

## COMPRESSION FLOW PERMEABILITY MEASUREMENT - A CONTINUOUS TECHNIQUE

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**SUMMARY:** A variety of permeability measurement techniques have been based upon pressure driven flow through fibrous reinforcing materials. An innovative method is presented here utilising compression driven flow, which allows for continuous measurement of permeability over wide fibre volume fraction ranges, in a single efficient test. The initial study has been limited to isotropic reinforcements, obtained permeability data being compared with values measured using a conventional method. Data has been collected from two styles of random mat, volume fraction ranging from 20 to 50% in a single test. Comparison between the two methods is very promising, validating further study and extension of this new technique. Larger errors have been noted at higher volume fractions, this effect seeming to be related to increasing mould clamping forces, and subsequent deflection of the upper platen.

**KEYWORDS:** Permeability Measurement, Compression Flow, Reinforcement, LCM.

### INTRODUCTION

The permeability of fibre reinforcements is vital material data required for the analysis of many composites manufacturing techniques. In most situations a detailed knowledge of the permeability tensor is required as a function of the reinforcement volume fraction,  $V_f$ . While a variety of techniques have been presented in the literature, the challenge remains to develop a fast and efficient method for measuring permeability as a function of  $V_f$ . Here we present a shift in the current thinking, developing a method based on compression driven flow in a porous media. This approach provides a number of advantages, including the ability to measure permeability as a function of  $V_f$  continuously, in a single efficient test.

Motivation for this technique is provided by the need for detailed permeability information to model mould filling in Liquid Composite Moulding (LCM) processes. LCM processes require the placement of fibre reinforcement within some form of closed mould. These fibrous structures, or preforms, provide the structural skeleton of the finished composite product. A thermoset resin is injected into the mould, infiltrating the preform. Practice has converged on modeling this process using Darcy's Law, permeability relating pressure gradients to local volume averaged fluid velocities.

The term LCM encompasses a growing list of mould filling processes, permeability- $V_f$  data being vital for the modeling of each variation. Parts manufactured using Resin Transfer Moulding (RTM) may exhibit sections at different  $V_f$ 's, or may have  $V_f$  varying continuously. LCM variations such as the Seeman Composite Resin Infusion Moulding Process (SCRIMP) and Injection/Compression Moulding (I/CM) exhibit preform  $V_f$ 's that vary with time. This is due to the potential for the cavity thickness to vary during mould filling, an effect that is inherent to a number of LCM processes.

An in-plane permeability measurement technique is being developed which utilises compression driven flow through a preform. As preform  $V_f$  is varied continuously during a test, permeability is determined as a truly continuous function. This method has the potential to provide a very fast and efficient test. In this paper we develop this method for the measurement of isotropic permeability, the extension to anisotropic permeability being the focus of current work. The relevant compression flow theory is presented, and the required flow model derived. Several measurements are made with the new technique on two common preform fabrics, the results being compared to a more traditional pressure driven flow method.

## EXISTING PERMEABILITY MEASUREMENT TECHNIQUES

### In-Plane Pressure Driven Flows

Many composite articles produced with LCM processes have wall thickness much smaller than their in-plane dimensions. It is often a relevant assumption that all resin velocities remain in the plane of the part, reducing mould filling problems to a 2D analysis. Under such conditions only the in-plane components of the permeability tensor are required. All previously reported in-plane permeability measurement methods have been based upon pressure driven flow in simple geometries. A sample of the preform is compacted between two flat plates to a desired thickness, and hence  $V_f$ . A test fluid is pumped into the mould at the injection gate, and either a radial or rectilinear flow is established in the preform. The fluid is pumped in at either a constant flow rate or injection pressure, internal fluid pressure, or flow front position data being recorded to determine the in-plane permeability tensor [1-6].

The vast majority of reported methods define the permeability tensor at a single  $V_f$  value, and must be repeated if permeability is required as a function of  $V_f$ . These tests must be repeated several times to fully characterize the preform material. Several authors have presented significant refinements to these methods, improving the speed of implementation, and allowing for determination of the directions of the principle permeability axes [7,8].

Recently, a method was introduced by Stadtfeld et al. which provides measurement of permeability at several  $V_f$ 's, in one continuous experiment [9]. A rectilinear flow geometry is used, an elaborate sealing system allowing for the characterization of a single preform at decreasing cavity thickness. This test is completed as a fully saturated flow, the test fluid filling the entire preform, before steady state flow is established. While permeability is found as a function of  $V_f$ , at least two full experiments are required to characterize a material, assuming the principle permeability directions are known prior to the experiments.

### Transient versus Steady State Flows

An important assumption required for the use of Darcy's Law in LCM simulations is that the preform is fully saturated once the resin flow front passes a position in the mould. Some

reinforcing fabrics display a region of progressive saturation behind the flow front, an effect which can be identified by permeability measurement. 'Transient' measurement techniques involve the progressive filling of a dry preform, expelling all air. 'Steady State' techniques involve the full impregnation of the preform with fluid, before flow is continued and flow rate versus pressure drop data gathered. While it can be argued that transient permeability measurements represent situations similar to actual LCM filling, it is debatable whether permeability values obtained are strictly valid if Darcy's Law is violated in the saturating region. The method detailed in this paper utilizes steady state flow, determining saturated permeability as a continuous function of  $V_f$ .

### COMPRESSION FLOW THEORY

The flow geometry is presented schematically in Fig. 1, while the physical mould is depicted in Fig. 2. Preforms are cut into circles of radius  $r_o$ , which are constrained between two rigid, parallel plates. Prior to the test, the preform is fully saturated with a test fluid of Newtonian viscosity,  $\mu$ . These plates are driven together at a predefined constant speed  $\dot{h}$ , the distance between the plates being defined as  $h$ . This compression flow drives fluid out of the preform, generating a fluid pressure field within. By monitoring the fluid pressure at the center of the mould,  $P_o$ , permeability can be determined as  $V_f$  is progressively increased. As the cavity thickness and hence  $V_f$  vary continuously through the test, a truly continuous measurement of isotropic permeability is achieved.

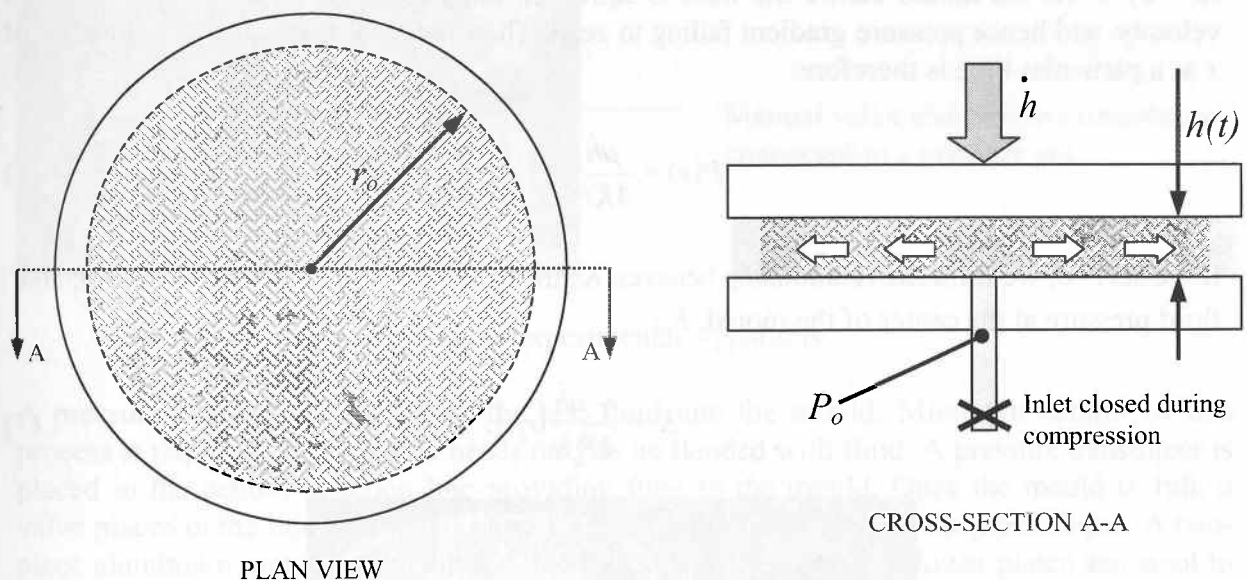


Fig. 1 Schematic of the flow domain. For the actual experiment, the lower platen was formed as a basin, to contain all fluid neatly within the mould.

We assume that all resin velocities will remain in-plane, and that the entire preform is fully saturated with the test fluid. Currently we assume isotropic permeability, resulting in axisymmetric pressure and flow fields. Resin flow is described by Darcy's Law in polar coordinates:

$$v_r = -\frac{K}{\mu} \cdot \frac{dP}{dr}, \quad (1)$$

where  $v_r$  is the radial volume averaged velocity,  $K$  is the isotropic permeability,  $P$  the fluid pressure, and  $\mu$  the fluid viscosity. The test fluid is assumed to be Newtonian. A “thickness-averaged” continuity equation is applied, written here in cylindrical coordinates:

$$\frac{1}{r} \frac{d}{dr} (hr v_r) = -\dot{h}, \quad (2)$$

where  $\dot{h}$  was defined earlier. Noting that the cavity thickness  $h$  is a constant at a given time, Eqns. 1 and 2 are combined to give the governing relationship for fluid pressure.

$$\frac{1}{r} \frac{d}{dr} \left( r \frac{dP}{dr} \right) = \frac{\mu \dot{h}}{Kh}. \quad (3)$$

This linear ODE can be integrated twice, and subjected to the following boundary conditions.

$$@ r = 0 \quad \frac{dP}{dr} = 0, \quad (4)$$

$$@ r = r_o \quad P = 0. \quad (5)$$

Eqn. 4 follows from the fact that no fluid is injected at the centre of the mould, and hence  $v_r(r=0)=0$ . At the mould centre the fluid is squeezed out equally in all directions, the fluid velocity, and hence pressure gradient falling to zero. The solution for pressure as a function of  $r$  at a particular time is therefore:

$$P(r) = \frac{\mu \dot{h}}{4Kh} (r^2 - r_o^2). \quad (6)$$

If we set  $r=0$ , we form the relationship between  $K$ , the specified parameters ( $\mu$ ,  $\dot{h}$ ,  $h$ ,  $r_o$ ), and fluid pressure at the center of the mould,  $P_o$ :

$$K = \frac{-\mu \dot{h}}{4P_o h} (r_o^2). \quad (7)$$

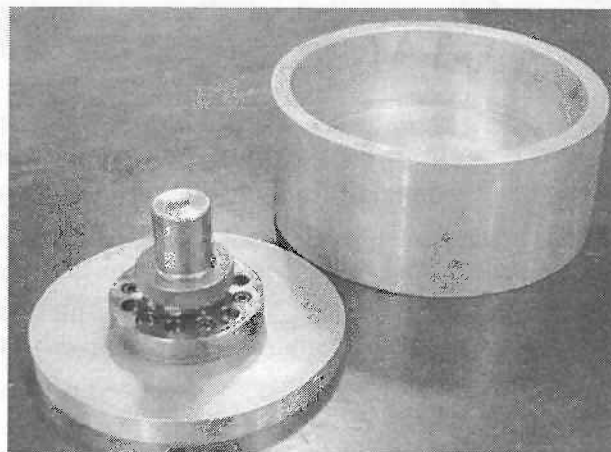


Fig. 2 Aluminium compression test mould.

## EXPERIMENTAL PROGRAM

### Experimental Equipment

The compaction flow permeability measurements require the ability to bring two parallel plates together, while recording  $h(t)$ , and supplying the force necessary to compress the sample to the required volume fractions. An Instron 1186 testing machine fitted with a 200kN load cell has been used. This instrument provides accurate control over platen position, while achieving  $V_f$  in excess of 60%. While an Instron is ideal for this method, the authors believe a simple purpose built rig will suffice. The current setup is shown in Fig. 3.

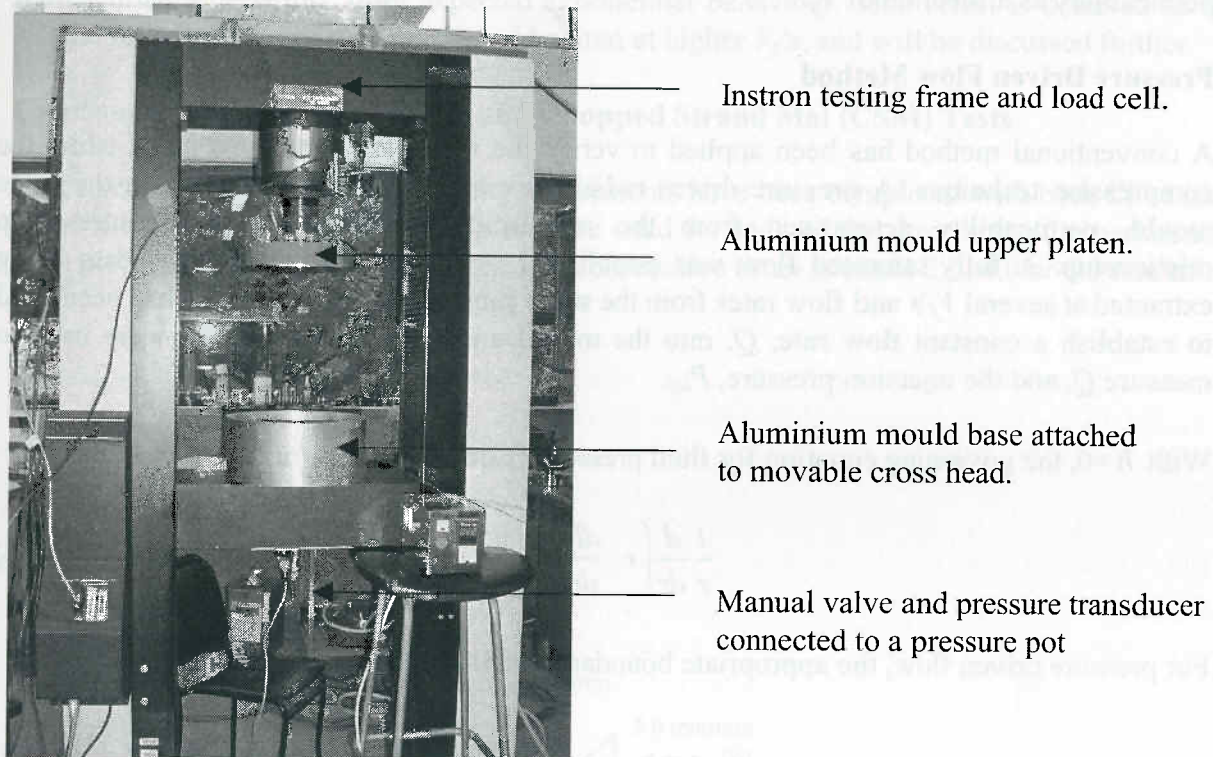


Fig. 3 Experimental Apparatus.

A pressure pot was used to drive the test fluid into the mould. Minimum control of this process is required, as the mould needs only to be flooded with fluid. A pressure transducer is placed in the central injection line providing fluid to the mould. Once the mould is full, a valve placed in the injection line is closed, ensuring no back flow into the pressure pot. A two-piece aluminium mould has been used, the lower portion acting as a lower platen and pool to contain the test fluid. The mould is pictured in Fig. 2.

### Materials

Two glass fibre preform materials were considered in this initial study, a  $450 \text{ g/m}^2$  continuous filament mat (CFM) and a  $430 \text{ g/m}^2$  chopped strand mat (CSM). These materials were chosen as they are commonly used as LCM preforms, and exhibit isotropic permeability tensors. The test fluid used to simulate resin was a glucose solution. This was chosen as it is water soluble and is therefore easily tailored to a desired viscosity. The solution used in this study had a viscosity of  $0.13 \text{ Pa}\cdot\text{s}$ , measured at  $25^\circ\text{C}$  on a Physica Rheolab viscometer.

## Compression Flow Method

Preform samples were cut into circles with a diameter of 270 mm (+/-2mm) and weighed before the test to accurately determine  $V_f$ . The samples are placed at the center of the mould, and the mould platens are brought together to a cavity thickness producing the initial volume fraction targeted. The test fluid is then injected slowly through the central injection gate, fully saturating the preform. The test is then commenced, the mould platens being forced together at a constant predetermined speed. Fluid pressure at the center of the mould is logged with time, and the final cavity thickness is determined by the maximum  $V_f$  required. All required data is logged to a data acquisition system, and Eqn. 7 is used to determine isotropic permeability as a function of  $V_f$ .

## Pressure Driven Flow Method

A conventional method has been applied to verify the initial measurements made using the compression technique. A pressure driven radial flow has been established within the same mould, permeability determined from the measured flow rate versus pressure drop relationship. A fully saturated flow was established in the mould, permeability data being extracted at several  $V_f$ 's and flow rates from the same preform. The pressure pot has been used to establish a constant flow rate,  $Q$ , into the mould and pressure transducers were used to measure  $Q$ , and the injection pressure,  $P_{inj}$ .

With  $\dot{h}=0$ , the governing equation for fluid pressure (Eqn. 3) reduces to:

$$\frac{1}{r} \frac{d}{dr} \left( r \frac{dP}{dr} \right) = 0. \quad (8)$$

For pressure driven flow, the appropriate boundary conditions are:

$$\textcircled{a} \quad r = r_i \quad \frac{dP}{dr} = -\frac{\mu Q}{2\pi r_i h K}, \quad (9)$$

$$\textcircled{a} \quad r = r_o \quad P = 0. \quad (10)$$

Note that a small hole of radius  $r_i$  must be cut in the centre of the preform to ensure all resin velocities remain in-plane. This is common practice with radial flow, pressure driven methods. Solving Eqn. 8 with the stated boundary conditions provides the required relationship for the calculation of  $K$ :

$$K = \frac{\mu Q}{2\pi h P_{inj}} \ln \left( \frac{r_o}{r_i} \right). \quad (11)$$

## RESULTS AND DISCUSSION

### Initial Feasibility Experiment

An initial experiment was performed using a polyester fibre mat, typically used as a resin bleeder fabric in autoclave processing. This material was selected because of the consistency

of structure maintained during repeated compression cycles. This was important to demonstrate the independence of permeability on compaction speed, while using the same preform sample.

Permeability measurements were made at compaction rates from 1.0 to 8.0 mm/min, covering  $V_f$ 's from 15 to 40%. The recorded pressure traces are shown in Fig. 4. As expected, doubling the compaction rate ( $\dot{h}$ ) produces a doubling of the measured fluid pressure. Applying Eqn. 7 these pressure traces have been converted into permeability data, and plotted on a logscale in Fig. 5. All six permeability curves collapse together, providing initial verification for this technique. A pronounced upturn in the permeability curves is noted at  $V_f$ 's greater than 30%, representing a departure from expected exponential behaviour. This effect is believed to be related to deflections of the upper mould platen at higher  $V_f$ 's, and will be discussed further.

### Continuous Filament Mat (CFM) and Chopped Strand Mat (CSM) Tests

Three sets of compaction permeability experiments have been completed with both CFM and CSM samples. Virgin preform samples were used for each of the three sets of experiments, each preform sample being compacted five consecutive times at either 2.0, 4.0 or 8.0 mm/min.

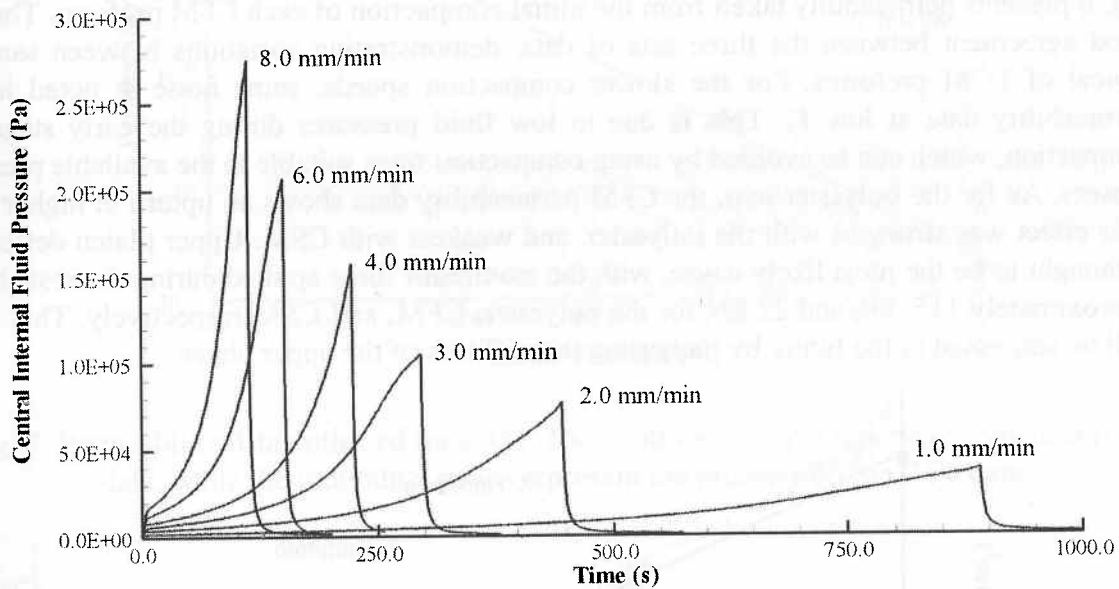


Fig. 4 Fluid pressure traces recorded during compaction of the polyester mat.

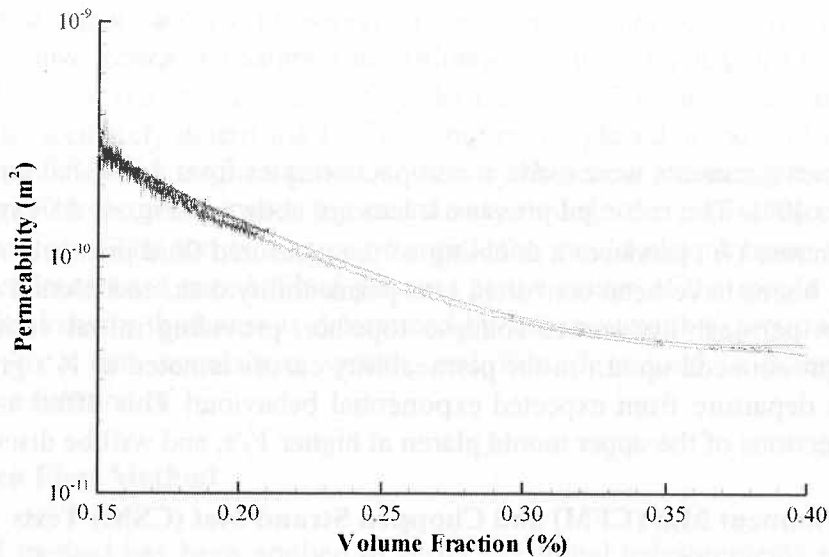


Fig. 5 Permeability data collected for the polyester mat during compaction tests.

### Continuous Filament Mat (CFM) Results

Fig. 6 presents permeability taken from the initial compaction of each CFM preform. There is good agreement between the three sets of data, demonstrating variations between samples typical of LCM preforms. For the slower compaction speeds, some noise is noted in the permeability data at low  $V_f$ . This is due to low fluid pressures during the early stages of compaction, which can be avoided by using compaction rates suitable to the available pressure sensors. As for the polyester mat, the CFM permeability data shows an upturn at higher  $V_f$ 's. This effect was strongest with the polyester, and weakest with CSM. Upper platen deflection is thought to be the most likely cause, with the maximum force applied during the tests being approximately 115, 80, and 22 kN for the polyester, CFM, and CSM respectively. This issue will be addressed in the future by increasing the stiffness of the upper platen.

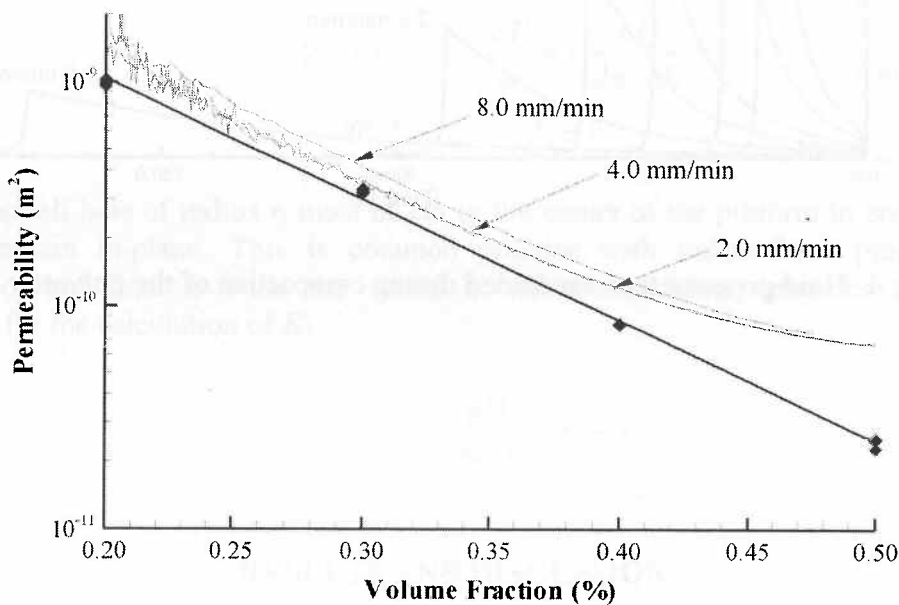


Fig. 6 Permeability data collected for CFM. The continuous traces represent compaction test data, while the individual points represent the pressure driven flow data.

Fig. 8a presents permeability data from five consecutive tests completed on a single preform at a compaction rate of 4.0 mm/min. The CFM demonstrated decreasing permeability with



successive compactions. This is most likely due to modification of the fibre architecture with successive compaction, emphasising the importance of using virgin preform samples in all permeability measurements.

### Chopped Strand Mat (CSM) Results

Fig. 7 presents permeability data obtained from the initial compaction of three CSM preforms. While there does appear to be a trend for higher permeability at greater compaction rates, the differences are small, and within expected variations for the material. The effect could be caused by increased upper platen deflection with increased compaction rate, this possibility being addressed in future studies. Fig. 8b presents the effect of repeated compaction on the permeability of the CSM. The biggest reduction in permeability is produced after the initial compaction, this material being less sensitive to repeated compaction than the CFM.

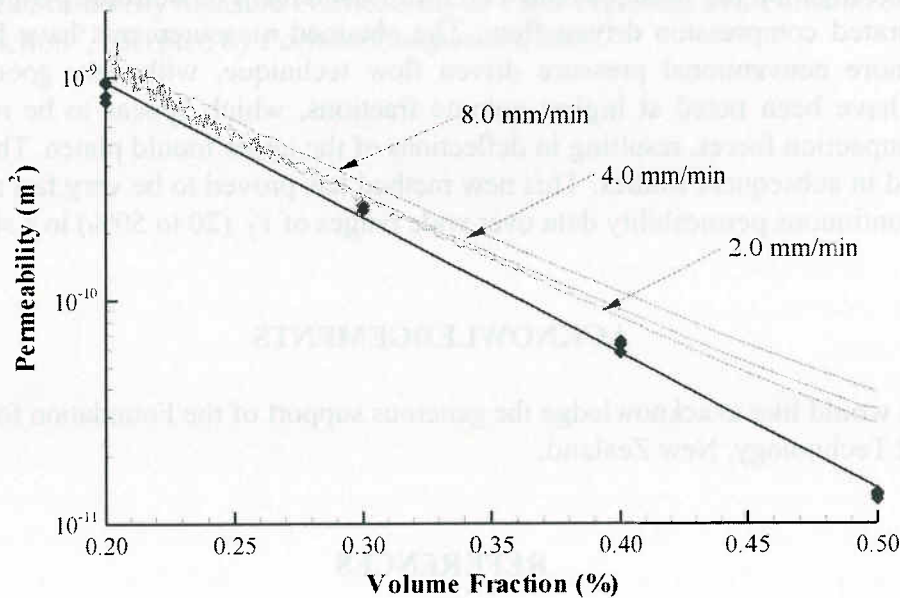


Fig. 7 Permeability data collected for CSM. The continuous traces represent compaction test data, while the individual points represent the pressure driven flow data.

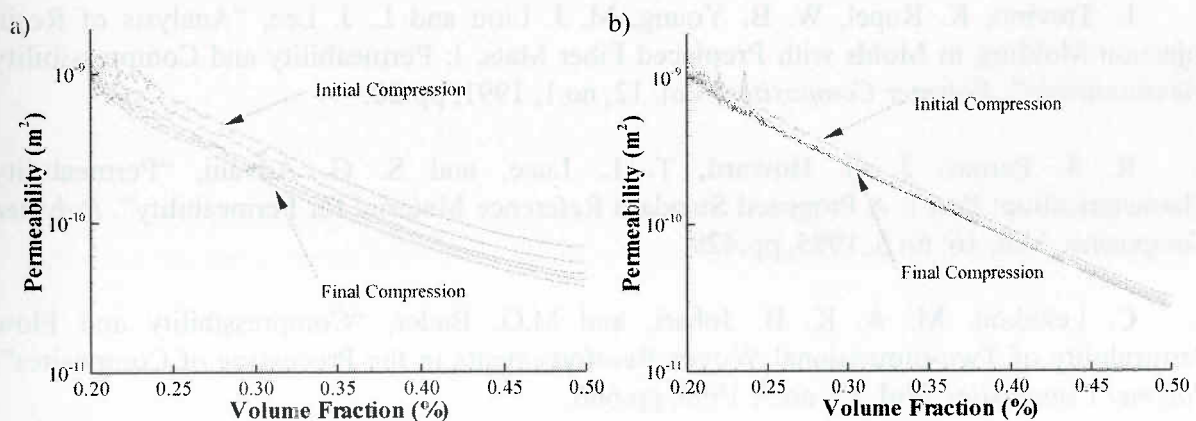


Fig. 8 Permeability data from consecutive compactions at 4.0 mm/min for a) CFM, b) CSM.

## Comparison with Conventional Measurement Method

Permeability measured using the conventional pressure driven flow method is presented in Figs. 6 and 7. Four  $V_f$ 's have been considered for comparison with the compaction measurement. The comparison is very good for both materials at  $V_f$ 's up to 40%. At higher  $V_f$ 's an upturn in the compaction curves is evident, deviating from the trend line placed through the conventional data. This effect is more pronounced for CFM, and is possibly more gradual for CSM. While the mould deflection issue must be addressed in the future, these initial comparisons provide the necessary verification for further investigation of this innovative technique.

## CONCLUSIONS

An initial investigation has been presented on the measurement of isotropic permeability using a fully saturated compression driven flow. The obtained measurements have been verified against a more conventional pressure driven flow technique, with very good agreement. Deviations have been noted at higher volume fractions, which appear to be related to the required compaction forces, resulting in deflections of the upper mould platen. This effect will be addressed in subsequent studies. This new method has proved to be very fast and efficient, providing continuous permeability data over wide ranges of  $V_f$  (20 to 50%) in a single test.

## ACKNOWLEDGEMENTS

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**ABSTRACT** The first step in the prediction of the permeability of fiber preforms is to solve the Stokes flow problem in domains with an arbitrary cross-section. This can be used to model annular flow parallel to a fiber or a preform as well as the radial flow flow. Here the partial flow problem is solved using a least squares approximation for the boundary conditions while minimizing the dissipative work of the fluid. The convergence of the method is tested for various particle factors for the boundary interpolation and for various number of collocation points for the boundary condition. Convergence is obtained easily. The algorithm is applied for a circular and a square flow domain. The flow rate in a circular domain can be determined in a closed form as well and is therefore used as a test case. The results of the square domain are compared with the results of a FE-analysis. The effect of the anisotropy of the fiber is analyzed. The results of the circular and square domain show similar behavior. The observed behaviour corresponds with the behaviour found in the literature. The method presented is fast and accurate. It can be used to analyze flow along and perpendicular to fibers. To compute the transverse flow only minor adaptations are required.

**KEYWORDS:** Minimized Dissipative Work, Radial Flow, Parallel Flow, Permeability, Anisotropy

**INTRODUCTION**

Resin Transfer Molding (RTM) is being applied increasingly as the manufacturing process of high performance composites. Consequently, significant research effort has been directed to resin filling and fibre impregnation during this type of liquid composite molding process. The main objective of this investigation is to improve the accuracy of RTM.

Radial flow analysis is used to help in finding the optimal process parameters. It considers the flow rate and inlet pressure, and inlet and exit and void fraction. Several process parameters are used to describe the flow. The flow rate is the dependent variable and the inlet and exit pressure are the independent variables. The permeability of the composite is a function of the fiber volume fraction. The permeability of the composite is a function of the fiber volume fraction. The permeability of the composite is a function of the fiber volume fraction.