COMPRESSION AND MICROSTRUCTURE OF GLASS FIBRE FABRICS IN THE PROCESSING OF POLYMER COMPOSITES

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ABSTRACT

The compression of typical glass fibre woven fabrics, namely plain, twill, satin, and noncrimped stitch-bonded fabric, was studied experimentally by performing a series of mechanical tests on dry and resin impregnated assemblies of fabrics. This was followed by microstructural studies of corresponding laminates cured under different degrees of compression. The experiments included investigations on the effects of applied pressure, speed of compression, fabric orientation, number of plies and different resin systems on fibre volume fraction, $V_f$, of the compressed assembly. It was found that the compression of dry fabrics followed a power-law relationship between pressure and $V_f$, where the power law index, $b$, was determined to be approximately equal to 10.3, 9.8 and 9.1 for assemblies consisting of plain, twill and 5 harness satin weaves, respectively. A mathematical analysis was performed for the viscoelastic compression of resin impregnated assemblies of fabrics and a model was developed incorporating the deformation of the fibre network and resin flow through the reinforcement. A methodology was devised for the geometrical representation of plain weaves in the microstructural analysis of cured laminates. The microstructural studies then provided data for the area and geometrical parameters of the yarn cross-section; the mean amplitude, wavelength and phase angle of the yarn waveform and the distance between plies at different compression pressures. Cross-sections of laminates with each of the considered fabrics were compared in terms of fibre area fractions, porosity and void content, for the different compression pressures. The aim for the microstructural analysis was to elucidate the mechanism of compression and to follow the development of fibre and pore structure under different degrees of compression. It was concluded that the compression of resin impregnated woven fabrics could be considered as a combination of four modes of deformation: (a) the elimination of a resin rich interlayer between adjacent layers of fabric; (b) the nesting of layers of fabric by slipping while under compression; (c) the deformation of the yarn waveform which results in the reduction of thickness of individual plies and (d) the compression and deformation of the cross-sections of individual yarns.
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1. INTRODUCTION
1.1 THE MANUFACTURE OF FIBRE REINFORCED COMPOSITES

There are today many processes for the manufacture of fibre reinforced composites; contact moulding, compression moulding of which dough moulding compound (DMC) and sheet moulding compound (SMC) are the two most common [Kendall et al (1992)], autoclave moulding and resin transfer moulding (RTM). These manufacturing processes have a variety of ways of introducing fibre reinforcement into the polymer matrix. It may be in the form of chopped strands as in compression moulding, resin impregnated reinforcement as in autoclave moulding or dry reinforcement preplaced into the mould before injection of the resin as in RTM.

1.1.1 CONTACT MOULDING

Contact moulding is the cheapest and most basic method of construction of fibre reinforced composites (FRC) and often uses a smooth waxed mould of lightweight construction (no need to withstand high pressures), to which a resin gel coat is applied. Usually between 0.5-2.0 mm in thickness, the gel coat's purpose is to accurately reproduce the surface of the mould and to provide a suitable surface finish for the moulding. Once the coat has gelled there are then two methods of incorporating the reinforcement.

The first method can use a variety of fabric reinforcements but the use of a chopped strand mat is the most common. This mat consists of random chopped strands held together with a suitable binder. The mat is cut to shape, laid on the gel coat and then coated with resin which is then hand rolled (see Figure 1.1) to give good impregnation of the mat, ensure the fibres are completely wetted out, remove the likelihood of voids and bubbles and to achieve a compact sheet. Once the first layer is complete the process is repeated until the desired thickness for the moulding has been achieved. The whole assembly is then left to cure until the moulding is dimensionally stable sufficiently to be removed from the mould. The main advantage of this hand lay-up method is that any fibre orientation can be achieved with the correct choice of reinforcement. Typical fibre volume fractions are in the region of 0.5.

The second variant consists of spraying a mixture of chopped strands and catalysed resin onto
the mould surface. This is obviously quicker than hand lay-up, and localised areas can be thickened at will. However the mass of composite still has to be rolled and compacted, and tolerances are only kept acceptable (but wide) by the use of skilled labour. Unfortunately the spraying restricts the orientation of the fibres to random planar resulting in a low fibre volume fraction of around 0.3.

![Diagram](image)

**Figure 1.1** Schematic illustrating the principle of hand lay-up in contact moulding.

### 1.1.2 COMPRESSION MOULDING

If more complex shapes are to be produced that fit strict dimensional tolerances, are needed quickly in large quantities and are to have a good surface finish then higher pressure techniques are used. Higher production pressures call for more robust and ultimately more expensive equipment. One such method is compression moulding.

#### 1.1.2.1 Sheet Moulding Compound

One type of feedstock used in compression moulding is a resin impregnated random glass fibre sheet, called a sheet moulding compound (SMC). The matrix is a thermosetting resin, usually unsaturated polyester. A maturation agent is added together with a particulate filler, of which the most common are calcium carbonate, magnesium hydroxide and aluminium trihydrate, to reduce cure shrinkage and improve surface finish.
The resin, maturation agent and filler are first compounded to form a slurry which is uniformly spread on to two thermoplastic backing films. Chopped glass strands are then sprinkled onto the surface of the slurry in the required proportions. The glass strands are typically chopped to a length of 25 or 50 mm and possess a hard size coating to prevent the fibres from fracturing during processing. The two layers are then placed together and passed through a series of rollers to impregnate the glass strands and form a sheet of SMC. The purpose of the maturation agent is to thicken the resin. A low viscosity is required initially to ensure fibre wet out, but once this has been achieved the viscosity needs to increase to stabilise the resin in the prepreg to allow handleability. The moulding process also requires a certain level of viscosity to maintain the uniform flow of fibres and resin during processing. By adding a maturation agent to the polyester a reaction effectively causes the polyester's chain length to increase resulting in an increase in the viscosity (over a period of a few days at room temperature) of the slurry.

For manufacturing the sheet of SMC is cut to shape (more than one layer may be used) and is placed in an open preheated split metal mould (see Figure 1.2). For optimum properties the SMC should cover 50-60% of the mould surface area. The mould is then closed under pressure and the charge flows to fill the cavity. The temperature of the mould during processing is within the range of 140-150°C with pressures in the range of 0.5-5.0 MPa. Once the cavity has been filled the final crosslinking within the matrix takes place ensuring the shape of the product remains fixed. The moulding is removed when still hot and any excess flashing removed. The total cycle time is normally in the range of 60-180 seconds.

![Figure 1.2](image_url) Compression moulding of SMC: (a) pieces of SMC are preplaced into the mould, (b) SMC flows to fill the mould cavity, (c) component ejected.
The orientation of the fibres are usually random in-plane, due to the nature of the manufacturing process; there is however a version of the SMC process which incorporates unidirectional reinforcement. The resulting fibre volume fractions for SMC are usually about 0.25.

The main advantages of SMCs are good surface finish, high heat deflection temperature and low cost. Their widest use is in the automotive industry for the manufacture of body panels which, in Europe, accounts for about 47% of SMC consumption [Gibson (1994)].

1.1.2.2 Dough Moulding Compound

Dough moulding compound (DMC) is another type of feedstock used in compression moulding. The feedstock is again a mixture of unsaturated polyester resin, chopped glass strands and mineral filler. Where DMC differs from SMC is that the fibres are oriented randomly in three dimensions, with a lower glass content and shorter fibre lengths (6-12 mm).

The resin is initially compounded with the filler and other additives and without the glass using a low intensity mixer. Then the glass reinforcement is added and the process continues until the glass reinforcement is completely wetted out. Care is taken so that no undue wearing of the chopped glass fibres occurs. The finished compound is then formed into dough-like logs which are wrapped in film to prevent loss of the styrene monomer from the polyester.

The compound can then be hot compression moulded in the same way as that of SMC. However more complex mouldings can normally be achieved with DMC compared to SMC due to a lower viscosity. DMC can also be injected under high pressures into a closed heated split mould. This does remove some of the randomness of the fibre orientation as the flow into the mould causes a certain amount of fibre alignment and can cause a significant loss to the fibre length. However, injection moulding does offer faster cycle times (20-50 seconds), increased levels of automation and higher quality surface finish.

The typical properties exhibited by DMCs are low mould shrinkage, good electrical properties and high deflection temperatures and this coupled with their low cost (lower than SMCs)
makes them ideal for electrical and automotive applications.

1.1.3 AUTOCLAVE MOULDING

Autoclave moulding is used primarily to produce high performance composite laminates for applications such as aerospace and racing car body shells. This is enabled because the fibres in the laminate can be arranged at predetermined angles best suited to the application in mind. This is achieved using prepregs of aligned or woven fibres preimpregnated with thermosetting (e.g. epoxy) or thermoplastic (e.g. PEEK) resins.

A prepreg is a thin sheet of fibres preimpregnated with a slightly crosslinked resin to hold the fibres in place. One way of constructing prepregs is to pull the fibres through a resin bath, remove excess resin and then wind the fibres helically on to a large drum. This action creates a thin walled tube of fibres which is cut along its length and removed from the drum. It is then placed into a heated press which flattens out the tube. The heat of the press causes a small amount of crosslinking to occur in the resin, removing stickiness, allowing it to be handled. The main advantage of using a prepreg, other than ease of handleability, is that the fibres can be preferentially oriented to the job in hand with high fibre volume fractions of the range 0.5 to 0.7 [Powell (1994)].

The prepregs can then be cut to their appropriate dimensions and stacked at the preferred angles for the application, ensuring a balanced structure. A typical lay-up for a unidirectional material would be $0^\circ/\pm 45^\circ/90^\circ/\pm 45^\circ/0^\circ$. The stack of prepreg is then placed on the tool surface ensuring that a layer of release film of silicone treated paper or polymer is placed between the mould surface and the prepreg. The prepreg is then covered by a porous release layer usually made from woven glass fibres treated with PTFE. Any excess resin passes through this permeable layer and is absorbed into the absorbent bleed pack. Resin bleed helps the consolidation of the laminate and also aids in the removal of entrapped air and residual volatile impurities. A non-stick gas permeable film is added, followed by a breather pack and the complete assembly is then encapsulated in a bag, so that the space between the outer membrane and the mould surface can be evacuated, and sealed. The breather pack, a porous membrane, is to ensure that the bag is evacuated uniformly. A schematic of the
assembly can be seen in Figure 1.3.

![Figure 1.3 Schematic of the typical autoclave setup for prepreg laminates.](image)

The bag is evacuated causing the outer membrane to push down on the laminate by atmospheric pressure. The whole assembly is then placed in an autoclave and pressurised to add to the consolidation pressure. Typical pressures of around 600 kPa are employed for epoxy composites [Jones (1994)]. Convection heating is used in the autoclave to heat the resin up to its cure temperature. Since the cure temperature is dependent on the resin system, it can be up to 200°C for epoxy but may be higher than 300°C for other thermosets. The cure schedule must be chosen to ensure the laminate heats at a steady rate and dwells may be necessary to enable thermal equilibration of the large thermal mass of the stack and tooling. The temperature causes the resin to first soften, whereby any excess resin flows into the bleeder pack and then the resin cures. Once the laminate has adequately cured the temperature is lowered, the autoclave depressurised, the assembly removed from the autoclave and the component removed from the mould.

### 1.1.4 RESIN FILM INFUSION

Instead of using a prepreg, resin film infusion (RFI) employs a laminate consisting of alternate layers of unimpregnated reinforcement and films of resin. The entire laminate is then heated to
soften the resin and then pressure applied to ensure that all the reinforcement is completely impregnated (see Figure 1.4). One of the main advantages of RFI when compared to RTM is shorter flow paths, the resin only has to infiltrate half the thickness of each reinforcement layer. The process therefore lends itself well to fabrics with a high fibre volume fraction and to high viscosity resins. Woven and noncrimp stitch-bonded fabrics are ideal, producing high fibre volume fraction laminates, and both thermoset and thermoplastic resin systems can be used in RFI. In comparison to prepreg processing, materials costs are reduced and there is an improvement of the through thickness properties [Rudd et al (1997)].

![Figure 1.4 Schematic of resin film infusion (RFI) showing: (a) alternate reinforcement and resin layers, (b) the impregnated reinforcement after the application of heat and pressure.](image)

1.1.5 RESIN TRANSFER MOULDING

Resin transfer moulding (RTM) has been used to mould large and thin structures such as wing skins, boat shells, and panels, etc. since the mid 1970s [Johnson (1987), Lee and Brew (1989), Um and Lee (1992)]. With the increasing acceptance of composite materials into the automobile industry RTM is being investigated for the production of both structural and semi-structural parts.

In RTM, shown schematically in Figure 1.5 [Rudd et al (1990a)], a liquid resin is injected,
under pressure, into a closed mould containing a dry fibre reinforcement preplaced in its preferred orientation. Typical resins used in RTM are epoxies, polyesters and phenolics. Once the reinforcement has been completely impregnated, it cures to form the completed composite product which is then removed from the mould.

Figure 1.5 A schematic showing the resin transfer moulding process.
RTM has many advantages over its competitors. As mould filling pressures are low, usually less than 7 bar (0.7 MPa), it enables the use of low stiffness shell moulds, such as glass reinforced plastic (GRP), which are of a very low cost when compared with the machining costs of steel moulds [Trevino et al (1991)]. Near net shaping methods can be carried out on a majority of parts eliminating the need for expensive finishing operations. Excellent part uniformity is achieved since the reinforcement is preplaced in the mould enhancing architectural control and structural integrity. RTM also has the ability of being able to preplace local reinforcement (cores and metals) into the mould prior to injection giving the advantage of consolidating several parts into one structure. This capacity for part integration eliminates the need for additional tooling and assembly functions. The resulting component from the moulding cycle is one of high quality having a good surface finish on both its upper and lower surfaces meaning there is less chance of air entrapment on the surface and no need for expensive finishing operations.

One of the main drawbacks of RTM, especially with regard to the automotive industry, is that its process cycle times are still too slow for high volume production of complex parts. At its present level it is only suitable for low volume production (less than 100,000 parts per annum). The reason for this is because of relatively long cycle times. The mould temperature in RTM is not excessively high which leads to slow curing times. This slow rate of reaction results in cycle times in the range of 15 to 20 minutes, far too slow for high volume production. Still its process cycle is far quicker than some manufacturing processes. The main advantage of RTM over autoclave moulding is that of cost of the manufactured component. The raw material costs for both processes are basically the same. However, manufacture of the prepreg for autoclave moulding is an expensive process resulting in costs in the order of £100 per kilogram for 0.125 mm thick prepreg. Autoclave moulding also has a long process cycle, around five hours, i.e. one part per day; this is compared to the twenty minute cycle time of RTM. The final cost then, of the component produced by the autoclave process, will be in the order of ten times more expensive than that of the RTM component.

However, instead of dismissing RTM as a process for high volume manufacture, ways are currently being looked at with regard to the use of heated moulds [Rudd et al (1990a)], resin preheating [Johnson et al (1995)] and hot setting polymer resins [Rudd et al (1990b)].
Johnson et al (1995) describes the main cause of extended cycle times as thermal quench at the point of injection. This is due to the cold resin entering the hot mould which has the effect of reducing the gate temperature. This reduction in temperature remains until impregnation is complete whereby the resin and mould begin to recover the heat loss. Johnson showed that by preheating the resin to an optimum temperature of 40°C the thermal quench was reduced. This reduced loss of mould temperature to the resin in turn reduced the resin viscosity and resulted in a reduced cycle time of around 24%. As the area around the gate is usually the last to cure it is this area that is predominantly responsible for the length of the cure cycle. Work has been carried out to adjust the resin chemistry at injection [Rudd (1996)]. Toward the end of injection a high reactivity catalyst (compared to a low reactivity catalyst that has been used for the majority of the injection) is added to the resin. This results in the last shot of the resin having faster gel times than the resin at the start of injection. If the levels of initiator are managed correctly then the resin throughout the mould is able to cure simultaneously and overall cycle times are reduced. A potential problem of increasing resin temperatures is the early onset of gelation of the resin during the impregnation phase increasing the risk of premature cure. An alternative is to heat the fibre reinforcement prior to injection [Wymer and Engel (1993), Rudd et al (1990a)]. By heating the reinforcement to the same temperature as the mould prior to resin injection, Rudd showed that both fill and gel times were reduced. This led to an overall reduction in the cycle time of approximately 30%.

1.2 THE USE OF REINFORCING FABRICS IN POLYMER COMPOSITES

1.2.1 REINFORCING FIBRES

A wide range of fibres and fibre architectures are used in the production of fibre reinforced composites (FRCs). The reinforcing fibres within a composite laminate play a large role in determining the mechanical properties and type of processing available. There are three main classes of high modulus material used for commercial applications: glass, carbon and aramid. Each of the class of materials has distinct advantages and disadvantages with respect to each other and so for commercial viability there has to be a compromise between optimum performance and acceptable cost.
1.2.1.1 Glass Fibre

The fibres most widely used in composites today are those manufactured from E-glass, so called because of their good electrical insulation properties. It was first produced for composite manufacture in the 1940s and for the last thirty years has been the standard material for FRCs with a usage approaching two million tonnes per year worldwide [Bader and Lekakou (1997)]. Made from a borosilicate glass compound its exact composition is dependent on the raw material available. Other types of glass fibres include S-glass, which exhibits better mechanical properties than E-glass, and C-glass which although has a high resistance to chemical corrosion has lower strength than E-glass.

Glass fibres have a relatively high strength, around 3 GPa, but a relatively low stiffness, around 70 GPa, when compared to carbon and some aramids. E-glass is the cheapest of the glass fibres and in the region of twenty times and eight times cheaper than carbon and aramid fibres respectively [Scardino (1989)]. Therefore for land and water applications where cost is the major design consideration, glass remains the preferred choice as it offers the best modulus per cost per weight value when compared to carbon and aramid fibres.

Glass fibres are manufactured as continuous filaments drawn from the molten glass raw material and are typically in the range of 5 - 30 µm in diameter. The filaments are cooled and formed into bundles commonly containing between 200 and 2000 individual filaments. As drawn glass bundles are susceptible to abrasion and moisture attack, a surface coating is applied known as a size. This size is generally applied in solution form and usually contains a polymer to coat and bind the filaments together in the bundle, a lubricant to reduce abrasion damage and increase handleability and a coupling agent which aids the filaments in adhering to the polymer matrix.

1.2.1.2 Carbon Fibre

Carbon fibres have been used in the manufacture of composites since around 1972 and the world carbon market is estimated today at 10,000 tonnes per year [Bader and Lekakou (1997)]. The fibres consist of carbon atoms aligned in the direction of the fibre axis resulting
in a high longitudinal tensile modulus. However this high alignment of the atoms in the longitudinal direction is also responsible for a low transverse tensile modulus which is in the region of only 3 - 10% of the longitudinal value.

Carbon fibres are produced from an organic precursor, usually polyacrylonitrile (PAN) or petroleum pitch, by thermal degradation. This process involves a series of high temperature heating stages which together with the type of precursor, the level of atomic alignment and the degree of conversion from polymer to carbon, is responsible for the final mechanical properties of the carbon fibre. There are three main groups of carbon fibre, (a) high modulus (HM or Type I) which have high a tensile modulus but a relatively low tensile strength, (b) high strength (HS or Type II) which have a higher tensile strength but lower modulus and (c) intermediate modulus (IM or Type III) which have a modulus in between that of HM and HS. Filament diameters are typically between 4-10 μm and supplied as continuous tows which generally contain 1000-12000 filaments [Rudd et al (1997)]. A size is not required for protection but for improved handleability and to promote adhesion with the matrix and is therefore usually an uncured resin compatible with the matrix.

The majority of carbon fibres produced are PAN fibres which generally have a lower tensile modulus, much higher strength and are much cheaper than pitch fibres. The main advantage over glass fibres is their much greater modulus, typically 200-350 GPa. Thus in the aerospace industry and other applications where the modulus to weight or stiffness to weight (specific modulus and specific stiffness respectively) is the major consideration, carbon is used almost exclusively.

1.2.1.3 Aramid Fibre

Aramid fibres, more readily recognised by the trade names 'Kevlar' and 'Twaaron', have been available in the commercial market since the early 1970s and have a structure based on aromatic polyaramids. Data for 1994 showed that in Europe, aramid fibres only represented approximately 0.1% of the total fibre consumption based on glass, carbon and aramid fibres [Bader and Lekakou (1997)].
Continuous aramid fibres are produced by the extrusion of a solution of poly paraphenylene terephthalamide (PPD-T). This results in fibres with a highly aligned structure in the direction of flow with long, straight, polymer chains oriented in the longitudinal direction. The fibres are generally available as yarns or rovings containing between 25-1000 filaments. Sizing is not required for many composite applications but can be applied to improve processing when certain resin systems are used.

Aramid fibres offer a tensile modulus between that of glass and carbon, around 100 GPa, very high tensile strength, around 3.6 GPa and low density. However the mechanical properties are extremely anisotropic, the compressive properties of the fibres are very poor and therefore the compressive strength is in the order of 20 % of the tensile strength. Aramid fibres exhibit excellent damage tolerance making them ideal for some forms of armour and other energy absorbing applications. If tensile strength-to-weight is extremely important in the application then aramid is the material of choice.

1.2.2 FABRIC STRUCTURE

In the majority of cases fibres need to be processed into an intermediate form of reinforcement before they can be used in the production of composite structures. The type of reinforcement chosen for a specific application is very important as it affects cycle times, determines fibre volume fractions, permeability and the final mechanical properties of the composite product. The discussion below describes some of the more common types of fabric reinforcement.

1.2.2.1 Chopped Strand Mat

Chopped strand mat (CSM) is manufactured by chopping continuous fibre rovings into short lengths which are then allowed to fall onto a moving conveyor belt. A binder is then applied either by spraying or in powder form to hold the fibres together and then the constituents are rolled between heated rollers for consolidation into a mat. Fibre lengths are generally between 10-75 mm and are typically made from glass. The CSM has a random in-plane isotropic structure with fibre volume fractions up to around 0.35. Infiltration is easy due to the random structure and low fibre volume fraction but tensile strength and modulus are relatively low.
1.2.2.2 Continuous Random Mat

A similar manufacturing process to CSM where, instead of chopped fibre rovings, a continuous roving is unwound onto a moving conveyor belt forming a pattern of overlapping random loops or swirls (continuous random mat is often known as swirl mat). A binder is applied, the material heated and then compressed between rollers to consolidate the mat. The fibre volume fraction tends to be a little lower than that of CSM, a typical maximum is in the range 0.30-0.35. This results in a similar tensile modulus but due to the continuous fibre structure the strength and toughness are increased with more consistent overall mechanical properties. The interlocking of the fibres reduces fibre displacement due to resin flow in processes like RTM and ensures that defects from tearing are reduced to a minimum. The open nature of the continuous random mat (CRM) results in a high permeability and means they are easily wetted out by liquid resins.

1.2.2.3 Woven Fabrics

Woven fabrics are made by interlacing yarns (twisted) or rovings (untwisted) at a 90° angle and are widely used in the manufacture of laminated structures. They are available in a variety of weights and weaves and exhibit good stability in the warp and weft directions. The simplest construction is the plain weave (see Figure 1.6) where each warp yarn passes alternately over then under each weft yarn allowing fibre volume fractions in the finished laminate of approximately 0.6. The yarns in the two orthogonal directions may be balanced or have up to 90% of the fibre in one direction (see Figure 1.6). In-plane strengths are much higher than for CSM and CRM, discussed earlier, having a maximum tensile modulus in the direction of the yarns and a minimum at 45°. Of all the weaves, plain is the most stable resulting in good handleability.

The mechanical properties of laminates made from woven fabrics vary considerably with weave type and the degree of crimp. Plain weave fabrics possess a high degree of crimp which can reduce the modulus by up to 15% compared with a similar fraction of straight fibres. The degree of crimp in twill weave fabrics (see Figure 1.6) is lower offering better drapeability and a more tightly packed structure. Satin weaves offer the least amount of crimp as the warp
yarns alternately go over several weft yarns (four and seven for a five and eight harness satin respectively, see Figure 1.6) before going under one. This also allows satin weaves to be very tightly packed and generally have much better mechanical properties than both the plain and twill weaves. The low resistance to shear of the satins also increases the amount of drapeability and therefore more complex mouldings can be achieved. However the more tightly packed the fabric is, the lower is its permeability and the fabric becomes more difficult to infiltrate. The high degree of shearing can lead to handling problems and also leads to poor in-plane shear resistance in the final laminate.

![Figure 1.6 Schematic of woven cloths showing, plain, twill and 5 harness satin weaves.](image)

**1.2.2.4 Triaxial Fabrics**

Conventional woven fabrics consist of a warp and weft yarn at 90° to each other where the warp yarn lies along the machine direction. For triaxially woven fabrics there are three sets of
yarns, two sets of warp yarns at ±30° and a weft yarn at 90° to the machine direction. This results in three sets of yarns which interlace at 60° angles improving planar isotropy, higher in-plane shear modulus and better handleability.

1.2.2.5 Knitted Fabrics

Knitted fabrics are made by interlooping one or more yarns together and can be suited to fit a wider range of applications than woven fabrics. The basic warp and weft knits (see Figure 1.7) have an open structure with a high degree of crimp but due to considerable extensibility in all directions have better drape properties than woven fabrics. By including laid-in, non-knitted warp yarns (or weft yarns) into the knit, stability can be achieved in one direction and extensibility in the rest. In the same way, the addition of both warp and weft yarns into a simple knitted fabric results in an extremely stable structure. Depending on the situation, a complete range of knits can be made in varying degrees of complexity by mixing different types of knits and yarns. Infiltration is usually easy due to the open structure but with low fibre volume fractions which results in laminates with only average mechanical properties. Knitted fabrics are ideally suited to the manufacture of cylindrical components such as pipes and aircraft radomes.

![Figure 1.7 Schematic showing the two basic knits: (a) warp knit, (b) weft knit.](image)

1.2.2.6 Braided Fabrics

Braided fabrics are constructed by intertwining yarns and, depending on the design of the braid, they can offer stability or drapeability. They are available in a wide variety of forms including hollow tubular, flat, solid square and irregularly shaped solids. By including yarns
that are laid into the braid system but are not actually intertwined, further forms such as stuffed tubular and stuffed flat can be constructed. These laid-in or stuffer yarns add stability to the braid when under tension in the direction of the yarn system but can do nothing for the poor stability in axial compression. Having better mechanical properties than knitted fabrics, due to less crimp, they are often preferred when high performance structures are required.

1.2.2.7 Three-Dimensional Fabrics

Regular composite laminates suffer from poor interlaminar strength as they are both weak in shear between the layers of the laminate and also in tension normal to the plane of the laminate. Structures for the aerospace industry were required that could withstand multidirectional mechanical stresses. The development of three-dimensional composite structures in the late 1960s sought to overcome this problem [Ko (1989)]. 3-D structural composites are fully integrated fibre assemblies possessing multiaxial in-plane and out-of-plane fibre orientation. This additional reinforcement in the through thickness direction makes the composite virtually delamination free. 3-D products are manufactured by weaving, knitting and braiding and can be formed into many complex shapes.

1.2.2.8 Non-Crimp Stitch Bonded Fabrics

The mechanical properties of a woven laminate are largely determined by the amount of crimp that the fabric possesses. A number of fabrics have been developed with virtually zero crimp which may have up to a 15 % higher load bearing efficiency than woven fabrics [Rudd et al (1997)]. These non-crimp stitch bonded fabrics consist of unidirectional layers of aligned rovings which have been bonded together by cross stitching with a very light yarn made usually of polyester. Four or more layers are bonded together in their preferred orientation, e.g. 0°, 90°, ±45°, to form a single fabric (see Figure 1.8).

Thus a whole range of fabrics can be produced with a variety of fibre orientations depending on the mechanical performance required. The other advantage is that laminate lay-up times are greatly decreased because for every layer of stitch-bonded fabric that is placed in the mould there are at least four unidirectional layers. These fabrics are more stable than the equivalent
woven structure with high fibre volume fractions in the range of 0.65-0.70. Also the stitch pattern can be varied to improve the drapeability of the fabric in excess of that obtained for unidirectional prepreg.

Figure 1.8 Schematic of a non-crimp stitch bonded fabric combining four unidirectional layers of different orientations.

1.3 SCOPE OF THIS STUDY

In many manufacturing processes of composite articles there will be a certain amount of compression acting on the fibre reinforcement. The major role of this compression is to achieve the consolidation of the plies within a composite laminate. Ply consolidation is established by a combination of mechanisms including compression of fibre reinforcement, flow of polymer resin and resin curing. This compression of the fibre reinforcement may be by means of an applied pressure (as in compression moulding and autoclave processing) to remove excess resin, eradicate air pockets and to help prevent void formation within the composite. The consolidation process also helps to increase, and make uniform the fibre volume fraction. In other processes such as in resin transfer moulding (RTM) there is a certain amount of compression experienced during the lay-up of the fibre reinforcement into the mould. This is further compounded when the mould is closed after the lay-up stage adding
more pressure to the fibre reinforcement [Rudd et al (1993)]. It is believed that this compression is substantial enough to increase the fibre volume fraction of the FRC and hence affect the permeability. This is of paramount importance as both the fibre volume fraction and the permeability of the composite structure ultimately determine its final mechanical properties.

The purpose of this study is to analyse experimentally the compressibility of fibre/resin systems for different types of woven fabrics and resins in terms of mechanical testing. Laminates are to be manufactured and the microstructure examined under different levels of compression. A technique for measuring a range of microstructural parameters for woven fabrics under compression is to be devised in order to investigate the different modes of compression. From the knowledge gained in the study a mathematical model is to be constructed describing the compression of resin impregnated woven fabrics in terms of both viscous, Newtonian and non-Newtonian resin flow.

More specifically the aims of this study are:

(a) To carry out systematically a series of compression tests on assemblies of various types of dry fabrics and study the effects on compression.

(b) To carry out systematically a series of compression tests on assemblies of various types of resin impregnated fabrics and study the effects on compression.

(c) To construct a mathematical model of compression of resin impregnated fabrics, to investigate the validity of this model in comparisons between theoretical and experimental data and to fit experimental data to suitable mathematical relations where appropriate.

(d) To examine the development of microstructure of laminates compressed at different levels of compression, for different types of fabrics and different types of resin.

(e) To develop a methodology to represent the cross-section of plain weaves in terms of mathematical, geometrical parameters which could be measured in the microstructural analysis of compressed laminates.
(f) To investigate the mechanisms and modes of compression with the aid of the microstructural analysis of compressed laminates.

The description of the next chapters is as follows:

Firstly Chapter 2 contains some relevant background information in the form of a literature survey.

Chapter 3 deals with the material and resin characterisation together with the experimental procedures used throughout the testing. It also includes the description of the methods employed for the determination of resin viscosity, laminate construction, mechanical compression testing and the image analysis of laminate microstructures.

In Chapter 4 a mathematical analysis is performed for the viscoelastic compression of resin impregnated assemblies of woven fabrics. A model has been developed incorporating the deformation of the fibre network and resin flow for the fibre/resin system.

Chapters 5 and 6 present the results and discussion regarding the compression experiments on assemblies of dry and resin impregnated reinforcement respectively. Numerous scenarios have been investigated including the repeated compression of the same assembly of plies, changing ply orientation and varying the compression speed to examine the effects on the compression data. Compression curves, compression hysteresis data and pressure relaxation data has been produced for a variety of fibre/resin systems. At the end of Chapter 6 theoretical predictions according to the model in Chapter 4 are compared with experimental data for the compression of an assembly of glass fibre woven plies impregnated with an epoxy resin.

Chapter 7 describes the microstructural image analysis of compressed plain woven laminates with two different resin systems. Specimens cured under a range of final compression loads were sectioned and their microstructure was examined using optical microscopy where different microstructural parameters were measured. The compression of the assembly of fibre cloths was modelled as a combination of four modes of deformation: elimination of the resin rich layers between plies, nesting between plies, deformation of the yarn waveform and
1. Introduction

decomposition of the yarn cross-section. The yarn cross-section was described as lenticular for
which the major and minor axes and the area were measured. The yarns in plain weaves were
modelled as sinusoidal waveforms for the definition of which measurements included
amplitude, wavelength and local phase angle. Nesting was assessed from the position of the
line through the midpoints of each yarn centreline and from local phase angles.

Chapter 8 goes further to discuss the microstructural comparison, in terms of fibre, porosity
and void area fraction, between different types of compressed fabrics, namely plain weave,
twill weave, 5 harness satin weave and noncrimped stitch-bonded fabric.

Finally Chapter 9 contains a summary of conclusions and recommendations for future work.
2. LITERATURE REVIEW
2.1 FABRIC ARCHITECTURE IN WOVEN FABRICS

There are many terms used to describe the different properties of fibre woven fabrics. The aim of this section is to explain some of the established definitions for fabrics.

A wide range of reinforcing materials are used in the production of fibre reinforced composites but there are three main classes for commercial applications: glass, carbon and aramid. Hybrid fabrics also exist which contain a combination of yarns of different reinforcing fibres. There are many possible material combinations, glass/carbon, glass/aramid, etc. which can exist in many different proportions; warp yarns of glass with weft yarns of carbon for example. The availability of hybrid reinforcement allows fabrics to be tailored for specific applications where certain mechanical properties need to be optimised.

The fibre bundle or strand is itself made up of a number of unidirectional fibres, and if used to weave a fabric in that state is known as a fibre bundle. However, it is common for two or three bundles to be used together to form a typical roving (untwisted) or to form a yarn (twisted). It is these yarns or rovings that are then woven usually at 90° to form the fabric.

The yarns along the length of the fabric are defined as warp yarns whilst the yarns perpendicular to them are defined as the weft or fill yarns. A major characteristic of the fabric is the number of warp yarns or ends per unit length compared with the number of weft yarns or picks per unit length. This is to help prevent confusion between two fabrics having the same density but different structures. For instance, one of the fabrics may be woven from large fibre bundles whilst the other is woven from smaller fibre bundles. By knowing the different number of ends and picks per unit length used in each of the fabrics, one can distinguish between the two. Fabrics having the same number of ends and picks, and with similar weights, are known as balanced therefore making all other fabrics unbalanced. In an unbalanced fabric the difference between the number of ends and picks may be small or large.

Three of the fabrics that have been used throughout this study are plain, twill and satin glass fibre weaves. Plain is used to describe the type of weave for a fabric where each warp yarn passes alternately over then under each weft yarn. Twill describes a woven fabric where each
warp yarn passes alternately over then under ‘two’ weft yarns. Finally the satin weave has warp yarns that alternately go over several weft yarns before going under one (see Figure 1.6).

A system has been devised that uses two geometrical parameters to describe a weave [Ishikawa and Chou (1982a, 1982b, 1983) and Chou (1985)] where the weave is characterised by the repeat pattern of the interlaced regions. A fill yarn is interlaced with every \( n_w \)-th warp yarn and every warp yarn is interlaced with every \( n_f \)-th fill yarn, where \( w \) and \( f \) refer to the warp and fill yarn respectively, whilst \( g \) signifies a geometric parameter. In most fabric weaves \( n_w = n_f = n_g \).

Industry standard orthogonal fabrics are generally confined to three types of structure, plain, twill, and satin weave. A plain weave fabric has \( n_g = 2 \), i.e. the fill is interlaced with every warp yarn and vice versa (see Figure 2.1a). Twill weaves have \( n_g = 3 \) (see Figure 2.1b), whilst any fabric with \( n_g \geq 4 \) is known as a satin weave (see Figures 2.1c and 2.1d). Plain weave fabrics have equal proportions of the warp and fill yarns on either side of the fabric. For all other fabrics, where \( n_w \geq 3 \), there is a dominant type of yarn associated with each surface.

![Examples of woven fibre fabrics](image)

**Figure 2.1** Examples of woven fibre fabrics: (a) plain weave (b) twill weave (c) 4 harness satin (d) 8 harness satin.
Thus for the accurate description of an individual woven fabric, six parameters must be defined in order to identify it as a unique cloth:

- fibre material
- normal or hybrid cloth
- weave type (plain, twill, satin, etc.)
- weight of the warp and weft yarn
- balanced or unbalanced
- weight of cloth per unit area (g/m²)
- number of picks and ends (/m).

2.2 THE CONSOLIDATION OF COMPOSITE LAMINATES

As mentioned earlier in the introduction, the purpose of consolidation is to remove air and excess of resin, to aid in the suppression of voids and to increase and make uniform the fibre volume fraction. Furthermore consolidation can be considered as a combination of three processes,

(a) Resin flow through porous media,
(b) Fibre deformation,
(c) Resin curing.

Gutowski et al (1986) stated that to completely describe the consolidation process all of these phenomena must be considered.

2.2.1 RESIN FLOW

The impregnation phase is an important step in the fabrication of fibre reinforced composites (FRCs) with reinforcement pre-laid into the mould (as in RTM), involving polymer fluid flow through the anisotropic porous media. This polymer flow is one of the main areas of concern when considering process control. Fluid flow through fibrous material can be divided into two

(1) flow in the plane of the material,

(x and y, see Figure 2.2),

(2) flow transverse to the plane of the material,

(z or through thickness direction, see Figure 2.2).

Figure 2.2 displays a model showing the designated in-plane and through thickness directions. Both through thickness flow and in-plane flow represent fluid flow through porous media. The two modes of flow may be significantly different when the fibrous material is structurally anisotropic.

Figure 2.2 Coordinate system and dimensions for an aligned fibre composite [Gutowski et al (1987a)].

Since the assembly of fibre cloths is highly anisotropic the values obtained for the permeabilities in the in-plane direction are different to the permeability obtained for the through thickness direction.

Clearly the rate at which a fluid penetrates the reinforcement and the shape of the advancing
fluid flow front are dependent on the rheology of the fluid and on the structure of the reinforcement. In RTM, due to the usually very small thickness of the reinforcement when compared to the width or length, the majority of work concentrates on the two dimensional in-plane flow of the polymer [Pillai and Advani (1994)]. In other processes, such as autoclave processing, flow in the through thickness direction is dominant.

2.2.1.1 Darcy's Law

The most widely used theory in the study of flow through porous media is Darcy's Law (see Equation (2.1)). Darcy states that;

'with a constant, homogeneous permeability of the porous medium and a constant viscosity of the passing fluid, there is a linear relationship between the pressure gradient and the superficial velocity for a steady state viscous flow'.

\[ u = \frac{K \, dP}{\mu \, dx} \]  

(2.1)

Here \( u \) is the superficial velocity, \( K \) the permeability, \( \mu \) the resin viscosity and \( \frac{dP}{dx} \) the pressure gradient in the fluid. The superficial velocity can be expressed in terms of the actual velocity, \( u_{\text{real}} \), by the relation

\[ u = e \cdot u_{\text{real}} \]  

(2.2)

Here \( e \) is the porosity of the porous medium available for fluid flow and \( u < u_{\text{real}} \). This law is proved to be valid in many engineering fields:

- soil mechanics
- petroleum engineering
- textile applications
- materials processing.
Darcy's Law has been used to describe polymer flow through the fibrous reinforcement with some success in various studies, both experimental and theoretical [Adams et al (1986), Gutowski et al (1987a) and other workers]. To achieve this two basic assumptions are made:

- flow is viscous dominant,
- fluid is Newtonian,

i.e. the shear stress of the fluid is directly proportional to the shear rate.

In general, although polymers behave in a non-Newtonian manner, by using the Newtonian fluid approximation many processes utilising thermosetting resins have shown reasonable results. However most of these cases involved a relatively low flow rate.

With new improvements in processing techniques there are many cases of polymer fluids passing through fibrous reinforcements at high flow rates. In these instances, the assumptions of Newtonian flow behaviour and viscous dominant flow may not be valid and non-Darcy flow behaviour is observed, i.e. the linear relationship between the fluid pressure gradient and flow rate is no longer valid. Other factors such as inertial effects, inhomogeneity in porosity and shear rate also become important.

Cai (1993) proposed a generalised flow model for non-Darcy flow, which considers inertial and viscous effects. Since flow is no longer viscous dominant, Cai used the Ergun equation to account for the inertial flow effect based on dimensional analysis.

\[
\frac{dP}{dx} = C_L \frac{\mu u (1-\varepsilon)^2}{\varepsilon^3} + C_T \frac{\rho u^2}{d_f} \frac{[1-\varepsilon]}{\varepsilon^3} \tag{2.3}
\]

Where \( C_L \) is a viscous flow constant, which is equivalent to the Kozeny constant in the Carman-Kozeny equation (see Section 2.2.1.2) and \( C_T \) is an inertial flow constant. \( C_L \) and \( C_T \) are determined experimentally. \( d_f \) is the fibre diameter, \( \varepsilon \) is the porosity and \( \rho \) is the fluid density. The fluid is still considered a Newtonian fluid of constant viscosity \( \mu \).
Christopher and Middleman (1965) used a modified form of Darcy's Law using a power law relation between velocity and pressure drop, verified by various experimental results, to allow for the non-Darcy flow behaviour of a non-Newtonian fluid.

\[
u = \left[ \frac{K}{M} \left( \frac{dP}{dx} \right)^n \right]^{\frac{1}{n}}
\] (2.4)

Equation (2.4) is applied to steady, viscous flow of a non-Newtonian fluid through a porous medium. The power law index \(n\) which is dimensionless determines the non-Newtonian behaviour of the polymer fluid. In their experimental work the value for \(n\) is typically 0.2 - 0.5 for the shear rates found in high speed flows and 0.75 - 1.0 for slow flows [Rosen (1993)]. \(M\) is a parameter depending on the porosity of the reinforcement.

Cai took the power law relation (2.4) together with the Ergun equation to form his generalised flow model.

\[
\frac{u \rho d_{f}}{\mu (1 - \varepsilon)} \left[ C_L + C_T \frac{u \rho d_{f}}{\mu (1 - \varepsilon)} \right] \left[ \frac{(1 - \varepsilon)^3}{\varepsilon^3} \right] = \left[ \frac{-dP}{dx} \frac{\rho d_{f}^3}{\mu^2} \right]^{\frac{1}{n}}
\] (2.5)

This relationship shows that there is no longer a linear relationship between the pressure gradient and the superficial velocity, as in the original Darcy relationship. The empirical constants are \(C_L, C_T\) and \(n\). This means that Cai's flow model may be used to evaluate more general flow situations in which both Darcy flow and non-Darcy flow are taken into consideration depending on the situation.

### 2.2.1.2 Permeability

The permeability, \(K\), in Darcy's law is one of the most used parameters in terms of the modelling of fluid flow through the reinforcement. This together with the viscosity of the resin determines the fluid flow characteristics during impregnation. Methods for the calculation of permeability in the different reinforcement structures vary considerably, but much work has been carried out to try and establish theoretical models and suitable experimentation techniques [Verheus and Peeters (1993)].
(i) Aligned and Continuous fibre bundles

The permeability can be determined using a form of the Carman-Kozeny equation, a permeability model for the flow of resin through a bed of unidirectional fibres. It was first presented by Williams et al (1974), (see Equation (2.6)), and is now the most cited work in this field. This model has been further studied and verified by Gutowski et al (1987b), Cai (1992), and other workers.

\[
K_x = \frac{(r_f)^2 (1 - V_f)^3}{4k_x V_f^2}
\]  

(2.6)

Here, \( K_x \) is the permeability in the x-direction (the fibre direction) as in Figure 2.2, \( r_f \) is the fibre radius, \( k_x \) is the Kozeny constant in the x-direction determined experimentally and \( V_f \) is the fibre volume fraction. Most studies agree to the effectiveness of this model for axial fluid flow although Cai (1992) points out that the values of the Kozeny constant vary in different experiments (see Table 2.1). Data scattering was observed in all these experiments, reflecting the status of the fibre bundle. Such factors as the wetting process, the fluid-reinforcement interface and a non-uniform fibre distribution all add to the results' variability.

<table>
<thead>
<tr>
<th>Reference</th>
<th>( k_x )</th>
<th>Test conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Williams et al (1974)</td>
<td>0.38 (initial water flow)</td>
<td>Porosity = 0.486</td>
</tr>
<tr>
<td></td>
<td>0.45 (water displaced by alcohol)</td>
<td>Pre-wet fibre beds (carbon)</td>
</tr>
<tr>
<td></td>
<td>0.35 (initial alcohol flow)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.39 (alcohol displaced by water)</td>
<td></td>
</tr>
<tr>
<td>Gutowski et al (1987b)</td>
<td>0.70</td>
<td>AS4 fibres (carbon)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Corn oil or silicone fluid</td>
</tr>
<tr>
<td></td>
<td></td>
<td>( V_f = 0.4 - 0.8 )</td>
</tr>
<tr>
<td>Lam and Kardos (1988)</td>
<td>0.35 (silicone oil)</td>
<td>Carbon fibres</td>
</tr>
<tr>
<td></td>
<td>0.68 (water)</td>
<td>( V_f = 0.57 - 0.75 )</td>
</tr>
</tbody>
</table>
There are many approaches for calculating permeability of the flow in the direction transverse to the fibres. Lam and Kardos (1988) and many others have used the same Carman-Kozeny model but with a different value for the Kozeny constant. This has shown to match experimental data within specific ranges of volume fraction. Experimental values obtained by Lam and Kardos (1988) for the Kozeny constant show the flow resistance in the transverse direction to be up to twenty times greater than the flow resistance in the direction of the fibres.

The major problem with using the same model for the transverse flow is that it does not take into consideration the situation when the fibre volume fraction reaches the maximum packing efficiency (available fibre volume fraction), hence stopping the transverse fluid flow. For ideal packing the maximum packing capacity is 0.79 for a square packing and 0.91 for a hexagonal packing. Under real conditions the maximum packing capacity is probably somewhere between these two values. So, as the fibre volume fraction increases toward the maximum packing capacity the resistance to transverse flow also increases due to the decreasing flow paths and to the gap resistance. It thus seems logical that the model used for flow along the fibre direction is inappropriate when considering transverse flow. This has led to work being carried out to modify the original Carman-Kozeny model to allow for the influences of maximum packing.

Gutowski et al (1987b) carried out simulated compression moulding and bleeder ply moulding of composites made from Hercules AS-4 graphite fibres impregnated with silicone oil, and studied the oil pressure history. The compression moulding experiment, at compression speeds of 0.05 and 0.1 mm per minute, ensured one dimensional flow along the fibre axis. Figure 2.3 shows the comparison of measured and predicted values of the resin pressure. As can be seen the unmodified form of the Carman-Kozeny model shows reasonable agreement to the measured results.

The results for Gutowski's simulation of bleeder ply moulding for transverse flow can be seen in Figure 2.4. The unmodified form of the Carman-Kozeny model was unable to predict the resin pressure decay exhibited in the experiment. However by adopting the following modified form of the model, which allows for the maximum packing capacity, good fitting of the data was obtained.
2. Literature Review

\[ K_z = \frac{r_f^2}{4k_z} \left( \frac{\sqrt{V_a}}{\sqrt{V_f}} - 1 \right)^3 \]  

(2.7)

Where \( K_z \) is the transverse permeability, \( k_z \) is the Kozeny constant in the transverse direction and \( V_a \) is the maximum packing capacity. This model effectively allows \( K_z \rightarrow 0 \) for \( V_f \rightarrow V_a \) where \( V_a \) is the fibre volume fraction for which all flow stops.

![Figure 2.3](image)

**Figure 2.3** Comparison of measured and predicted values of the resin pressure for resin flow along the fibre direction in compression moulding [Gutowski et al (1987b)].

A comparison of the models for transverse permeability used by Lam and Kardos (unmodified Carman-Kozeny relation) and that proposed by Gutowski (modified Carman-Kozeny relation) and confirmed by experimentation can be seen in Table 2.2, where the permeability has been normalised as \( K_z/r_f^2 \). The comparison is also illustrated in Figure 2.5. From the plots it can be seen that the difference between the two models in the 0.5 to 0.6 \( V_f \) region is relatively small but becomes more significant as the fibre volume fraction reaches 0.7 and above. Cai (1992) argued that in RTM, since fibre volume fractions are generally in the lower range, either of the models can be used.
2. Literature Review

Figure 2.4 Comparison of measured and predicted values for flow transverse to the fibres in compression moulding [Gutowski et al (1987b)]

Table 2.2 Comparison of the normalised permeability in the transverse direction.

<table>
<thead>
<tr>
<th>$V_f$</th>
<th>$K_x/t_f^1$</th>
<th>$K_x/t_f^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Lam and Kardos ($k_g=11.0$)</td>
<td>Gutowski ($k_g=0.2$, $V_f=0.8$)</td>
</tr>
<tr>
<td>0.45</td>
<td>$1.867 \times 10^2$</td>
<td>$1.667 \times 10^2$</td>
</tr>
<tr>
<td>0.50</td>
<td>$1.136 \times 10^2$</td>
<td>$8.938 \times 10^3$</td>
</tr>
<tr>
<td>0.55</td>
<td>$6.846 \times 10^3$</td>
<td>$4.455 \times 10^3$</td>
</tr>
<tr>
<td>0.60</td>
<td>$4.040 \times 10^3$</td>
<td>$1.983 \times 10^3$</td>
</tr>
<tr>
<td>0.65</td>
<td>$2.306 \times 10^3$</td>
<td>$7.337 \times 10^4$</td>
</tr>
<tr>
<td>0.70</td>
<td>$1.252 \times 10^3$</td>
<td>$1.920 \times 10^4$</td>
</tr>
<tr>
<td>0.75</td>
<td>$6.313 \times 10^4$</td>
<td>$2.133 \times 10^5$</td>
</tr>
</tbody>
</table>

Cai (1993) developed his own relationship for the permeability based on his generalised flow model represented by Equation (2.5) where subscript $g$ defines generalised permeability.
This relationship thus takes into consideration not only the properties of the fibrous reinforcement, such as fibre diameter and the porosity, as in the Carman-Kozeny relationship, but also the viscosity of the polymer fluid and the process variables such as the pressure gradient or flow velocity. This means that if in an experimental flow situation a permeability, $K_d$, is calculated using Darcy’s Law and a permeability, $K_g$, is calculated by using Cai’s relationship, the values for $K_d$ and $K_g$ may be substantially different. This is because $K_d$ has been calculated using Darcy’s Law and has not taken into consideration the possible non-Newtonian behaviour of the fluid and the inertial flow effects, whereas Cai’s $K_g$ has. This then illustrates that by using Darcy’s Law in a non-Newtonian fluid situation significant error may be introduced.
(ii) Woven and Cross-Ply Fibrous Structures

For structural applications it is more usual for preforms to be made from woven or cross-ply fibre cloths. This is because of their superior performance on damage tolerance. These preforms are therefore set up in a certain way, woven usually in a 0°, 90° arrangement, whilst in a braided structure a variation of angles are commonly used. Work by Lam and Kardos (1989) considered the permeability of firstly aligned fibres and secondly woven fabrics. In the woven structure there are two flow cases, in-plane flow and the flow transverse to the fibre plane. They again used the unmodified Carman-Kozeny relationship but with different values for the Kozeny constant. Lam and Kardos considered the unidirectional and the 0°, 90° woven as the two extreme structures. Therefore the permeability of a woven structure with any other cross-woven angles can be expressed using the permeability value of the two extreme cases.

An assembly of unidirectional fibre plies is considered where each ply has been laid up at an angle $\theta$ with respect to the previous ply. The proposed relationship [Lam and Kardos (1988)] for the Kozeny constant is:

\[
(k_x)_{\theta\theta} = (k_x)_{\text{uni}} \cos^2 \theta + (k_x)_{090} \sin^2 \theta
\]  

\[
(k_z)_{\theta\theta} = (k_z)_{\text{uni}} \cos^2 \theta + (k_z)_{090} \sin^2 \theta
\]  

Where $\theta$ is defined as the angle between the fibres in successive plies, the subscript $x$ refers to in-plane flow, $z$ refers to transverse flow, $(k_x)_{\text{uni}}$ and $(k_z)_{\text{uni}}$ are the Kozeny constants for a unidirectional fibre bed in the in-plane and transverse directions respectively and $(k_x)_{090}$ and $(k_z)_{090}$ are the Kozeny constants for a 0°, 90° bed of fibres.

The permeability of assemblies of woven fabrics depends on the structural characteristics of fabric and more specifically on the pore size distribution, pore shape and tortuosity of the flow path. Several workers [Adams and Rebenfeld (1987), Summerscales (1993), Griffin et al (1995), Shafi and Neitzel (1996)] have reached the above conclusions in their measurement of the permeability of assemblies of different types of fabrics: plain, twill and satin. In particular, it was found that it is possible to design flow enhancing fabrics with uneven pore distribution. This was achieved [Griffin et al (1995), Guild et al (1996)] by incorporating a bound tow
periodically in the fabric structure where this tow was less flattened during weaving than the rest of the yarns. However, this locally increased porosity creates resin rich areas in the composite product which might degrade mechanical properties [Basford et al (1995)].

2.2.2 COMPRESSION OF RESIN IMPREGNATED REINFORCEMENT: RESIN FLOW AND FIBRE DEFORMATION

As mentioned previously in this chapter Gutowski et al (1987a, 1987b) described consolidation as the combination of both, resin infiltration, and fibre deformation of the fibrous reinforcement. They argued that resin flow during the moulding process cannot be accurately described unless the fibre deformation is also taken into account. Previous work on compression moulding had only considered resin flow out of the composite ignoring the elastic effects of fibre deformation due to resin pressure [Springer (1982) and Loos and Springer (1983)]. Gutowski’s theory for unidirectional laminates, based on the Voigt viscoelastic model (see Figure 2.6), modelled the fibre reinforcement as a porous, non-linear elastic medium filled with a viscous liquid.

![Figure 2.6 Voigt viscoelastic model displaying how the applied pressure is shared between the resin and the fibres.](image)

The idea is that when pressure is applied normal to the flat laminate it is applied to both the resin and the fibre. The Voigt viscoelastic model has a spring element which represents the nonlinear elastic deformation of the fibres and a damper element representing the viscous resin. According to Gutowski’s model, there is no deformation initially and so the applied pressure is...
carried solely by the resin. As the pressure causes the resin to flow out of the laminate the reinforcement will begin to compact, compressing the fibres and increasing the fibre volume fraction, $V_f$. By doing so, some of the applied pressure is transferred to the fibres, consequently reducing the applied pressure on the matrix.

The model taken to its extremities would result in all the resin flowing out of the laminate and the pressure on the resin tending to zero. This results in the composite's maximum fibre volume fraction being completely determined by the deformation of the fibres, having nothing to do with the resin flow process. In reality there will be several restrictions to the flow process (e.g. high resin viscosity) meaning that the applied pressure will only ever be partly transferred to the fibres. Figure 2.2 shows the type of the composite structure used as the basis of the mathematical model of Gutowski (1987a, 1987b), a flat laminate of continuous aligned fibres.

Thus the applied pressure $P$ transverse to the fibre plane must be split into an average resin pressure $\bar{p}_r$, and an average effective stress in the fibre network $\sigma$.

\[
P = \sigma + \bar{p}_r. \tag{2.11}\]

Here $\sigma = \bar{p}_f \cdot a_f$ where $\bar{p}_f$ is the average fibre pressure and $a_f$ the area volume fraction. To obtain a relationship describing the transverse stiffness of the fibre network (see Equation (2.12)) Gutowski assumed that all fibres act as bending beams with numerous contact points due to the natural waviness of the fibre and also due to fibre misalignment. As the laminate is compressed more contacts between the fibres will be established. To describe this behaviour of the fibres a relationship for $\sigma$ was derived

\[
\sigma = A_s \frac{\left( \frac{V_f}{V_{f_0}} - 1 \right)}{\left( \frac{1}{V_f} - \frac{1}{V_a} \right)^4}. \tag{2.12}\]

Here $A_s$ is the spring constant, $V_{f_0}$ is the original fibre volume fraction and $V_a$ the available fibre volume fraction (i.e. the maximum fibre volume fraction possible) so that at
\[ V_f = V_{f0}, \ \alpha = 0, \text{ and as } V_f \to V, \ \alpha \to \infty. \] Cai and Gutowski (1992) defined the spring constant as

\[ A_s = 3\pi \frac{E}{\left( \frac{L}{a_1} \right)^4} \tag{2.13} \]

where \( E \) is the bending stiffness of the fibre and \( L \) and \( a_1 \) are the span length and span height respectively of the fibre beam based on the buckling arch concept (see Figure 2.7).

Figure 2.7 Schematic of the buckling arch model for a slightly curved fibre [Cai and Gutowski (1992)].

Fibre compression experiments were carried out to confirm Equation (2.12) [Gutowski et al (1986)] and took the form of compressing bundles of approximately aligned fibres impregnated with a light oil at compression speeds of 0.05 and 0.1 mm per minute. Fibre alignment representative of commercially available prepregs was established by dissolving out the resin from epoxy prepregs and then impregnating them with oil. The compression results of the fibre bundles in their drained state (resin pressure = 0) showed them to be approximately elastic i.e. once the load had been removed, the fibre bundle fully recovered from the incurred deflection. The results showed that for typical fibre volume fractions the fibre bundles can carry significant loads when the resin pressure within the composite is much less than the applied pressure. Graphs constructed for applied pressure versus fibre volume fraction from both the experimental data and the bending theory model showed good correlation.

The fibre volume fraction also has an effect on the permeability of the composite. Gutowski
used the Carman-Kozeny relation (Equation (2.6)) to calculate the permeability. By further manipulation of equations for fibre and resin continuity and of Darcy's Law (Equation (2.1)) a relationship was derived for the pressure distribution within the resin.

\[
K_x \frac{\partial^2 \bar{p}_r}{\partial x^2} + K_z \frac{\partial^2 \bar{p}_r}{\partial y^2} + \mu \frac{\partial V_f}{\partial t} = 0
\]  

(2.14)

Where, \( K_x \) and \( K_z \) are the permeabilities, \( \bar{p}_r \) the average resin pressure, \( \mu \) the viscosity of the resin and \( V_f \) the rate of change in fibre volume fraction.

Equation (2.14) was then simplified for one dimensional flow, and by using Equation (2.12) for the fibre stiffness and Equation (2.6) for the permeability, Gutowski derived a final equation showing how the applied pressure is shared between the fibres and the resin.

\[
P = A_s \frac{\left( \frac{V_f}{V_a} - 1 \right) \left( \frac{1}{V_f} - \frac{1}{V_a} \right)}{\left( \frac{1}{V_f} - \frac{1}{V_a} \right)^4} + \frac{4}{3} \frac{\mu k_x r_f^2}{r_f^3 (1-V_f)^3} \frac{V_f}{V_f}
\]  

(2.15)

In this relationship, \( P \) is the total applied pressure to both the resin matrix and the fibres and \( a \) is the length of a rectangular laminate. Thus the first part of the relationship describes the pressure responsible for the fibre deformation whilst the second part is responsible for the resin viscous effects. If the rate of compression of the laminate is very slow then \( V_f \) tends to zero and the resin viscous effects become negligible which, in turn, increases the load carried by the fibres.

Lekakou et al (1993, 1996a) continued the work of Gutowski with regard to the deformation of fibre reinforcement in composites. Their area of research differed in that the work considered the compression of woven cloths rather than that of unidirectional aligned fibres. Gutowski's viscoelastic model is used for the transfer of pressure between fibre and resin and elastic beam theory used to model the basic element in a woven cloth. The following relation between fibre pressure and fibre volume fraction was finally derived:
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\[ \bar{p}_f = \frac{V_f^2 (V_f - V_{f_0})(2.75 \times 10^{-2})a_1a_2}{(a_1V_f - (3.9 \times 10^{-5}) + (1.64 \times 10^{-5}))^3} \]  

Equation (2.16)

Where \( \bar{p}_f \) is in atmospheres, the numerical values are derived from geometrical parameters of the fabric used in the experimentation. \( V_{f_0} \) is the initial fibre fraction before compression and \( a_1 \) is a factor in the range of 48 to 192, depending whether the contact points of the fibre bundles can or cannot slip respectively. \( a_1 \) is an empirical compressed height associated with the decrease in the span length of the beams. This is due to the assumption that the number of contact points of the fibre bundles increases with compression. Hence the span length of crossing beams decreases with increasing compression.

Compression experiments were carried out on twenty layers of glass fibre woven cloth impregnated with 60% sugar solution. The agreement between the experimental results at low compression speeds and data derived from Equation (2.16) was shown to be good. Hence the model can be used to determine the fibre volume fraction as a function of applied pressure in the elastic compression of woven cloths.

Servais et al (1997) used the power law relationship, derived by Toll and Manson (1994) (see section 2.2.3) for non-impregnated random planar fibre networks, and applied it to the compression of a fibre network impregnated with molten resin. They showed that the power law exponent \( b \) was unchanged at \( b=5 \) for compression speeds ranging from \( 10^3 \) to \( 5 \times 10^3 \) mm/s and that the relationship successfully predicted the loading response for a range of experimental data. Viscous effects of the resin were observed for compression speeds above \( 10^3 \) mm/s. Williams et al (1996) have also made preliminary investigations into the compression of resin impregnated reinforcement using the power law relationship for the Resin infusion under flexible tooling (RIFT) method. Possible lubricating effects of the plain weave reinforcement by the flowing resin were observed but a model describing the compression process of the fibres and resin has not yet been formulated.

The case of compression of glass mat thermoplastics is more complicated since it involves flow of both polymer melt and fibres [Kotsikos et al (1996)]. Wang and Gutowski (1991) constructed a model for the flow of fibres and melt in the compression moulding of continuous
aligned fibre reinforced thermoplastics. The total applied compression pressure is then consumed on the flowing system where the viscosity of such systems, including thermoplastic melt and fibres, is considerably high. It is possible that at high compression rates, the required total pressure is reduced [Wakeman et al (1995)], due to a possibly considerable decrease of viscosity. It must be mentioned at this point that such systems are not included in the present study.

2.2.3 COMPRESSION OF DRY REINFORCEMENT

Toll and Manson (1994) introduced a set of micromechanical formulations for the compressive response of assemblies of dry elastic particles (fibres). The range of equations introduced are based upon the theory that any static load applied to an assembly of particles (fibres) will be transferred across contact points between the fibres. The contact point between two fibres is defined as a point contact, unless the contacting fibres are very close to being parallel in which case it is defined as a line contact. In this way any assembly can be defined by a finite number of points interconnected by deformation units.

A deformation unit is used to describe the deformation mechanism of a particular fibre assembly. Parameters used in the model are the number of deformation units per unit volume and the height and compliance of the deformation unit as functions of the fibre volume fraction.

They described their fibres as approximately straight and non-parallel. To this end the deformation mechanism will be that of a fibre segment which is being bent between points of contact with other fibres. As the composite is further compressed, the fibre segment will encounter similar fibre segments and form new contact points and new, smaller fibre segments. This mechanism of deformation will happen at a greater rate with increasing values of fibre volume fraction and thus the rate of encounter between fibre segments will be proportional to the fibre volume fraction. As a short segment will be stiffer than a long one, and the number of segments increases at an ever increasing rate, the compressive force per unit area will be a non-linear function of the fibre volume fraction.
Toll and Manson (1994) began with the following general equation into which the relationship for the compression of the chosen defined deformation unit is substituted.

\[
\bar{p}_f = \int_0^{\frac{dV}{V_f}} \frac{\eta h^2}{V_f s} dV_f
\]  

(2.17)

Where \( \bar{p}_f \) is the vertical force per unit area, \( \eta \) is the number of deformation units per unit volume, \( h \) is the height of the deformation unit, \( V_f \) is the fibre volume fraction and \( s \) is the compliance of a deformation unit. Only elastic deformation is accounted for, thus the compression process is considered fully reversible.

Toll defines his model for a deformation unit for the case of contact points formed by fibre-fibre crossover, as a fibre segment supported by two other fibres and loaded by a third somewhere between the two supports. He uses previously established assumptions to define a deformation unit for a 3-dimensional (3D) wad as a loose wad of wool under compression [Van Wyk (1946)] and a deformation unit for a planar fibre network as a planar mat of dispersed, non-bundled straight fibres. Equations (2.18) and (2.19) are for 3D wads and Equations (2.20) and (2.21) are for planar fibre networks.

\[
\bar{h} = \bar{x}
\]  

(2.18)

\[
\bar{L} = 2\bar{x}
\]  

(2.19)

\[
\bar{h} = d
\]  

(2.20)

\[
\bar{L} = 2\bar{x}
\]  

(2.21)

Where \( \bar{L} \) and \( \bar{x} \) are the mean free length of a fibre deformation unit and the mean contact point spacing along the fibre respectively. By substituting these relationships into Equation (2.17) and integrating, a power law equation is established for the two cases. The final equations of Toll and Manson were formulated as follows:
For 3D wads:

$$\bar{p}_f = cEV_f^3$$  \hspace{1cm} (2.22)

and for planar fibre networks:

$$\bar{p}_f = cEV_f^5$$  \hspace{1cm} (2.23)

The factor $c$ is the only adjustable parameter and accounts for the crimp density, orientation and the unknown geometry and constraints on the fibre segments. Resulting values from these relationships were compared with experimental data and were shown to be in good agreement. A deformation unit was then suggested for fibre bundles but was made difficult by the uncertain nature of the contact geometry. In conclusion, when fibres were planarly oriented the resulting exponent was $b=5$ and when the fibres were oriented in all directions the resulting exponent is $b=3$. When fibres were nearly parallel the free segment length decreased faster than the contact point spacing, and the exponent $b$ could assume any positive value in excess of 3, which is what Toll suggested for fibre bundles.

Experimental data of pressure versus fibre volume fraction for weaves by Gauvin and Chibani (1988), Quinn (1990) and Kim et al (1991) were also fitted to the power law with $b=7$, 9 and 11 respectively (see Figure 2.8). Pearce and Summerscales (1995) fitted their experimental data to $b=5$ for a single layer of plain woven fabric and $b=7-8$ for assemblies of up to five layers of the same material. In the same study Pearce and Summerscales subjected assemblies of dry woven reinforcement to a series of loading cycles. They reported that at the end of loading a certain amount of pressure relaxation occurred. They concluded that this pressure relaxation was due to the internal translation and rearrangement of fibres within the reinforcement rather than a global expansion of the assembly.

Compressibility measurements have also been carried out by Trevino et al (1991) on several different types of glass fibre mats. They also found that the fibre reinforcement had a viscoelastic compression behaviour. When the mats were compressed to a given thickness, the pressure required to maintain that thickness decayed logarithmically to a pressure as much as
40\% lower than the maximum pressure. Most of this pressure decay was found to occur in the first ten seconds after the maximum pressure had been reached.

![Log-log compression curves for mats and weaves indicating the values of the power law exponent $b$ [Toll and Manson (1994)].](image)

Carnaby and Pan (1989) produced a theoretical model for the prediction of the full hysteresis curve for the compression and recovery of random wool fibre assemblies. They incorporated a mechanism for fibre-fibre slip into the model to explain the hysteresis in deformation. In their model, compression of the fibre assembly causes bending energy to be stored in the deformed fibres. Once the external load is removed the release of the bending energy causes the fibres to recover. However, the stored bending energy is not enough to overcome the interference and frictional restraints at the contact point between fibres and so the fibres are prevented from reaching their original configuration. The assembly still contains some strain energy due to the bent fibres and is locked in, due to the frictional constraints. If the assembly is again compressed, the stress-strain curve will follow a new path, different from the original, resulting in higher fibre volume fractions. Comparisons between experimental and theoretical results were shown to be in good agreement.

Matsudaira and Qin (1993) also produced experimental data for the compression and recovery of fabrics. They split both the compression and recovery curve of the loading cycle as a function of deformation into three steps. The first and third step of the compression curve and
the first step of the recovery were fitted to linear relationships. The second step of the compression and recovery curves were fitted to exponential relationships. A regression constant in the first step of the compression curve was related to the bending of fibres on the surface of the fabric and also to the type of yarn structure, twisted or untwisted. One of the regression constants from the second step of the compression curve was related to the hardness in compression due to the effect of friction between fibre-fibre contact. Another regression constant in the third step of the compression curve was related to the material of the fibre and also used to explain the initial lateral modulus of the fibres. They also concluded that once the third step of the recovery curve had been reached there was no chance of instantaneous recovery taking place.

Another micromechanical model using the fibre contact approach was developed by Batch and Macosko (1988). They proposed a two-stage model for fibre compaction by dividing the compression of fibres into two regimes: Hookean and non-Hookean, the difference between them being the amount of fibre contact. Unlike the ‘buckling arch’ approach for fibre deformation used by other workers (including Van Wyk and Gutowski), Batch uses a ‘fibre contact’ model which decreases elasticity as fibres bend to touch each other between two stationary points. In the first regime, Hookean, which occurs at low fibre volume fractions, the compression obeys Hooke’s Law and Batch derives the relationship

\[ P = K_0 (V_f - V_0) \]  

(2.24)

where

\[ K_0 = \frac{3\pi E}{(L/d_f)^4 V_0} \]  

(2.25)

\(K_0\) (or \(K_n\)) is the fibre spring constant in the Hookean regime, where \(L\) is the distance between fibre contacts, \(d_f\) the fibre diameter and \(E\) is Young’s modulus. However, once the fibre bends enough to make contact with another fibre, the resistance to deformation increases due to the third point of contact and the model enters the non-Hookean regime. The spring constant, no longer in the Hookean regime, increases dramatically due to the increasing fibre-fibre contact. Batch therefore adjusts the spring constant to take into consideration the increasing fibre-fibre contact.
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contact

\[ K_{NH} = \frac{K_H}{1 - (m/m_\infty)} \]  \hspace{1cm} (2.26)

so that the spring constant in the non-Hookean regime, \( K_{NH} \), approaches infinity as \( m \) approaches \( m_\infty \). \( m \) is the length of the fibre contact which increases toward the ultimate contact length, \( m_\infty \), with increasing fibre deformation. Equation (2.26) is then substituted into Equation (2.24) in place of \( K_0 \), taking into account that \( K_H \) is equivalent to \( K_0 \), and results in the model for fibre compression in the non-Hookean regime.

\[ P = \frac{K_0}{1 - (m/m_\infty)}(V_f - V_0) \]  \hspace{1cm} (2.27)

The model was compared with published data of other workers for the compression of aligned and random fibres and for a woven fabric. Batch showed that values for \( L/d_f \) were approximately 50% lower for the random mats when compared to the aligned fabrics, which is to be expected due to the increased number of fibre-fibre contacts in random mats. However, values for \( L/d_f \) for well aligned and poorly aligned fibres showed no difference and \( L/d_f \) for the woven fabric was only 25% higher. Batch suggested that this may point to the roving microstructure having a greater influence on the deformation than the gross roving alignment. He finally concluded that the results justify the use of the Hookean model alone for the deformation of mats.

2.2.4 MICROSTRUCTURAL ANALYSIS OF COMPRESSED LAMINATES

Yugartis et al (1993) manufactured laminates reinforced with layers of plain woven fabric of high crimp and measured microstructural parameters from cross-sections. Measurements were made with the aid of in-house image analysis software and included inclination angles and crimp angles to describe the shape of the longitudinal yarns and angle match between inclination angles of adjacent yarns to establish the degree of yarn nesting (see Figure 2.9). A three-dimensional reconstruction of a yarn section from available measurements showed local irregularities and local distortions of the waveform. It was still possible, however, to construct sinusoidal fits through the data points for various yarns.

![Image of yarn with definitions of measures](image)

**Figure 2.9** Definitions of measures of yarn shape and nesting: inclination angle, crimp angle and angle match [Yugartis et al (1993)].

Yugartis (1995) continued the investigation of the plain weave laminate microstructure to measure possible microcrack structure. In a similar study Jortner (1992) noticed that across ply compaction during lamination of an assembly of resin impregnated plain woven plies did not significantly change the wavelengths of the yarns. However he noted that the laminate compression can alter crimp angles substantially which distorts the waveform and that nesting patterns are quite variable and complex. None of the studies made an attempt to measure the height, width or cross-sectional areas of the warp and weft yarns.

Simacek and Karbhari (1996) compressed an assembly of resin impregnated glass plain weave
plies from an uncompacted state to a maximum fibre volume fraction of 0.58. They measured a variety of mean geometric parameters of a unit cell based on the fabric architecture. These measurements of the laminate microstructure showed that the warp and weft yarns were compressed from 0.60 mm to 0.56 mm and from 0.68 mm to 0.44 mm respectively. They also showed experimental data that indicated that the width of the weft yarn increases whilst the width of the warp yarn decreases for increasing applied pressure. They suggested that the decrease in the width of the warp yarn was due to the nesting effects of the yarns. No description of the experimental methods for the compression or measurement of the geometric parameters were given and so the accuracy of the results cannot be determined.

2.3 POLYMER RHEOLOGY

The term rheology was invented by E.C. Bingham in 1929 and was originally defined as 'the deformation and flow of matter [Tanner (1985)]. This is a very broad field of which the study of polymeric fluids is only a small part. However if improvements are to continue to be made in the fields of both polymer and polymer composite processing then a firm understanding of polymer rheology is required.

There are times when some polymers exhibit similar behavioural characteristics to solids, at other times they are like liquids. Polymers in the rubbery region have simultaneously the attributes of both solids and liquids. In reality most polymers at some point will have the characteristics of both solids and liquids and are often termed viscoelastic. However, there is a common factor between solids, liquids and all the materials that lay between these two extremes; if a force or stress is applied to any of the materials they will deform or strain. This deformation make take place instantaneously or over a period of time. When the relationships between stress, strain and time are expressed mathematically they are known as rheological equations of state (r.e.s) [Brydson (1981)]. Most models represent idealisations of actual material behaviour. Whorlow (1992) stressed that real materials do not necessarily fit the behavioural patterns indicated by these models. The idealisation is sometimes valid to a high degree of accuracy for a certain stress range but other times it may represent only an approximation of the true behaviour. It is important to recognise the limitations of the r.e.s but irrespective of the type of fit, they provide an insight about the effects of stress on the material.
2.3.1 NEWTONIAN FLUIDS

The stress-deformation behaviour of a Newtonian fluid is best described by considering the model in Figure 2.10. Two parallel plates of area \( A \) are separated at a distance \( r \) by the Newtonian fluid. \( r \) is very small in comparison to the length and width of the plates. A shear force \( F \) is applied to the top plate which moves with a uniform velocity \( v \) whilst the bottom plate remains stationary. The arrows between the plates indicate the velocity of the various layers of fluid relative to the top and bottom plate velocities. The shear stress \( \tau \) is then \( F/A \) and the rate of shear or shear rate \( \dot{\gamma} \) is \( dv/dr \).

\[ \tau = \mu \left( \frac{dv}{dr} \right) \quad (2.28) \]

\[ \therefore \tau = \mu \dot{\gamma} \quad (2.29) \]

where \( \mu \) is the constant of proportionality and is called viscosity. Viscosity has been defined as ‘the resistance of a material to irreversible positional change of its constituent volume elements and the concomitant conversion of mechanical energy into heat’ [Lenk (1968)]. However a more common definition for viscosity is ‘a measure of the resistance to flow that a fluid offers when it is subjected to shear stress’. For a Newtonian fluid the viscosity is constant and this

![Figure 2.10 Shear deformation of a Newtonian Fluid. Shear stress \( \tau = F/A \) and the shear rate \( \dot{\gamma} = dv/dr \).](image-url)
can be illustrated by examining the flow curve for a Newtonian fluid, a graph of shear stress against strain rate. As can be seen in Figure 2.11 the flow curve is a straight line through the origin, the slope being equal to the viscosity. As previously mentioned, the viscosity of a Newtonian fluid remains constant irrespective of the shear stress. This is perhaps more easily illustrated by examining the graph of viscosity against shear rate (see Figure 2.12). Examples of typical Newtonian fluids, within experimental accuracy, are water, most aqueous solutions, organic liquids and liquid metals.

2.3.2 NON-NEWTONIAN FLUIDS

Many real materials, especially polymer melts and solutions, do not exhibit the simple characteristics of Newtonian fluids. This ‘non-Newtonian’ behaviour means the relationship between shear stress and shear rate is no longer linear and that the viscosity of a non-Newtonian fluid is not constant for a given temperature and pressure but depends on the rate of shear. There are three broad categories of non-linear fluids:

(a) *Time-independent fluids*: fluids for which the shear rate at any point is some function of the shear stress at that point and depends on nothing else.

(b) *Time-dependent fluids*: more complex systems for which the relation between shear stress and shear rate depends on the time the fluid has been sheared and on its previous history.

(c) *Viscoelastic fluids*: systems which have the characteristics of both solids and fluids and exhibit partial elastic recovery after deformation.

For the purpose of this thesis only the first category of fluids needs examining.

2.3.2.1 Time-independent, non-Newtonian Fluids

Fluids whose properties are independent of time may be described mathematically by the rheological equation
\[ \dot{\gamma} = f(\tau) \]  

(2.30)

which implies that the shear rate at any point in the fluid is a function of the shear stress at that point. Depending on the nature of the function in Equation (2.30), time-independent fluids can be divided into three distinct types:

(a) Bingham plastics
(b) pseudoplastic fluids
(c) dilatant fluids.

Typical flow curves for these three fluids are shown in Figure 2.11 along with the typical linear relation of Newtonian fluids.

![Figure 2.11 Shear stress (\(\tau\)) versus shear rate (\(\dot{\gamma}\)) flow curves for Bingham body, dilatant fluids and pseudoplastic fluids compared to a Newtonian fluid.](image)

The Bingham model represents a solid, not a fluid, and is only used when flow occurs. The flow curve (see Figure 2.11) is a straight line with an intercept \(\tau_y\) on the stress axis. \(\tau_y\) is the yield stress and is the amount of stress that must be exceeded before flow begins. The slope of the flow curve is defined as the plastic viscosity, \(\mu_p\). Thus the material behaves as an elastic solid for stresses less than the yield stress and for greater stresses.
2. Literature Review

\[ \dot{\gamma} = \frac{1}{\mu_p} \left( \tau - \tau_y \right) \quad \text{when} \quad \tau \geq \tau_y \quad (2.31) \]

Although \( \mu_p \) is known as the plastic viscosity, the Bingham model rarely represents plastics, either solid or molten polymers. However it is convenient in practice because some fluids such as clay slurries, oil paints, greases and toothpaste approximate the type of behaviour closely enough. A simplified explanation of Bingham plastic behaviour is that the fluid at rest has a sufficiently rigid three-dimensional structure to withstand any stress less than the yield stress. If this stress is exceeded the structure collapses and the fluid behaves as a Newtonian fluid under a shear stress \( \tau - \tau_y \). When the shear stress returns below the yield stress the structure reforms.

\textit{Pseudoplastic fluids} show no yield value and there is no constant of proportionality between shear stress and shear rate. Therefore instead of the term viscosity, which is used for Newtonian fluids, the ratio of shear stress to shear rate is known as the \textit{apparent viscosity}, \( \mu_a \).

A typical flow curve (see Figure 2.11) shows that the ratio of shear stress to the shear rate, the apparent viscosity, falls progressively with shear rate. One simplified explanation for the pseudoplastic behaviour of a material is that with increasing shear rate the molecules of the structure become more aligned. At rest, the molecules are in a completely random and entangled state but with the increasing shear rate the molecules become aligned along their major axes in the direction of flow and the viscosity decreases. Polymer melts and solutions are usually pseudoplastic fluids [Middleman (1968)].

Many equations have been proposed to describe the behaviour of a pseudoplastic fluid but the most widely used relationship is the power law, sometimes referred to as the Ostwald-de Waele equation [McKelvey (1962)].

\[ \tau = C (\dot{\gamma})^n \quad (2.32) \]

\( C \) and \( n \) are constants where \( C \) is a measure of the consistency of the fluid, the higher the value for \( C \) the more viscous the fluid; \( n \) is the power law flow index of the fluid and is a measure of the degree of non-Newtonian behaviour. Thus the greater the deviation of \( n \) from unity the
more pronounced the non-Newtonian behaviour of the fluid.

The apparent viscosity can also be expressed in terms of the two power law constants $C$ and $n$. By definition

$$\mu_a = \frac{X}{\gamma} \quad (2.33)$$

so combining Equations (2.32) and (2.33) gives

$$\mu_a = C(\gamma)^{n-1} \quad (2.34)$$

By plotting curves of apparent viscosity versus shear rate (see Figure 2.12) one can easily illustrate the dependence of apparent viscosity on the shear rate. When $n$ is less than unity the viscosity of the fluid decreases with increasing shear rate. Hence for a power law fluid when $n<1$ the fluid is pseudoplastic and when $n>1$ the fluid is dilatant. When $n=1$ the power law, Equation (2.32), reverts back to the model for a Newtonian fluid (see Equation (2.29)).

![Figure 2.12 Apparent viscosity-shear rate curves for a dilatant fluid ($n>1$), a Newtonian fluid ($n=1$) and a pseudoplastic fluid ($n<1$).](image)
Dilatant fluids behave in the opposite manner to pseudoplastic fluids. They are similar in that they have no yield stress but the viscosity of a dilatant fluid increases with increasing shear rate (see Figure 2.11 and 2.12). The power law, Equation (2.32) is still applicable but the flow index $n$ is larger than unity. Concentrated suspensions of solids have been used to describe this type of behaviour. When the concentrated suspensions are at rest the solids are at their maximum packing capacity and there is just enough liquid to occupy all the interstices or voids. At low rates of shear the liquid acts as a lubricant for the moving particles and as a result the stresses are low. When the shear rate is increased the particles become less densely packed and the material expands or 'dilates' increasing the number of interstices. As there is now insufficient liquid to fill all the interstices, the liquid can no longer act as a lubricant for the flow of particles and so the applied stresses need to be much larger. Hence the increase in the volume of this structure causes the viscosity to increase rapidly with increasing rates of shear.

2.3.3 EFFECTS OF TEMPERATURE AND CURING ON VISCOSITY OF POLYMER RESINS

Choosing a suitable cure cycle for the processing of polymer composites is vital to the cost effective manufacture of high quality composite parts. The curing cycle affects the final mechanical and physical properties of the composite and should thus be selected accordingly [Loos and Springer (1983)]. Important parameters for consideration include, temperature, time at temperature, pressure, dwell times and heating and cooling rates.

The viscosity of the resin is mainly dependent on temperature and the degree of curing. To minimise cycle times an optimal flow rate is desired [Lee and Um (1993)] and in most cases concerning polyesters, epoxies and phenolics, the aim is to ensure resin flows at the minimum value of viscosity (see Figure 2.13) so that consolidation and suitable void removal occurs before significant curing has taken place [Frank-Susich (1993)].

Figure 2.13 shows a typical resin curing cycle where the viscosity decreases with increasing temperature until curing begins whereby the viscosity increases.
The non-Newtonian character of the resin is often described as a power-law as follows [Lekakou and Richardson (1988)]:

\[ \mu = \mu_0(T, X) |\dot{\gamma}|^n \]  

(2.35)

where \( T \) is the temperature, \( X \) is the degree of cure as fractional conversion, \( \dot{\gamma} \) is the shear rate and \( n \) is the power-law index. The function \( \mu_0(T, X) \) can be decomposed into temperature, \( \mu_0(T) \), and conversion, \( \mu_0(X) \), dependent terms:

\[ \mu_0(T, X) = \mu_0(T) \mu_0(X) \]  

(2.36)

An exponential temperature dependence of viscosity exists and takes the form:

\[ \mu_0(T) = A_n \exp \left( \frac{E_v}{RT} \right) \]  

(2.37)

where \( A_n \) is a constant, \( E_v \) is the viscosity activation energy and \( R \) is the universal gas constant. \( \mu_0(X) \) can be expressed as [Castro and Macasko (1982)]:

Figure 2.13 Resin cure cycle showing the change in viscosity as a function of time.
2. Literature Review

\[ \mu_0 x = \left[ \frac{X_G}{X_G - X} \right]^{-A_1 + B_1 X} \tag{2.38} \]

where \( X_G \) is the conversion at the gelling point and \( A_1 \) and \( B_1 \) are constants. Equation (2.38) is suitable for polyesters, epoxies and phenolics.
3. EXPERIMENTAL MATERIALS
AND PROCEDURES
3.1 INTRODUCTION

Initially this chapter describes the materials used throughout this study, including systems of both reinforcement and matrix and also characterisation studies. The chapter then continues with the experimental procedures dealing with the construction and processing of polymer matrix glass laminates and the consequent compression testing. Laminates have been constructed using three different kinds of woven glass fabric, a noncrimped stitch-bonded glass fabric and three different resin systems. Compression experiments have been carried out on assemblies of both dry and resin impregnated reinforcement. The effect of varying the number of plies in an assembly, changes in ply orientation, repeated compression of the same reinforcement, varying the maximum target loads applied to the laminates and changes in the speed of compression have all been explored. The experimental work then goes further to investigate changes in the microstructure of the laminate as it undergoes different types of deformation under varying compression. Hence, the image analysis procedures and measuring techniques employed in the examination of the laminate's microstructure are presented and discussed in this chapter.

3.2 MATERIALS

A variety of laminates were produced using different types of reinforcement and several resins. Three of the fabrics were of woven glass and the final type of reinforcement used was a noncrimped stitch-bonded fabric. The resins used consisted of two different types of polyester and an epoxy. A complete description of firstly the fabrics and secondly the resins is given below.

3.2.1 FABRICS AND FABRIC CHARACTERISATION

The three different types of woven fabrics used for the construction of the polymer matrix composites were plain, twill and 5 harness satin. These were purchased from Fothergill Engineered Fabrics and all the specifications (see Table 3.1) have been taken from Fothergill's glass fabrics technical data sheet [Fothergill Engineered Fabrics (1997)]. However sections of
the fabrics were weighed and the diameter of the fibres were measured using optical microscopy to confirm the accuracy of the data. The noncrimped stitch-bonded glass fabric is not a weave, but four unidirectional layers of aligned rovings with a 0°, 45°, -45°, 90° orientation. The four layers are bonded together by cross stitching with a very light yarn to form a single ply.

### Table 3.1 Fabric specifications of the three woven fabrics used in the construction of polymer matrix composites.

<table>
<thead>
<tr>
<th>Weave type</th>
<th>Code Number</th>
<th>Material</th>
<th>Warp Yarn (tex)</th>
<th>Weft Yarn (tex)</th>
<th>Ends/10 mm</th>
<th>Picks/10 mm</th>
<th>Thickness’</th>
<th>Areal Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plain</td>
<td>Y0212</td>
<td>E-glass</td>
<td>136g x 3 EC9</td>
<td>136g x 3 EC9</td>
<td>6.7</td>
<td>6.3</td>
<td>0.48 mm</td>
<td>0.546 kg/m²</td>
</tr>
<tr>
<td>Twill</td>
<td>Y0185</td>
<td>E-glass</td>
<td>68g x 2 EC9</td>
<td>68g x 2 EC9</td>
<td>11.8</td>
<td>11.8</td>
<td>0.28 mm</td>
<td>0.331 kg/m²</td>
</tr>
<tr>
<td>5 harness satin</td>
<td>Y0227</td>
<td>E-glass</td>
<td>22g x 3 EC7</td>
<td>22g x 2 EC7</td>
<td>22.4</td>
<td>21.3</td>
<td>0.23 mm</td>
<td>0.297 kg/m²</td>
</tr>
</tbody>
</table>

* Nominal thickness values from Fothergill Engineered Fabrics Technical Data

### 3.2.1.1 Plain Weave

The plain weave fabric, Y0212, has identical warp and weft yarns consisting of three bundles twisted together which are made from continuous E-glass fibres 9 µm in diameter. Both yarns have the same tex of 136 g. The amount of ends and picks per cm are so close that the cloth can be considered as virtually balanced.

### 3.2.1.2 Twill Weave

This fabric is also made from continuous E-glass fibres 9 µm in diameter. Again the warp and weft yarns are identical but in this case both yarns only consist of two bundles twisted together. The yarn tex is half the weight of the plain weave yarns at 68 g and the approximate
3. Experimental Materials and Procedures

3.2.1.3 5 Harness Satin

Of the three fabrics this has the finest yarns consisting of three bundles twisted together with a fabric thickness of only 0.23 mm. The continuous E-glass fibres have a smaller diameter of 7 μm and the yarn tex values are much smaller at 22 g. The yarns are very densely packed together with 22.4 ends per cm and 21.3 picks per cm indicating a slightly unbalanced fabric.

3.2.1.4 Noncrimped Stitch-bonded

As mentioned previously the noncrimped stitch-bonded (NCSB) glass fabric used in this study comprises four unidirectional layers of aligned rovings, made from continuous E-glass, with a 0°, 45°, -45°, 90° orientation. The areal weight is unsurprisingly higher than that of the wovens, 0.853 kig/m², due to the fact that each layer of the NCSB has four unidirectional layers. The cross-section of the unidirectional rovings can be described as elliptical where the major axis value varies for each of the four plies due to the type of cross stitch. The 0° layer has the largest major axis at 3.0 mm and the highest tex of 1243 g. The 90° layer has an intermediate value for the major axis of approximately 1.5 mm and a tex value of 482 g. Finally the ± 45° layers have the smallest values for the major axis and tex with approximately 1.2 mm and 290 g respectively. The number of ends for each of the layers is thus 2.5 per cm, 5.2 per cm and 7.0 per cm for the 0°, 90° and ± 45° respectively.

3.2.2 RESINS AND RESIN CHARACTERISATION

The resins employed in this study included two different cold curing polyester systems and a hot curing epoxy system. Regarding the two types of polyester that were used in this study, the first was suitable for general purpose applications and the second was specific to RTM. In attempts to find a resin more able to impregnate the woven glass fabrics used in the study a
hot curing matrix system was used. A commercial epoxy resin was chosen that had been tried and tested successfully in the RTM process [Thirion et al (1988)].

3.2.2.1 Polyesters

The first resin used for the compression experiments was Crystic 471 PALV unsaturated polyester, preaccelerated with cobalt napthanate from the Scott Bader Company Ltd. The addition of a preaccelerator to the polymer allows the curing of the resin to take place at room temperature. The monomer styrene is also present (39-43% by weight) in the dual role of solvent and curing agent. It enables the resin solution to cure from the liquid state into a solid by the formation of styrene crosslinks between the polyester chains. Curing begins by adding an initiator to the solution which produces the free radicals that initiate the copolymerisation of the styrene with the unsaturated polyester. The initiator used, also from Scott Bader, was 'Catalyst M', an organic peroxide, methyl ethyl ketone peroxide (MEKP). Preliminary experiments of curing in bulk were carried out for all resin systems, including variations in composition. Table 3.2a shows the approximate time to gel for the Crystic 471 resin system for various amounts of initiator (these are nominal values determined from experimentation).

<table>
<thead>
<tr>
<th>Table 3.2 Curing times with different concentrations of initiator for:</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Crystic 471 PALV  b. Crystic 781 PA at 25 °C.</td>
</tr>
<tr>
<td>a. Crystic 471</td>
</tr>
<tr>
<td>50 g</td>
</tr>
<tr>
<td>50 g</td>
</tr>
<tr>
<td>50 g</td>
</tr>
<tr>
<td>50 g</td>
</tr>
<tr>
<td>50 g</td>
</tr>
<tr>
<td>50 g</td>
</tr>
<tr>
<td>50 g</td>
</tr>
<tr>
<td>50 g</td>
</tr>
</tbody>
</table>

Whereas the first polyester, Crystic 471 PALV, was for general purpose processing and applications the second, Crystic 781 PA also from Scott Bader Company Ltd, was specifically
for use in resin transfer moulding. It is also an unsaturated polyester resin in styrene solution and preaccelerated with cobalt naphthanate. The same initiator, 'Catalyst M', was also used to begin curing. The main difference between the two polyester resins was that the Crystic 781 had a lower viscosity, 200-300 mPa s at 25 °C. This makes impregnation of dry reinforcement easier in processes such as RTM. As can be seen from Table 3.2b the times to gel for the Crystic 781 are much longer than that for Crystic 471. The lower viscosity coupled with the reduction in the viscosity build-up made the resin impregnation of the reinforcement much quicker during the lay-up stage of the laminates in this study.

3.2.2.2 Epoxy

The epoxy used was Araldite LY 564 with Hardener HY 2954 from Ciba Polymers. It is a hot curing epoxy matrix system based on a bisphenol A epoxy resin with a reactive diluent and a cycloaliphatic amine hardener. It is suitable for various production processes to produce high performance composite parts. Its relatively low initial mix viscosity, see Table 3.3, means that it is suitable for the hand lay-up procedure used for the compression testing in this study (see section 3.4 Laminate Construction). As the temperature is raised the resin viscosity falls, allowing this epoxy to be used for RTM.

Table 3.3 Initial mix viscosities for Araldite LY 564 with Hardener HY 2954 [Ciba Polymers data sheet (1994)].

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Initial Mix Viscosity (mPa s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>500-700</td>
</tr>
<tr>
<td>40</td>
<td>200-300</td>
</tr>
<tr>
<td>60</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 3.4 shows approximate times to gel for small batches of the epoxy system (approximately 50 g) for different concentrations of hardener at two different temperatures. The recommended mix ratio of Araldite LY 564 to Hardener HY 2954 is 100:35 parts by weight (pbw) [Ciba Polymers instruction sheet (1994)].
### Table 3.4 Curing cycle for Araldite LY 564 with various concentrations of Hardener HY 2954 at two different temperatures.

<table>
<thead>
<tr>
<th>Araldite LY 564 (pbw)</th>
<th>Hardener HY 2954 (pbw)</th>
<th>Temperature (°C)</th>
<th>Gel Time (mins)</th>
<th>Cure Time (mins)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>35</td>
<td>50</td>
<td>38-40</td>
<td>60-65</td>
</tr>
<tr>
<td>100</td>
<td>40</td>
<td>50</td>
<td>36-38</td>
<td>60-65</td>
</tr>
<tr>
<td>100</td>
<td>45</td>
<td>50</td>
<td>34-36</td>
<td>55-60</td>
</tr>
<tr>
<td>100</td>
<td>35</td>
<td>70</td>
<td>15-17</td>
<td>30-35</td>
</tr>
<tr>
<td>100</td>
<td>40</td>
<td>70</td>
<td>15.5-16</td>
<td>30-35</td>
</tr>
<tr>
<td>100</td>
<td>45</td>
<td>70</td>
<td>15-15.5</td>
<td>25-30</td>
</tr>
</tbody>
</table>

### 3.3 RHEOMETRY OF RESINS

The viscosity of resin is the typical rheological property used in the studies of infiltration of fibrous media. The two most common types of instrumentation for studying the flow properties of polymer melts are rotational viscometers and capillary viscometers [Brydson (1981)]. For both types of viscometer there are many variants on the basic method. In the current investigations a Brookfield Synchro-Lectric Viscometer was used which is of the rotational type (see Figure 3.7). In this basic type of rotational viscometer the fluid is placed in a beaker and is sheared by a solid concentric cylinder or spindle. The shear rate is generally proportional to the speed of rotation (see Table 3.5). By measuring the torque required for rotation, the viscosity of the fluid can be determined under shear conditions.

For the accurate measurement of viscosity in a Brookfield viscometer, three prerequisites must be followed [Brookfield Viscometer Handbook]:

(a) The viscometer should be perfectly level while in use.

(b) When taking a reading from the viscometer the spindle should be perfectly centred within the test sample container. The spindle should also be immersed in the fluid exactly to the line marking on the spindle shaft.
(c) The viscometer has been calibrated for the immersion of a spindle in a 600 ml glass beaker with an internal diameter of approximately 83 mm (3.25 inches). Increasing or decreasing the diameter of the beaker will result in different viscosity ranges.

Table 3.5 Shear rate, shear stress and viscosity for several viscometers in steady flow [Grulke (1994)].

<table>
<thead>
<tr>
<th>Viscometer</th>
<th>Shear Rate ($\gamma$)</th>
<th>Shear Stress ($\tau$)</th>
<th>Viscosity ($\mu$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Narrow gap concentric cylinder</td>
<td>$\frac{r_0 \Omega}{r_0 - r}$</td>
<td>$\frac{T}{2 \pi r_0^2 L}$</td>
<td>$\frac{T(r_0 - r_1)}{2 \pi r_0^3 \Omega_1 L}$</td>
</tr>
<tr>
<td>Wide-gap concentric cylinder</td>
<td>$\frac{2 \Omega}{n(1 - B^{2n})}$</td>
<td>$\frac{T}{2 \pi r_1^2 L}$</td>
<td>$\frac{Tn(1 - B^{2n})}{4 \pi r_1^2 \Omega_1 L}$</td>
</tr>
<tr>
<td>Rotating cylinder in large volume</td>
<td>$\frac{2 \Omega_1}{n}$</td>
<td>$\frac{T}{2 \pi r_1^2 L}$</td>
<td>$\frac{Tn}{4 \pi r_1^2 \Omega_1 L}$</td>
</tr>
</tbody>
</table>

$r_0 =$ beaker radius, $r_1 =$ cylinder radius, $r_1 < r < r_0$, $B = r/r_0$, $L =$ length of cylinder, $\Omega =$ rotation speed, $\Omega_1 =$ angular velocity, $n =$ power law exponent, $T =$ torque

3.3.1 POLYESTER

The apparatus was set up as shown in Figure 3.1, ensuring that the three conditions for accurate readings of the equipment had been followed. Tests were carried out on Crystic 471 PALV with three different amounts of the initiator ‘Catalyst M’, 0.28 %, 0.32 % and 0.40 % by weight (bw). Viscosity readings were taken for a range of speeds between 0.5 and 50 rpm at 10 minute intervals. Readings continued to be taken until the resin had reached its gelation stage at which point the equipment was cleaned and the test repeated with a different concentration of initiator.

The results were plotted on log-log graphs of viscosity against frequency of rotation. The power law equation is used to represent the relation between viscosity and shear rate

$$\mu = C(\gamma)^{n-1} \quad (3.1)$$
where $\mu_a$ is the apparent viscosity, $\dot{\gamma}$ is the shear rate, $C$ is the consistency and $n$ is the power law constant. As is illustrated in Table 3.5 the shear rate in rotational viscometers is generally proportional to the frequency of rotation. Information was obtained on the rheology of the polyester resin at different concentration levels of initiator. The nature of the resin, whether Newtonian, pseudoplastic or dilatant was determined from the slope of the curve of viscosity versus speed of rotation. What was also apparent from the curves was the viscosity build-up of the resin due to the onset of gelation.

![Diagram of rheology analysis apparatus](image)

Figure 3.1 Schematic of the apparatus used for the rheology analysis of both the Crystic 471 PALV and the Araldite LY564 resin systems.

### 3.3.2 EPOXY

The viscosity test was performed for the system of Araldite LY564 resin mixed with Hardener HY 2954 characterised in section 3.2.2.2. The mix ratio of araldite to hardener was 100:35 parts by weight (pbw). As the epoxy curing cycle is dependent on temperature, viscosity tests were carried out on the same mix ratios of epoxy to hardener but at different temperatures, 20, 30, 40 and 50 °C. Thus the apparatus for the testing of the epoxy system was set up in the same way as for the polyester but this time with the addition of a water bath. The resin and hardener were preheated separately to the required temperature before mixing. In the
meanwhile the water within the tank was heated to the same temperature as that of the resin and thereafter kept constant by a heating element in the tank. Once the resin and hardener had been mixed the beaker containing the resin was partially submerged in the water and the viscosity tests were carried out.

Viscosity readings were taken for a range of frequencies of rotation between 0.5 and 100 rpm again at 10 minute intervals. Log-log graphs were plotted of viscosity against frequency of rotation so the viscosity build-up at different curing temperatures could be compared and the nature of the resin determined.

### 3.3.3 RESULTS OF VISCOMETRY

#### 3.3.3.1 Polyester - Crystic 471

From the slope of the curves (see Figures 3.2 to 3.4) one can instantly realise the pseudoplastic nature of the polyester resin. This was further established by performing regression analysis on the curves to calculate the power law index $n$. If the value for $n$ is less than 1 the polyester is considered pseudoplastic. Exponent $n$ values were calculated for the resin's initial mix viscosity and found to be 0.49, 0.51 and 0.48 for Crystic 471 with 0.28, 0.32 and 0.40 % bw initiator respectively (see Table 3.6). It can be concluded from Table 3.6 that $n=0.5$ approximately for all concentrations of initiator up to the gel time.

As can be seen from both the curves (see Figures 3.2 to 3.4) and the viscosity values in Table 3.6 obtained from the flow experiments, the time to gel increases with decreasing amounts of initiator as one would expect, approximately 40, 60 and 70 minutes for concentrations of 0.40, 0.32 and 0.28 % bw initiator respectively. What can also be determined from the assembled data is that the viscosity build-up is relatively sudden. For example the Crystic 471 with an initiator concentration of 0.32 % bw had a constant viscosity of 6000 mPa s at 0.5 rpm up to 50 minutes into the curing cycle. Then at 60 minutes the viscosity had rapidly increased to 6600 mPa s and timing was stopped at 65 minutes when the viscosity readings went off scale as gelation occurred.
Table 3.6 Values for the apparent viscosity, $\mu_a$, at $\Omega=1$rpm and power law constant $n$ taken at intervals during the curing cycle of Crystic 471 with different concentrations of initiator.

<table>
<thead>
<tr>
<th>Time (mins)</th>
<th>0.28% bw initiator</th>
<th>0.32% bw initiator</th>
<th>0.40% bw initiator</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\mu_a$ (mPa s)</td>
<td>$n$</td>
<td>$\mu_a$ (mPa s)</td>
</tr>
<tr>
<td>10</td>
<td>4000</td>
<td>0.49</td>
<td>4000</td>
</tr>
<tr>
<td>20</td>
<td>3900</td>
<td>0.51</td>
<td>3800</td>
</tr>
<tr>
<td>30</td>
<td>3900</td>
<td>0.51</td>
<td>3800</td>
</tr>
<tr>
<td>40</td>
<td>4000</td>
<td>0.50</td>
<td>3800</td>
</tr>
<tr>
<td>50</td>
<td>4000</td>
<td>0.51</td>
<td>3800</td>
</tr>
<tr>
<td>60</td>
<td>4000</td>
<td>0.51</td>
<td>4600</td>
</tr>
<tr>
<td>70</td>
<td>4700</td>
<td>0.57</td>
<td></td>
</tr>
</tbody>
</table>

= no measurement possible due to the onset of gelation

Figure 3.2 Plots of viscosity versus frequency of rotation taken at 10 minute intervals during the curing cycle of Crystic 471 with 0.28% bw initiator.
3. Experimental Materials and Procedures

Figure 3.3 Plots of viscosity versus frequency of rotation taken at 10 minute intervals during the curing cycle of Crystic 471 with 0.32% bw initiator.

Figure 3.4 Plots of viscosity versus frequency of rotation taken at 10 minute intervals during the curing cycle of Crystic 471 with 0.40% bw initiator.
3.3.3.2 Epoxy - Araldite LY 564

From the viscosity data in the plots of viscosity versus frequency of rotation of Figures 3.5 to 3.8, one can see no real change in the viscosity with increasing frequency of rotation. This indicates a Newtonian polymer which is further confirmed from the regression analysis of the plots (see Table 3.7).

Table 3.7 Values for the apparent viscosity, \( \mu_a \), at \( \Omega=1 \text{rpm} \) and the power law constant \( n \) taken at intervals during the curing cycle of Araldite LY 564 at different temperatures.

<table>
<thead>
<tr>
<th>Time (mins)</th>
<th>20 °C</th>
<th>30 °C</th>
<th>40 °C</th>
<th>50 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \mu_a ) (mPa s)</td>
<td>( n )</td>
<td>( \mu_a ) (mPa s)</td>
<td>( n )</td>
</tr>
<tr>
<td>10</td>
<td>1000</td>
<td>1.00</td>
<td>600</td>
<td>1.03</td>
</tr>
<tr>
<td>20</td>
<td>1200</td>
<td>0.98</td>
<td>400</td>
<td>1.05</td>
</tr>
<tr>
<td>30</td>
<td>1200</td>
<td>0.98</td>
<td>400</td>
<td>1.05</td>
</tr>
<tr>
<td>40</td>
<td>1200</td>
<td>1.00</td>
<td>400</td>
<td>1.04</td>
</tr>
<tr>
<td>50</td>
<td>1200</td>
<td>1.01</td>
<td>400</td>
<td>1.06</td>
</tr>
<tr>
<td>60</td>
<td>1200</td>
<td>1.02</td>
<td>400</td>
<td>1.06</td>
</tr>
<tr>
<td>70</td>
<td>1300</td>
<td>1.02</td>
<td>500</td>
<td>1.05</td>
</tr>
<tr>
<td>80</td>
<td>1400</td>
<td>1.02</td>
<td>600</td>
<td>1.05</td>
</tr>
<tr>
<td>90</td>
<td>1400</td>
<td>1.04</td>
<td>600</td>
<td>1.07</td>
</tr>
</tbody>
</table>

= no measurement possible due to the onset of gelation

From the initial mix viscosity of the epoxy at 20, 30, 40 and 50 °C the values for the power law exponent \( n \) were found to be 1.00, 1.03, 1.03 and 1.04 respectively, where \( n=1 \) for a Newtonian fluid. The small variations in the value of the power law exponent \( n \) from unity are due to the increase of the apparent viscosity with increasing cure during the time taken to vary the rotation speed and take the measurements at each time interval and not due to any nonNewtonian characteristic of the epoxy resin. The initial mix viscosity values obtained from the testing compare very well to the product data sheets supplied by Ciba Polymers [Ciba Polymers Instruction Sheet (1994)]. The viscosity data in the graphs illustrate well the viscosity build-up during the curing cycle and also the decrease in the initial mix viscosity with increasing temperature.
3. Experimental Materials and Procedures

**Figure 3.5** Plots of viscosity versus frequency of rotation taken at 10 minute intervals during the curing cycle of Araldite LY 564 at 20 °C.

**Figure 3.6** Plots of viscosity versus frequency of rotation taken at 10 minute intervals during the curing cycle of Araldite LY 564 at 30 °C.
Figure 3.7 Plots of viscosity versus frequency of rotation taken at 10 minute intervals during the curing cycle of Araldite LY 564 at 40 °C.

Figure 3.8 Plots of viscosity versus frequency of rotation taken at 10 minute intervals during the curing cycle of Araldite LY 564 at 50 °C.
3.4 LAMINATE CONSTRUCTION

The initial set of laminates were constructed to try and establish a feel for the hand lay-up process. It was a useful procedure for estimating the time to impregnate different layers of the reinforcement, to ascertain whether the fibres were being suitably wetted out, and for confirming the curing times for the resin used.

A square aluminium template, 100 mm x 100 mm, was cut to speed up the process of cutting out many square sections of the glass fabric. Once the fabric had been cut it was noticed that the edges frayed quite considerably, approximately 20 mm toward the centre of the square section. This was not of immediate concern as the final sample would be cut from the centre of the laminate, an area unaffected by the fraying of the fabric.

A square glass plate (170 mm x 170 mm x 4 mm) was placed on a flat surface and covered with a single sheet of a silicone coated polyester release film (pink Melinex). The release film was approximately 200 mm x 200 mm to prevent any over spill of resin during the processing coming into contact with the glass. A generous amount of the mixed resin (polyester and initiator) was then brushed onto an area of the plate with a half inch soft bristled brush. The first layer of glass fabric was then carefully placed on top ensuring that any loose yarns due to fraying had been removed. The fabric was then encouraged to wet out by gentle pressure of the brush. Once the fabric was completely wetted the procedure was repeated until all the layers of the glass reinforcement had been laid up. A second layer of release film was then placed on top of the assembly of plies and a glass plate on top of that. A known load of approximately 25 kg was then applied to compress the laminate, and the whole apparatus left to cure in a vented cupboard (see Figure 3.9).

A slightly different procedure was carried out to produce the resin impregnated reinforcement that was to be used in the compression tests. 150 mm squares of the fabric were initially marked out using masking tape. This meant that when the fabric was cut to size through the tape it was prevented from fraying. This ensured less wastage of the fabric and a homogenous structure.
3. Experimental Materials and Procedures

**Figure 3.9** Schematic showing the compression of an assembly of resin impregnated cloths between two glass plates.

The first layer of the reinforcement was placed into the base of a Teflon coated aluminium tray and impregnated with the resin using a soft brush. Ensuring that the first layer of the reinforcement had completely wetted out, the next dry layer was added on top, aligning the warp and weft yarns, and the procedure repeated. Once all the plies had been impregnated a layer of silicone coated polyester release film was placed on top to ensure no adhesion between the uppermost ply and the compression platen during the compression test to follow.

Care also had to be taken with regard to the curing cycle of the resin. The correct mixture of polyester and initiator was used so that there was enough time to carry out both the impregnation of the assembly of dry reinforcement and the compression test. If gelation were to occur anywhere locally in the laminate before the end of compression the results would be unreliable due to local inhibition of compression and inhomogeneous total compression.

### 3.5 COMPRESSION TESTING

All of the compression experiments were carried out using the same Instron 1195 universal testing machine, fitted with either a 5 kN or 100 kN load cell. A steel circular platen of 120 mm diameter (specifically constructed for the purpose of these compression tests) was fitted directly to the crosshead of the Instron.
Depending on the maximum load to be used in the compression test the load cell was calibrated to either 2, 5 or 10% full scale. The Instron's chart recorder, measuring displacement against applied load, was set to a constant chart speed of 50 mm/minute, see Figure 3.10. Again, depending on the test, the crosshead speed of the Instron was set to either 0.05, 0.1, 0.5 or 1.0 mm/minute. This would ensure that an increasing load was applied to the reinforcement under a constant crosshead speed.

![Chart Displacement vs Load](chart-displacement-vs-load.png)

**Figure 3.10** A typical trace from the Instron's chart recorder monitoring load against chart displacement.

The circular compression platen was then slowly brought into contact with the base platen until a force registered on the chart recorder. The compression platen was then raised until the force was reduced to zero. This position was taken to be the zero datum point and the two Instron dials, measuring crosshead displacement, were then set to zero. An additional dial gauge was fitted directly to the crosshead to confirm displacement readings, this was also zeroed. The distance between the compression and base platen would now be recorded by three independent sources. The platens were then separated a suitable distance to allow the reinforcement to be placed directly onto the base platen.
3.5.1 DRY REINFORCEMENT

The glass fabric was first cut into a number of 150 mm square sections. The layers of reinforcement were stacked and placed between the platens of the Instron. The crosshead was then slowly lowered until the compression platen was almost touching the uppermost layer of the assembly. The chart recorder and the crosshead were then started simultaneously. Once the target load had been reached the crosshead and chart recorder were stopped. The final distance between the platens, representing the final thickness of the compressed assembly of dry reinforcement, was then recorded. Using these results the fibre volume fraction for the assembly could be calculated at any instant during the compression. Graphs of pressure versus fibre volume fraction were then plotted for the compression of the assembly of dry reinforcement.

A series of compression tests were carried out on assemblies of 1-6, 8, 10, 15 and 20 plies for different loading conditions. The alignment was such that the warp and weft yarns were matched as best as possible from one layer to the next. Compression tests were also carried out on reinforcement laid up at alternating 0/45° orientation and on layers of reinforcement that had been previously compressed, to see if this had any affect on the compression curves of the reinforcement. It was ensured that between repeated compression tests on the same assembly of dry reinforcement, the plies were separated after each test and reassembled. This was to allow recovery of any elastic deformation of the fibres, to negate any preferential nesting that the layers of reinforcement may have acquired under loading and to make the experiment reproducible. The effect of the compression speed was also investigated by performing compression tests at different constant crosshead speeds, namely 0.05, 0.1, 0.5 and 1.0 mm/min.

3.5.2 RESIN IMPREGNATED REINFORCEMENT

3.5.2.1 Compression without Resin Curing

The assembly of resin impregnated reinforcement was constructed as described in section 3.4. No initiator was added to the resin before impregnating the reinforcement and hence, these
studies include the viscous resin effects on compression but no curing effects. Besides, as microstructural examination of the compressed laminate was not required at this stage there was no need to cure the laminate in-situ.

The tray containing the impregnated cloths was placed on the base platen and the crosshead lowered until the compression platen was almost in contact with the surface of the release film, see Figure 3.11. It is worth noting that the zero datum point in this case was set up so that the base of the tray was zero, and not the base platen.

![Figure 3.11 Schematic of the apparatus used in the compression tests.](image)

Again both the chart recorder and the crosshead were started simultaneously and stopped once the target compression load had been reached. The value of the crosshead displacement was recorded, which represented the compressed thickness of the laminate from the three dial gauges.

As mentioned previously in Chapter 2, when pressure is applied to the laminate it is to both the resin and the fibres. As the pressure increases and the resin flows out of the laminate the fibres begin to compact, transferring more of the pressure to the fibres and away from the resin. To examine the viscous effects of the resin during compression a series of tests were carried out where the target pressure remained unchanged but three different crosshead speeds of the Instron were selected, namely 1 mm/min, 0.1 mm/min and 0.05 mm/min. It is expected from
previous studies on the compression of uni-directional laminates [Gutowski et al (1987a, 1987b)] (see Chapter 2) that decreasing the speed of compression would decrease the pressure contribution to the viscous resin flow and increase correspondingly the pressure transferred to the fibres. This should result in a shift of the compression curves for assemblies of reinforcement impregnated with resin to the compression curves obtained for similar assemblies of dry reinforcement. Thus the final thickness of the laminate was recorded in each test from the three dial gauges monitoring crosshead displacement. The data was then assembled as graphs of pressure as a function of thickness or fibre volume fraction.

3.5.2.2 Compression with Resin Curing

Again the assembly of resin impregnated reinforcement was constructed as described in section 3.4. This time initiator was added to the resin before impregnating the reinforcement so that the laminate could cure. The assemblies of resin impregnated reinforcement were then compressed under a constant compression speed until a maximum target pressure had been reached. The crosshead then remained in place until the laminate had completely cured in-situ. Once cured, the crosshead was raised, the laminate removed, a sample cut from the centre, and the actual thickness of the laminate measured using a micrometer screw gauge. The results were used to plot graphs of pressure versus fibre volume fraction.

Compression tests were carried out on assemblies of 10 and 20 resin impregnated plies at a constant crosshead speed of 1 mm/min whilst the loading conditions were varied to a series of maximum target loads, ranging from 0.05 to 20 kN corresponding to a pressure range of 4.4 to 1768 kPa (0.04 to 17.4 bar). In all tests the warp and weft yarns were aligned from one layer to the next as best as possible.

As the polyesters used to impregnate assemblies of dry reinforcement were cold curing systems, the laminate was able to cure in-situ at room temperature. Crystic 471 PALV and Crystic 781 PA were mixed with 0.28 % and 1.70 % bw initiator respectively which would result in gel times of approximately 70 and 80 minutes respectively. However as an epoxy, a hot curing resin system, was used as the matrix in a number of the laminates a hot plate was added to the compression apparatus (see Figure 3.12). The use of the epoxy was to examine
the effects of different resin viscosities on both the compression curves and the laminate microstructure. The hot plate contained three heating rods connected to a temperature controller offering the ability to rapidly change the system temperature. The bottom of the hot plate was covered with a ceramic insulating material, to prevent heat loss into the load cell, and placed on the base platen. The hot plate was then preheated to 40 °C, the same temperature as the initial temperature of the epoxy matrix, before the tray containing the resin impregnated plies was placed on top. The compression platen, also covered with an insulating layer, was then lowered into position and the compression test was carried out. Once the target load had been reached and the crosshead stopped, the hot plate was heated up to 100 °C and the laminate was allowed to cure in-situ under pressure. Finally the cured laminate was removed and post cured for three hours at 160 °C. The epoxy mixture of Araldite LY564 to Hardener HY 2954 was at 100:35 pbw ratio, resulting in approximately a gel time over 60 minutes at 40 °C. Given that the preparation time for mixing of reactants and lay-up of the resin impregnated plies was approximately 30 minutes for both polyester and epoxy systems and that the viscosity of both systems is low before gelling, curing effects could be considered negligible during compression.

Figure 3.12 Schematic of the apparatus used for the compression of epoxy resin impregnated assemblies showing the addition of a hot plate and temperature controller.
3.5.3 CALCULATIONS

It was assumed that changes in the cross-sectional area of the laminate were negligible, therefore the fibre volume fraction at any instant during compression can be calculated using the following expression:

\[
V_f = \frac{N p_a}{\rho_f H}
\]  

(3.2)

where \(V_f\) is the fibre volume fraction, \(N\) is the number of cloths, \(H\) is the total thickness of the laminate and \(\rho_a\) and \(\rho_f\) are the areal density of the fabric and the fibre density (in this case E-glass) respectively. Once the pressure, \(P\), at any point during the compression had also been calculated, a graph of pressure versus fibre volume fraction was plotted for the compression test (see Chapters 5 and 6). The pressure during the compression test can be determined as

\[
P = \frac{F}{A}
\]  

(3.3)

where \(F\) is the load applied to the laminate during compression and \(A\) is the cross sectional area of the compression surface which is determined by the size of the compression platen, in this case a circular area of diameter 120 mm.

According to suggestions by Toll and Manson (1994) (see Chapter 2) the elastic deformation of assemblies of planar fibre networks can be represented by the power law relation

\[
P_f = a V_f^b
\]  

(3.4)

Thus plotting log-log graphs of pressure versus fibre volume fraction should produce a straight line of gradient \(b\) and y-intercept \(a\).
3.6 LAMINATE CHARACTERISATION

3.6.1 SAMPLE PREPARATION

Once a compressed, resin impregnated assembly of reinforcement had been completely cured it was marked up for the removal of a sample from the centre of the laminate, 10 mm x 5 mm. This was then cut from the laminate using a diamond saw. The thickness of the sample was measured using a micrometer before being mounted in *Epofix*, a transparent epoxy cold curing resin. The sample was then polished in cross-section (see Table 3.8) ready for inspection by optical microscopy. However as the refractive indexes of polyester, epoxy and glass are similar it was difficult to determine the glass/matrix interface when the samples were inspected under the microscope. The samples, once polished, were therefore etched in hydrofluoric acid fumes for approximately 30 seconds. The hydrofluoric acid is an extremely corrosive fluorinating agent which attacks the glass fibres on the surface of the sample and highlights the glass in the matrix. An Axiophot optical microscope was used to photograph the entire cross-section of the sample. The resulting pictures were assembled together as a mosaic to form an accurate representation of the sample surface from which measurements of various geometric parameters and feature examination could be made.

3.6.2 VOLUME FRACTION BURN OFF TESTS

To confirm the fibre volume fraction calculations (see section 3.5.3) a series of burn off tests were performed. Four small sections were cut, approximately 10 x 10 mm, from different parts of the cured laminate. Four empty ceramic crucibles with lids were then individually weighed. One sample was then placed into each of the crucibles and the sample, crucible and lid were weighed together. The crucibles containing the samples were then placed in a furnace preheated to 600 °C for one and a half hours whereby the resin in the sample was completely burned off, leaving only the glass fibres. On removal the crucibles were allowed to cool and then the crucible, lid and the glass fibres were reweighed. The fibre volume fraction of each of the four samples could then be determined from the relationship.
3. Experimental Materials and Procedures

\[ V_f = \frac{(C - B)}{\rho_f} - \frac{(A - C)}{\rho_m} + \frac{(C - B)}{\rho_f} \]  \hspace{1cm} (3.5)

where \( A \) is the mass of the crucible and the sample (pre burn off), \( B \) is the mass of the crucible, \( C \) is the mass of the crucible and the glass fibres (post burn off), \( \rho_f \) is the density of the fibres and \( \rho_m \) is the density of the matrix. Once the fibre volume fraction had been calculated for all four of the samples they were averaged to obtain the mean fibre volume fraction of the laminate.

Table 3.8 Polishing program for polyester and epoxy matrix samples mounted in Epofix.

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3. Experimental Materials and Procedures

3.6.3 MICROSTRUCTURAL IMAGE ANALYSIS OF COMPRESSED LAMINATES

The microstructural image analysis of laminates processed under different maximum compression loads, which has been carried out systematically in this study, is an important contribution in the area of processing of woven fabric laminates and in the area of their micromechanical analysis. The main aim of this area of investigations is to accurately determine the fundamental geometric parameters of the woven fabrics under compression and the modes of deformation during compression. This knowledge of microstructural data is important when considering the modelling of structure changes of woven fabrics which have implications in processability studies (change of permeability), and also for the micromechanical analysis of woven fabric composites. By examining the through thickness microstructures of different assemblies of resin impregnated reinforcement loaded to various compressive forces, one is able to view the changing geometry of the laminate, the changing fabric and fibre structure and the varying pore structure and size during the compression cycle, i.e. at different levels of pressure and for different magnitudes of compressed laminate thickness.

3.6.3.1 Through Thickness Geometry

As explained earlier in section 3.6.1 the photographs taken of the polished sample were assembled together to form a mosaic. From this mosaic of pictures one was able to accurately measure various geometric parameters of the through thickness of the sample. Figure 3.13 shows a schematic of a segment taken from the cross-section of the plain weave laminate indicating the regions measured.

The proposed analysis concerns the examination of plain weaves and for the purpose of this investigation a glass plain weave fabric, Y0212, was used (see section 3.2.1). In the presented studies, the warp yarns are represented by the elliptic cross-sections in Figure 3.13 and the weft yarns are the longitudinal yarns. The height, h, and the width, w, representing the ellipse minor and major axis respectively, of all the warp yarns were measured, together with the x and y co-ordinates of the centre of each ellipse.
3. Experimental Materials and Procedures

The mosaic of photographs was then converted into a digital image and further analysed using UTHSCSA *Image Tool* software [Barett *et al* (1995)]. The image analysis yielded area measurements of the cross-sections of the warp yarns where the technique of summing up pixels was employed without involving any assumptions about the shape of the cross-sections.

The weft yarn waveform was considered sinusoidal, described by the relation

\[ y = a \sin \left( \frac{2\pi x}{\lambda} + \phi \right) \]  (3.6)

**Figure 3.13** A segment from a cross-section of plain weave laminate indicating some of the geometric parameters for measurement.
where $a$ is the amplitude and $\lambda$ the wavelength of the yarn waveform respectively and $\phi$ is the phase angle. It was not possible to examine the weft yarns across their thickness since the thickness, $t$, and other features of its perimeter would depend at which location across its width the yarn was sectioned. To overcome this problem the centrelines of the longitudinal weft yarns were drawn and each centreline was assumed to form a sinusoidal wave which would represent the yarn waveform. Three best fit lines were then drawn through various points of the centreline for each yarn: (a) maxima, (b) minima and (c) mid-points (see Figure 3.14).

![Figure 3.14](image-url)

**Figure 3.14** Schematic presenting the microstructural measuring technique and the measured parameters.
The distance, $2a$, between the lines of maxima and minima represented twice the amplitude of the yarn waveform. The distance between the intersections of the centreline with the mid-point line represented half the wavelength, $\lambda$, of the yarn waveform. The distance, $d$, between the midpoint lines of consecutive weft yarns represented the distance between plies. The phase angle, $\phi$, between consecutive plies was determined from the relative horizontal shift between the maxima of consecutive waveforms and the average wavelength for each of the longitudinal weft yarns. In this way the average phase angle between each of the consecutive layers of cloth was established for the entire laminate.

In comparison with the present technique, Yugartis et al (1993) measured the inclination and crimp angles of weft yarns, rather than their amplitude and wavelengths. These measurements of angles might have been distorted due to local irregularities of the shape of the yarns. The presented technique of the generalised sinusoidal approximation, calculates the amplitude of the waveform from best fit data, which avoids erroneous effects due to the localised irregularities of the yarns. The technique is also compatible with approximations employed in the latest micromechanical models [Naik and Ganesh (1994)].

This chapter then has characterised both the fabrics and resins that are to be used throughout this study. Viscometry tests have determined the pseudoplastic and Newtonian nature of polyester Crystic 471 and epoxy Araldite LY 564 respectively. The systematic compression testing of both dry and resin impregnated reinforcement has been described. Finally the chapter introduces a technique for measuring a range of geometric parameters for woven fabrics under compression.
4. MATHEMATICAL DESCRIPTION OF COMPRESSION
4. Mathematical Description of Compression

4.1 INTRODUCTION

Two types of compression experiments are covered in this thesis: (a) compression tests of assemblies of dry reinforcement and (b) compression tests of assemblies of resin impregnated reinforcement. This chapter focuses on a theoretical description of compression, incorporating the general case of viscoelastic compression of resin impregnated reinforcement. Past studies on resin impregnated assemblies of unidirectional fibres [Gutowski et al. (1986 and 1987a)] and the results of the present study on resin impregnated fabrics (see Chapter 6) lead to the conclusion that the degree of compression depends on the compression speed which was initially interpreted [Gutowski et al. (1986 and 1987a)] as the result of viscoelasticity. No such dependence on the compression speed has been observed in the compression of dry fabrics (see Chapter 5) which has been explained as a result of the absence of resin flow.

The viscoelastic model of compression included in this chapter is split into a component for the deformation of the fibre network and a component for the resin flow and addresses various issues of viscoelasticity for the fibre/resin system. It has been assumed that any curing during compression can be neglected for slow reacting systems and, hence, no curing kinetics are included in the model of this study. The model of viscoelastic compression applies directly to the compression of resin impregnated reinforcement. In the case of the compression of dry reinforcement the component of viscous resin flow is omitted from the model. The investigation considers a Newtonian and a non-Newtonian resin applicable to the epoxy and polyester systems, respectively, used in this study. The comparisons between experimental and theoretical results from the model are presented at the end of Chapter 6.

4.2 COMPRESSION OF FIBRE/RESIN SYSTEM

Figure 4.1 presents a diagram of the modelled assembly which corresponds to the experimental setup. The resin impregnated reinforcement is placed between two disc type compression platens and compression is applied by the downward displacement of the top platen at a constant compression speed whilst the lower platen remains stationary. A model is formulated and examined for the viscoelastic compression of the composite system.
This next section presents the construction and development of the model. According to the currently proposed model, the total compression force, $F$, is split between the fibres and the resin as it has also been suggested in the viscoelastic Voigt-type model of Gutowski et al (1987a):

$$F = F_f + F_m$$ (4.1)

where subscripts $f$ and $m$ refer to fibres and matrix, respectively.

If $A$ is the compression area of the fibre/resin system normal to the compression force, forces are replaced by the corresponding pressures.

Define

$$P_f = \frac{F_f}{A}$$ (4.2)

and

$$P_m = \frac{F_m}{A}$$ (4.3)
4. Mathematical Description of Compression

It follows

\[ P = P_f + P_m \]  \( (4.4) \)

The volume of the fibre/resin system under compression is

\[ VOL = \pi R^2 H \]  \( (4.5) \)

at a radial distance \( R \) and thickness \( H \). As the volume changes during compression it is assumed that any lateral expansion of cloths (in the radial direction) is negligible and, hence, only the reduction in thickness contributes to the volume change. This is in agreement with previous experimental studies [Jortner (1992)] and with work carried out in this study on the microstructural analysis of the compression of assemblies of plain woven cloths. The next stage is to consider the response to compression of each component of the composite assembly, i.e. the fibres and the resin.

4.2.1 COMPRESSION OF DRY REINFORCEMENT

The aim of this section is to suggest a relation for the determination of the pressure component, \( P_f \), acting on the fibres as is described by Equation \( (4.4) \). According to suggestions by Toll and Manson (1994) which have also been validated by experimental studies in the next chapter and also by other workers, [Gauvin and Chibani (1988), Kim et al (1991), Pearce and Summerscales (1995) and Quinn (1990)], the elastic deformation of assemblies of planar fibre networks can be represented by the power-law

\[ P_f = a V_f^b \]  \( (4.6) \)

The constant \( a \) includes a factor from the beam theory that can vary between 48 to 192, depending whether the fibre/beams can fully slip (supported beam) or cannot slip (packed beam) at contact points. Hence if \( a \) and \( b \) are determined from compression tests on dry cloth assemblies, \( a \) may increase or decrease up to four times in the compression of resin impregnated reinforcement. This would depend upon whether the added resin reduces
slippage between fibres due to its high viscosity or increases slippage due its lubricating properties, respectively.

**4.2.2 RESIN FLOW THROUGH A POROUS MEDIUM DURING COMPRESSION**

The compression process is generally represented as a quasi-steady state process where inertia terms are ignored. Therefore no convection terms are present in the resin flow equations, which is a reasonable assumption given the relatively low compression speeds applied in the experimental procedures of this study that also result in low resin flow speeds.

The resin flowrate at any radial position \( r \) within the composite system is given by the rate of change of resin volume within the reinforcement due to compression:

\[
Q_m(r) = \frac{d(VOL_m)}{dt} = \frac{d(nr^2H(1-V_f))}{dt}
\]  (4.7)

where \( V_f \) is the fibre volume fraction and \( (1-V_f) \) is the resin volume fraction. Complete impregnation of the reinforcement by resin is assumed in this study without considering air voids or other types of voids within the resin. By taking into account the assumption of absence of lateral expansion of reinforcement during compression, *Equation (4.7)*, becomes

\[
Q_m(r) = nr^2 \frac{d(H(1-V_f))}{dt}
\]  (4.8)

The compression process is considered to cause in-plane resin flow which is then modelled by relations of the type of Darcy's flow through a porous medium where in-plane, isotropic permeability is assumed.

**4.2.2.1 Viscous, Newtonian behaviour**

In this section the liquid resin is considered to behave as a viscous Newtonian fluid of constant viscosity. The model includes in-plane resin flow which, in the cylindrical system of
co-ordinates in Figure 4.1, lies in the radial direction; no angular effects exist due to axisymmetry. Darcy’s law is then written as

\[ Q_m(r) = -2 \pi r H \frac{K}{\mu} \frac{dP_m}{dr} \]  

(4.9)

where \( K \) is an in-plane isotropic permeability and \( \mu \) is the resin viscosity.

By combining Equations (4.8) and (4.9)

\[ \frac{d[H(1 - V_f)]}{dt} = -\frac{2 KH}{\mu r} \frac{dP_m}{dr} \]  

(4.10)

If it is assumed that the change in fibre fraction is directly related to the change in laminate thickness where any changes in the areal fraction of each cloth are neglected, the fibre volume fraction can be expressed as

\[ V_f = \frac{N \rho_a}{\rho_f H} \]

(4.11)

where \( N \) is the number of woven cloths in the compressed assembly and \( \rho_a \) and \( \rho_f \) are the areal density of cloth and the density of the fibre material (e.g. E-glass) respectively; it follows

\[ \frac{d[H(1 - V_f)]}{dt} = \frac{d}{dt} \left[ H \left( 1 - \frac{N \rho_a}{\rho_f H} \right) \right] \]

\[ = \frac{1}{\rho_f} \frac{d}{dt} \left( \rho_f H - N \rho_a \right) \]

\[ = \frac{1}{\rho_f} \frac{d}{dt} \left( \rho_f H \right) - \frac{1}{\rho_f} \frac{d}{dt} \left( N \rho_a \right) \]  

(4.12)
As it has been assumed that there is no lateral expansion of the assembly there is no change in the areal density ($\rho_0$) with time and therefore the second half of Equation (4.12) goes to zero and becomes

\[
\frac{d[H(1-V_r)]}{dt} = \frac{1}{\rho_f} \frac{d(\rho_f H)}{dt}
\]

\[
= \frac{\rho_f}{\rho_f} \frac{dH}{dt}
\]

\[
= \frac{dH}{dt}
\]  

(4.13)

By combining Equations (4.10) and (4.13) the following relation is derived.

\[
\frac{dP_m}{dr} = -\frac{\mu r}{2KH} \frac{dH}{dt}
\]  

(4.14)

which is integrated

\[
\int_{r}^{r_{\text{front}}} dP_m = \int_{r}^{R} \frac{\mu r}{2KH} \frac{dH}{dt} r dr
\]  

(4.15)

and becomes

\[
P_{m, r} - P_{m, \text{front}} = \frac{\mu}{4KH} \frac{dH}{dt} (R^2 - r^2)
\]  

(4.16)

where $P_{m, r}$ and $P_{m, \text{front}}$ is the resin pressure at position $r$ and at the flow front (at position $R$), respectively. Equation (4.16) gives the pressure distribution along the radial direction which leads to resin flow during compression. Integration of the pressure over the compression area will result in the component of the compression force that generates the resin flow, noting that $P_{m, \text{front}} = 0$. 

Compression and Microstructure of Glass Fibre Fabrics in the Processing of Polymer Composites
4. Mathematical Description of Compression

\[ F_m = \int_0^{2\pi} \int_0^R (P_{m,r} \cdot r^2) \, dr \, d\theta \]  \hspace{1cm} (4.17)

This practice of pressure integration over the compression area in order to determine the compression force, is commonly applied in any type of flow due to compression as for example in creeping, viscous, squeezing flow between two circular disks [Bird et al (1987)].

After substituting the pressure from Equation (4.16), Equation (4.17) is integrated:

\[ F_m = \frac{\pi \mu R^4}{8KH} \frac{dH}{dt} \]  \hspace{1cm} (4.18)

By dividing \( F_m \) by the composite compression area \( A = \pi R^2 \), the mean pressure component acting on the matrix (as defined by relation (4.3)) can be derived:

\[ P_m = \frac{\mu R^2}{8KH} \frac{dH}{dt} \]  \hspace{1cm} (4.19)

Equation (4.19) expresses the pressure component acting on the matrix as a function of the compression speed, \( \frac{dH}{dt} \), and can replace \( P_m \) in Equation (4.4) for a resin with viscous, Newtonian behaviour.

4.2.2.2 Viscous, non-Newtonian behaviour

Christopher and Middleman (1965) suggested the following relation for one-dimensional, Darcy's law to be applied to power-law, non-Newtonian fluids:

\[ U_{mp} = \left( \frac{K}{M} \frac{dP_m}{dx} \right)^{1/n} \]  \hspace{1cm} (4.20)

where \( U_{mp} \) is the superficial velocity, \( n \) is the power-law index, \( K \) is the permeability and \( M \) is a parameter depending on the rheology of fluid and the porosity, pore size and structure of the porous medium. Hayward and Harris (1989) used a similar but simpler relation.
where \( C \) is the consistency of the power-law fluid. Relation (4.20) was found to be the most suitable relation in this model from the trial-and-error procedure to fit predictions with experimental data.

The resin flowrate during the compression of the fibre/resin system between two circular discs is then given by the relation

\[
Q_m(r) = -2\pi r H \left( \frac{K}{M} \frac{dP_m}{dr} \right)^{1/n} \tag{4.22}
\]

By combining Equations (4.8) and (4.22)

\[
\pi r^2 \frac{d[H(1-V_f)]}{dt} = -2\pi r H \left( \frac{K}{M} \right)^{1/n} \left( \frac{dP_m}{dr} \right)^{1/n}
\]

therefore

\[
\frac{d[H(1-V_f)]}{dt} = -2H \left( \frac{K}{M} \right)^{1/n} \left( \frac{dP_m}{dr} \right)^{1/n} \tag{4.23}
\]

Further combination of Equations (4.13) and (4.23) gives

\[
\frac{dH}{dt} = -2 \frac{H}{r} \left( \frac{K}{M} \right)^{1/n} \left( \frac{dP_m}{dr} \right)^{1/n}
\]

therefore

\[
\frac{dP_m}{dr} = -\frac{M}{K} \left( \frac{r}{2H} \right)^n \left( \frac{dH}{dt} \right)^n \tag{4.24}
\]

which is integrated

\[
\int_{r_0}^{r} dP_m = -\frac{K}{r} \frac{M}{2^n K^n} \left( \frac{dH}{dt} \right)^n r^n \, dr \tag{4.25}
\]
and becomes

\[ P_{m,r} - P_{m,\text{front}} = \frac{M}{(n+1)2^a H^n K} \left( \frac{dH}{dt} \right)^n \left( R^{n+1} - r^{n+1} \right) \] (4.26)

Integration of Equation (4.26) over the compression area \( A \) yields the compression force component causing the viscous resin flow.

\[
F_m = \frac{M 2\pi}{(n+1)2^a H^n K} \left( \frac{dH}{dt} \right)^n \int_0^R (R^{n+1} - r^{n+1}) r \, dr
\]

\[
= \frac{M 2\pi}{(n+1)2^a H^n K} \left( \frac{dH}{dt} \right)^n \left[ \frac{R^{n+3}}{2} - \frac{r^{n+3}}{n+3} \right]_0^R
\]

\[
= \frac{M 2\pi}{(n+1)2^a H^n K} \left( \frac{dH}{dt} \right)^n \left[ \frac{R^{n+3}}{2} - \frac{R^{n+3}}{n+3} \right]
\]

\[
= \frac{M \pi R^{n+3}}{2^n (n+3) H^n K} \left( \frac{dH}{dt} \right)^n
\] (4.27)

After dividing Equation (4.27) by the composite compression area \( A \), the compression pressure on the matrix is derived

\[ P_m = \frac{M R^{n+3}}{2^n (n+3) H^n K} \left( \frac{dH}{dt} \right)^n \] (4.28)

4.3 CONCLUSIONS

According to the described theoretical analysis in this chapter the constructed model of viscoelastic compression can be further classified into the following types of general equations which are applicable to the compression of a particular subsystem.
4. Mathematical Description of Compression

(a) Compression of assemblies of dry fabrics:

\[ P_f = aV_f^b \]  \hspace{1cm} (4.29)

(b) Compression of assemblies or resin impregnated fabrics where the resin behaves as a Newtonian fluid (e.g. epoxy in this study):

\[ P = aV_f^b + \frac{\mu R^2}{8KH} \frac{dH}{dt} \]  \hspace{1cm} (4.30)

(c) Compression of assemblies of resin impregnated fabrics where the resin behaves as a non-Newtonian power-law fluid (e.g. polyester in this study):

\[ P = aV_f^b + \frac{MR^{n+1}}{2^n(n+3)KH^n} \left( \frac{dH}{dt} \right)^n \]  \hspace{1cm} (4.31)
5. COMPRESSION TESTING OF DRY REINFORCEMENT
5.1 INTRODUCTION

During the lay up stage of Resin Transfer Moulding the reinforcement is subjected to a certain amount of compression when the mould is closed. The purpose therefore of the compression testing on dry reinforcement is to simulate this type of compression. Three types of glass fabric were tested in this manner: plain weave (YO212), twill weave (YO185) and 5 harness satin weave (YO227).

The first stage of the compression testing included a comprehensive set of tests for plain weave under a variety of different conditions. Compression tests were performed on assemblies of dry plain weave reinforcement that consisted of assorted numbers of plies between one and twenty. These assemblies were loaded to different maximum target pressures between 0.088 and 0.88 MPa and compressed at various rates of compression ranging between 0.05 and 1.0 mm/min. Tests were also performed on assemblies of plain weave plies aligned warp yarn to warp yarn and weft yarn to weft yarn (0°), assemblies of plain weave plies aligned at an orientation of 0°/45° and on assemblies of plain weave plies that had been previously compressed. In the second stage of testing assemblies of twill weave and 5 harness satin weave plies were also submitted to compression testing at different compression speeds and to a maximum pressure of 1.77 MPa.

5.2 PLAIN WEAVE

Figure 5.1 is a graph of pressure versus fibre volume fraction for the compression of three different assemblies of 5 layers of dry reinforcement at a compression rate (crosshead speed) of 0.5 mm/min. In all the graphs of compression data in Chapters 5 and 6 the values for the fibre volume fraction, $V_f$, have been calculated according to Equation (3.2), based on corresponding values of thickness, $H$, of the assembly of reinforcement under compression. The three curves in Figure 5.1 represent the compression of the assemblies to three different maximum target pressures 0.088, 0.44 and 0.88 MPa. Comparison of fibre volume fractions at 0.088 MPa shows a maximum difference of 0.0134.
5. Compression Testing of Dry Reinforcement

Crosshead speed = 0.6 mm/min  
Number of plies = 5

Maximum pressure
- 0.088 MPa
- 0.44 MPa
- 0.88 MPa

Figure 5.1  Plots of pressure versus fibre volume fraction for three assemblies of 5 dry plain weave plies compressed to different maximum pressures.

Figures 5.2 and 5.3 show graphs of pressure versus fibre volume fraction for the compression of assemblies of 10 and 20 layers of dry reinforcement respectively. In each graph there are three curves labelled run 1, run 2 and run 3. Each run represents a single compression test on a completely fresh assembly of plies i.e. reinforcement that had undergone no previous mechanical testing. For the purpose of this thesis an assembly of fresh plies will be defined as ‘original’. For each run the assemblies of original plies were compressed to a maximum pressure of 0.88 MPa at a compression rate of 1.0 mm/min. The purpose of these tests was to examine the level of repeatability one could expect from the compression experiments. The values obtained for the final fibre volume fraction from each of the three compression tests on assemblies of 10 plies were compared and differed by a maximum of 0.0228. Similarly of the three compression tests on assemblies of 20 plies, the final fibre volume fraction values differed by a maximum of 0.013.
Figure 5.2 Plots of pressure versus fibre volume fraction for the compression of three assemblies of 10 dry plain weave plies to 0.88 MPa.

Figure 5.3 Plots of pressure versus fibre volume fraction for the compression of three assemblies of 20 dry plain weave plies to 0.88 MPa.
An assembly of 5 original plies was compressed to a maximum pressure of 0.88 MPa at a compression rate of 0.5 mm/min. Once the target pressure had been removed the five layers of reinforcement were separated and then restacked in the same order and orientation (0°) as before (following the procedure outlined in section 3.5.1). The assembly was compressed again to the same maximum pressure, 0.88 MPa, and at the same compression rate, 0.5 mm/min, as the previous test. An original assembly of 5 plies was stacked in a 0°/45° sequence and compressed to 0.88 MPa at the same compression rate of 0.5 mm/min. By carrying out this series of tests one was able to examine the effects on fibre volume fraction that resulted from repeated compression of the same assembly of plies and also any effects that resulted from altering the ply orientation of an assembly. Figure 5.4, a graph of pressure versus fibre volume fraction, shows the compression curves for the three tests.

![Graph showing pressure versus fibre volume fraction](image)

**Figure 5.4** Plots of pressure versus fibre volume fraction for the compression of three assemblies of 5 dry plain weave plies that have undergone three different testing conditions, 'original', 'repeat 1' and '0/45'.

'Repeat 1' in the graph legend is used to describe an assembly of cloths that has been compressed once before. '0/45' represents an assembly of plies that have been stacked such
that the orientation of the warp and weft yarns from one layer are at an angle of ±45° from the warp and weft yarns of an adjacent layer. In this compression test, for example, the stacking sequence for the 5 plies was 0°/45°/0°/45°/0°. The term 'original' has been described earlier in this section.

The difference in final fibre volume fraction between the compression of the original plies to that of the plies compressed for a second time (repeat 1) was 0.011. The values for the final fibre volume fractions of the original plies at 0° orientation and the original plies at 0°/45° orientation differed by 0.027.

The same three compression tests were repeated on assemblies of 10 dry plies to the same target pressure and the same compression rate (see Figure 5.5). The difference in final fibre volume fractions from the original oriented at 0° to that compressed for a second time and that compressed at 0°/45° were 0.015 and 0.006 respectively.

![Figure 5.5 Plots of pressure versus fibre volume fraction for the compression of three assemblies of 10 dry plain weave plies that have undergone three different testing conditions, 'original', 'repeat 1' and '0/45'.](image)

Crosshead speed = 0.5 mm/min
Maximum pressure = 0.88 MPa
Number of plies = 10
This line of investigation was taken further by compressing original assemblies of 10 and 20 plies to a maximum pressure of 0.88 MPa at a compression rate of 1.0 mm/min. Each assembly of plies was then compressed a further 2 times following the procedure in section 3.5.1.

The runs on assemblies of 10 plies showed that repeated compression of the same assembly of plies resulted in a maximum variation of 0.017 in the final fibre volume fraction when compared to the final fibre volume fraction obtained by compressing the original assembly of plies (see Figure 5.6). Similarly for assemblies of 20 plies, the maximum variation between the final volume fractions for repeated compression to that of the original was 0.019 (see Figure 5.7).

![Figure 5.6 Plots of pressure versus fibre volume fraction for the repeated compression of an assembly of 10 dry plain weave plies to 0.88 MPa.](image)

Four different original assemblies of 10 dry plies were compressed to a maximum pressure of 0.88 MPa at different compression speeds, 0.05, 0.1, 0.5 and 1.0 mm/min. Figure 5.8 shows the four compression curves for pressure versus fibre volume fraction illustrating the effects of the different compression speeds. On comparing the final fibre volume fractions of the four tests, using the 0.05 mm/min compression curve as the baseline, there was a maximum difference of 0.0085, which can be considered within the repeatability error (see Figure 5.2).
5. Compression Testing of Dry Reinforcement

Crosshead speed = 1.0 mm/min
Target pressure = 0.88 MPa
Number of plies = 20

Figure 5.7 Plots of pressure versus fibre volume fraction for the repeated compression of an assembly of 20 dry plain weave plies to 0.88 MPa.

Crosshead speed
- 0.05 mm/min
- 0.1 mm/min
- 0.5 mm/min
- 1.0 mm/min
Target pressure = 0.88 MPa
Number of plies = 10

Figure 5.8 Plots of pressure versus fibre volume fraction for the compression of four original assemblies of 10 dry plain weave plies at different compression speeds, 0.05, 0.1, 0.5 and 1.0 mm/min.
The investigation continued by studying the effect of the number of plies on the compression data. A series of compression tests were conducted on original assemblies of dry reinforcement where the number of plies in each assembly varied between 1 and 20. Tests were performed on assemblies consisting of 1, 2, 3, 4, 5, 6, 8, 10, 15 and 20 plies (see Figure 5.9). Each test involved the compression of the assembly to a maximum target pressure of 0.88 MPa at a compression rate of 1.0 mm/min. For each assembly of plies the compression test was performed three times ensuring original layers of reinforcement were used each time. At the end of each compression test (i.e. once the target pressure had been reached) the final thickness of each assembly was determined together with the final fibre volume fraction. In this way comparisons were made between the number of plies within an assembly and its final fibre volume fraction. The apparent individual ply thickness was also calculated by dividing the final thickness of each assembly by the number of plies within it.

![Figure 5.9 Plots of pressure versus fibre volume fraction presenting the compression curves for assemblies of dry plain weave reinforcement, each with a different number of plies.](image)

*Figure 5.9* Plots of pressure versus fibre volume fraction presenting the compression curves for assemblies of dry plain weave reinforcement, each with a different number of plies.

*Table 5.1* shows the mean values for the final thickness, apparent ply thickness and the final fibre volume fraction for each of the assemblies tested in this way. The resulting final fibre
volume fraction from the compression of a single ply was much lower than for the final fibre volume fractions of assemblies with two or more plies. The mean final fibre volume fraction for a single ply, was 0.06 lower than that for an assembly of two plies. There then appeared to be an intermediate mean final fibre volume fraction for assemblies of two, three and four plies where the $V_f$ only varied by 0.01. With assemblies of between five and twenty layers the final mean fibre volume fraction seemed to stabilise with a maximum variation of 0.026. From the results of mean apparent single ply thickness there is a reduction from 0.420 mm, for the compression of a single ply, to 0.345 mm per ply for the compression of an assembly of twenty plies.

Table 5.1 Results from the compression of assemblies of dry plain weave fabric, each with a different number of plies, to a maximum pressure of 0.88 MPa. The data shows mean values for assembly thickness, apparent ply thickness and fibre volume fraction.

<table>
<thead>
<tr>
<th>Number of plies</th>
<th>Final assembly thickness (mm)</th>
<th>Apparent ply thickness (mm)</th>
<th>Final assembly fibre volume fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.420</td>
<td>0.420</td>
<td>0.507</td>
</tr>
<tr>
<td>2</td>
<td>0.753</td>
<td>0.377</td>
<td>0.567</td>
</tr>
<tr>
<td>3</td>
<td>1.107</td>
<td>0.369</td>
<td>0.577</td>
</tr>
<tr>
<td>4</td>
<td>1.477</td>
<td>0.369</td>
<td>0.577</td>
</tr>
<tr>
<td>5</td>
<td>1.800</td>
<td>0.360</td>
<td>0.592</td>
</tr>
<tr>
<td>6</td>
<td>2.143</td>
<td>0.357</td>
<td>0.596</td>
</tr>
<tr>
<td>8</td>
<td>2.773</td>
<td>0.347</td>
<td>0.614</td>
</tr>
<tr>
<td>10</td>
<td>3.540</td>
<td>0.354</td>
<td>0.602</td>
</tr>
<tr>
<td>15</td>
<td>5.275</td>
<td>0.352</td>
<td>0.606</td>
</tr>
<tr>
<td>20</td>
<td>6.895</td>
<td>0.345</td>
<td>0.618</td>
</tr>
</tbody>
</table>

It has already been shown that no permanent deformation of the plain weave reinforcement occurs through repeated compression, if the plies are separated and reassembled between each subsequent compression test. The next stage then involved examining the effect on the compression curves if the assembly of plain weave plies were repeatedly compressed, without separating and reassembling the plies. *Figure 5.10* displays the hysteresis loops for three consecutive compression tests on an assembly of 20 layers of dry plain weave reinforcement. As can be seen from *Figure 5.10* when the assembly of reinforcement is unloaded to zero
pressure the recovery curve does not return to the initial thickness of the assembly of 11.10 mm but returns to 9.46 mm. As the assembly is once again compressed to the target pressure the compression curve does not follow the original path but is instead shifted to a smaller assembly thickness. On recovery the second hysteresis loop returns to a lower assembly thickness of 9.10 mm. The third compression cycle imitates the second but this time the shift to lower values of assembly thickness is much less, from 9.10 mm to 9.05 mm.

![Figure 5.10 Hysteresis loops showing plots of pressure versus assembly thickness for three consecutive compression tests of an assembly of 20 dry plain weave plies compressed to a maximum target pressure of 0.88 MPa.]

5.3 TWILL WEAVE

Two original assemblies of 10 dry twill weave plies, oriented at 0°, were compressed to the same maximum pressure of 1.77 MPa but at different compression speeds, 0.05 and 1.0 mm/min (see Figure 5.11). Comparison of the final fibre volume fractions resulted in a difference of 0.014, which can really be considered within the repeatability experimental error.
Maximum pressure = 1.77 MPa
Number of plies = 10

Crosshead speed
- 0.05 mm/min
- 1.0 mm/min

Fibre volume fraction

Figure 5.11 Plots of pressure versus fibre volume fraction for the compression of two assemblies of 10 dry twill weave plies at different compression speeds.

Target pressure = 1.77 MPa
Number of plies = 10

Crosshead speed
- 0.05 mm/min
- 1.0 mm/min

Fibre volume fraction

Figure 5.12 Plots of pressure versus fibre volume fraction for the compression of two assemblies of 10 dry 5 harness satin weave plies at different compression speeds.
5.4 5 HARNESS SATIN WEAVE

As with the assemblies of twill weave, two assemblies of 10 dry 5 harness satin weave plies, oriented at 0°, were compressed to 1.77 MPa at different compression speeds 0.05 and 1.0 mm/min (see Figure 5.12, previous page). The difference in final fibre volume fractions was 0.006, which again can be considered within the repeatability experimental error.

5.5 POWER-LAW RELATION FOR COMPRESSION

Plots of the compression data on log-log graphs of pressure versus fibre volume fraction led to the conclusion that the compression testing of assemblies of dry reinforcement followed the power law relation

\[ P = aV_f^b \]  (5.1)

(see section 3.5.3). The values for \( a \) and \( b \) were then determined by regression analysis of the compression data.

Comparisons were made between assemblies of reinforcement with different numbers of plies (see Figure 5.13), assemblies of reinforcement compressed at different compression speeds (see Figures 5.14 to 5.16) and also between different types of fabric (Figures 5.17). Table 5.2 summarises the results obtained for the variety of compression tests described above including the values for \( a \) and \( b \).

The three types of woven fabrics exhibit similar values for \( b \) but they reach different final fibre volume fractions at the maximum pressure. The satin weave is compressed more than the than the twill as expected because of its flat structure. In these results, it looks that the plain weave can also be compressed to high fibre volume fractions of around 0.65; this might be attributed to nesting effects as will be seen in Chapter 7.
5. Compression Testing of Dry Reinforcement

Figure 5.13 Log-log plots of pressure versus fibre volume fraction for the compression of assemblies of dry plain weave fabric, containing 5, 10 and 20 plies.

Figure 5.14 Log-log plots of pressure versus fibre volume fraction for the compression of assemblies of 10 dry plain weave plies at different compression speeds.
Figure 5.15 Log-log plots of pressure versus fibre volume fraction for the compression of assemblies of 10 dry twill weave plies at different compression speeds.

Figure 5.16 Log-log plots of pressure versus fibre volume fraction for the compression of assemblies of 10 dry 5 harness satin weave plies at different compression speeds.
Figure 5.17 Log-log plots of pressure versus fibre volume fraction for the compression of assemblies of 10 dry plain, twill and 5 harness satin weave plies.

Table 5.2 A summary of results from the compression tests on a variety of original assemblies of dry glass woven reinforcement.

<table>
<thead>
<tr>
<th>Weave type</th>
<th>Number of plies</th>
<th>Compression speed (mm/min)</th>
<th>Maximum fibre volume fraction</th>
<th>Pressure (MPa)</th>
<th>b</th>
<th>α   (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>plain</td>
<td>5</td>
<td>1.0</td>
<td>0.582</td>
<td>0.88</td>
<td>10.22</td>
<td>237.19</td>
</tr>
<tr>
<td>plain</td>
<td>10</td>
<td>1.0</td>
<td>0.595</td>
<td>0.88</td>
<td>10.20</td>
<td>175.32</td>
</tr>
<tr>
<td>plain</td>
<td>20</td>
<td>1.0</td>
<td>0.605</td>
<td>0.88</td>
<td>10.48</td>
<td>169.72</td>
</tr>
<tr>
<td>plain</td>
<td>10</td>
<td>0.05</td>
<td>0.599</td>
<td>0.88</td>
<td>10.00</td>
<td>150.80</td>
</tr>
<tr>
<td>plain</td>
<td>10</td>
<td>0.1</td>
<td>0.595</td>
<td>0.88</td>
<td>10.18</td>
<td>174.51</td>
</tr>
<tr>
<td>plain</td>
<td>10</td>
<td>0.5</td>
<td>0.600</td>
<td>0.88</td>
<td>10.34</td>
<td>173.96</td>
</tr>
<tr>
<td>twill</td>
<td>10</td>
<td>0.05</td>
<td>0.576</td>
<td>1.77</td>
<td>9.85</td>
<td>433.47</td>
</tr>
<tr>
<td>twill</td>
<td>10</td>
<td>1</td>
<td>0.584</td>
<td>1.77</td>
<td>9.80</td>
<td>360.82</td>
</tr>
<tr>
<td>5 HS</td>
<td>10</td>
<td>0.05</td>
<td>0.619</td>
<td>1.77</td>
<td>9.09</td>
<td>144.40</td>
</tr>
<tr>
<td>5 HS</td>
<td>10</td>
<td>1</td>
<td>0.623</td>
<td>1.77</td>
<td>9.10</td>
<td>136.44</td>
</tr>
</tbody>
</table>
5.6 DISCUSSION

The results of the compression of an 'original' assembly of plain weave plies oriented at 0° (warp yarn to warp yarn and weft yarn to weft yarn) showed no significant difference in performance to that of an original assembly of plies stacked in a 0°/45° sequence. The reason why this may be the case is due to the way in which the individual plies of the assemblies were laid up. For an assembly oriented at 0° the warp and weft yarns of one ply were aligned as best as possible to that of the warp and weft yarns of the ply directly below. If this was done perfectly the peaks and troughs from one ply could nest with the peaks and troughs of the plies directly above and below. In this case a high degree of nesting between plies would occur when pressure is applied to the assembly. However, if the orientation of an individual ply differs by even a few degrees from the one above or below the chances of matching peaks and troughs from one ply to the next become very slim. This second case results in a far lower degree of nesting between plies on compression of the assembly.

For the assembly oriented at 0° no attempt was made to match undulations in the fabric, only to align warp and weft yarns as best as possible. Also as the assembly lay-up was carried out by hand it is inevitable that there is going to be some degree of misalignment between the plies. The result then, on the compression of the assembly oriented at 0° is the second case, a low degree of nesting. The assembly oriented at 0°/45° does not pretend to match undulations in the fabric between plies and therefore may result in a similar degree of nesting when compression takes place. This then explains why the difference in fibre volume fractions between the assemblies oriented at 0° and the assemblies oriented at 0°/45° is on average only 0.019.

Compression of assemblies of dry plain weave plies that had been previously compressed and reassembled (following the procedure outlined in section 3.5.1) resulted in data comparable to the compression of the original assembly of plies where the fibre volume fractions at 0.88 MPa differed by a maximum of 0.0188. This would seem to indicate that there is no permanent deformation of the plies due to repeated loading (up to 0.88 MPa) and that they undergo complete elastic recovery after they have been reassembled, for at least three compression cycles.
The elastic behaviour of the plain weave dry reinforcement is further illustrated by the compression of original assemblies at different compression speeds ranging between 0.05 and 1.0 mm/min. From the results there is no significant change in the final fibre volume fractions by varying the compression speed where the maximum difference was only 0.0085. Similarly compression tests on assemblies of dry twill and 5 harness satin weave at compression speeds of 0.05 and 1.0 mm/min showed no significant difference in final fibre volume fractions to a pressure of 1.77 MPa.

On the other hand the existence of the hysteresis loops indicates that strain energy is being stored in the fibres as they are deformed in the nesting between consecutive layers of fibre reinforcement on compression. This is in agreement with previous studies on the compression of fibre assemblies [Carnaby et al (1989)]. When the pressure is released the fibres are allowed to recover. However the strain energy is not enough to overcome all the frictional restraints of the fibre-fibre contacts of the nested fabric which results in the assembly being unable to return to its original thickness. Subsequent loading increases the amount of nesting between fibres and the amount of fibre-fibre contacts, reducing the assembly thickness still further. As the assembly tends toward its maximum packing capacity the amount of fibre rearrangement and nesting is greatly reduced. This limits the amount of fibre deformation possible and hence the amount of stored strain energy, which in turn decreases the size of subsequent hysteresis loops.

According to Equation (3.2), regardless of the number of dry plies in an assembly, if two assemblies with different numbers of plies are compressed by the same percentage thickness their final fibre volume fractions will be the same. If this is the case, and, if the compression of dry fabric reinforcement is to follow the power-law (Equation (3.4)) then does the number of plies within an assembly affect the values for the power law constants $\alpha$ and $b$? Some typical values determined from compression testing are shown in Table 5.2 where $b = 10.22, 10.20$ and 10.48 for assemblies of 5, 10 and 20 glass plain weave plies respectively. This would suggest that $b$ is unaffected by the number of plies within an assembly of the same reinforcement type. This further suggests then that applying the same pressure to two assemblies, containing different numbers of plies, but consisting of the same plain woven reinforcement, will result in the same final fibre volume fraction for each assembly. Using the
examples from Table 5.2 the difference in final fibre volume fraction of assemblies of 5, 10 and 20 plain weave plies is 0.024, well within the repeatability experimental error.

However, this does not hold true for the compression of assemblies of less than 5 plies. The results have shown (see Table 5.1) that there is a relatively large difference in the fibre volume fractions of assemblies containing less than 5 plies. This is likely to be due to the nesting effects of the plain weave reinforcement on compression. Comparison of the apparent ply thickness’ show a decrease with increasing number of plies up to a total of 5 plies. The apparent ply thickness for an assembly of two plies is substantially less than for that of a single ply as two of the fabric surfaces are able to rearrange and nest during compression. As the number of plies increases so does the opportunity for a higher percentage of the assembly to nest and hence decrease the apparent ply thickness. However from the results it can be seen that the edge effects become negligible with 5 or more plies in an assembly and both the apparent ply thickness and the final fibre volume fraction stabilise.

Pearce and Summerscales (1995) also carried out compression experiments on assemblies of dry glass plain weave reinforcement with between 1 and 5 plies. They also used an Instron 1175 universal testing machine to compress the assemblies up to a maximum pressure of 0.3 MPa. They however reported the opposite situation, that the apparent ply thickness increased with an increasing number of plies. This was explained by suggesting that the frictional forces between fabric layers would prevent the plies from nesting and therefore each additional ply would contribute to a greater apparent ply thickness than the single ply in isolation. These results seem unlikely as the high compression pressures are more than likely overcome the frictional forces between plies. One explanation for these results may be the small degree of nesting that might have been associated with the type of plain weave fabric tested by Pearce and Summerscales (1995).

The value for $b$ determined from regression analysis for the plain weave reinforcement compressed to 0.88 MPa was on average 10.2 and irrespective of the number of plies (between 5 and 20) or the compression rate (between 0.05 and 1.0 mm/min). Average values of $b$ for the twill and 5 harness satin weave again irrespective of compression rate were 9.8 and 9.1 respectively. Toll and Manson (1994) concluded from theoretical analysis that when fibres are
planarly oriented (dispersed non-bundled straight fibres) compression follows the power law with $b=5$; when fibres are assembled into bundles planarly orientated $b$ could be any value greater than 3. Gauvin and Chibani (1988), Quinn and Randall (1990) and Kim et al. (1991) fitted their experimental data for weaves to the power law with suggested values for $b$ of 7, 9 and 11 respectively. Pearce and Summerscales (1995) determined experimentally a range of values of $b$ between 4.8 and 8.8 for assemblies of glass plain woven fabric with different numbers of layers ranging between one and five.

The results of the compression testing have shown that dry woven reinforcement undergoes nonlinear elastic deformation when a pressure, normal to the assembly, is applied. The fibre volume fraction is unaffected by ply orientation or compression speed between 0.05 and 1.0 mm/min. No permanent deformation of the plies was observed for assemblies loaded (up to 0.88 MPa), reassembled and loaded once more for up to three compression cycles. However, consecutive compression tests on the same assembly of plain weave reinforcement, that has not separated and reassembled between compression tests, results in a hysteresis history which increases the fibre volume fraction of the assembly with each successive loading cycle. Values for the power-law exponent $b$ have been determined for plain, twill and 5 harness satin weaves. What seems apparent from the results of this study and previous studies by other investigators is that the number of plies, when greater than 5, within an assembly and the compression rate do not affect values for $b$. $b$ is dependent on factors such as the type of weave (plain, satin, etc.), weave specification including degree of crimp and material type amongst others. Thus if an accurate database of compression data is to be established then separate experimentation will need to be carried out for different types of fabric reinforcement.

Such compression data will be very useful, primarily in the case of RTM. As the mould will need to be closed to a specified moulding thickness, the required clamping pressure (and hence clamping force) can be calculated from compression graphs. The knowledge of clamping pressure is important for the stage of resin injection. If the injection pressure of resin exceeds the clamping pressure, local deformation of the reinforcement is expected at the gate which may result in resin flowing over the reinforcement rather than through it [Trevino et al. (1991), Parnas et al. (1996)].
6. COMPRESSION TESTING OF RESIN IMPREGNATED REINFORCEMENT
6.1 INTRODUCTION

The purpose of the compression testing of resin impregnated reinforcement is to simulate the compression of assemblies of fabrics in autoclave processing or the compression of reinforcement impregnated with binder in the preforming stage of RTM. The majority of the assemblies of resin impregnated plies that were tested were allowed to cure under pressure in the Instron universal testing machine. The reason for this was twofold. Firstly the final thickness of a cured laminate could be easily obtained by removing the laminate from the Instron testing machine and measuring the final thickness with a micrometer screw gauge. In this way the final thickness of the laminate was absolute without any sources for error. The second reason for curing the assemblies in-situ was to allow for microstructural examination of the laminates at a later stage.

Some of the compression experiments did not require the final laminates for microstructural examination. In these cases non-curing resin (resin without the initiator) was used to impregnate the layers of reinforcement so that no excessive time was consumed by having to allow the laminate to cure in-situ. The resin used for this type of compression test was the polyester Crystic 471 without the addition of the initiator ‘Catalyst M’.

The tests include investigations of the effects of the following parameters on compression: pressure, compression rate, number of plies, repeated loading cycles, type of resin and type of fabric. Three resins were investigated: polyester Crystic 471, polyester Crystic 781 and epoxy Araldite LY 564. Four types of glass fabrics were tested: plain weave (YO212), twill weave (YO185), 5 harness satin weave (YO227) and a noncrimped stitch-bonded fabric.

6.2 REINFORCEMENT IMPREGNATED WITH NON-CURING RESIN

Compression experiments were performed on several assemblies of plain weave plies impregnated with non-curing polyester Crystic 471 resin where the rate of compression was varied between 0.05 and 1.0 mm/min. This was to establish whether or not the reduction of compression speed had any effect on the fibre volume fraction of the laminate.
Compression tests were performed on separate assemblies of twenty plies at three different compression speeds, 0.05, 0.1 and 1.0 mm/min. The final pressure for each of the tests was 0.88 MPa. Figure 6.1 shows the compression curves for the three tests compared to a compression curve for twenty dry plies compressed to the same target pressure, 0.88 MPa, at a compression speed of 1.0 mm/min.

![Compression Test Curves](image)

**Figure 6.1** Experimental data for the compression of assemblies of 20 non-curing polyester resin (Crystic 471) impregnated plies at compression speeds of 0.05, 0.1 and 1.0 mm/min compared to an assembly of 20 dry plies compressed at 1.0 mm/min.

The curve corresponding to dry reinforcement is generally located at higher fibre volume fractions for the same value of pressure. This is expected since all the applied pressure is transferred to the fibres. The curves corresponding to the resin impregnated reinforcement are shifted to lower fibre volume fractions for the same values of pressure. This is justified by the fact that the applied pressure is now split between a component transferred to the fibres and a pressure component utilised for resin flow. Therefore, in reality less pressure is available for the compression of fibre reinforcement. As the compression rate is increased the pressure component available for resin flow (see Equation 2.15) increases, resulting in further shifts of the curve to lower fibre volume fractions.
6.3 REINFORCEMENT IMPREGNATED WITH CURING RESIN

Identical assemblies of resin impregnated plies (resin with initiator or curing agent) were compressed to a range of predetermined maximum target pressures and allowed to cure under pressure. With the aid of optical microscopy, each of the cured laminates was then examined in turn to collect information on the microstructure of the laminate at each stage of compression.

Compression tests were firstly carried out in this way on the assemblies of plain weave reinforcement under a variety of conditions. Tests were conducted on assemblies of one, ten and twenty resin impregnated plies to a range of maximum pressures between 0.088 and 1.77 MPa. Three different resins were used; two types of polyester, Crystic 471 and Crystic 781, and an epoxy, Araldite LY 564. The second stage of compression testing consisted of impregnating assemblies of twill weave, 5 harness satin weave and noncrimped stitch-bonded glass fabric with polyester, Crystic 471 PALV, and compressing them to a series of maximum target pressures between 0.88 and 1.77 MPa.

Table 6.1 shows the mean fibre volume fractions resulting from resin burn-off tests of the plain, twill and 5 harness satin weave fabrics compared to the fibre volume fractions obtained from Equation (3.2). The maximum difference in fibre volume fraction between the two methods is only 0.0082 validating the use of Equation (3.2) as an accurate means of determining laminate fibre volume fractions.

Table 6.1 Comparison of fibre volume fractions obtained from resin burn-off tests and from Equation (3.2), for plain, twill and 5 harness satin weaves.

<table>
<thead>
<tr>
<th>Weave</th>
<th>( V_f ) (from Equation 3.2)</th>
<th>Mean ( V_f ) (from burn-off tests)</th>
</tr>
</thead>
<tbody>
<tr>
<td>plain</td>
<td>0.5729</td>
<td>0.5682</td>
</tr>
<tr>
<td>twill</td>
<td>0.5802</td>
<td>0.5758</td>
</tr>
<tr>
<td>5 harness satin</td>
<td>0.6081</td>
<td>0.6163</td>
</tr>
</tbody>
</table>
6.3.1 PLAIN WEAVE

Compression tests were carried out on assemblies of ten and twenty plies impregnated with polyester resin, Crystic 471 PALV, to a range of target pressures between 0.088 and 1.77 MPa at a compression speed of 1.0 mm/min. Figures 6.2 and 6.3 show the compression curves for tests on ten and twenty resin impregnated plies respectively. Differences between the curves are the result of two effects:

(a) they illustrate the degree of repeatability in these type of experiments.

(b) The fibre volume fraction at each point of every curve was calculated from the corresponding laminate thickness for the pressure at that point. Thickness values however, were obtained only at the maximum target pressure of every curve by measuring the thickness of the cured laminate. Laminate thicknesses after curing were found to be smaller than thicknesses before curing, due to resin contraction. According to the methodology followed in the calculation however, the thickness at the intermediate points of every curve were evaluated from the final thickness of the cured laminate, the compression rate and the compression time. Therefore a certain error is involved due to the resin contraction of the cured laminate. This error is translated to differences between the resulting curves corresponding to different maximum pressures.

In Figure 6.1 assemblies of non-curing resin impregnated plies compressed at different speeds, 0.05, 0.1 and 1.0 mm/min, were compared to the compression of an equivalent assembly of dry plies at 1.0 mm/min. The data obtained for the compression of twenty resin impregnated plies at a speed of 1.0 mm/min which had been allowed to cure was also added to this set of compression curves in Figure 6.4. As can be seen from the graph the cured assembly has a higher fibre volume fraction than that of the equivalent assembly impregnated with the non-curing resin and compressed at the same speed of 1.0 mm/min, due to resin shrinkage. A maximum difference in the final fibre volume fraction of 0.02 was observed between the non-curing and cured curves corresponding to a resin shrinkage of about 5 % which is expected in polyesters.
Figure 6.2 Plots of pressure versus fibre volume fraction for the compression of assemblies of 10 polyester Crystic 471 impregnated plies to a range of target pressures between 0.088 and 0.88 MPa.

Figure 6.3 Plots of pressure versus fibre volume fraction for the compression of assemblies of 20 polyester Crystic 471 impregnated plies to a range of target pressures between 0.088 and 1.77 MPa.
Compression tests were then carried out on single layers of reinforcement impregnated with the Crystic 471 polyester resin. The range of target pressures used in the tests were identical to the ones used for the compression of assemblies of twenty resin impregnated plies (see Figure 6.3). The compression speed of 1.0 mm/min also remained the same. For each target pressure three compression tests were performed, each time on an original ply, and then allowed to cure under pressure in the Instron testing machine.

*Table 6.2* shows the mean final thickness values obtained for each of the single plies at the different target pressures along with their fibre volume fractions. The apparent single ply thickness was then calculated from the compression of assemblies of twenty resin impregnated plies by dividing the final assembly thickness at each target pressure by the number of plies in the assembly, see Table 6.2. This process was repeated to obtain the apparent single ply thickness for the assembly of ten resin impregnated plies (see Figure 6.2) and all the values...
6. Compression Testing of Resin Impregnated Reinforcement

were then plotted on a graph of pressure versus thickness, see Figure 6.5. All thickness values correspond to cured laminates.

Table 6.2 Comparison of the single ply thickness data obtained from the compression of a single ply in polyester resin (Crystic 471) to that of the apparent ply thickness of an assembly of 20 plies compressed to the same target pressures.

<table>
<thead>
<tr>
<th>Pressure (MPa)</th>
<th>Mean single ply thickness (mm)</th>
<th>Assembly fibre volume fraction</th>
<th>Apparent ply thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.08</td>
<td>0.55</td>
<td>0.479</td>
<td>0.445</td>
</tr>
<tr>
<td>0.26</td>
<td>0.51</td>
<td>0.529</td>
<td>0.403</td>
</tr>
<tr>
<td>0.44</td>
<td>0.50</td>
<td>0.539</td>
<td>0.395</td>
</tr>
<tr>
<td>0.61</td>
<td>0.49</td>
<td>0.555</td>
<td>0.384</td>
</tr>
<tr>
<td>0.88</td>
<td>0.48</td>
<td>0.573</td>
<td>0.372</td>
</tr>
<tr>
<td>1.77</td>
<td>0.46</td>
<td>0.626</td>
<td>0.341</td>
</tr>
</tbody>
</table>

Figure 6.5 Plots of pressure versus thickness comparing compressed single ply thickness to that of the apparent single ply thickness from the compression of assemblies of 10 and 20 polyester resin (Crystic 471) impregnated plies.
As one would expect both the apparent ply thickness of the assemblies of ten and twenty plies and the mean ply thickness from the compression of a single impregnated ply decrease with increasing pressure. However, whilst the apparent ply thickness for the assemblies of ten and twenty plies are very similar, the mean ply thickness for the compression of the single plies are on average 28% thicker. Nesting effects in the assemblies of 10 and 20 plies are most likely to be the reason for this difference as in the case of compression of dry reinforcement in Chapter 5.

Throughout the compression testing of assemblies of resin impregnated plies an interesting phenomenon was observed. Once the target pressure of a compression test had been reached the crosshead on the Instron testing machine was stopped. The pressure on the reinforcement continued to be monitored and instead of the pressure remaining constant it began to decrease (stress relaxation phase) and in some cases it reached zero. To investigate this effect further a compression test was conducted on an assembly of twenty resin (polyester Crystic 471) impregnated plain weave plies. The assembly was initially compressed to a target pressure of 0.84 MPa whereupon the crosshead was stopped but the pressure on the reinforcement continued to be closely monitored. Once the pressure had fallen to 0.75 MPa the compression of the plies was resumed until the target pressure had once again been reached (0.84 MPa in this case) at which point the crosshead was stopped once again. This procedure was repeated a total of three times. Graphs were plotted for pressure versus fibre volume fraction (see Figure 6.6) and for pressure versus time (see Figure 6.7).

Both figures indicate a viscoelastic response of the resin impregnated laminates and the stress relaxation effect. From Figure 6.6 it can be seen that each loading cycle increases the assembly fibre volume fraction. The initial compression of the assembly to the target load of 0.84 MPa resulted in an assembly fibre volume fraction of 0.5791. After the three loading cycles had been completed the assembly fibre volume fraction had increased to 0.5842. It was also observed that the time taken for the pressure to fall from 0.84 to 0.75 MPa increased with each loading cycle (see Figure 6.7). On the first loading cycle it took 67.2 seconds for the pressure to reduce from 0.84 to 0.75 MPa compared to 5992.8 seconds for the same pressure reduction after the third loading cycle, thus the relaxation time increases with each subsequent loading cycle.
6. Compression Testing of Resin Impregnated Reinforcement

Target pressure = 0.84 MPa
Crosshead speed = 0.5 mm/min

Number of plies = 20

Figure 6.6 Plots of pressure versus fibre volume fraction for the compression of an assembly of 20 polyester resin (Crystic 471) impregnated plies that has undergone repeated loading cycles.

Figure 6.7 Plots of pressure versus time for the compression of an assembly of 20 polyester resin (Crystic 471) impregnated plies that has undergone repeated loading cycles.
It was also observed from the compression experiments on assemblies of ten and twenty resin impregnated plies that the time taken to reach the maximum target pressure increases with increasing numbers of plies (see Table 6.3). This is expected since both assemblies were compressed at the same rate but had different initial thicknesses.

<table>
<thead>
<tr>
<th>Target pressure (MPa)</th>
<th>Time taken to reach target pressure</th>
<th>Time increase (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10 plies</td>
<td>20 plies</td>
</tr>
<tr>
<td>0.18</td>
<td>16.8</td>
<td>40.8</td>
</tr>
<tr>
<td>0.27</td>
<td>26.4</td>
<td>60.0</td>
</tr>
<tr>
<td>0.35</td>
<td>34.8</td>
<td>73.2</td>
</tr>
<tr>
<td>0.44</td>
<td>40.8</td>
<td>82.8</td>
</tr>
<tr>
<td>0.53</td>
<td>45.6</td>
<td>91.2</td>
</tr>
<tr>
<td>0.62</td>
<td>49.2</td>
<td>98.4</td>
</tr>
<tr>
<td>0.71</td>
<td>54.0</td>
<td>104.4</td>
</tr>
<tr>
<td>0.80</td>
<td>56.4</td>
<td>109.2</td>
</tr>
</tbody>
</table>

The time increase percentages in Table 6.3 represent the percentage increase in the time taken to reach the target pressure for an assembly of twenty plies compared to that of an assembly of ten plies. From these results the time taken to reach the target pressure for an assembly of twenty plies is on average 2.09 times longer than that for an equivalent assembly of ten plies.

As mentioned previously in section 3.2.2 two other resins were used for the manufacture of some of the glass fibre laminates, another polyester, Crystic 781 PA, and an epoxy, Araldite LY 564. Both of these resins have similar viscosities at the working temperatures used in these experiments. Crystic 781 was used at room temperature with a viscosity of 200-300 mPa s and the Araldite LY 564 was used at 40 °C also with a viscosity 200-300 mPa s. This range of viscosity is lower than the viscosity of polyester Crystic 471.

Assemblies of twenty polyester resin (Crystic 781) impregnated plain weave plies were compressed to a variety of target pressures between 0.088 and 0.88 MPa at a compression speed of 1.0 mm/min. Figure 6.8 shows the corresponding compression curves for pressure versus fibre volume fraction.
Figure 6.8 Plots of pressure versus fibre volume fraction for the compression of assemblies of 20 polyester resin (Crystic 781) impregnated plies to a range of maximum target pressures between 0.088 and 0.88 MPa.

Similarly assemblies of twenty epoxy resin (Araldite LY 564) impregnated plain weave plies were compressed to a variety of target pressures between 0.088 and 1.77 MPa at a compression speed of 1.0 mm/min. Figure 6.9 shows the corresponding compression curves for pressure versus fibre volume fraction.

As both the Crystic 781 and the Araldite LY 564 had considerably lower viscosities than that of the Crystic 471 there was a need to examine the effect this may have on the fibre volume fractions of the compressed assemblies. Therefore a comparison was made between the compression data obtained from the testing of assemblies with the Crystic 471 matrix and the compression data from assemblies with the Crystic 781 and Araldite LY 564 matrices. Figure 6.10 shows the compression data for three assemblies of twenty plain weave plies impregnated with Crystic 471, Crystic 781 and Araldite LY 564 respectively and compressed to a maximum target pressure of 0.88 MPa.
6. Compression Testing of Resin Impregnated Reinforcement

**Figure 6.9** Plots of pressure versus fibre volume fraction for the compression of assemblies of 20 epoxy Araldite impregnated plies to a range of maximum pressures between 0.088 and 1.77 MPa.

**Figure 6.10** Plots of pressure versus fibre volume fraction comparing the compression of 3 assemblies of 20 plain weave plies impregnated with Crystic 471, Crystic 781 and Araldite LY 564.
The final fibre volume fraction of the assembly impregnated with polyester Crystic 471 varies with the equivalent assembly impregnated with the polyester Crystic 781 at the target pressure of 0.88 MPa by 0.0126. Similarly, comparison between the final fibre volume fraction of the assembly impregnated with polyester Crystic 471 to the equivalent assembly impregnated with the epoxy Araldite LY 564 shows a variation at the target pressure of 0.88 MPa to be 0.0031.

6.3.2 TWILL WEAVE

Compression tests were conducted on assemblies of twenty twill weave plies impregnated with polyester, Crystic 471, resin. Assemblies were compressed to a range of target pressures between 0.088 and 1.77 MPa at a constant compression speed of 1.0 mm/min (see Figure 6.11).

![Figure 6.11 Plots of pressure versus fibre volume fraction for the compression of assemblies of 20 twill weave polyester resin (Crystic 471) impregnated plies to a range of maximum target pressures between 0.088 and 1.77 MPa.](image-url)
6.3.3 5 HARNESS SATIN WEAVE

Assemblies of twenty 5 harness satin weave plies were impregnated with polyester Crystic 471 and compressed to a range of target pressures between 0.088 and 1.77 MPa at a constant compression speed of 1.0 mm/min (see Figure 6.12).

![Figure 6.12 Plots of pressure versus fibre volume fraction for the compression of assemblies of twenty 5 harness satin weave plies impregnated with polyester resin (Crystic 471) to a range of maximum target pressures between 0.088 and 1.77 MPa.]

6.3.4 NONCRIMPED STITCH-BONDED FABRIC

Assemblies of four layers of reinforcement (each of the layers contained four unidirectional plies oriented at 0°, 45°, -45°, 90°) impregnated with polyester Crystic 471 were compressed to four different maximum target pressures: 0.088 MPa, 0.44 MPa, 0.88 MPa and 1.77 MPa. The four compression curves show excellent repeatability and reach a maximum final fibre volume fraction of 0.6502 at 1.77 MPa (see Figure 6.13).
As a final comparison the compression curves for assemblies of twenty plain, twill and 5 harness satin weaves impregnated with polyester resin (Crystic 471) and compressed to 1.77 MPa were plotted on the same graph of pressure versus fibre volume fraction (see Figure 6.14). The final fibre volume fractions for the plain and twill weaves are very similar at 0.626 and 0.631 respectively. The final fibre volume fraction for the 5 harness satin weave however is noticeably higher at 0.668 which is due to the flatter structure of the fabric, the fact that the resin escapes easily and is not trapped (no macro-pores caused by high crimp) and the lubricating effect of resin which facilitates the compression of satin weave. On the other hand, resin, especially in the plain weave, can be trapped inside the macro-pores (which already exist in this high crimp fabric). The trapped resin cannot escape and shifts the packing fibre volume fraction to higher pressures and lower values of fibre volume fraction.
6. Compression Testing of Resin Impregnated Reinforcement

Maximum pressure = 1.77 MPa
Crosshead speed = 1.0 mm/min
Number of plies = 20

Figure 6.14 Plots of pressure versus fibre fraction for the compression of assemblies of 20 resin (Crystic 471) impregnated plain, twill and 5 HS weave plies to a target pressure of 1.77 MPa.

Figure 6.15 Log-log plots of pressure versus fibre fraction for the compression of assemblies of 20 resin (Crystic 471) impregnated plain, twill and 5 HS weave plies to a target pressure of 1.77 MPa.
The same three compression curves were then plotted on a log-log graph of pressure versus fibre volume fraction (see Figure 6.15) to establish whether or not assemblies of resin impregnated plies would follow the same power law relation as the assemblies of dry plies (see Equation 3.4). By performing a linear regression analysis on the compression data values of $b$ for the plain, twill and 5 harness satin weave were found to be 10.96, 8.81 and 7.28 respectively. However, it is apparent from Figure 6.15 that the data did not fit on a straight line on the log-log plot as well as in the case of the dry reinforcement.

**6.4 DISCUSSION**

It has been shown that compression of assemblies of dry reinforcement at different compression speeds between 0.05 and 1.0 mm/min had no significant effect on the curves of pressure versus fibre volume fraction (see section 5.2). However, this is not the case when compressing assemblies of resin impregnated reinforcement. Comparison of assemblies of dry reinforcement with assemblies of resin impregnated reinforcement that have been compressed to the same target pressure and at the same compression speeds show that the assemblies of dry reinforcement have a higher fibre volume fraction. This is expected as all the applied pressure on the dry reinforcement is transferred directly to the fibres. In resin impregnated reinforcement the applied pressure is split between a pressure component transferred to the fibres and a pressure component transferred to the resin (see Equation 2.15). In this equation the pressure component producing the elastic deformation of fibres depended on the fibre volume fraction, $V_f$, whereas the pressure component causing resin flow depended not only on $V_f$, but was also proportional to the change of fibre volume fraction with time, $V_f$. So at very low compression speeds the viscous pressure component is negligible and the total applied pressure tends to be borne wholly by the fibres. This was illustrated by the compression of assemblies of resin impregnated plies at different compression speeds (see Figure 6.1). As the compression speed was reduced from 1.0 to 0.05 mm/min the $V_f$ increased from 0.561 to 0.586 due to the decrease in the viscous effects and more pressure being transferred to the fibres. This is in qualitative agreement with the results by Gutowski *et al* (1987b) of the compression of oil impregnated unidirectional assemblies of carbon fibre.

A comparison was made between assemblies of twenty plies impregnated with non-curing resin
and compressed at a speed of 1.0 mm/min to that of identical assemblies that had been impregnated with curing resin and compressed at the same speed. The thicknesses of the assemblies impregnated with the non-curing resin were taken as soon as the target pressure had been reached. The assemblies impregnated with the curing resin however, were allowed to cure in-situ under pressure and then the thicknesses measured using a micrometer screw gauge. The thicknesses of the cured specimens were somewhat less than that of the uncured specimens (see Figure 6.4). This is assumed to be due to resin shrinkage during the curing of the laminate. As confirmation a test was performed on an assembly of plies impregnated with curing resin and compressed to a predetermined target pressure. The final thickness of the laminate was then measured before cure using the Instron’s two dial gauges and the dial gauge mounted directly on the crosshead (see section 3.5.2.1). Once the laminate had cured it was removed from the Instron and the thickness measured using a micrometer screw gauge. The results showed that the thickness of the cured specimen was indeed smaller than that of the laminate in its uncured state. This is then responsible for an increase in the final fibre volume fraction on curing of approximately 0.02. The significance of this means that if compression curves (plots of pressure versus fibre volume fraction) of assemblies are calculated based on the data obtained from a cured laminate, then for them to be an accurate representation of the laminate during compression they must be shifted to the left on a fibre volume fraction axis by approximately 0.02. However this is also within the repeatability error of the compression experiments.

In all of the compression experiments involving assemblies impregnated with resin there was the situation that once the target pressure had been reached and all crosshead movement stopped, the pressure acting on the laminate decreased, sometimes to zero.

From the compression curves, Figures 6.2 and 6.3, it can be seen that the increase in fibre volume fraction with each pressure step decreases as the overall fibre fraction increases toward the fibre maximum packing capacity. When the pressure is initially applied to the laminate it is carried by both the uncured resin and the fibres. The continual increase in pressure further compresses the assembly and forces resin out of the system transferring more of the pressure to the fibres. The strain energy within the assembly is translated directly to the individual fibres as bending strain. As the pressure continues to increase there is also an increase in the
distortion of the fibres. This increase in the number and area of fibre-fibre contacts results in an increase in the fibre volume fraction and an increase in bending energy within the fibres. As more and more fibres come into contact with each other the increasing bending strain energy offers increasing resistance to the externally applied pressure. Thus the rate of increase of the fibre volume fraction with pressure slows as it tends toward the optimum packing capacity of the laminate.

Figures 6.6 and 6.7 showed that once the maximum target pressure had been reached and the downward movement of the compression platen stopped, there was an immediate decrease in the pressure acting on the reinforcement. Due to the elastic nature of the fibres, when the compression platen is stopped some of the distorted fibres begin to relax allowing a certain amount of fibre rearrangement to occur. This fibre rearrangement slowly releases the strain energy that has been stored in the system by the compression thus reducing the amount of pressure on the compression platen. As the compression platen has remained stationary during the period of fibre relaxation the fibre volume fraction has remained constant, see Figure 6.6. The pressure in the compression test was allowed to relax to 0.75 MPa at which point the compression of the reinforcement was resumed until the target pressure was reached once again. The result of this was to increase the fibre volume fraction. What is important to note from Figure 6.7 is that each consecutive loading cycle increases the fibre volume fraction which in turn reduces the amount of fibre rearrangement possible. Thus an increase in the fibre volume fraction of 0.0051 resulted in an increase of the relaxation time from 67.2 to 5992.8 seconds.

The Maxwell model [Young and Lovell (1991)] consisting of a spring and dashpot in series can be used to describe the stress relaxation as

\[
\sigma = \sigma_0 e^{-t/\tau_0}
\]  

(6.1)

where \(\sigma\) is the stress, \(\sigma_0\) is the stress at time zero, \(t\) is the time taken to reach \(\sigma\) and \(\tau_0\) is the relaxation time for each cycle. Table 6.4 shows the relaxation times for both the dry and resin impregnated plain weave reinforcement.
Table 6.4 Relaxation times, $\tau_0$, for the loading cycles of assemblies of both dry and polyester resin (Crystic 471) impregnated plain weave reinforcement

<table>
<thead>
<tr>
<th>$\frac{\sigma}{\sigma_0}$ = 0.89</th>
<th>resin impregnated reinforcement</th>
<th>dry reinforcement</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\tau_{01}$</td>
<td>$0.58 \times 10^3$ secs</td>
<td>$1.40 \times 10^3$ secs</td>
</tr>
<tr>
<td>$\tau_{02}$</td>
<td>$6.59 \times 10^3$ secs</td>
<td>$17.10 \times 10^3$ secs</td>
</tr>
<tr>
<td>$\tau_{03}$</td>
<td>$51.43 \times 10^3$ secs</td>
<td></td>
</tr>
</tbody>
</table>

*Figure 6.16* compares the loading cycles for the compression of dry plain weave reinforcement to that of the loading cycles of the polyester resin (Crystic 471) impregnated plain weave reinforcement.

*Figure 6.16* Plots of pressure versus time comparing the compression of an assembly of 10 polyester resin (Crystic 471) impregnated plain weave plies that has undergone repeated loading cycles to that of an assembly of 10 dry plain weave plies under the same loading conditions.
As can be seen from both Table 6.4 and Figure 6.16 the relaxation times are in the order of 2.5 times longer for the assembly of dry plies under pressure than for the resin impregnated plies. Earlier work has shown from the compression hysteresis history (see Figure 5.10) that continual reloading of an assembly of dry reinforcement does not allow the complete recovery of the fibres even when the applied pressure is returned to zero. This, in association with Figure 6.16, indicates viscoelastic behaviour of the dry fibre reinforcement, regarding pressure relaxation and hysteresis effects. On the other hand earlier work has shown that no permanent deformation of the dry reinforcement occurs up to pressures of 0.88 MPa (see section 5.2) if the layers of fibre reinforcement are separated and reassembled. This implies that the main reason for the viscous pressure relaxation in dry reinforcement must be the nesting mode of compression since nesting is disturbed when the layers of reinforcement are reassembled.

It would appear from the comparison of assemblies of ten and twenty plain weave plies impregnated with the polyester Crystic 471 matrix, that compressing them to the same target pressure results in the same fibre volume fraction. If this were the case, a comparison of the apparent ply thicknesses of both assemblies of ten and twenty plies should result in the same values. As can be seen from Figure 6.5 in the results, the apparent ply thickness values are very close, within 0.014 mm. If these values are normalised with the compressed thickness of the assembly of twenty plies this is a difference of only 2.7%. What Figure 6.5 also shows is the comparison of the apparent ply thicknesses of assemblies of ten and twenty plies with the final thickness of a single resin impregnated ply compressed to the same target pressures. What is immediately apparent is that the single ply thicknesses are much larger than that of the apparent ply thicknesses for the assemblies of ten and twenty plies. Using the same normalisation procedure, the single ply thicknesses are between 24 and 35% larger. The reason for this difference is that within an assembly of multiple layers of reinforcement there will be a certain amount of nesting that takes place between plies. When the plies are being laid up to form an assembly there is little chance that they will be perfectly aligned from one ply to the next. As a consequence when pressure is applied to the resin impregnated assembly the plies will quickly come into contact with each other allowing the peaks from one ply to nest within the troughs or interstices of the plies directly above and below. The magnitude of the nesting that takes place for a given applied pressure together with the fibre deformation ultimately determines the final thickness of the compressed laminate and hence the apparent ply
It follows then that the amount of nesting is dependent on such parameters as applied pressure, type of reinforcement, reinforcement structure, fibre/fabric orientation and whether or not the fibres are lubricated. One might expect nesting between layers of reinforcement within an assembly of plies consisting of lubricated fibres to be the dominant mode of compression, due to the fact that fibre rearrangement is easier for lubricated fibres because of lower frictional forces on fibre-fibre contact. By examining the microstructure of samples taken from assemblies of resin impregnated plies one should be able to get some idea of the amount of nesting and fibre deformation that takes place and to assess which mode of deformation is dominant (see Chapter 7).

The rate of compression for all the experiments was constant throughout the cycle. This means that for a specific time during the compression tests on the assemblies of ten and twenty resin impregnated plies, the crosshead has moved exactly the same distance. In other words both assemblies have decreased by the same thickness. As a result the volumes of both assemblies are also decreased. However, because the assembly of ten plies has a smaller initial volume than that of the assembly of twenty plies, its percentage volume decrease for the same decrease in thickness is far greater. Consequently the smaller assembly increases in fibre volume fraction far quicker than its counterpart. This then explains why increasing the number of plies increases the time to reach its target load.

There is then a linear relationship between the time taken for each assembly to reach its target load and the number of plies within the assembly. Examining the data, the larger assembly in the compression tests had double the number of layers and should therefore take approximately twice as long to reach the target load. The time taken in fact is on average 2.09 times longer (see Table 6.3).

Three types of resin were used in the manufacture of the laminates, two polyesters - Crystic 471 and Crystic 781 and an epoxy - Araldite LY 564. The Crystic 781 and the Araldite LY 564 had similar resin viscosities, 200-300 mPa s, at the working temperatures used in the compression testing. However the viscosity of the Crystic 471 was much higher, 600-6000
mPa s, when used at 25 °C. One might expect the compression of reinforcement impregnated with the Crystic 471 resin to result in laminates of lower fibre volume fraction due to the increased viscous effects of the resin. The compression curves for assemblies of twenty plain weave plies impregnated with the three different resins and compressed to the same target pressure at the same compression speed were compared in Figure 6.10. As can be seen from the graph the higher viscosity of the Crystic 471 has not resulted in the expected increase of the viscous effects at a compression speed of 1.0 mm/min. In fact the maximum difference in the fibre volume fraction at the target pressure of 0.88 MPa is only 0.003. This would seem to indicate that viscous effects are not significant at a compression speed of 1.0 mm/min and therefore no increase in the viscous pressure component was experienced for Crystic 471. Either a higher viscosity resin or faster compression speed may result in an increase of the viscous effects and hence a lower final fibre volume fraction but this has not been investigated in this study.

Comparisons were also made between the three different woven glass fabrics used in the compression tests, plain, twill and 5 harness satin weave. Figure 6.14 shows the compression curves for laminates constructed from the three fabrics, all with the same number of plies and compressed at the same compression speed of 1.0 mm/min to the same maximum target pressure of 1.77 MPa. The slope of the curve is a good indication of the increase in fibre volume fraction over a given pressure range. A steep curve indicates a lower final build up of fibre volume fraction than a compression curve with a more gentle slope. The compression curve for the plain weave assembly has the steepest slope and also results in having the lowest final fibre volume fraction of 0.626. This is to be expected as the plain weave fabric has a higher crimp than the other two fabrics and the highest potential for resin entrapment in the macro-pores. The compression curve for the 5 harness satin weave has the least steep slope resulting in the highest final fibre volume fraction of 0.668. Again this is to be expected as the 5 harness satin weave has a flatter fabric structure with very low crimp enabling a higher packing capacity and easy resin flow and escape. The compression curve for the twill weave has an intermediate slope indicating a more rapid increase in fibre fraction for increasing pressure when compared to the 5 harness satin weave compression curve but not as large an increase as the plain weave compression curve. Again this is a direct result of the structure of the fabric, less crimp than the plain weave but more crimp than the 5 harness satin weave.
6.5 COMPRESSION OF RESIN IMPREGNATED PLAIN WOVEN CLOTHS: COMPARISON BETWEEN THEORETICAL AND EXPERIMENTAL RESULTS

This next section involves the application of the mathematical models proposed in Chapter 4 and their validation on the basis of the experimental data. The selected case studies include the compression of twenty layers of plain weave fabric, Y0212, (i) dry, i.e. not impregnated with any resin; (ii) impregnated with an epoxy (curing system), Araldite LY 564, and compressed at a speed of 1.0 mm/min.

6.5.1 RESULTS

First the different parts of the model are validated. According to the model the total applied pressure, \( P \), is split between the fibres and the resin as described by Equation (4.4). The pressure component applied to the fibres, \( P_f \), is determined by a power-law relation, Equation (4.6), whereas the pressure component applied to the resin, \( P_m \), is derived from a viscous, Darcy's flow model including deformable porous medium as is described by Equation (4.19) for a Newtonian resin or by Equation (4.28) for a non-Newtonian, power-law resin. The empirical constants \( a \) and \( b \) in Equation (4.6) have been determined from a best fit procedure of data from the compression of 20 layers of dry plain weave fabric compressed at a speed of 1.0 mm/min (see section 5.5), as

\[
\alpha = 169.7 \text{ MPa} \quad \text{and} \quad b = 10.48.
\]

Figure 5.12 shows the curve of best fit and the corresponding experimental data of dry compression.

Equation (4.19) has then been applied for the compression of 20 layers of plain weave fabric impregnated with the epoxy resin to estimate the resin pressure. Since epoxy has been considered as a Newtonian fluid with measured viscosity (see Section 3.3.2), there is no uncertainty about fitting an empirical rheological constant in this case. Equation (4.28) for the
resin pressure of a non-Newtonian fluid (polyester in this study) contains an extra empirical constant, $M$, which depends on both resin rheology and the degree of compression of reinforcement. Equations (4.19) and (4.28) contain the permeability, $K$, of the assembly of woven cloths. In both equations, $K$ has been considered as a function of the fibre volume fraction, $V_f$, according to the Carman-Kozeny relationship [Williams *et al* (1974)]

$$K = \frac{r_f^2}{4k_0} \frac{(1 - V_f)^3}{V_f^2}$$

(6.2)

where $r_f^2/4k_0$ can be determined from experimental data [Lekakou *et al* (1996)] for assemblies of plain weave fabric, Y0212, as $r_f^2/4k_0 = 9.88 \times 10^{-10} \text{ m}^2$.

![Figure 6.17](image_url)

**Figure 6.17** Validation of the model (Equations (4), (6) and (19)) for assemblies of both 20 dry and epoxy resin (Araldite LY 564) impregnated plies.

**Figure 6.17** illustrates that the curve predicted (prediction 1) by the applied model configuration is close to the data of dry compression and that the resin pressure has not raised the curve to the data of wet compression at 1.0 mm/min. This indicates that the predicted resin pressure component at a compression speed of 1.0 mm/min is negligible in comparison to the...
pressure needed for the compression of fibre cloths. As a result, other sources for pressure consumption have been investigated in the configuration of the model. Due to the fact that the results for Equation (4.19) are unsatisfactory it was felt that to pursue with the validation of Equation (4.28) for a non-Newtonian resin, which has two empirical constants rather than one, and therefore more uncertainty, was futile.

6.5.2 DISCUSSION OF THEORETICAL RESULTS

In the comparison between theoretical and experimental results it was concluded that the predicted pressure for the resin flow was insufficient to raise the total pressure to the values of the experimental data. As both Equations (4.19) and (4.28) for the pressure of resin flow contain a value for the in-plane permeability, \( K \), they are to a certain extent dependent on the cross section and the tortuosity of the flow paths in the reinforcement. The tortuosity is defined as \( \left( \frac{L_e}{L} \right)^2 \) where \( L_e \) is the average effective or real distance of the flow path along which the fluid flows and \( L \) is the shortest distance in the direction of flow. Therefore since both the tortuosity and cross section of the flow paths are dependent on the fibre fraction, fibre topology and the dimensions and shape of the fibres the compression of the reinforcement will play an important role in determining the permeability.

From the micrographs of the compression of assemblies of plain weave fabric and from the examination of the microstructure (see Chapters 8 and 7 respectively) there is no evidence to suggest that the tortuosity of the flow paths are greatly increased when the compression of the reinforcement is increased from 88 to 884 kPa. However from area fraction data (see Chapter 8) the cross section of the flow paths are reduced as the porosity area fraction of the reinforcement decreases from 0.23 at 88 kPa to 0.08 at 884 kPa. The result of this would undoubtedly decrease the permeability of the reinforcement.

Work by Lekakou et al (1996a and 1996b) showed in a series of permeability experiments that the Carman-Kozeny relationship (see Equation (6.2)) could be used to describe the dependence of permeability on porosity in plain weave, YO212, fabric (the same fabric that is used in this study). They also concluded that increasing the flow rate increased the in-plane permeability and gave as one of the values of flow rates, \( Q_{in}(r) \), used in the testing, at a fibre volume
fraction of 0.5, to be $6.76 \times 10^{-7}$ m$^2$/sec. Using Equation (4.8) the flow rate of resin through the reinforcement for this study was calculated at the same fibre volume fraction as $Q_m(r)=9.42 \times 10^{-8}$ m$^3$/sec at $r=60$ mm and compression rate of 0.5 mm/min. This is a factor of ten lower than the flow rate obtained by Lekakou et al. (1996a and 1996b) and therefore suggests that the permeability values for this study might actually be lower than the permeability values that they obtained in their experiments. By fitting Equation (4.19) to the experimental data for the compression of 20 layers of epoxy impregnated plies (see prediction 2 in Figure 6.17) the permeability was calculated, assuming the Carman-Kozeny relationship is valid, and indeed found to be lower at $K=5.5076 \times 10^{-12}$ m$^2$. This is a factor of two lower than the permeabilities determined by Lekakou et al. (1996a and 1996b).

It would seem then that for the predicted resin pressure component to match the experimental data the permeability needs to be very small. What also must be considered in this study is that the compression on the reinforcement is continually increasing toward a predetermined target pressure whilst simultaneously the resin flows. This differs from the permeability experiments of Lekakou et al. (1996a and 1996b) where the fibre volume fraction and degree of compression were maintained constant in each experiment during resin flow. So whereas in the permeability experiments of Lekakou the structure of the reinforcement was approximately static, in this study the structure and fibre volume fraction changed dynamically during the compression experiments. This results in a continuous reduction of pore size as the pressure increases and rapidly closes down flow channels which would certainly reduce the permeability. If the resin is unable to find alternative flow paths quick enough it may well become trapped within the remaining pores as the pressure tends towards $P_{\text{max}}$. Thus the entrapment of resin within the dynamically changing pore system would go some way to suggesting how to increase the pressure component of the model. It should be noted that both Darcy’s Law and the Carman-Kozeny relationship apply to quasi steady state flow whereas during the compression of the reinforcement in this study the flow is far from steady because the pore size is continually decreasing and pore channels can close abruptly.

Work by Lekakou et al. (1993) concerned the compression of assemblies of plain woven reinforcement impregnated with a 60% sugar solution of relatively low viscosity, 57 mPa s. A compression test was first carried out at a low compression rate of 0.05 mm/min at which the
viscous resin flow effects are expected to be kept to a minimum and so the compression should resemble the compression of dry reinforcement (see Figure 6.18).

![Compression of assemblies of 20 plain weave plies impregnated with a 60% sugar solution at 5 mm/min and 0.05 mm/min [Lekakou et al (1993)].](image)

Figure 6.18 Compression of assemblies of 20 plain weave plies impregnated with a 60% sugar solution at 5 mm/min and 0.05 mm/min [Lekakou et al (1993)].

A packing fibre volume fraction was observed at approximately $V_f = 0.65$. When the compression rate was raised to 5 mm/min the packing pressure was observed much earlier at approximately $V_f = 0.52$. No such difference is observed in the compression of dry reinforcement when the rate of compression is changed. So, since the sudden rise in pressure cannot be justified from the normally expected packing fraction of dry reinforcement or from the expected pressure for resin flow according to Equation (4.19), it may well be due to resin entrapment in the rapidly changing pore structure. More work is therefore needed to investigate the values for permeability of reinforcement during transient deformation conditions. It must also be mentioned at this point that the relation between permeability and fibre volume fraction in assemblies of fabrics might be affected by the packing fibre volume fraction, in a similar manner as in Equation (2.7) which applies to the transverse permeability of uni-directional fibre assemblies. In this case, the packing fraction will have to change with compression rate according to Figure 6.18, which will also affect the permeability.
7. MICROSTRUCTURAL IMAGE

ANALYSIS OF COMPRESSED LAMINATES
7.1 INTRODUCTION

This chapter presents the results from the microstructural analysis of assemblies of glass plain weave reinforcement. Various geometric parameters of a plain weave fabric have been measured for different degrees of laminate compression. An accurate picture of microstructural data is required if the structure changes of woven fabrics during compression are to be accurately modelled.

In this study a thorough examination of the through thickness microstructure of assemblies of plain weave reinforcements has been performed. Assemblies of plain weave fabric impregnated with resin were compressed at a speed of 1.0 mm/min up to a target pressure and allowed to cure whilst the corresponding target laminate thickness remained constant. Samples were taken from the compressed laminates for microstructural analysis. Figure 7.1 shows a schematic of a cured laminate indicating the co-ordinate geometry of the sample cross-section for the purpose of the microstructural analysis. Comparisons have been made between assemblies impregnated with a polyester resin (Crystic 471) to assemblies impregnated with an epoxy resin (Araldite LY 564).

![Schematic of a cured laminate indicating the cross-section co-ordinate geometry](image)

Figure 7.1 Schematic showing a polished sample taken from the cured laminate indicating the cross-section co-ordinate geometry used in the microstructural analysis.
7.2 PLAIN WEAVE - POLYESTER MATRIX

As a result of the compression testing, samples were taken from a series of cured laminates that had been compressed to a range of target pressures between 0.0044 and 1.77 MPa at a constant compression speed of 1.0 mm/min. Each of the cured laminates consisted of twenty glass fibre plain weave plies assembled warp to warp and weft to weft and impregnated with polyester Crystic 471. After polishing and etching the samples, the through thickness of the sample was examined under an optical microscope before taking photographs of the entire cross-section of the sample. A number of geometrical parameters were then measured including, the height and width of the elliptic warp yarns and the amplitude, wavelength and phase angle of the waveform of the weft yarns (see Figures 3.13 and 3.14).

The first sets of results to be examined were the values obtained for the height, $h$ and width, $w$ of the elliptic warp yarn cross-sections for samples compressed to 0.0044 and 0.88 MPa. Combining this data with the $x$ and $y$ co-ordinates of the centre of each warp yarn cross-section a series of graphs were plotted. Firstly 3-dimensional surface plots were created for $h$ and $w$ with respect to their co-ordinate positions (see Figures 7.2a to 7.5a). Then secondly, spectral plots were made which represented $h$ and $w$ as a series of colourmapped bands, different colours representing the different values for $h$ and $w$, in relation to their position within the sample cross-section (see Figures 7.2b to 7.5b).

Both the 3-dimensional surface plots and the spectral plots are not particularly useful for accurately determining absolute values for either the height or width. However at a glance one is able get a feel for the range of values represented in each case. Comparison of Figures 7.2 and 7.3, which are the plots of height, $h$, shows an approximate range of values between 0.26 - 0.34 mm for the 0.0044 MPa sample and an approximate range of values between 0.21 - 0.30 mm for the 0.88 MPa sample. On first impressions this indicates a decrease in the height of the warp yarn cross-section with increasing pressure up to 0.88 MPa. Again comparing Figures 7.4 and 7.5, which are the plots of the width, $w$, show approximate ranges between 1.04 - 1.26 mm and 1.04 - 1.35 mm for the 0.0044 and 0.88 MPa samples respectively. This would seem to indicate an increase in the width of the elliptic cross-section as the target pressure is increased from 0.0044 to 0.88 MPa.
Figure 7.2 (a) 3-dimensional surface plot (b) spectral plot of the values of height, h, of the elliptic warp yarn cross-sections against the x and y co-ordinates of the centre of each yarn cross-section for a laminate compressed to 0.0044 MPa at a compression speed of 1.0 mm/min.

* Expressed in mm (as measured from the mosaic of photographs)
Figure 7.3 (a) 3-dimensional surface plot (b) spectral plot of the values of height, \( h \), of the elliptic warp yarn cross-sections against the \( x \) and \( y \) co-ordinates of the centre of each warp yarn cross-section for a laminate compressed to 0.88 MPa at a compression speed of 1.0 mm/min.
Figure 7.4 (a) 3-dimensional surface plot (b) spectral plot of the values of width, $w$, of the elliptic warp yarn cross-sections against the x and y co-ordinates of the centre of each warp yarn cross-section for a laminate compressed to 0.0044 MPa at a compression speed of 1.0 mm/min.
Figure 7.5 (a) 3-dimensional surface plot (b) spectral plot of the values of width, \( w \), of the elliptic warp yarn cross-sections against the \( x \) and \( y \) co-ordinates of the centre of each warp yarn cross-section for a laminate compressed to 0.88 MPa at a compression speed of 1.0 mm/min.
The primary purpose of the 3-dimensional surface plots and spectral plots was however to
determine whether or not the deformation of the warp yarn cross-sections was homogeneous
throughout the sample. There was particularly the question of whether the top layers are
compressed to the same degree as the bottom layers. The information that could be obtained
from the plots was that the deformation seemed to be random throughout the specimens with
no obvious patterns or trends appearing.

Further results of the height and width of the warp yarn cross-sections were recorded for the
remaining samples. Results were also recorded for the area of the warp yarn cross-sections.
*Table 7.1* presents the change of the various mean geometric parameters for the pressure range
4.4 to 1768 kPa as measured from the mosaic of the assembled micrographs.

*Table 7.1* Mean geometric parameters for a range of laminates compressed to target pressures
between 4.4 and 1768 kPa. Each laminate consisted of 20 layers of plain weave glass reinforcement
in a polyester Crystic 471 matrix.

<table>
<thead>
<tr>
<th>Pressure $P_{max}$ (kPa)</th>
<th>Height $h$ (mm)</th>
<th>Width $w$ (mm)</th>
<th>Area $A_{x}$ (mm$^2$)</th>
<th>Amplitude $2a$ (mm)</th>
<th>Distance $d$ (mm)</th>
<th>Wavelength $\lambda$ (mm)</th>
<th>Apparent ply thickness (mm)</th>
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<tbody>
<tr>
<td>4.4</td>
<td>0.31</td>
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<td>0.25</td>
<td></td>
<td>2.98</td>
<td>0.341</td>
</tr>
</tbody>
</table>

Figures 7.6 and 7.7 present the size distributions of the minor and major axes (height and
width), respectively, of the cross-sections of the warp yarns at different degrees of
compression. If flattening of the yarns occurred as the compression pressure increased, the
distribution of the minor axis would be expected to shift respectively to lower values whereas
the distribution of the major axis would display a corresponding shift to higher values.
Figure 7.6 Size distributions of the minor axis, h, of the cross-section of plain weave warp yarns at different degrees of compression.
Figure 7.7 Size distributions of the major axis, \( w \), of the cross-section of plain weave warp yarns at different degrees of compression.
As is shown in Table 7.1 a decrease in the mean value of the minor axis is observed as the compression pressure is first raised from 4.4 to 88 kPa. Figure 7.6 also shows a noticeable shift in the size distribution to lower values for the minor axis in this first compression step indicating some warp yarn compression. However, as is shown in Table 7.1 there is not a corresponding increase in the size of the major axis when the compression pressure is increased from 4.4 to 88 kPa but in fact a slight decrease. It also corresponds to an area decrease for the yarn cross-section for the pressure rise from 4.4 to 88 kPa. This could be attributed to various reasons:

(i) this first step of compression is expected to correspond to considerable nesting between layers of reinforcement during which the yarn cross-sections are squeezed;

(ii) The warp yarns might not be exactly normal to the sectioning plane and errors might have occurred due to possible small changes of yarn orientation as the layers of reinforcement are compressed;

(iii) there is a wide statistical variation of structural parameters within the examined specimens.

Not until the compression pressure is raised to 442 kPa is there a clear shift of the size distribution of the major axis of yarn cross-section to higher values (see Figure 7.7). A definite increase of the major axis is observed for an increase of the compression pressure from 884 to 1768 kPa which amounts to a noticeable shift of the size distribution to higher values. Similarly over the same pressure range there is a small decrease of the minor axis illustrated by a skewing to lower values of the size distribution. Both of these results imply that at high pressures there is a definite level of compression and flattening of warp yarn cross-sections.

The mean cross-sectional area, $A_{w}$, of the warp yarns experiences a first decrease as the compression pressure is increased from 4.4 to 88 kPa and a second decrease as the pressure is further raised from 265 to 442 kPa. Figure 7.8 presents the cross-sectional area distribution curves for the warp yarns. A wide cross-sectional area distribution is observed at $P_{\text{max}} = 4.4$ kPa, indicating the wide statistical variation of structural parameters in the layers of fabric.
Figure 7.8 Size distributions of the cross-sectional area, $A_{wy}$ (mm$^2$) of plain weave warp yarns at different degrees of compression.
when they are laid up under minimum pressure. The distribution becomes narrower and is shifted to smaller values of area as the pressure is increased to 442 kPa. While the average value for the area does not change above this pressure, a certain degree of skewing of the distribution curve toward lower values of $A_w$ can be seen as the pressure is increased to 1768 kPa. The overall results indicate a stepwise compaction of fibres within the yarns as the compression pressure is raised corresponding to an increase of the fibre volume fraction inside the yarns.

The yarn waveform was considered sinusoidal, described by the relation

$$y = a \sin \left( \frac{2\pi x}{\lambda} + \phi \right)$$  \hspace{1cm} (7.1)

The mean amplitude of the yarn waveform decreased from $2a=0.31$ mm under a compression pressure of 4.4 kPa to $2a=0.25$ mm under a compression pressure of 1768 kPa, as shown in Table 7.1. From the examples of the distributions of the amplitudes in Figure 7.9 one can see a definite shift to lower values as $2a$ decreases with increasing $P_{\text{max}}$. This is consistent with observations of the current study of literature [Yugartis et al (1992) and (1995)] made on the samples where the waveform of the weft yarns became deformed and changed crimp angles with increasing pressure.

With decreasing amplitude one might expect a comparable increase in the wavelength as the weft yarn waveform flattens. However no trend could be concluded in the change of the wavelength of the yarn waveform for increasing maximum compression pressures (see Table 7.1 and Figure 7.10). In general there was a wide distribution of wavelengths as shown in Figure 7.10. The wavelength values seemed to remain approximately the same over the increasing pressure range. The numerous resin rich areas of the laminate at lower compression pressures allow the yarn waveforms to nest together in the through thickness direction comparatively easily. The nesting process allows the waveform to compress and, hence, the amplitude decreases. On the other hand if the waveform wavelength is to increase in the in-plane direction it must travel a tortuous path due to the high degree of nesting. Due to the large levels of friction caused by the numerous fibre-fibre contacts during nesting and waveform deformation, the chances of any waveform expansion were highly unlikely. In this
Figure 7.9  Size distributions of the amplitude (2a) of the waveform of plain weave weft yarns at different degrees of compression.
Figure 7.10 Size distributions of the wavelength of the waveform of plain weave weft yarns at different degrees of compression.
situation then where an increase in the yarn waveform is not possible but the amplitude is still decreasing one must expect either, a certain amount of deformation and flattening of the peaks of the waveform, or the waveform is shifting into available interstices in the direction of the warp yarns (i.e. transverse to the direction of the weft yarn waveform), or a combination of both. There is evidence from the micrographs to suggest that flattening of the waveform peaks is indeed occurring. However due to the opaque nature of the cured laminates any optical examination to determine movement of the waveform in the warp yarn direction proved fruitless and is thus open to speculation.

Unfortunately not enough measurements of the phase angle, $\phi$, could be taken, that included all twenty plies from the micrographs (see Appendix I), to plot representative distribution curves. From the data that was obtained there was evidence that the phase angle between plies followed a random distribution under all compression pressures indicating that the cloths were laid up at random phase differences between each other.

*Figure 7.11* presents the changes in the apparent ply thickness, mean ply thickness and the average distance between plies for laminates that have been compressed to different maximum target pressures. The apparent ply thickness, $H_{apparent}$, is calculated by dividing the thickness of a laminate by the number of plies it contains. The distance, $d$, between consecutive plies is defined as the distance between consecutive midpoint lines each of which is formed by drawing a line of best fit through the midpoints of each weft yarn centreline (see *Figure 3.14*). The ply thickness, $H_{ply}$, is taken from the microstructural measurements as

$$H_{ply} = \max (2h, 4a)$$

(7.2)

The difference between $H_{ply}$ and $H_{apparent}$ is that $H_{ply}$ is a measure of the actual ply thickness whereas, as mentioned previously, $H_{apparent}$ is determined by dividing the laminate thickness by the number of plies. This means that the value of $H_{apparent}$ will be dependent on the presence of any resin rich interlayers or nesting between the plies.

A comparison between the mean ply thickness from the microstructural data and the mean distance between plies gives further indications about certain modes of compression. In
theory, if a number of dry cloths were perfectly stacked (see Figure 7.12a), warp to warp and weft to weft, at $\phi = 0$, and no applied pressure, $d$ should be equal to $H_{piy}$.

If $H_{piy}$ is smaller than $d$ a resin rich layer should exist between plies (see Figure 7.12b) whereas if $H_{piy}$ is larger than $d$ the plies have nested with each other without a resin free layer. In this case it seems that nesting generally occurs as is shown in Figure 7.11. A certain amount of nesting occurs on average between consecutive fabric layers even at low compression and as the compression increases the amount of nesting increases since the change in $d$ is greater than the change in $H_{piy}$. Most of the nesting seems to have been completed at a pressure of around 0.8 MPa since the difference between $d$ and $H_{piy}$ does not significantly change thereafter. There is some evidence from the micrographs (see Figure 7.13) that indicates a resin rich layer may be occasionally present between two plies at the low compression pressure of 4.4 kPa although, as Figure 7.11 shows, it is not present on average.
7.3 PLAIN WEAVE - EPOXY MATRIX

As mentioned previously in section 6.3.1, compression experiments were performed on assemblies of plain weave plies impregnated with Araldite LY 564, an epoxy resin. Comparisons were made between compression curves for assemblies impregnated with polyester Crystic 471 and the epoxy Araldite LY 564 in order to examine the viscous effects of the two resins. Initial results showed that there was no significant difference in the characteristics of the compression curves (see Figure 6.10, section 6.3.1). The next stage was to refine the investigations into the microstructural parameters for compressed epoxy and polyester laminates. Therefore as a comparison with corresponding polyester laminates,
Figure 7.13 Imaged processed sections of cured specimens of assemblies of plain weave fabric, impregnated with polyester Crystic 471, compressed to various degrees of compression: (a) 4.4 kPa (b) 88 kPa (c) 265 kPa (d) 442 kPa (e) 884 kPa (f) 1768 kPa.
samples were sectioned from manufactured laminates consisting of assemblies of plain weave reinforcement impregnated with the epoxy resin that had been compressed to three target pressures, 0.088, 0.44 and 0.88 MPa.

Initially the height, \( h \) and width, \( w \) of the elliptic warp yarn cross-sections were recorded for the three samples. Figures 7.14 and 7.15 illustrate the size distributions for the minor and major axis (height and width) of the warp yarn cross-sections respectively. As is shown in Table 7.2 there is a decrease in the mean value of the minor axis as the compression pressure is increased from 88 to 442 kPa.

<table>
<thead>
<tr>
<th>Pressure ( P_{\text{max}} ) (kPa)</th>
<th>Polyester (Crystic 471)</th>
<th>Epoxy (Araldite LY 564)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Height ( h ) (mm)</td>
<td>Width ( w ) (mm)</td>
</tr>
<tr>
<td>88</td>
<td>0.290</td>
<td>1.154</td>
</tr>
<tr>
<td>442</td>
<td>0.282</td>
<td>1.169</td>
</tr>
<tr>
<td>884</td>
<td>0.289</td>
<td>1.193</td>
</tr>
</tbody>
</table>

This reduction is confirmed by Figure 7.14 which shows a shift to lower values of the size distribution of the minor axis. For the same pressure increase there is a corresponding increase in the major axis of the warp yarn, see Table 7.2, and there is a noticeable shift to higher values of the size distribution, see Figure 7.15. The compression pressure increase from 442 to 884 kPa shows a much smaller decrease in the mean value of the minor axis than for the previous step. This is illustrated by the skewing, rather than a shift, of the minor axis size distribution to lower values. Concurrently there is only a slight increase in the major axis dimensions resulting in a skew of the size distribution to higher values. The data then indicates compression of the warp yarn as the compression pressure is increased from 88 to 884 kN as expected. A comparison between epoxy and polyester laminates in Table 7.2 yields a certain absolute difference between the respective parameters at all pressure levels, including the low pressure of 88 kPa. This is first expected as a result of the statistical deviation of the values of these parameters.
Figure 7.14 Size distributions of the minor axis, $h$, of the cross-section of plain weave warp yarns at different degrees of compression. Samples taken from assemblies of plain weave reinforcement impregnated with epoxy, Araldite LY 564.
Figure 7.15 Size distributions of the major axis, w, of the cross-section of plain weave warp yarns at different degrees of compression. Samples taken from assemblies of plain weave reinforcement impregnated with epoxy, Araldite LY 564.
parameters in fabrics. On the other hand, the first step of yarn deformation at a pressure change from 88 to 442 kPa is higher in epoxy than in polyester laminates. So it can be concluded at this stage that the yarn cross-section can deform a little easier in reinforcement impregnated with epoxy Araldite LY 564 than with the polyester Crystic 471, possibly due to the lower viscosity of epoxy. However, this mode of deformation is not very significant in the compression mechanism at low pressure levels, where nesting generally dominates. So the difference is not reflected in the general compression curves.

Further investigation of the microstructure was carried out by examining the amplitude and wavelength data for the samples compressed to target pressures of 88 and 884 kPa. The mean amplitude (2a) of the yarn waveform exhibited a decrease of 0.01 mm as the pressure increased from 88 to 884 kPa, as shown in Table 7.3. This is consistent with the amplitude decrease seen in the assemblies impregnated with the polyester Crystic 471.

<table>
<thead>
<tr>
<th>Pressure (kPa)</th>
<th>Polyester (Crystic 471)</th>
<th></th>
<th>Epoxy (Araldite LY 564)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Amplitude 2a (mm)</td>
<td>Wavelength λ (mm)</td>
<td>Amplitude 2a (mm)</td>
<td>Wavelength λ (mm)</td>
</tr>
<tr>
<td>88</td>
<td>0.27</td>
<td>2.95</td>
<td>0.26</td>
<td>2.83</td>
</tr>
<tr>
<td>884</td>
<td>0.26</td>
<td>2.89</td>
<td>0.25</td>
<td>2.82</td>
</tr>
</tbody>
</table>

As discussed previously in this chapter (see section 7.2) the mean wavelength data resulting from the compression of plain weave assemblies impregnated with polyester Crystic 471 remained approximately the same over the increasing pressure range. The mean values of the wavelength obtained for the assemblies impregnated with the epoxy, Araldite LY 564 also seem to indicate no significant change in the wavelength as the compression pressure was raised from 88 to 884 kPa (see Table 7.3).
7.4 DISCUSSION: MECHANISM AND MODES OF COMPRESSION

From the microstructural studies it can be envisaged that the compression of woven fabrics can be modelled as four modes of compression (see Figure 7.16). Compression mode 1 includes the elimination of a resin rich layer between layers of the reinforcement. In compression mode 2 the layers of fibre reinforcement nest closer by slipping while under compression. As they are impregnated with resin an equivalent amount of resin is expected to be expelled. In compression mode 3 the fibre yarns are deformed by decreasing the amplitude of yarn waveform in which case the thickness of individual plies is reduced. In compression mode 4 the fibre yarns are individually compressed and deformed. Another mode in the compression of the laminate is the resin flow through the fibres which occurs during all modes of the reinforcement compression. All compression modes of the reinforcement may occur simultaneously although some of them may be more significant within a certain pressure range.

The microstructural data of the presented work indicated that mode 2 is dominant over a wide range of pressures in the low and intermediate pressure regime whereas modes 3 and 4 are stepwise and become significant at high pressures. Nesting is related to both the type of reinforcement and to the geometrical parameters of that reinforcement and also to resin rheology. In the presented experimental work, nesting was present even at low pressures of 4.4 kPa. During nesting and changes in the yarn waveform, the number of contact points between fibres increases. This leads to a type of stepwise compaction of the yarn cross-section, as they are squeezed between plies vertically and between neighbouring fibre contact points laterally.

The development of fibre structure during compression is important both from the processing and micromechanics point of view. Nesting reduces the macro-pores available for resin macro-flow between bundles and the resin rich areas in the laminate. Compaction of bundle cross-section reduces the permeability of the bundles in the resin micro-flow [Lekakou et al (1996c)]. Both effects can be evaluated from the microstructural analysis of laminates compressed to different degrees of compression.
Figure 7.16 Schematic showing the compression of woven fabrics as a combination of four modes of compression: Mode 1 - the elimination of a resin rich layer between layers of the reinforcement. Mode 2 - the layers of fibre reinforcement nest closer by slipping while under compression. Mode 3 - the fibre yarns are deformed by decreasing the amplitude of yarn waveform reducing the thickness of individual plies. Mode 4 - the fibre yarns are individually compressed and deformed.
8. MICROSTRUCTURAL COMPARISON BETWEEN DIFFERENT TYPES OF COMPRESSED FABRICS
8. Microstructural Comparison Between Different Types of Compressed Fabrics

8.1 INTRODUCTION

The flow of resin through fibre reinforcement is an important process in resin transfer moulding. It can be generally regarded as macro flow between fibre yarns and micro flow within the fibre yarns [Lekakou et al. (1996c)]. Macro flow through the fibre reinforcement, with regard to the direction of flow, can be considered according to two main classes of processes. In the first class, an example being autoclave processing, the resin flow is approximated as one-dimensional, where the resin flows through the fibre reinforcement in the through thickness direction into the surrounding bleeder layers. In the second class of processes, as in resin transfer moulding, the resin flow is approximated as two-dimensional in-plane flow in which the resin spreads in-plane during the macro infiltration of the reinforcement. In this case, the thickness is usually very small in comparison with the other two dimensions and resin flow along the thickness direction occurs very quickly. Hence, the flow rate is controlled primarily by the in-plane macro infiltration.

As mentioned previously in section 2.2.1 the rate of flow is commonly described using both Darcy's law and the Carman-Kozeny equation. Thus the rate of flow is a function of the resin viscosity, the pressure gradient across the fibre bed and the permeability of the fibre reinforcement. The permeability is a measure of a porous medium's 'resistance' to fluid flow and is dependent on the fibre radius and the porosity of that medium. More specifically the permeability depends on the cross-section and tortuosity of the flow paths within the fibre reinforcement, which in turn depend on the fibre fraction, fibre topology and the dimensions and shape of the fibres.

Therefore, when considering macro flow through fibre reinforcement there is an important relationship between the flow and the porosity of the reinforcement. Adams et al. (1986) considered the porosity of woven fabrics as two main pore systems. The first of the two pore systems is associated with the regions bordered by adjacent yarns in each weave direction, i.e. the interstices. Figure 8.1 shows these type of pores denoted as A, with the distance between yarns or 'mesh size' denoted as M. The second of the pores systems occurs at the crossover point between warp and weft yarns and is denoted as B in Figure 8.1. These pores are usually the smaller of the two and therefore offer more resistance to fluid flow. It should also be noted
that as the mesh size of the reinforcement increases the size of the pores at the yarn crossover points, B, also increases. Pores of type A control mainly the macro flow along the thickness direction whereas pores of type B control mainly the macro flow of in-plane infiltration processes. Therefore small changes in the structure of the reinforcement can have dramatic effects on flow properties.

![Figure 8.1 Schematic representation of pore systems in woven fabrics that are operative during in-plane fluid flow [Adams et al (1986)].](image)

As the flow in resin transfer moulding is two-dimensional in-plane flow, the pore type of interest is that of B, the pores at the yarn crossover points. These are the types of pores illustrated in the micrographs of the sections of compressed laminates presented in section 8.2. Such micrographs have been examined so far (sections 7.2 and 7.3) for microstructural parameters to characterise compression. In this section these micrographs are examined in terms of pores, available to resin flow, and void formation. A comparison is made between four types of fabric: plain weave, twill weave, 5 harness satin weave and a noncrimped stitch-bonded fabric.
In this section area fractions were determined for fibres, macro-porosity and void content from micrographs of samples taken as a result of the compression testing. The micrographs in Figures 8.2-8.3 and 8.6-8.7 show the through thickness section of the laminates compressed to different maximum target pressures. Table 8.1 shows the resulting area fractions for plain weave (PW), twill weave (TW), 5 harness satin weave (5HSW) and the noncrimped stitch-bonded fabric (NCSB) at different degrees of compression.

The plain and twill weave exhibit similar porosity area fractions at a compression pressure of 88 kPa. This value is for the total area porosity and does not take into consideration the size and frequency of the pores that make up this figure. Thus two different types of reinforcement having the same porosity fraction does not indicate that the permeability for both types of reinforcement will be the same. The structure of the reinforcement and the size of the yarns will determine the size and shape of the pores and hence the permeability. Figures 8.2a and 8.3a shows the through thickness microstructure of assemblies of plain weave and twill weave fabrics respectively, both compressed to the same target pressure of 88 kPa. The yarns of the plain weave are larger than that of the twill weave and this coupled with nearly twice the
Figure 8.2 Micrographs showing the through thickness microstructure of assemblies of plain weave glass/epoxy laminates compressed to a maximum pressure of: (a) 88 kPa (b) 884 kPa.
Figure 8.3  Micrographs showing the through thickness microstructure of assemblies of twill weave glass/polyester laminates compressed to a maximum pressure of: (a) 88 kPa (b) 884 kPa.
amount of ends and picks for the twill weave (6.7 x 6.3 and 11.8 x 11.8 per cm for plain and twill respectively) results in the plain weave having fewer but much larger pores. On this evidence it would seem that the plain weave would have a higher value for the permeability due to the wider flow paths. However if the two fabrics had identical mesh sizes (the number of ends and picks equal) and the same yarn dimensions then another difference between the two fabrics would be the pore sizes at the yarn crossover points due to crimp and fabric structure. Figure 8.4 illustrates that per repeating unit, the plain weave has four small pores of equal size, while the twill weave has two pores of that size and one larger pore. Permeability experiments by other workers [Adams et al (1986), Adams and Rebenfeld (1987), Chan and Hwang (1991)] have shown that this results in a greater permeability for the twill weave when compared to an otherwise identical plain weave reinforcement. This must be the result of a complex selectivity process for flow between different sizes of macro pores, taking into account the tortuosity of the flow path.

![Figure 8.4 Pore systems in a plain weave fabric and in a 2/1 twill weave fabric.](image)

Another consideration with regard to weave structure is when the reinforcement is no longer balanced i.e. there is a different number of ends to picks, altering the mesh sizes in the warp and weft direction. Figure 8.5 shows an example of a plain weave fabric where there is more picks than ends. It would appear from the pore systems that the predominant direction of flow
would be that of warp (machine) direction due to the larger pore sizes. However this does not take into consideration the tortuosity of the flow path which is also much greater in the warp direction than in the weft (machine) direction. Thus in an unbalanced weave structure there are two competing factors. Experiments by other workers [Adams et al (1986) and Shafi and Neitzel (1996)] have shown that higher permeabilities resulted in the flow paths with less tortuosity indicating that the number of crossover points per unit length is the controlling factor for in-plane flow.

Table 8.1 and Figures 8.2b and 8.3b show that as the compression pressure is increased to 884 kPa the porosity area fraction of the plain weave decreases to 0.08 whereas the twill weave only reduces to 0.15. This is probably due to the layers of the plain weave reinforcement being able to reach a higher degree of nesting than that of the twill weave.

The 5 harness satin weave also exhibits a similar porosity area fraction to that of the plain and twill weave (see Table 8.1 and Figure 8.6). The pore sizes are much smaller again even when compared to the twill weave. This is partially due to a slight decrease in the size of the yarn dimensions but primarily due to the increase in the number of ends and picks (22.4 x 21.3 per
Figure 8.6 Micrographs showing the through thickness microstructure of assemblies of 5 harness satin weave glass/polyester laminates compressed to a maximum pressure of: (a) 88 kPa (b) 1768 kPa.
cm). The decrease in pore size would probably result in the 5 harness satin having the lowest permeability of the three weaves discussed. However the 5 harness satin weave also has the least tortuous flow paths so any decrease in the permeability due to the decreasing pore size will be offset to a certain extent by this conflicting factor. The decrease of the porosity area fraction with increasing pressure (see Figure 8.6b) is as expected due to the low crimp of the reinforcement. It is not quite as low as the plain weave which is probably due to the inability of the 5 harness satin weave to nest with adjacent layers very well due to the low level of crimp.

The noncrimped stitch-bonded fabric is not a weave, but four unidirectional layers of aligned rovings (0°, 45°, -45°, 90°) bonded together by cross stitching with a very light yarn to form a single ply. Figure 8.7 shows the through thickness section of such a laminate consisting of four plies (sixteen unidirectional layers in total) at three different degrees of compression. These laminates allow higher fibre volume fractions [Bader and Lekakou (1997)] than woven fabrics, are unaffected by influences of crimp and are more stable than simple weaves so less distortion occurs. The relatively high value of the porosity area fraction at a compression pressure of 88 kP (see Table 8.1 and Figure 8.7a) is due to the large dimensions of the rovings, a fewer number of ends and picks, non-unidirectional fibre orientation in each layer and low crimp and therefore no nesting. So this would indicate a high level of permeability at a \( P_{\text{max}} \) of 88 kP. As the pressure is increased to 1768 kP the layers of the reinforcement adjust together well due to the low crimp ensuring that there are no resin layers and reducing the porosity area fraction dramatically leaving almost no pores for resin flow (see Figure 8.7c). The only reason for the porosity area fraction not decreasing further is due to the remaining pores between rovings in a single ply. These would be difficult to reduce further as the plies are stitched together inhibiting the movement of the rovings and combined with zero crimp also means any form of nesting is greatly reduced. When permeability is considered in such fabrics one should also consider the effects of fibre orientation on permeability.

Control of voids is very important in the production of polymer composites [Wood and Bader (1994) amongst others]. Void formation is related to resin impregnation and more specifically to viscous flow and wetting properties of resin related to the reinforcement [Mahale et al (1992), Lundstrom (1996)]. The formation of voids can be attributed to many factors but
Figure 8.7  Micrographs showing the through thickness microstructure of assemblies of noncrimped stitch-bonded glass/polyester laminates compressed to a maximum pressure of:

(a) 88 kPa, (b) 884 kPa, (c) 1768 kPa.
the main source in the compression testing carried out in this work has been due to entrapped air from the mixing and layup stages of the experimentation. The size of the voids are dependent on available pore size and to their position along the flow path. As can be seen from Table 8.1 and Figure 8.2a the plain weave reinforcement at a relatively low compression pressure of 88 kPa has the largest void area fraction and also contains the largest sized voids. This plain weave sample has the largest pore size of all the samples and thus has the largest voids. Once the plain weave assembly has been raised to a compression pressure of 884 kPa, there is a decrease of the void area fraction as they are forced out of the system and the size of the voids have decreased with decreasing available pore size. Once compression of the assembly has begun during the compression testing the only way for the entrapped air to escape is along the in-plane flow paths. The tortuosity of the flow path then is an important factor when considering the eradication of the voids. The plain weave fabric has the more tortuous flow path and thus it is more difficult for the entrapped air to flow out of the system which is confirmed by the void area fraction data. The twill weave has the next most tortuous flow paths and there is a corresponding reduction in the void area fraction. As the flow paths of the different fabrics continue to be less tortuous, the 5 harness satin weave is next, where there is a corresponding reduction toward zero of the void area fraction. The noncrimped stitch-bonded reinforcement has no crimp and thus very little tortuosity. However it has large pores at low pressures, resulting in medium voids at low pressures. At the high compression, air escapes through the non-tortuous path, resulting in the noncrimp stitch-bonded fabric having low voidage.
9. CONCLUSIONS
AND RECOMMENDATIONS
FOR FURTHER WORK
9.1 CONCLUSIONS

The focus of this study has been on the compression and development of microstructure of glass fibre fabrics during the processing of polymer composites. Work has been carried out in the following areas:

(a) Mechanical compression testing of assemblies of dry and resin impregnated fabrics for different types of fabrics, including a plain weave, a twill, a 5 harness satin and a non-crimp stitch bonded fabric.

(b) Construction of a mathematical model of the compression of resin impregnated fabrics and comparison between theoretical and experimental results.

(c) Microstructural analysis of laminates compressed to different maximum pressures in order to follow the evolution of fibre and pore structure during compression for the four types of fabrics mentioned above. Furthermore, the plain weave was selected as a representative fabric for which geometrical parameters of its structure under different degrees of compression were measured in the microstructural analysis in order to try to elucidate the mechanism of compression. For this purpose, a methodology was devised for the geometrical representation and quantitative characterisation of plain weave in the microstructural analysis.

On the basis of the measured microstructural parameters under different degrees of compression, it was concluded that the compression of plain weaves can be considered as a combination of four modes of deformation: Mode 1 involves the elimination of a resin rich layer between adjacent fabric layers. Mode 2 involves the nesting of layers of fabric. Mode 3 involves the deformation of the yarn waveform. Mode 4 involves the deformation of the yarn cross-section. In the present study assemblies of a high-crimp plain weave were considered, which displayed considerable nesting even at pressures as low as 4.4 kPa. Mode 2 was the dominant mode of deformation over a wide range of pressures at low and intermediate level. Modes 3 and 4 became significant at relatively high pressures.
Mechanical compression testing was carried out on assemblies of (a) dry fabrics and (b) resin impregnated fabrics. In general, the fibre volume fraction increased non-linearly with increasing pressure while the rate of increase of fibre fraction decreased as it tended toward its maximum packing capacity. Compression of assemblies of dry fabrics was generally independent of the speed of compression, for the tested types of weaves, namely plain weave, twill and 5 harness satin, and for compression speeds ranging between 0.05 and 1 mm/min. In contrast, the compression curves of assemblies of resin impregnated plain weaves shifted to lower fibre volume fractions, away from the compression of dry assemblies, as the compression speed was raised. This resulted in higher pressures being required for compression of wet assemblies to a certain fibre volume fraction, as the speed of compression was increased.

In the compression of assemblies of plain weave, differences in ply orientation had no significant effect on compression. Compression of assemblies of dry plain weave plies, which were previously compressed and reassembled, resulted in data comparable to that of the original compression, suggesting that there was no permanent deformation of fibres once nesting had been eradicated. On the other hand, consecutive compression cycles on the same assembly of dry weaves, which were not separated and reassembled between cycles, resulted in hysteresis loops, which suggests that the stored strain energy was not sufficient to overcome the frictional restraints at the fibre-fibre contacts and reverse the nesting process. The assembly was compressed more and more with subsequent loading cycles up to a limit corresponding to the fibre packing fraction. As the packing fraction was approached the hysteresis loops became smaller as further nesting was not possible.

The compression data for assemblies of dry fabrics could be fitted into a power law relation between pressure and fibre volume fraction. Compression was not significantly affected by the number of plies if this number was greater than five. The value of the empirical constants of the power law compression for assemblies of dry fabrics depends generally on the type of weave, where the power law exponent, $b$, was found to be equal to 10.3, 9.8 and 9.1 respectively for the plain weave, twill and 5 harness satin fabrics that were tested. This suggests independent compression tests are needed for each type of fabric. If the number of plies was less than five, it affected the compression data due to the effects of the flat area of compression platens in contrast to the wavy surface of fabrics.
The compression of resin impregnated fabrics was of viscoelastic nature with respect to two aspects: (a) The compression curves depended on the speed of compression; this fact was associated with the presence of viscous resin. (b) Pressure relaxation tests and repeated loading cycles manifested the viscoelastic nature of the fibre/resin system comprising the elastic deformation of fibres, the irreversible nesting of fabrics and the irreversible resin flow.

The type and viscosity of resin did not seem to have any significant effect on the compression data of assemblies of resin impregnated plain weaves, where an epoxy and two types of polyester were used in the tests. This seems to suggest that the viscous resin flow may not have much influence on the total required pressure for compression for the range of tested viscosities and compression speeds. Compression depends on the type of fabric: resin impregnated satin was compressed gradually to the highest fibre fraction where plain weave and twill were compressed to a lower fibre fraction at a compression speed of 1 mm/min. Maximum fibre fractions seem to be associated to both maximum packing capacity of fabric due to its structure and the ability of the resin to flow out of the macro-pores quickly before the pores close and the resin gets entrapped. Satin weave has a flat structure which favours close packing and easy resin flow. Non-crimp stitch bonded fabrics were also compressed to high fibre fraction. In the case of plain weave, although it is possible that assemblies of dry plain weaves can nest close together, resin impregnated assemblies of the tested plain weave suffer from resin entrainment in the macro pores as they come to nest, and do not reach high maximum fibre fractions. Twills do not generally nest very close, even when dry, and also suffer from resin entrainment.

A mathematical model was constructed for the viscoelastic compression of assemblies of resin impregnated fabrics, where the total applied pressure was split into two components: (a) a pressure component causing the non-linear elastic deformation of fibre yarns and (b) a pressure component causing the viscous flow of resin. One of the model parameters was the permeability of reinforcement which changes during compression. In a comparison between model predictions and experimental data of compression of assemblies of epoxy impregnated plain weaves, the predicted pressure for resin flow at relatively high compression speeds was very small compared to the measured pressure difference between the pressure required for the wet compression and that for the dry compression. This was attributed to the change of
permeability during compression where the pore structure, size and shape changes dynamically. This may cause flow channels to close down and, if compression takes place at high speeds, the resin might not be able to find alternative flow paths sufficiently quickly and, hence, become entrapped. This leads to a reduction of permeability and an increase of the required pressure. The conclusion from the comparison between theoretical and experimental data was that the permeability of reinforcement needs to be measured under dynamic conditions of changing pore structure and size.

Assemblies of resin impregnated fabrics were compressed up to a target pressure and were left to cure under constant compression, thereafter. Laminates had a smaller thickness after curing due to resin shrinkage (about 5% resin shrinkage in polyesters). Laminates cured under different degrees of compression were subjected to microstructural analysis to follow the mechanism of compression and the corresponding structure development. A comparison between epoxy and polyester laminates did not reveal significant differences in the evolution of their respective microstructural parameters during compression, apart from a slightly easier compaction of the epoxy impregnated warp yarns due to the lower viscosity of epoxy compared to the viscosity of polyester.

Laminates of the different types of tested fabrics cured at different degrees of compression were sectioned through their thickness and compared in terms of general microstructure and overall fibre area fraction, pore area fraction (available for resin flow) and void area fraction. Assemblies of plain weave, twill and 5 harness satin displayed an overall pore area fraction between 0.23 and 0.27 at low pressures: assemblies of plain weave or twill presented many relatively large macro-pores whereas assemblies of satin weave presented a continuous structure of relatively flat pores. The non-crimp stitch bonded fabric contained relatively thick rovings which led to large macro pores at low pressures. Regarding voidage, the plain weave was associated with the highest void area fraction and the 5 harness satin weave with the lowest void area fraction. As the pressure increased a corresponding decrease of voids was observed: Assemblies of non-crimp stitch bonded fabric contained the highest pore area fraction due to its thick rovings and its inability for nesting. From the other three, assemblies of plain weave displayed quite low pore area fraction due to their good ability for nesting. However, their voidage was still high due the high voidage they originally had at low
pressures. The pore size, shape, structure and tortuosity of flow paths observed in the microstructural analysis can be associated qualitatively with the expected in-plane permeability of various types of fabrics.

9.2 RECOMMENDATIONS FOR FUTURE WORK

Recommendations for future studies include the following suggestions:

Permeability tests are needed at dynamically varying pore structure and at low resin speeds, comparable to those occurring in the compression experiments. The main source of uncertainty in the described model of viscoelastic compression has been the value of permeability and its change during compression. Measurements of permeability, so far, have been carried out under constant pore structure and pore size. A technique needs to be devised for measurement of permeability under changing pore structure and a corresponding theoretical analysis needs to be developed.

The present microstructural analysis focused on one plane of cross-section, including the through thickness direction and the overall direction of weft yarns. Lateral movement of weft yarns was not possible to observe due to the poor visibility of layers underneath the laminate surface when examined by a microscope. It is suggested to incorporate a thin wire into the weft yarns which might make observations easier by means of X-ray techniques, for example.

Data from the microstructural analysis can be used in future theoretical modelling of in-plane permeability or in micromechanics. With regards to permeability, there has been recently special interest in macro and micro-impregnation [Lekakou et al (1996)]. This provides a good opportunity to extract microstructural data regarding size of macro pores and micro-porosity (inside yarns) which would aid in the more accurate prediction of macro and micro-permeabilities.
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