate into the end piece by more than a few millimetres. The delamination reduced the stiffness of the test piece which in turn reduced the effective stress significantly, the first failure event therefore terminated the test and complete separation of the test piece did not occur. The waisted test piece failed initially by tensile failure at the surface, followed by delamination when the crack had penetrated about 0.5 mm or less.

2.5 SHEAR STRENGTH DETERMINATION

The interlaminar shear strength of the composite and the shear strength of the resin were determined by the following methods.

2.5.1 DETERMINATION OF THE INTERLAMINAR SHEAR STRENGTH (ILSS)

Two methods were used for ILSS determination.

2.5.1.1 Short beam test

This is the most simple method to perform and it consists of bending a beam in which $1/d$ is small enough to produce an interlaminar shear failure on the neutral plane. The ILSS was calculated from the elastic theory equation:

$$\tau_{SB} = \frac{3P}{4bd}$$

The results are shown in Fig. 46 for both composite systems.

Examination of the failed test pieces revealed that failure occurred by flexure rather than by interlaminar shear on the neutral plane. (Figs. 47-48). Shear failure was only observed in composites containing more than 0.6 $V_f$ type I untreated fibres in the 828/HPA/BDMA epoxy resin system. This method is therefore unsuitable for the determination of ILSS of most of the composites examined.
Figure 46  Short Beam Shear Strength versus $V_f$
FIG. 47  X 1000
Type I/828 Composite 0.6\(\nu\)_f
Short beam interlaminar fracture

FIG. 48  Type II/828 Composite 0.6\(\nu\)_f
Short beam interlaminar fracture
2.5.1.2 Direct single shear (D.S.S.) test

This is an alternative to the short beam test and it consists of propagating a crack on a predetermined interlaminar plane. The specimens were secured in air grips on an Instron table model testing machine and tested at a crosshead speed of 1 mm/min. Good alignment of the test piece is important since rotation of the specimen may affect the value of shear strength obtained (Fig. 50). The shear strength was calculated from the equation

\[ \tau_{D.S.S.} = \frac{P}{b l} \]

where \( l \) = length of fracture surface.

The average results of D.S.S. tests carried out on the HTS/DLS 60 prepreg material for different types of test piece shown in Fig. 49 are given in Table 3. The specimen in Fig. 49a was used to determine the shear strength across the prepreg layers. Attempts were made to reduce the effect of the stress concentration at the root of the notch in the above specimen by drilling a hole (Fig. 49b). The radius of curvature of the notch was significantly reduced, however the values obtained for DSS were very similar to those using the specimen in Fig. 49a. A third test piece (Fig. 49c) was used to determine the shear strength in the resin-rich region between the prepreg layers. In this case the values of DSS were lower than those obtained with the other test pieces and the scatter of the results was wider. It is very difficult to cut the specimen so that failure always occurs in the resin-rich region, this may explain the wide scatter of results.

D.S.S. tests were also carried out on Morganite fibre - 828/HPA/BDMA epoxy resin composites using the specimen in Fig. 49a. The results are shown in Fig. 50, the value of TDSS decreased with fibre V\(_f\) for untreated fibre composites while for the treated fibre composites TDSS was independent of V\(_f\) up to 0.6 - 0.7 V\(_f\), above this value however it fell rapidly.

S.E.M. examination of the fracture surfaces revealed that the fibres were usually coated with resin in the treated fibre composite, but in composites containing type I untreated fibres the fracture surface was clean.

High and low frequency fatigue tests were also performed on the Avery and Instron machines respectively and the results are shown in Fig. 51. The S-N curve is steeper than that obtained in the bend tests, there is also a negligible frequency effect over the frequency range examined (i.e. 4 - 3000 cycles/min).
Fig. 49  Test pieces for the Direct Single Shear test

<table>
<thead>
<tr>
<th>SPEC. NO.</th>
<th>$l_{mm}$</th>
<th>$d_{mm}$</th>
<th>$T_{max}$</th>
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<tbody>
<tr>
<td>8/2/B3</td>
<td>3.2</td>
<td>2.65</td>
<td>370 N/m²</td>
</tr>
<tr>
<td>7/2/B5</td>
<td>4.3</td>
<td>2.70</td>
<td>65.0</td>
</tr>
<tr>
<td>1/1/B6</td>
<td>4.7</td>
<td>2.50</td>
<td>67.5</td>
</tr>
<tr>
<td>1/1/H5</td>
<td>3.7</td>
<td>2.40</td>
<td>61.7</td>
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</table>

Table 3 A

<table>
<thead>
<tr>
<th>SPEC. NO.</th>
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<th>$d_{mm}$</th>
<th>LOAD N</th>
<th>$T$ MN/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td>7/2/B7</td>
<td>5.5</td>
<td>2.7</td>
<td>970</td>
<td>65.0</td>
</tr>
<tr>
<td>4/1/AB</td>
<td>4.2</td>
<td>2.34</td>
<td>650</td>
<td>66.1</td>
</tr>
<tr>
<td>1/1/AB</td>
<td>3.5</td>
<td>2.35</td>
<td>495</td>
<td>60.0</td>
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</tbody>
</table>

Table 3 B

<table>
<thead>
<tr>
<th>SPEC. NO.</th>
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<th>$l_{mm}$</th>
<th>$T$ MN/m²</th>
</tr>
</thead>
<tbody>
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<td>2.75</td>
<td>3.76</td>
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<tr>
<td>1/1/AB</td>
<td>3.33</td>
<td>3.68</td>
<td>57.8</td>
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<td>9/2/AB</td>
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<tr>
<td>9/2/AB</td>
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<td>59.3</td>
</tr>
<tr>
<td>9/2/AB</td>
<td>4.95</td>
<td>2.90</td>
<td>37.5</td>
</tr>
<tr>
<td>9/2/AB</td>
<td>5.30</td>
<td>3.20</td>
<td>60.0</td>
</tr>
</tbody>
</table>

Table 3 C

Results of the Direct Single Shear tests
Fig. 50 Direct Single Shear Strength versus Volume Fraction
Fig. S1 Direct Single Shear fatigue
2.5.2. **DETERMINATION OF RESIN SHEAR STRENGTH**

Two methods were used to determine the resin shear strength and the results are shown in Fig. 52.

### 2.5.2.1 Punch shear test

This test is widely used in the adhesives industry to determine the shear strength of the epoxy resin systems. It consists of punching a disc from a resin sheet. The rig is shown in Fig. 53 and the test was performed on an Instron testing machine at a crosshead speed of . The resin shear stress is given by the equation

\[ \tau_{PS} = \frac{P}{2\pi r d} \]

- \( P \) = load to failure
- \( r \) = radius of the disc
- \( d \) = thickness of the disc

The results are shown in Fig. 54 of the punch shear test and are in good agreement with the shear strength obtained in the D.S.S. test.

### 2.5.2.2 Plane strain compression test

In this test a resin sheet is compressed between two flat dies. The tests were carried out on an Instron machine at a crosshead speed of 0.05 cm/min. A calibration curve was obtained by performing the test on the dies and the supporting rig without the specimen. The test was repeated with the specimen and the strain in the specimen was calculated by subtracting the deflection at any load in the first curve from the deflection at the same load in the second curve with the aid of an Instron Data Logger System 2415. A true stress-true strain curve was plotted

\[ \sigma = \frac{P}{BW} \]

- \( B \) = die width
- \( W \) = die thickness

\[ \epsilon = \frac{d(P) - d_1(P)}{T} \]

- \( d(P) \) = deflection on curve 2 at load \( P \)
- \( d_1(P) \) = deflection on curve 1 at load \( P \)
- \( T \) = specimen thickness

2.6 **IMPACT RESISTANCE**

The impact resistance of the material was measured on a miniature Charpy impact tester with a hammer velocity of 3 m/s. The energy absorbed by the specimen was calculated from the decrease in the kinetic energy of...
Fig. 53  Punch shear test rig
\[ J = \frac{1}{2} m (V_0^2 - V_f^2) \]

\( J \) = energy absorbed by the specimen

\( V_0 \) = velocity of the pendulum without a specimen

\( V_f \) = velocity of the pendulum with a specimen

The pendulum velocity was measured electronically using a reference marker which activated a photocell attached to the pendulum. The effect of the following parameters on impact resistance was investigated:

2.6.1 (i) Effect of volume fraction

The effect of \( V_f \) on the impact resistance of the composite is shown in Fig. 55. The relationship is linear with \( V_f \) up to 0.5 \( V_f \), above this value however the increase in impact resistance is less marked.

2.6.2 (ii) Effect of temperature

Specimens of the prepreg material were placed in the temperature chamber for \( \frac{1}{4} \) hour - 1 hour and then quickly tested. The results are shown in Fig. 56. The amount of energy required to break the specimen was given by two almost horizontal straight lines, the higher value being 70 - 90 KJ/M² and the lower value about 40 KJ/M². The largest values were obtained at the lowest temperatures (-70° C -30° C).

A diagrammatic representation of the fracture modes at various temperatures is given in Fig. 57. At low temperatures there was extensive delamination with a narrow region of compressive damage. Above -30° C the extent of compressive damage gradually increased with temperature but there was a reduction of delamination. Figs. 58 - 60 are low magnification S.E.M. photomicrographs of impact specimens tested at -30° C, +30° C and +50° C which illustrate the variation in the width of the compressive zone. At higher magnifications the high and low temperature failures look very similar, Figs. 61 and 62 illustrates the typical fibre failure in the compressive region and clumped fibre pull-out is shown in Figs. 63 - 62.

2.6.3 Effect of \( 1/d \)

Specimens of different thickness (2 mm - 12 mm) were tested at a constant span \( L \). Three specimens were tested at each thickness and the
Fig. 56 Miniature Charpy type pendulum impact tests with an impact velocity of 3m/s
FIG. 57  IMPACT FAILURE MODES

FIG. 58  
T = -30°C

FIG. 59  
T = 30°C

THE EFFECT OF TEMPERATURE ON THE FAILURE MODE DURING IMPACT

FIG. 60  
T = 50°C
IMPACT FAILURES

FIG. 61  
*T = 50°C*
INTERFACIAL REGION

FIG. 62  
*T = 30°C*
COMPRESSIVE REGION

FIG. 63  
*T = -30°C*
CLUMPED PULL-OUT IN THE TENSILE REGION

FIG. 64  
*T = -10°C*
RESIN ADHESION THE TENSILE REGION
results are given in Fig. 65. In some cases it was found that the 5J hammer did not break the specimen and therefore a 15J hammer was used. The effect of changing hammers was investigated by testing specimens of intermediate thickness with both hammers and the difference was found to be negligible.

At high values of 1/d (78) the value for impact energy absorbed was independent of 1/d. At lower values however the amount of energy required to break the specimen rose very rapidly. The values for impact energy are therefore not necessarily comparable at different 1/d ratios.

2.6.4 Effect of notches

Specimens of different thicknesses were notched using either a 0.006 inch wide diamond cutting wheel or a special V notch cutting wheel attached to a standard milling machine. Some of the V-notches were sharpened with a scalpel to increase the stress concentrating effect of the notch. The results are given in Fig. 66 for the Morganite - 828/HPA/BDMA composite system (in terms of \( \frac{1}{d} - d_n \) where \( d_n \) is the notch depth). All of the specimens examined exhibited a predominantly clumped pull-out type of failure and parallel splitting was observed in most cases at the root of the notch.
Fig. 65 Unnotched impact at Various L/d Ratios
Fig. 66 Notched Impact at various L/(d-d) Ratios
CHAPTER 5 \ DISCUSSION

3.1 INTRODUCTION

In this section the effect of using high strength i.e. high modulus, carbon fibres to reinforce relatively weak resin matrices is discussed. The factors affecting the flexural, shear, impact and fatigue properties and also the suitability of conventional testing techniques are considered.

3.2 FLEXURAL STRENGTH

The flexural strength of the composites was calculated using the 3-point bend test. According to elastic theory analysis the tensile stress in the direction of the fibres is given by:

\[ \sigma_Z = \frac{M\gamma}{I} \]

where \( M \) is the bending moment
\( \gamma \) is the distance from the neutral plane
\( I \) is the moment of inertia of the beam

The deflection of the beam \( d \) is given by:

\[ d = \frac{PL^3}{48EI} \]

where \( E \) is the Young's modulus of the material

The tensile and compressive stresses are therefore greatest at the outer surfaces of the beam and an increase in the deflection of the beam produces an increase in these stresses. When either the tensile or compressive strength of the composite is exceeded failure occurs. Since the fibres are much stronger than the matrix nearly all of the stress is borne by them and the strength of the composite is a function of the fibre \( V_f \).

This is given by a law of mixtures relationship (56):

\[ \sigma_{\text{composite}} = \sigma_f V_f + \sigma_m (1 - V_f) \]

\( \sigma_m \) is the tensile stress borne by the matrix when the composite is strained to its U.T.S.

In practice the failure process is usually more complicated than that described above. The concentration of stress beneath the central loading roller tends to cause local plastic deformation in the resin which eventually
results in a buckling failure of the fibre in this region (Fig. 18).
The compressive buckling damage causes a redistribution of stress which
causes premature failure of the composite. It is possible to reduce the
likelihood of compression initiated failure as follows:

(i) The L/d of the beam may be increased. The flexural stress in the
composite is given by the equation:

$$\sigma = \frac{3PL}{2bd^2}$$

An increase in L/d reduces the load necessary to produce a given
flexural stress and therefore the tendency to produce the compressive
buckling failure is also reduced (Refs. 78 and 79).

(ii) The 4-point bend test may be used instead of the 3-point test (72).
The load necessary to produce a given stress is then borne by two rollers
instead of one and the local stress concentration is significantly reduced.
The results of the 4-point bend tests carried out on HTS/DLS 60 prepreg
material indicate that compressive buckling was not completely suppressed
unless backing plates were used. The results obtained using aluminium alloy
backing plates were fairly close to those predicted in the rule of mixtures
equation (Ref. 56). The 4-point bend test is therefore a more satisfactory
test for flexural strength determination than the 3-point test providing
that local indentation can be avoided.

(iii) The yield strength of the resin also affects the flexural strength of
the composite. Values obtained in the long beam 3 -point bend tests (L/d = 20)
carried out at temperatures ranging from -50° C to +70° C indicate that at
high temperatures the resin was deformed much more easily and the compressive
buckle therefore occurred at a lower load. At very low temperatures (-50°C
and -30° C) however the yield strength of the resin was much higher and the
fibres failed in tension on the outer surface of the beam instead of buckling
beneath the central loading roller. The value obtained for flexural strength
of the composite max was very close to the law of mixtures prediction and
this is again associated with a suppression of compressive buckling.

It may be concluded therefore that although the strength of the fibres
control the maximum attainable flexural strength, the actual strength may be
limited in the 3-point bend test by the mechanical behaviour of the resin.
The resin properties affect the failure mode and if compressive buckling
occurs the value of flexural strength is reduced. It would therefore be
beneficial to use highly crosslinked resin systems (Ref. 35) which remain
elastic up to the fibre failure strain in order to achieve optimum flexural
3.3 INTERLAMINAR SHEAR STRENGTH

The previous section dealt with the determination of flexural strength using the 3-point bend test at \( \ell/\delta \) of approximately 20. Shear stresses are also produced in the beam during bending, these stresses are zero at the outer surface of the beam and maximum at the neutral plane. The maximum shear stress at the neutral plane is given by the equation:

\[
\tau_{SB} = \frac{3 \, P}{4bd}
\]

\( \tau_{SB} \) is the interlaminar shear strength of the short beam.

Combining the equations for \( \sigma \) and \( \tau_{SB} \), the relationship \( \frac{\sigma}{\tau_{SB}} = \frac{2L}{\ell d} \) is obtained, it should be possible therefore to alter the failure mode by changing \( \ell/d \). If the ratio of the flexural strength to interlaminar shear strength is greater than \( \ell/d \) failure should occur by interlaminar shear on the neutral plane of the beam. The 3-point bend tests used to calculate \( \tau_{SB} \) were carried out at an \( \ell/d \) of 5.

In practice premature buckling similar to that described in the previous section occurred (Ref.72 & 73) rather than interlaminar shear failure and visual observation of the broken test pieces indicated a flexural failure initiated in the compressive region. The flexural strength of the composite decreased as \( \ell/d \) was reduced because larger loads were required to produce an equivalent stress in the shorter beams and therefore the stress concentration effects around the loading roller were more severe. In order to obtain a shear failure \( \ell/d \) would therefore have to be rather less than \( \frac{\sigma}{\tau_{SB}} \) (Ref. 78 and 79). It is extremely difficult to produce a true interlaminar shear failure in a composite which has an interface strength of more than about 3% of its flexural strength because the \( \ell/d \) would have to be impractically low. The only composites which failed by interlaminar shear on the neutral plane were those containing more than 0.6 \( V_f \) untreated type I fibres and in this case \( \tau_{SB} \) is about 30. There was some delamination in the resin-rich regions of the HTS/DLS 60 prepreg material but since it did not usually occur on the neutral plane or extend the whole length of the plane, it was not considered therefore to be a true interlaminar shear failure. There are two main causes of delamination, (a) fibres which obstruct the main crack travelling across the beam and divert it parallel to the fibres, this is presumably due to the stress...
distribution around the crack tip, and (b) buckled fibres at the base of the compressive failure zone which open up cracks parallel to the fibres.

Interpretation of short beam results is difficult even for composites which failed by interlaminar shear on the neutral plane. The failure includes contributions from both resin and fibre/matrix interface and the value of ILSS therefore depends on the relative amounts of these contributions. Consider a section of the material perpendicular to the fibre direction in which the fibres are arranged in a square array (Fig. 67). The shear force on the neutral plane is borne by fibre/matrix interface and resin which have surface areas equivalent to \( \frac{W}{x} \) and \( x \) respectively. As the fibre \( V_f \) is increased the interfibre distance \( x \) is reduced (Fig. 68). Theoretical curves of shear strength at different \( V_f \)'s are shown in Fig. 69 for assumed fibre/matrix interface strengths \( \gamma_i \) of 10, 20, 30 and 40 MN/m², and the calculated resin shear strength of approximately 40 MN/m². The actual values of ILSS obtained for the short beams which failed by interlaminar shear are also shown in this Fig. as well as the results obtained from the DSS tests.

The crack path length is increased by the presence of the fibres so that for quite low values of \( \gamma_i \) the shear strength of the composite would be higher than that of the resin. Zero interface strength would lead to a rapid decrease in the composite shear strength as the fibre \( V_f \) is increased, becoming zero, at \( V_f = 0.8 \), i.e. when the fibres touch in the assumed geometry. Visual observation of failed test pieces from the short beam and DSS tests revealed that the untreated fibre composites failed along the interface (Fig. 47), whereas the treated fibre composites failed predominantly in the resin, although there was again evidence of some interfacial failure (Ref. 81). This would imply that the interfacial strength of the untreated fibre composites was about 10 MN/m² and about 30 MN/m² for the treated fibre composites i.e. similar to the shear strength of the resin.

In the composites examined interlaminar shear failure was only observed in the short beam test when the fibre/matrix interface strength was very low and this is a very serious limitation on the usefulness of this test. The DSS failure on the other hand always occurred on a predetermined plane and it is therefore much easier to interpret the results of this test. The disadvantage of this test is the stress concentration at the root of the notch, attempts to reduce the effect of this stress concentration by reducing the radius of curvature of the notch tip were unsuccessful. In the DSS test the stress is automatically concentrated in the resin and in the fibre/matrix interface.
FIG.67 FIBRES ARRANGED IN A SQUARE ARRAY

FIG.68 EFFECT OF $V_f$ ON INTERFIBRE DISTANCE
In most practical cases the notch radius is large compared to the interfibre distance and increasing the radius of the notch had very little effect on the value of interlaminar shear stress obtained. The presence of the fibres presumably affects the stress distribution around the notch.

Visual observation of the failed DSS test pieces showed that the untreated fibre composites failed along the interface whereas the treated fibre composites failed predominantly through the resin (Ref. 77), although there was some evidence of interfacial failure. The value of $\gamma_1$ for treated fibre composites is therefore probably slightly higher than that indicated in Fig. 69.

In the composites examined it is evident that interlaminar shear failure only occurs in the short beam test if the fibre matrix interface is very weak and this imposes a serious limitation on the use of this test. The DSS test failure always occurs on a predetermined plane and it is therefore qualitatively easier to interpret. The stress concentration effects are a disadvantage although for practical applications the values obtained for $\gamma$ DSS are a conservative estimation of the true $\gamma$ ILSS and therefore composite efficiency is sacrificed rather than composite safety when using these values. Neither of the above tests can therefore be guaranteed to give reliable values of interlaminar shear strength but they do give some indication of likely failure modes of the composite and possible ways of improving their interlaminar shear properties.

3.4 IMPACT RESISTANCE

The energy absorbed by the composite during impact is affected by contributions from both fibre and resin. In the composites examined both the fibre and resin are brittle, the energy required to produce new surface is small. The impact results however show that the energy absorbed by the composite can be very high depending on the failure mode.

In the Charpy test failure occurred in one of the following ways (Ref. 92):-

(a) In a brittle manner, the energy required to produce new surface being provided by the strain energy stored in the beam, this is given by the equation:

$$ U = \frac{1}{2} \frac{\sigma^2}{2E} V_f L b d $$

If the stored energy is greater than the surface energy required, the excess is dissipated in the form of heat, vibration and kinetic energy of the test piece. The excess cannot however be returned to the pendulum and it is therefore impossible to calculate the amount of excess energy involved and
this constitutes a major disadvantage of the Charpy test.

(b) In some cases the crack progressed in a series of steps at a slower rate than the pendulum. The cross-section and stiffness of the beam were progressively reduced and the pendulum continued to transfer energy to the test piece. The initial damage in the 'progressive' failure was usually the compressive buckle mentioned in the previous section and there was also a contribution to energy absorption from fibre/matrix debonding and pull-out.

(c) Composites which have very poor fibre/matrix bonding properties usually failed by multiple shear in which cracks propagated parallel to the fibre direction. The composite virtually disintegrated and the energy required to break it was very high because of the large amount of new surface created.

A number of factors were found to affect the failure mode during impact, (Refs. 3, 5, 8, 11, 91, 92) these include:

3.4.1 Fibre type

The strain energy of type II high strength fibres is about four times that of type I high modulus fibres and this is reflected in the impact results (Fig. 55). For applications in which impact resistance is more important than modulus type II fibre composites should be used (Refs. 92 and 93).

3.4.2 Fibre surface treatment

The fibre surface treatment affects the fibre/matrix interface properties and hence the failure behaviour of the composite (Refs. 3, 94, and 95). The untreated fibre composites have low interlaminar shear strengths and they usually failed by multiple shear and the absorption of a large amount of energy whereas the treated fibre composites usually failed in a progressive manner. It appears therefore that a reduction of fibre/matrix bond strength improves the impact properties of the composite. It should be noted however that the Charpy test gives no indication of the threshold energy necessary to initiate damage and this may be extremely important for practical applications in which the component must also retain its strength and stiffness.

3.4.3 Fibre volume fraction

At low fibre Vf's the failure was usually brittle and the strain energy stored in the specimen was greater than that required to produce new surface. As the fibre Vf was increased the beam tended to fail in a progressive manner, usually initiated on the compressive side of the beam. At high fibre Vf's the
the interfibre distance is very small and there is therefore a greater likelihood that voids will be present due to the incomplete wetting of fibre during moulding and this favours the multiple shear failure mode. Unfortunately the strength and stiffness of the beam are significantly reduced at high fibre $V_f$'s and this tends to offset the increased impact resistance due to multiple shear.

3.4.4 L/d ratio

The L/d ratio affects the relative flexural and shear stresses set up in the beam and this influences the failure mode of the composite (Refs. 90 and 91). At high L/d the failure was flexural and the Charpy results indicate that the energy absorbed is independent of L/d above L/ds7. Below this value the impact resistance rises sharply, partly due to multiple shear failure and partly due to the physical restraint on the hammer. Unfortunately in the composites examined the standard Charpy configuration is near the critical L/d ratio and comparison of the results obtained at different L/d's must be treated with great caution.

3.4.5 Mechanical properties of the resin

At high temperatures the resin was deformed much more easily and this caused fibre buckling in the compressive region which resulted in a 'progressive' type of impact failure. The impact resistance was highest at very low temperatures and once again it is emphasized that a highly cross-linked brittle resin is better than a more flexible one for greatest impact resistance. At low temperatures the failure occurred in a progressive manner but there was also some delamination. The delamination should not be confused with multiple shear since it is usually the result of diversion of the crack travelling perpendicular to the fibres rather than shear.

3.4.6 Effect of notches

The presence of a notch perpendicular to the fibre direction usually caused some delamination at the root of the notch. The energy absorbed by the notched composite has two components; the first is the energy required to produce new surface and the second is the energy absorbed during the failure of the rest of the beam. In practice the effect of delamination at the root of the notch was insufficient to cause a significant change in the energy absorbed by the composite (Figs. 65 and 66), this may not however be the case in other composite systems. The composites examined may be regarded as notch insensitive, this is an important advantage for their use in practical applications.
3.4.7 Manufacturing conditions

In the 0.6 Vf type II treated fibre composites which failed in a progressive manner it was found that delamination occurred more readily in the well-defined resin-rich regions of the HTS/DLS 60 prepreg material than in the Morganite - 828/HPA/EIM composite system.

It should be emphasized that great care is required when interpreting the results of Charpy impact tests. The strain energy imposes a lower limit on the value of impact energy absorption which can be recorded and this is important if the composite fails in a brittle manner. The effect of L/d on the impact resistance makes comparison of results obtained at different L/d very difficult. Despite these shortcomings however it has been possible to investigate the effect of various parameters on the impact behaviour of the composite using the Charpy test. It appears that for a given fibre Vf the impact resistance depends on the type of fibre used as reinforcement and on the mode of failure of the beam and the factors which affect it.

3.5 THE FATIGUE BEHAVIOUR OF C.F.R.P.

The effect of fatigue on the HTS/DLS 60 prepreg material was investigated using several different types of test. The following were investigated:

3.5.1 Long and short beam 3-point bend fatigue

The values obtained from the three-point bend fatigue tests show that the fatigue strength is about 75% of the static strength after 10 cycles. As in the static tests failure in both the long and short beams occurred initially by compressive buckling beneath the central loading roller at room temperature. The results of these tests must therefore be treated with great caution (Ref. 113).

3.5.2 Four-point bending in reversed loading

The advantage of the four-point bend test was that it could be carried out in reversed bending as well as unidirectional bending. There was understandably a much more pronounced fatigue effect (Ref. 113) in reversed bending, the strength after 10 cycles being only 30% of the static strength. The failure occurred by delamination on or near the neutral plane, this is very surprising since the relative shear stress should be zero on the neutral plane in 4-point bending. This effect may have been due to the constraint of the aluminium end pieces. The drastic effect of reversed bending in this test emphasizes the need for reliable reversed bending data since the practical
implications of these results are very important if these composites are to be used for applications where they may be subjected to reversed cycling stresses.

3.5.3 Effect of temperature on fatigue behaviour

The effect of temperature is very similar to that observed in the static tests. At high temperatures resin flow beneath the central loading roller causes premature buckling in both the long and short beam 3-point bend specimens. The resin yield strength increases as the temperature is decreased and this is reflected in the fatigue strength of the composites. There is a greater tendency toward delamination during fatigue than in the corresponding static tests (Ref. 81) which may be due to local damage in the resin-rich regions of the prepreg material. However, the failure process usually occurs fairly rapidly in these tests and there is very little evidence of progressive crack growth. The tendency toward delamination during fatigue is an important consideration for the use of these composites in practical applications because in some cases they were split up completely into the original prepreg layers.

3.5.4 Direct single shear fatigue

The short beam 3-point bend test failed in flexure rather than by interlaminar shear, the DSS test was therefore used to investigate the effect of fatigue on the interlaminar shear strength. The values were between 60 and 75% of the equivalent short beam results and the effect of fatigue was slightly greater in the DSS test probably due to the stress concentrating effect of the notch. There was however no significant effect of either frequency or reversed loading on the DSS results obtained.

None of the fatigue tests used was entirely satisfactory. The difficulties encountered during static tests were enhanced during fatigue and consequently the results obtained should be treated with caution (Ref. 113). In all of the tests it was found that fatigue in either unidirectional or reversed loading reduced the failure strength, in some cases quite drastically. The fatigue strength was a maximum at low temperatures, indicating again the desirability of using a relatively brittle resin matrix.

3.6 CONCLUSIONS

The following conclusions can be drawn from the results of the experiments carried out on the carbon fibre/epoxy resin systems.

(1) The 3-point bend test for flexural strength determination must be carried out with very large L/d ratios otherwise failure occurs by compressive buckling beneath the central loading roller. Compressive buckling causes premature failure of the beam and although the fibre strength and fibre
\( V_f \) limit the theoretical strength of the composite the actual strength is controlled by the mechanical properties the resin. It is therefore desirable to use resin systems with a high yield strength in order to attain the maximum efficiency from the composite material. The 4-point bend test is better than the 3-point test because the local stress concentration is reduced, it may be necessary however to use backing plates on the specimen in order to suppress compressive buckling completely.

(2) Neither of the tests used to determine the ILSS of the composite was very satisfactory. The short beam test is the standard method for ILSS determination but nearly all of the short beam tests failed by compressive buckling because of the very high local load beneath the central roller. Only composites with very low fibre matrix interface strengths failed by interlaminar shear. The results emphasize the necessity for careful examination of the failure mode since the values obtained for ILSS are meaningless if the first failure event is compressive buckling.

The D.S.S. test is an improvement on the short beam test because the failure occurs on a predetermined interlaminar plane. The stress concentrating effect of the notch reduces the value of ILSS but the value obtained is a conservative estimate and this is a more useful value than that derived from the short beam test.

(3) Temperature has a significant effect on the results and failure modes observed in all of the experiments. At high temperatures plastic deformation occurs relatively easily and this leads to premature failure by compressive buckling. The measured properties in all of the tests were lowest at these high temperatures. At low temperatures the yield strength of the resin increases and failure in the beam tests usually occurs by a combination of tensile failure on the outer surface of the beam and delamination. The maximum value for composite properties may be obtained by using a highly cross-linked brittle resin matrix. The drastic effect of temperature especially on flexural strength is likely to cause great difficulties when the composites are used for practical applications. Great attention must therefore be paid to changes in failure mode which occur over the operating temperature range.

(4) The impact properties are affected by the type of reinforcement, the \( V_f \) of reinforcement, the fibre/matrix interface strength and other factors which affect the failure mode of the composite. The \( L/d \) affects the failure mode and therefore the impact resistance. At high \( L/d \) (\( >6 \)) the impact energy absorption is virtually independent of \( L/d \) and failure occurs by flexure while at low \( L/d \) there is an increase in multiple shear and also a greater physical restraint is imposed on the hammer, both of which combine to produce
a rapid rise in energy absorption. The variation of impact resistance with L/d means that great care must be taken when comparing results obtained at different L/d.

There is no evidence of notch sensitivity because the stress concentration at the root of the notch is reduced by delamination parallel to the fibres. The impact resistance is greatest at low temperature when the resin matrix is relatively brittle. The Charpy test is not entirely satisfactory especially when failure occurs in a brittle manner because the excess strain energy in the beam cannot be returned to the pendulum.

(5) The bending strength is reduced by repeated loading. A similar failure mode occurs in the static and fatigue tests although there is more delamination during fatigue. Temperature has a similar effect on fatigue results as in other tests and the advantage of using a brittle resin is again emphasized. At high temperature it is very difficult to obtain a satisfactory failure criterion because of the large amount of plastic deformation in the specimen. None of the tests used for the determination of fatigue strength was entirely satisfactory. The most drastic reduction in composite fatigue strength occurs in 4-point reversed bending. The ILSS of the composite is also reduced by repeated loading, the effect being slightly more pronounced in the D.S.S. test. In all of the tests carried out, frequency has very little effect over the frequency range 4-3000 cycles/min.

(6) Manufacturing variables affect the failure mode of the composite. Delamination occurs most frequently in the prepreg material because of the resin-rich layers. This may be advantageous for high impact energy absorption because more energy is required to produce new surface. Delamination occurs as a result of diversion of the main crack travelling perpendicular to the fibres which reduces the stress concentration at the tip of the crack. Unfortunately at higher temperatures the resin-rich regions become an area of weakness because plastic deformation occurs and this leads to premature fibre buckling in the compressive region. In the wet lay-up material it is noticeable that the strength of the composite is drastically reduced at high fibre Vf. This is probably due to the difficulty of manufacturing void-free composites at these high fibre volume fractions. Complete wetting of the fibre is not usually obtained when the interfibre distance is very low and the voids tend to be elongated and therefore lead to delamination. Unfortunately delamination occurs by shear at fairly low stress and although the impact resistance is high due to multiple shear type of failure, the strength properties are usually poor and the composites, unless they contain treated fibres, are not as good for practical applications as those which contain 0.6 Vf.
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