Permeability Measurements: In Plane and through the Thickness

Maarten Labordus

Center of Lightweight Structures TUD-TNO
Kluyverweg1, 2629 HS Delft, The Netherlands
Corresponding Author's e-mail: M.Labordus@ind.tno.nl

SUMMARY: Liquid molding processes like RTM and vacuum infusion are becoming more and more state-of-the-art in the composite industry. Resin flow simulation software for these processes has also been around for many years. One of the key input parameters required for a good flow simulation is the permeability of the fiber reinforcement. No standard, general accepted permeability test has been established yet. Currently, most composite research institutes have developed their own permeability test set-up. Methods based on visual tracking of the resin flow front with circular or even spherical are notoriously inaccurate and sensitive to small variations in preform dimensions. The Center of Lightweight Structures TUD-TNO has developed permeability measuring devices for accurate in-plane and through-the-thickness permeability measurements following the conditions for Darcy's Law as close as possible. This paper describes the experimental set-up and gives some results.

KEYWORDS: Permeability, in-plane, through-the-thickness, liquid molding, vacuum infusion, RTM, permeameter, flow simulation

INTRODUCTION

The filling stage of the resin transfer molding process is considered to be an analogy of ground water flow though soil. A mathematical relation to describe this process, the (viscous) flow through porous media, was formulated by Darcy in 1856. Darcy's law is now generally applied to model RTM. Applied to a one-dimensional flow Darcy's law is:

$$Q(t) = \frac{k(x)A(x)}{\eta(t)} \frac{dp(x,t)}{dx}$$

with:

Q(t): the time-dependent volumetric flow rate (m^3/s)
η(t): the time-dependent fluid viscosity (Pa.s)
A(x): the surface area of the cross section of the mold (m^2); A(x) can depend on the position x in the mold
dp(x,t)/dx: the pressure gradient in the flowing resin (Pa/m)
k(x): the apparent reinforcement permeability (m^2)

During mold filling, when a resin is injected with a constant pressure in an isotropic reinforcement, the flow direction is constant; the flow velocity decreases in time. In a steady state fluid flow, the flow rate is constant with respect to the magnitude and direction.

In a rectangular mold cavity (A(x)=A) containing a reinforcement material with a permeability independent from x, the pressure gradient dp(x,t)/dx equals the linear pressure drop Δp/Δx. With a model viscous fluid, the viscosity is $\eta(t)=\eta$. Based on this equation a method is developed to determine the permeability of reinforcement materials, under the assumptions that a steady state flow method is adequate to model a (non-steady state) filling process, thus neglecting interfacial phenomena (e.g. fiber wetting, impregnation) and that the reinforcements are isotropic, or, in case of woven (orthotropic) fabrics, the reinforcements are characterized in the principal directions. An apparatus is developed in which the permeability of a (preformed) reinforcement material is measured in a steady state flow of a model fluid instead of a (curing) resin. The cavity of this apparatus contains an adjustable top side so the cavity can be adjusted to the thickness h of the preform. Also, the permeability of a non-preformed reinforcement can be determined as a function of the fiber volume fraction by repeatedly varying the cavity thickness h, thus compressing the reinforcement, and measuring the permeability. The volumetric flow rate Q of the model fluid is determined by measuring the mass of the fluid flown through the mold during a given time interval divided by the density of the model viscous fluid. With assumptions described, the permeability is calculated by:

$$k = \frac{\eta}{bh} \frac{\Delta m}{\Delta t} \frac{1}{\rho} \frac{\Delta x}{\Delta P}$$

EXPERIMENTAL SET-UP

The experimental set-up consists of the CLS permeameter, a plastic fluid container, an electronic scale and a personal computer with a data-acquisition system. It is placed in a laboratory with a temperature control (22±0.5°C). The reinforcements are placed in the permeameter cavity. The cavity height is adjusted to the desired level, in order to obtain the correct fiber volume content in the preform. The container with the glycerol-water mixture is pressurised to a pressure of 0.4 bar. After opening the valve at the fluid inlet, the fluid flows through the reinforcement and fills the container on the scale.

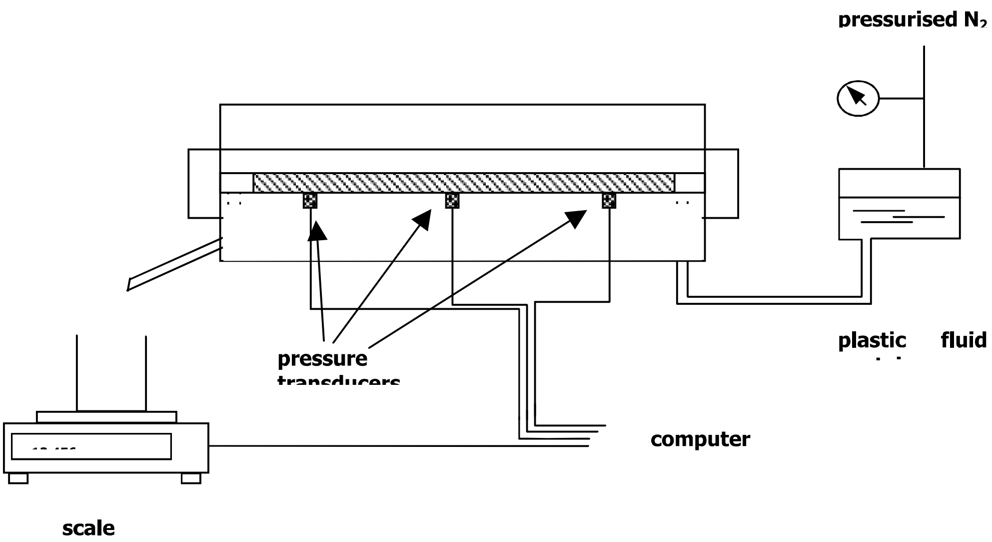

Figure 1: In-plane permeameter

A custom made LabView data acquisition program processes the output signal from the scale, the time interval Δt (s) (read from the PC clock) and the output signals from the pressure transducers. The program automatically determines when the flow has stabilised by analysing the statistical properties of the pressures and mass flow in time. From the data acquired during a period of stable flow, the mass flow Δm/Δt (g/s) and pressure gradient Δp/Δx are calculated with a best-fit method. From given values of accuracy and the statistical properties of the stable flow, an estimated error for the permeability is calculated as well. If the operator desires so, the height of the mold cavity can be changed and a new test run is started.

For permeability tests through the thickness, a similar approach was followed. From literature, it was known that the through the thickness permeability could be 5 to 10 times lower than the in plane permeability. The effect of short-cut flows around the perform would therefore also be 5 to 10 times more significant. It was decided to build a set-up with two separate regions. A central measuring region and a circumferential region where the flow was discarded. The layers of reinforcements were placed in an aluminium casing were they were clamped between two aluminium honeycomb layers, supported by two sheets of metal mesh. This configuration allowed for an almost undisturbed uniform feeding of the complete surface of the reinforcement. The pressure was measured in the cavity in the casing just above and below the reinforcement in the central measuring region. The height of the casing could be adjusted to be able to measure permeability's for a range of fiber volume contents.

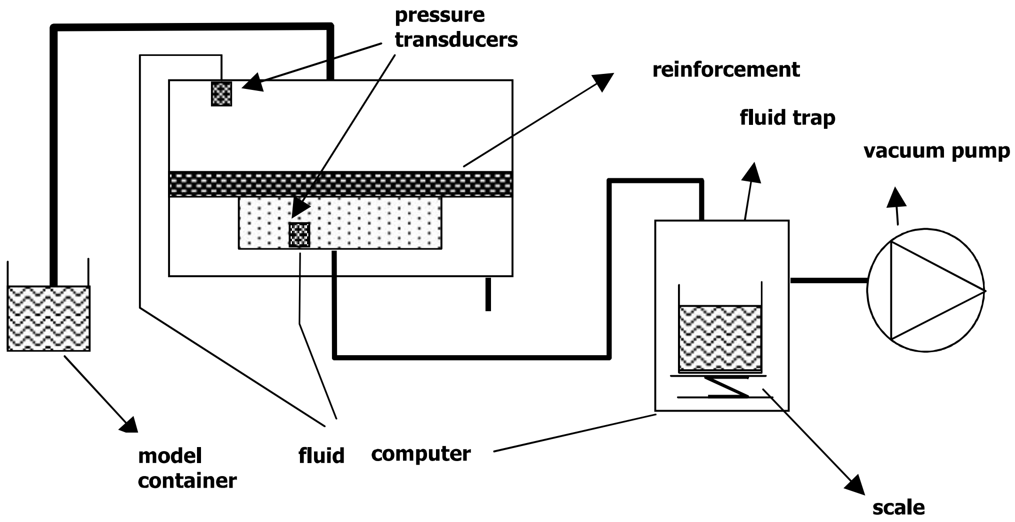

Figure 2: Through-the-thickness permeameter

RESULTS

The results of the permeability measurements are listed in table 1. In the third column the direction of cutting from the fabric is listed; $(0)_{10}$ indicates that 10 layers, cut lengthwise from the fabric (0^{o} direction), are stacked. As can be seen, the results vary greatly for different types of fiber reinforcements. Materials like Unifilo or Injectex have relatively high permeabilities and allow for fast fill times when used in infusion processes.

Other materials like normal fabrics or non-crimp fabric show a much lower permeability. To be able to use these materials in an infusion process, they need to be combined with materials with a higher permeability like Injectex fabric or Unifilo. Another common procedure is to use a sacrificial flow enhancing material like a Newbury infusion membrane or Airtech flownet placed on top of the laminate in combination with a peelply. When a laminate is infused in such a way, the resin actually flows over the laminate and then impregnates it through the thickness. As can be seen from the permeability test results, the through thickness permeability for non-crimp fabrics is indeed much lower than the in plane permeability. This causes problems when infusing thick laminates.

Table 1: Test data

#	Material	Lay-up	Vf	Direction	Permeability (m2)	Standard deviation (m2)
1	Unifilo 750	--	29	In-plane	6.9 10-10	3 10-12
2	Injectex EF630	$(0)_{10}$	42	In-plane	2.3 10-10	7 10-13
3	G947 E3910	$(0)_{25}$	60	In-plane	2.1 10-11	2 10-13
4	E3836	$(0)_{28}$	61	In-plane	1.3 10-11	1 10-13
5	G904	$(0)_{13}$	44	In-plane	1.1 10-10	2 10-12
6	E3833	$(0)_{13}$	55	In-plane	5.6 10-11	1 10-12
7	Injectex E3795	$(0)_{13}$	55	In-plane	9.7 10-11	5 10-13
8	Injectex EF630	$(90)_{12}$	50	In-plane	2.6 10-10	2 10-12
9	6K T700 600	$(+45)_{5}$	60	In-plane	1.8 10-11	2 10-13
10	6K T700 600	$(+45)_{5}$	60	Through-the-thickness	3.2 10-12	3 10-13
11	12K T700 603	$(0)_{5}$	60	In-plane	4.8 10-11	3 10-13
12	12K T700 603	$(90)_{5}$	60	In-plane	2.1 10-11	4 10-13
13	12K T700 603	$(90)_{5}$	60	Through-the-thickness	6.8 10-12	5 10-13

CONCLUSIONS

The following is concluded:

- The measurement of the permeability of a reinforcement sample in the permeameters is a simple and fast routine. The permeameters have turned out to be adequate apparatus for measuring permeability's of reinforcement materials.
- The precision of the permeameter method is at minimum 1% during an experiment. The precision decreases to at least 6% by repeating measurements on the same preform. If the permeability of several identical preforms of an equal number of layers of the same fabric is measured, the precision will further decrease due to unknown variations in the fabric samples and the unknown influence of the preforming stage.
- The assumption is correct that a steady state flow method with which the permeability is measured is adequate to model a (non-steady state) filling process. The adequacy is proved by the agreement between the calculation of the propagation of errors and the relative standard deviation (coefficient of variation) of the within-experiment results (both 1%)
- It is recommended that, additionally, the permeability of the fabrics should be measured over a wide range of fiber volume fractions using the adjustability of the permeameter. By curve-fitting with the (modified) Kozeny-Carman equation the permeability data are more conclusive and applicable than the discrete data from the measurements of this work.

REFERENCES

1. R.S. Parnas et al., "Report on Manufacturing Polymer Composites by Liquid Molding", NISTIR 5373, September 20-22, 1993, N.I.S.T., Gaithersbrug
2. J.C. Miller, J.N. Miller, "Statistics for analytical chemistry", Ellis Horwood Ltd., Chichester, 3rd edition, 1993
3. A.E. Schedegger "The physics of flow through porous media", University of Toronto Press, 1957, p.104-105