COMPRESSIBILITY AND PERMEABILITY OF FIBER REINFORCEMENTS FOR PULTRUSION

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Abstract
The aim of this work is to characterize fiber reinforcements used in the pultrusion process in terms of compressibility and permeability. The compressibility of single and multiple layers of reinforcement are obtained by applying a defined compression load ramp to the material positioned into a tool mounted to a universal testing machine. The results are used to estimate the number of rovings which can be fed to the process for a given die cross section as well as quantity and type of planar reinforcements. A tool with a central injection nozzle is employed for the permeability measurements. The tool allows the monitoring of unsaturated as well as saturated flow through pressure transducers located at different positions within the cavity. The principal permeability parameters in the plane K_1 *and* K_2 *are obtained. The results provide important input data for pultrusion process simulations.*

1. Introduction

Fiber packages for pultrusion are usually composed of roving and continuous filament mat layers -CFM-; lately so called complex reinforcements (composed of a combination of chopped strand mat and/or surface veil and woven or non-crimp fabric layers) have also become attractive depending on the application, as they have the potential to improve mechanical properties in directions other than the profile main axis of orientation.

In RTM, planar reinforcements are usually cut according to the part shape being molded and laid into the mold before closing and injecting the resin. These reinforcements are frequently bound together, e.g. by means of powder binders or yarns, in order to attain some degree of form stability. The pultrusion process, on the other hand, is characterized by the use of a multilayered laminate structure composed of roving layers aligned to the profile longitudinal direction and planar reinforcements. As consequence of this laminate structure, it is important to precisely position the rovings in order to achieve a uniform fiber distribution across the layer, i.e. avoiding resin-rich regions and other regions with very high fiber volume content. The positioning is usually achieved by means of threading the rovings through guiding plates with hole patterns specific to the profile cross section being produced.

The importance of assessing the compressibility of fiber reinforcements becomes apparent when one is concerned with a part design composed of layers of different reinforcement materials. In order to define a stack structure and/or a processing window, the compressibility of each of the fiber reinforcements must be known (i.e. the fiber volume content as a function of compaction pressure). Especially for the pultrusion process, one needs to estimate the number of rovings which are needed to fill a given cross section, according to the compaction pressure and the number and type of planar reinforcements present in the profile [1,2].

The permeability of fiber reinforcements is an important parameter for process simulation and the purpose of better process understanding. The determination of the permeability is a widespread characterization technique for the resin transfer molding (RTM) process. There is a range of methods for determining permeability, depending on whether one is concerned with the permeability parameter in the planar or in the transverse orientation. For in-plane permeability, experimental techniques can be divided into linear and radial flow, each one having intrinsic advantages and disadvantages. Furthermore, the permeability can be obtained from unsaturated or saturated flow. In unsaturated flow, a liquid is injected through the reinforcement while the flow front position vs time is recorded. In saturated state, liquid constantly flows through the already wetted-out reinforcement, the permeability being determined from the absolute pressures at different positions and the volumetric flow rate [3].

Experimental efforts for characterizing reinforcements specific for pultrusion have been limited. A revived development trend towards closed injection pultrusion can be currently observed. Hereby, the fiber wet-out takes place in a specifically designed injection and impregnation chamber directly attached to the die, as opposed to the widespread method of guiding the fibers through a resin bath. The challenging aspect of designing such an injection and impregnation cavity is that a complete fiber wet-out must be achieved in a confined volume where the reinforcement layers are in a compacted condition. In this case, the suitability of a particular design is strongly dependent on the reinforcements being processed (and their respective permeabilities), as well as on resin viscosity and processing parameters, e.g. injection pressure and pulling speed [4].

2. Materials and methods

2.1. Materials

The following reinforcements were characterized in terms of their compressibility: glass fiber direct rovings 399A-AE with 4.800 tex (3B Company, Belgium) and CFM layers of 300 g/m² from two different suppliers (M8643 from 3B Company and Conformat® N720 from Superior Fibers, LLC, Ohio, US). Silicone oil (Korasilon® M 1000, Kurt Obermeier GmbH & Co. KG, Germany) with a dynamic viscosity of about 1 Pa.s at room temperature was used as soaking fluid for measurements of reinforcements in wetted-out condition.

Permeability measurements were performed on direct rovings 399A-AE with 4.800 tex and CFM M8643 of 300 g/m² (both materials supplied by 3B Company). Silicone oil (Korasilon® M 100) with a dynamic viscosity of about 0,1 Pa.s at room temperature was used as permeating fluid.

2.2. Tool design and methods

For measuring the compressibility, a tool with a 180 x 346 mm² cavity is employed. The tool for measuring the permeability has a cavity of 396 x 396 mm² and a central injection nozzle. Both tools are completely made of steel, in order to avoid deflections (and consequently local volume content variations), especially when applying the relatively high compaction pressures common in pultrusion. The tools have lateral shearing edges and both ends open to the atmosphere. This allows a roving fiber package to be positioned in a similar way as it would be in a pultrusion die, with the individual rovings stretched and in parallel orientation. The tools are mounted to a universal testing machine.

2.2.1 Compressibility measurements

For measuring the compressibility, the reinforcement material was cut to the cavity dimensions, laid into the tool and then compressed between the two tool halves. When measuring rovings, the positioning was achieved by means of guiding plates similar to the ones used in the pultrusion process. The roving layer is composed of 159 roving ends. Measurements of CFM were performed on single and double layers. A mat/roving/mat laminate structure typically processed in pultrusion was measured, as well as layers wetted out with silicone oil. The measurement assembly with a layer of rovings already in place is shown in Figure 1(a).

The load program was controlled at a displacement rate of 1 mm/min, and the curve force vs displacement was recorded, which was then converted to pressure vs fiber volume content. The material was compressed up to a compression force of 100 kN, which corresponds to 1,64 MPa compaction pressure. The relaxation behavior discernible in dry fiber reinforcements [1] was not examined for the purpose of this analysis.

2.2.2 Permeability measurements

For the permeability measurements, the cavity thickness is fixed by placing a stack of steel strips of defined thickness laterally between the tool halves. The tool is closed with the reinforcement material inside, such that the upper tool half prevents the material from shifting. Directly thereafter, a hole is cut through the reinforcement by a punching tool with diameter 10 mm directly over the outlet of the central injection nozzle. The testing machine presses the two tool halves until the distancing stack can no longer be moved, setting thus the cavity thickness.

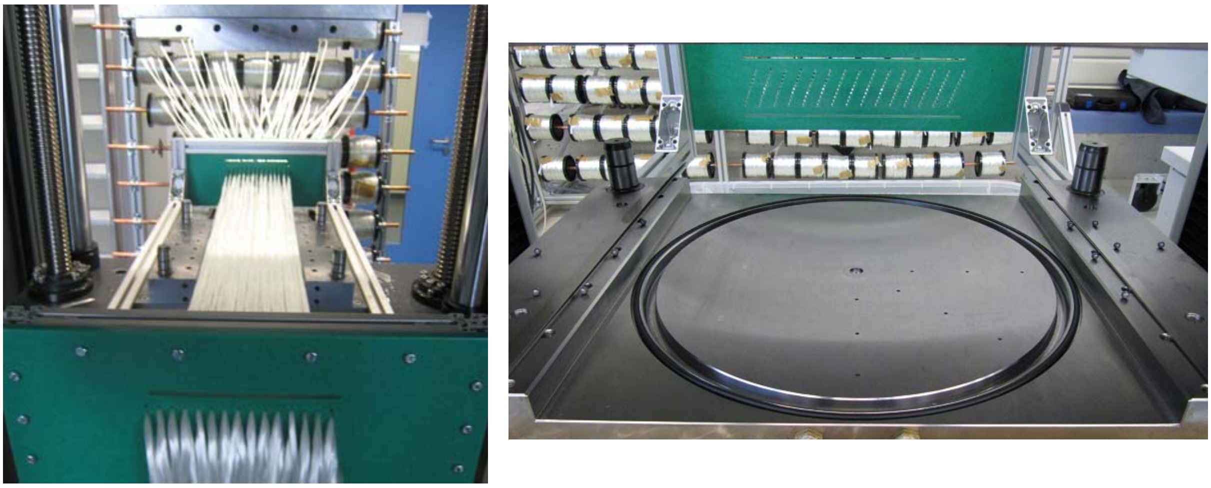
Silicone oil is injected with constant pressure through the nozzle. Injection under constant pressure is achieved by a pressurized pot. The flow front is detected when reaching pressure transducers located at different positions within the cavity. 10 transducers are mounted to the tool. A picture of the open tool is shown in Figure 1(b).

The sensitivity of the detection method was previously assessed by comparing visual flow and transducer response, and was determined to be below 2 mm, which was the difference between visual observation and detection of flow front by the transducer.

The permeability of CFM was measured for fiber contents of 17, 22 and 24% Vol. The fiber volume contents were chosen according to a compaction pressure range typically processible in pultrusion ($7x10^4$ – $4x10^5$ Pa). The material was cut from the roll to the tool dimensions and stacked to 4 pieces. The orientation relative to the roll was kept constant when stacking and laying into the tool (see Figure 4). A total of 10 measurements were performed for each fiber volume. Injection pressure was set to $6x10^5$ Pa.

Rovings were measured for a fiber content of 60% Vol. 300 roving ends were placed in the tool in a similar way as shown in Figure 1(a) and the cavity thickness was set to 2,25 mm. A compaction force of about 140 kN was applied to close the tool. Injection pressure was set to $1x10^5$ Pa for flow front measurements in unsaturated state and then increased to $5\ x10^5$ Pa for saturated flow measurements.

The analysis proposed by Weitzenböck et al [5] is employed to obtain the principal permeability parameters in the plane K_1 and K_2 as well as the angle between the principal permeabilities and the axes of the coordinate system for unsaturated flow. The absolute pressure values at different positions in the cavity are used to determine the permeability of a roving layer in a saturated flow condition. In order to find the permeability parameters, the steady-state flow is simulated in CFD software (Ansys CFX). An optimization function is implemented to find K_1 and K_2 which minimizes the error between simulated and measured pressures for a group of 3 pressure transducers.



(a) (b)

Figure 1(a) Assembly for measuring the compressibility of a roving layer; **(b)** Permeability measurement tool showing locations of pressure transducers, distancing stack for setting cavity thickness and rovings guiding plate.

3. Results and discussion

3.1. Compressibility

The curves of compaction pressure (P) vs. fiber volume content (V_f) for rovings and CFM are shown in Figure 2. Experimental data is found to be well fitted by a logistic function such as equation (1).

$$V_f = \frac{A_1 - A_2}{1 + (P/P_0)^b} + A_2 \quad (1)$$

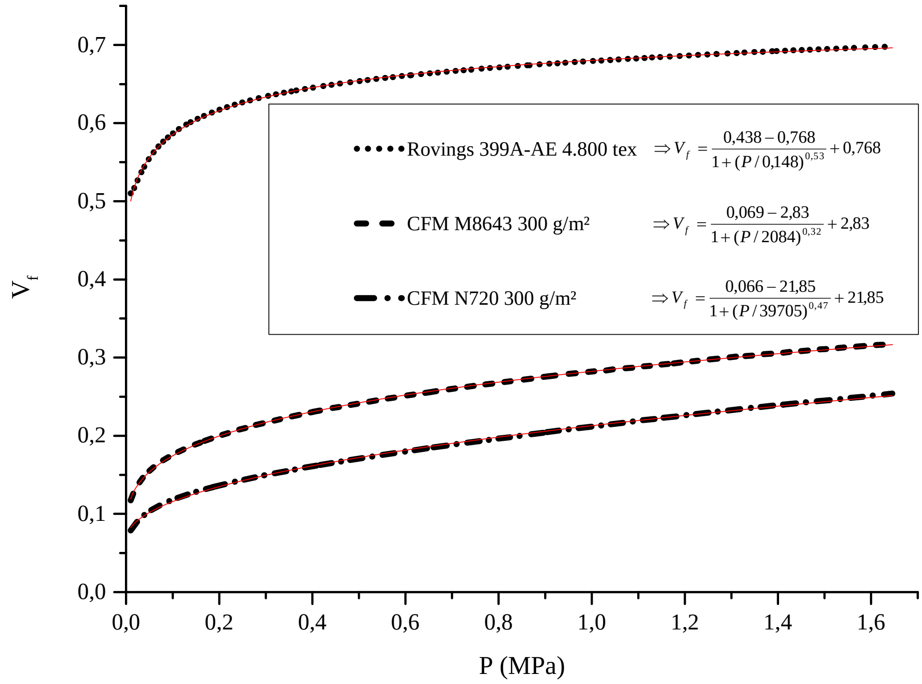


Figure 2 Compressibility curves for rovings and CFM. The fitted curves corresponding to each material are plotted as red lines.

The compression behavior of rovings can be described as relatively insensitive to variations in the applied mechanical pressure. Already a small variation in the number of rovings ends being fed to the pultrusion die can have a significant impact on processing stability; if the fiber volume is too low, resin deposition is likely to occur in the die, if it is too high, pulling the bundle through the die will not be possible.

CFM on the other hand are generally more compressible, and are effectively applied in pultrusion processing to extend the processing window by balancing variations in the rovings layer. It is also noteworthy that the compressibility of apparently similar materials may be very different, as clearly seen for the CFM from different suppliers.

The nesting effect present in a multilayer laminate is also represented by comparing the measured curve of laminate thickness (s) vs. compaction pressure (P) for a mat/roving/mat stack and the added curves of the same layers (see Figure 3). By nesting, one understands the interpenetration of fibers between layers. Due to nesting, there will be an error of about 10% in the cavity thickness (and consequently in the average fiber volume content) when using the single curves of the materials to calculate the multilayer laminate structure. In fact, a lower number of rovings will be determined than actually needed to achieve the same laminate thickness at the given compaction pressure.

The effect of wetting out the reinforcements was also investigated. For low pressures (0 – 0,1 MPa), the wet-out curve is close to the theoretical curve of added layers. That means that at this pressure range, wetting-out of reinforcements compensates the nesting effect. For the pressure range up to 0,6 MPa, the compressibility curves of the wet-out and dry reinforcements approximate each other, as the compaction pressure effectively squeezes out the soaking fluid. In this pressure range, the error incurred by adding up the layers must be taken into consideration. It should be noted however, that dependence is expected between profile structure and the deviations discussed above, for example in the case of a thick laminate (over 4 mm) in which more than 2 layers of CFM are present, or when working with a resin of high viscosity.

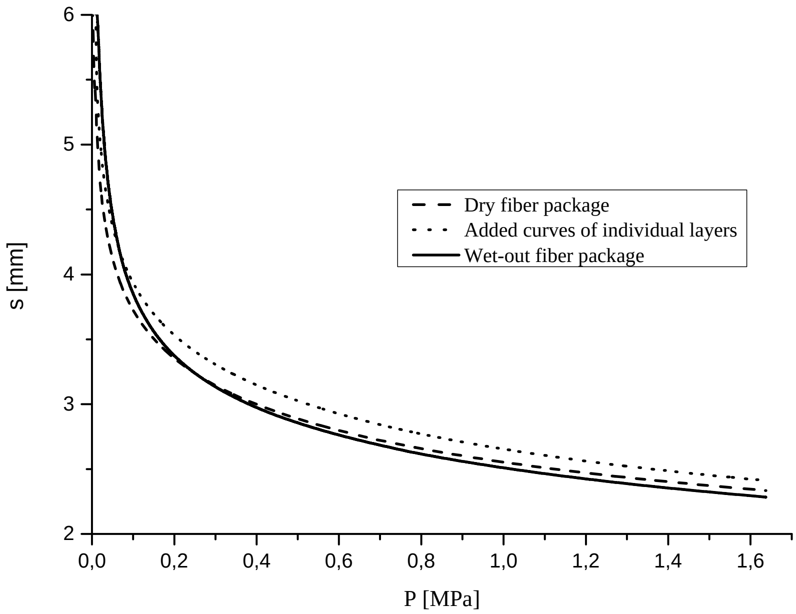


Figure 3 Compressibility measurements on multilayer stacks composed of 1 x CFM N720 300 g/m² / 80 rovings / 1 x CFM N720 300 g/m² and comparison with value obtained by the added curves of the individual layers.

3.2 Permeability

The permeability results for the CFM layers are shown in Figure 4. It is interesting to note that with increasing fiber volume content, the accuracy of the measurement clearly improves for K_1 and K_2. One possible explanation for this behavior might be the fact that for lower volume contents, the fluid flows not only through the bulk region of the mats, but also to some extent between the mat and the tool wall. These local variations may be suppressed when the fiber stack is subjected to a higher compaction pressure, resulting thus in flow only through the porous material.

On the other hand, the scattering of the determined angle between the principal permeabilities and coordinate system increases with increasing fiber volume content. This might arise from the inherent fluctuation of specific weight in CFM materials. A specific weight with ± 10% fluctuation is typically declared for this kind of material. These local variations may result in scattering on the orientation of main permeabilities between the measurements. Contrary to the general assumption that CFM is an isotropic material, a discernible anisotropic permeability was observed, with the permeability in the transverse direction relative to the roll winding direction higher than in the parallel orientation.

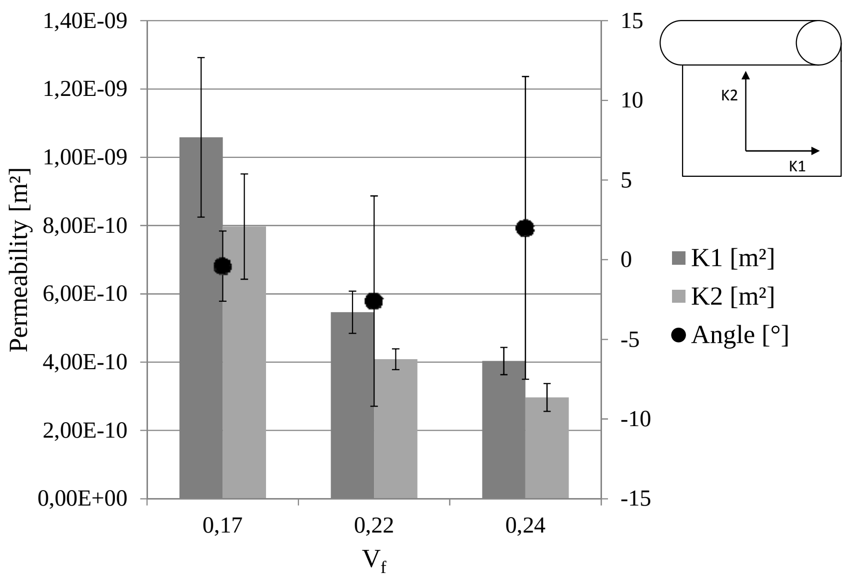


Figure 4 Results of permeability measurements on CFM M8643 of 300 g/m².

Measurements of roving layers showed a higher degree of anisotropy than predicted by models such as proposed by Gebart [6], and experimentally determined by Schell et al [7]. These studies presented for a bundle of parallel oriented fibers a difference between longitudinal and transverse permeability of around one degree of magnitude. Such a difference lies within the limits of the measurability by the method of Weitzenböck et al [5] and the tool configuration. Higher degrees of anisotropy however result in the testing fluid reaching the third pressure transducer in the longitudinal before the first transducer in the transverse direction. This flow behavior prevents an analysis of the unsaturated flow.

For the saturated flow, it is necessary that a steady state is reached where the pressure distribution over the measurement region no longer varies with time. The permeability parameters are obtained by an optimization function implemented in Ansys CFX Workbench. This optimization procedure evaluates the minimum residuals between 3 measured and simulated pressure values located in the principal axes x and y, and at an angle of 45° to the principal axes. It is assumed that the principal permeabilities coincide with the principal axes, that is $K_1=K_x$, $K_2=K_y$. The results obtained for three measurements, as well as values predicted by Gebart [6] for the nominal fiber diameter of 24 µm are shown in table 1.

	Permeability [m²]		
	Calculated values	**Gebart, quadratic fiber packing**	**Gebart, hexagonal fiber packing**
K_1	(1,32±0,09)x10^{-11}	1,44x10^{-11}	1,55x10^{-11}
K_2	(1,45±0,70)x10^{-13}	1,82x10^{-12}	3,36x10^{-12}

Table 1 Calculated values for longitudinal (K_1) and transversal (K_2) permeability parameters of glass fiber rovings of 4.800 tex.

From the development of the pressure profiles as well as the calculated permeability values, it is clear that the longitudinal permeability is much higher than the transverse one. The longitudinal permeability is close to the model prediction, while the transversal flow results in a lower value than expected. One assumption is that the discrepancy might be due to an imperfect parallel orientation of the fibers. Stacked rovings create regions with higher volume content, which effectively prevents transverse flow. Twisting and undulations are characteristic and very discernible in direct glass fiber rovings, which might contribute to obstruction of flow. Furthermore, these characteristics make a perfect stretching of the fibers more difficult, accentuating the problem of fiber stacking. Nevertheless, the measurement assembly reproduces to a large extent a typical configuration of a pultrusion die in-feed. The experimental results obtained are thus considered to be representative of flow conditions through a roving layer prevailing in pultrusion processing.

4. Conclusions and outlook

This study focused on the characterization of fiber reinforcements used in the pultrusion process. The results of the compressibility measurements are applied regularly as a basis to calculate suitable laminate structures for processing; hereby it is possible to determine the desired type, width and number of planar reinforcements or rovings for a known profile section. Permeability measurements show a good reproducibility for mats, especially for relatively high fiber volume content. For the measurements on roving layers, a longitudinal permeability in reasonable agreement to available data and models in the literature was found, while the transversal permeability was lower than expected. More measurements on roving layers are needed to assert the reproducibility of the method.

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