FLOW INDUCED SAMPLE DEFORMATIONS IN OUT-OF-PLANE PERMEABILITY MEASUREMENT

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Abstract

Efficient design of Liquid Composite Molding (LCM) process variants providing out-of-plane impregnation requires accurate knowledge of the related permeability of the preform. During this kind of processes, the out-of-plane flow is shown to induce a non-homogeneous hydrodynamic compaction of the textile stack, making it difficult to assess the permeability to certain fiber volume contents. The aim of this study is to validate a numerical model which describes saturated out-of-plane flow including hydrodynamic compaction and calculates the apparent permeability at different pressure differences. For this, the model introduces permeability values from low injection pressures (0.5 bar) with presumably negligible hydrodynamic compaction coupled with data on the compaction behavior gained from tests on a universal testing machine. The simulation results are then compared to experimental results obtained with a permeability measurement system allowing online-monitoring of stack compaction. The comparison between permeability values predicted by simulation and measured in experiments shows small deviation of 0.2 to 30% for three different textiles (glass fiber woven fabric, non-crimp fabric and random matt) investigated at different FVC ranging from 35 to 56% and injection pressures ranging from 0.5 to 5.6 bar.

1. Introduction

In order to make LCM processes more attractive for mass production, understanding impregnation behavior of engineering textiles is essential. The injection in out-of-plane direction leads to complex hydrodynamic compaction of the stack, as illustrated in Figure 1: At the onset of injecting the fluid, but prior to textile impregnation the fluid pressure compacts the unsaturated textile; As the textile is saturated the part is inhomogeneously compacted as the fluid pressure decreases over the flow length. The unsaturated part is however continuously compacted by the injection pressure. Once the stack is completely saturated, the fluid pressure decreases of the complete stack height and the complete stack is inhomogeneously compacted. After removal of the injection pressure (no fluid flow) the layers relax again until homogeneous compaction is reached. This inhomogeneous compaction during an injection in out-of-plane-direction has also been described e.g. by Michaud et al. [1] and Springer [2].

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As demonstrated, this complex compaction is occurring during out-of-plane permeability measurement, thus making it difficult to account the measured permeability to certain fiber volume content. In a joined project the Institut für Verbundwerkstoffe GmbH and the ETH-Zürich intend to develop a novel experimental-simulative approach for out-of-plane permeability characterization. The target is to use simulation to model the actual FVC conditions during the experiment and then correct for the permeability – FVC allocation.

In a first step, a model for the saturated flow case is developed followed by the introduction of permeability values. The permeability values considered were allow injection pressure (0.5 bar) with presumably negligible hydrodynamic compaction as determined experimentally. In order to validate the simulation, the numerical results are then compared to experimental results obtained with a permeability measurement system allowing online-monitoring of stack compaction.

2. Modeling

The fiber bed is modeled as a fully saturated compressible porous media impregnated with a constant viscosity fluid under isothermal conditions. The fluid flow is considered in the out-of-plane direction assuming even distribution with no in-plane impregnation. The model is implemented in COMSOL Multiphysics, it describes the time dependent interaction between fluid flow and fiber bed deformation. The model geometry is presented in Figure 2, it is considered as a 1D problem with a fixed size and number of elements of 100 over the thickness. It defines a constant cavity height and number of layers determining nominal fiber volume fraction. Inlet and outlet pressures are considered constant with the fiber-bed experiencing a compaction load introduced from the bottom and defined as a free edge. This allows deformation in the z direction, unlike the top end which is considered as fixed.
Governing equations

Fluid flow through porous media is widely investigated in the literature, it is generally modeled by solving Darcy’s equation (1) [4], which defines the fluid velocity as a function of the gradient pressure:

\[
\langle v \rangle = -\frac{k_z}{\mu} \nabla (p^\Phi)
\]  

where \( \langle v \rangle \) is the volume averaged fluid velocity through the porous medium, \( k_z \) is the out of plane permeability, \( \mu \) the fluid dynamic viscosity, \( \Phi \) the fiber-bed porosity and \( \nabla (p^\Phi) \) is the gradient of the pore-averaged fluid pressure that drives the flow. The compaction behavior of the fiber-bed is described using Hook’s (2)

\[
\sigma = E(vf, t) \cdot \varepsilon = E(vf) \cdot \frac{dw}{dZ}
\]  

where \( \sigma \) is the effective stress applied on the fiber-bed, \( E \) is the Young’s modulus which depends on the FVC \( vf \) and \( \varepsilon \) is the strain defined as a gradient in the Lagrangian description where \( w \) is the displacement in \( z \) direction and \( Z \) is the Lagrangian coordinate.

An important relation, which has to be taken into account in the flow-induced deformation of fibrous reinforcement, is Terzhagi’s principle (3) [3]. The effective stress formulation states that at any point in the laminate the following relation must be satisfied:

\[
p_{app} = \langle p \rangle^\Phi - \langle \sigma \rangle
\]

where \( p_{app} \) is the total pressure applied on both fiber-bed and the fluid, \( \langle p \rangle^\Phi \) is the pore-averaged fluid pressure and \( \langle \sigma \rangle \) is the effective stress applied on the preform.

In order to describe fluid flow in a saturated and deformable porous media, the previous equations are coupled together with the stress equilibrium equation and solved for the mass conservation equations to derive the general transport equation (4) [5].

\[
\frac{\partial \Phi}{\partial t} + \nabla \left( \Phi \frac{\partial (w)}{\partial t} e_z \right) = \nabla \left( \frac{k_z}{\mu} \nabla (p^\Phi) \right)
\]  

3. Materials & Methods

3.1. Material

In the experiments, three different textile architecture were tested, a twill 2/2 woven fabric (Hexcel 1202) with 290 g/m² (WF), a random mat (PDFibreGlass Group MA 111-300) with 300 g/m² and a non-crimp biaxial (± 45°) fabric (Saertex X-E-444) with 444 g/m² (NCF) have been used along with the silicone oil test fluid (XIAMETER PMX-100 from DOW CORNING) with a viscosity of 100 mPas at a temperature of 20°C. The viscosity of the test fluid depending on the temperature was determined with the Rheometer from Anton Paar, Modell MCR302 in a temperature range of 15-40°C. In order to determine the real areal weight of the used textiles, each fiber stack sample was weighed before starting
permeability measurements. Knowing the number of layers, the total weight and the cross-section area of the samples, the average single layer heights were calculated to obtain the true FVC in the permeability experiments. The result of the measurement of the area weight of the used textiles shows a small variation in a range between 2.2% for the random mat, 0.17% for the WF and 0.1% for the NCF. Also the deviations of the theoretical areal weight to the measured area weight just have a small range between 1.5% for the WF, 0.43% for the NCF and 0.03% for the random mat.

3.2. Compaction measurements

In order to extract the wet compaction curves required for the simulation experiments, the three different materials were compressed in a fully wetted state, using a Zwick Materialprüfung 1474 universal material testing machine. This was accomplished using two round load aluminum plates with a diameter of 120 mm, where the lower plate is fixed and the upper one attached to a movable crosshead where a 100 kN load cell is mounted to measure the reaction force. The experiments were controlled via the Zwick Roell testXpert III test software using displacement controlled testing. In order to perform wet compact experiments, the setup introduces an oil bath with an aluminum base plate and transparent sidewalls, made from PMMA (acrylic glass). The oil bath is filled gradually by letting the test fluid flow slowly in the container from the bottom to the top, allowing a complete wetting of textile. Stepwise compaction tests were performed to obtain the quasi-static compaction curves needed to understand the saturated flow induced deformation [6]. The compaction of the textile sample was implemented using a constant velocity of 4 mm/min up to a defined FVC. This was followed by holding until the relaxation stabilized, in which the holding pressure was then measured. The sample is than further compacted repeating the same process for higher FVC presented in Table 1.

<table>
<thead>
<tr>
<th>Textiles</th>
<th>Step 1</th>
<th>Step 2</th>
<th>Step 3</th>
<th>Step 4</th>
<th>Step 5</th>
<th>Step 6</th>
<th>Step 7</th>
<th>Step 8</th>
<th>Step 9</th>
</tr>
</thead>
<tbody>
<tr>
<td>Woven</td>
<td>40%</td>
<td>42%</td>
<td>44%</td>
<td>50%</td>
<td>52%</td>
<td>54%</td>
<td>56%</td>
<td>58%</td>
<td>59%</td>
</tr>
<tr>
<td>NCF</td>
<td>40%</td>
<td>42%</td>
<td>44%</td>
<td>50%</td>
<td>52%</td>
<td>54%</td>
<td>56%</td>
<td>58%</td>
<td>59%</td>
</tr>
<tr>
<td>Random Mat</td>
<td>24%</td>
<td>26%</td>
<td>28%</td>
<td>30%</td>
<td>34%</td>
<td>36%</td>
<td>38%</td>
<td>40%</td>
<td>41%</td>
</tr>
<tr>
<td>Holding time (s)</td>
<td>200</td>
<td>200</td>
<td>200</td>
<td>500</td>
<td>500</td>
<td>500</td>
<td>1000</td>
<td>1000</td>
<td>1000</td>
</tr>
</tbody>
</table>

The quasi-static compaction curves are extracted by joining the ends of each relaxation step defined by the red dots in Error! Reference source not found..b, and the experiment has been performed on 5 different samples comprising of 20 layers for each of the considered textiles.

**Figure 3:** Illustrative graphs for stepwise compaction showing how to obtain quasi static compaction curves (red dots) a. compaction evolution over time. b stepwise compaction curve

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3.3 Permeability and hydrodynamic compaction measurements

For measuring permeability in out-of-plane direction and also the total stack compaction, the measurement system “HyKoPerm” [7], developed by the IVW shown in Figure 4 was employed. The system monitors pressure difference between in- and outlet, lower layer displacement and volume flow of the test fluid. With the known variables, the volume flow $Q$, the cavity height $\Delta L$, the cross-section surface $A$ and the viscosity of the fluid $\mu$, the out-of-plane permeability $k_z$ can be calculated with Darcy’s law (5)[4].

$$Q = -\frac{k_z \cdot \Delta p \cdot A}{\mu \cdot \Delta L}$$  \hspace{1cm} (5)

Figure 4: Measurement system “HyKoPerm” for measuring the permeability of saturated engineering textiles in out-of-plane direction incl. total stack compaction monitoring.

The pre-compaction of the fiber stacks to FVC is realized with two hole plates. These hole plates are highly permeable for the test fluid compared to the used textiles and have a high mechanical stiffness. The exact cavity height can be adjusted (adjustment tolerance of ± 0.05 mm) with spacers between the case and the cover of the measurement cell. Squeeze seals contain the fiber stack to the outside and limit the measurement area to an elliptical cross section of 15,000 mm². The pressure difference is monitored by pressure sensors at the in- and outlet. To measure the total stack compaction, the lower hole plate is moveable and slightly spring-loaded to ensure constant contact to the lowest layer due to hydrodynamic compaction. Three linear variable differential transformers (LVDTs) track the displacement.

The permeability is measured saturated, requiring the fiber stack to be impregnated before the measurement at a very low pressure (< 0.5 bar) to prevent hydrodynamic compaction from the injection pressure. Before measuring, the fiber stacks have been compacted five times to the final FVC. This pre-compaction reduces the influence of compaction history, which is a result of handling of fiber and increases reproducibility. The experiments have been carried out with three different nominal FVC for each textile: 35%, 39% and 42% FVC for the random mat; 46%, 48% and 53% FVC for the WF; 46%, 52% and 56% FVC for the NCF. The measurements have been carried out at different pressure levels staring with a very low pressure difference of 0.05 bar for the random mat and 0.1 bar pressure difference for the WF and NCF – the permeability obtained at this pressure was used as input for the simulation as it was assumed the hydrodynamic compaction is negligible. Subsequently injection pressure was increased in steps (pressure difference) of 0.25 bar with the WF and NCF and 0.05 bar with the random mat (due to the better permeability) up to a maximal pressure difference between 0.2 and 2.5 bar between in- and outlet, depending on textile and FVC. The FVC can permeability measured at these higher pressures were used for validation. Figure 5 exemplarily shows a result: hydrodynamic stack compaction

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occurs every time the pressure difference is increased.

**Figure 5:** Pressure difference to displacement due to hydrodynamic compaction, with Seartex X-E-444 non-crimp glass fiber biaxial fabric (444 g/m²), with a FVC of 56.3% and 11 layers.

4. Results

4.1. Compaction behavior

Figure 6 shows the quasi-static wet compaction curves resulting from the stepwise loading, for the twill 2/2 woven, the biaxial (±45°) NCF and the random mat fabrics. The results of the 5 different samples are averaged for each fabric then fitted with an exponential curve, those curves are used in the next step for the flow simulation as an input data.

The presented compaction curves show a nonlinear behavior, where an increase in FVC with the applied stress is observed. The mat fabric is less compressible than the other two fabrics which are comparable, this indicates that for a certain injection pressure, the hydrodynamic pressure will affect more the woven than the NCF followed by the mat samples.
4.2. Validation of the simulation model

**Error! Reference source not found.** shows the numerical and experimental results of the permeability evolution as a function of the fiber volume fraction for the three fabric architectures. In these graphs we consider four measurement points for each FVC with the following pressure differences \{0.1, 0.2, 1 and 2\} bar respectively for the WF and the NCF, and \{0.05, 0.1, 0.25, and 0.5\} bar for the random mat.

![Figure 7: Comparison between numerical and experimental results of the permeability function of FVC and pressure differences a. woven, b. NCF and c. random mat](image)

Generally, the match between simulation and experiment is getting better with increasing nominal FVC and at a low injection pressure. This corresponds to the expectations as increasing FVC and decreasing injection pressure both reduce influence of hydrodynamic compaction. Figure 8 presents the difference between measured and calculated permeability and FVC as a function of the applied pressure difference.

![Figure 8: Difference between experimental and numerical results of permeability and FVC at the used nominal FVC and pressure differences](image)

The discrepancy between numerical and experimental results is at its lowest at low pressure differences. This is explained by the resulting low hydrodynamic compaction; in which the fiber remains relatively fixed and thus the simulation can reproduce rather precisely the experiment. This is not the case with high pressure differences, especially at low nominal FVC where high deformations take place leading to an increase of FVC. It is to mention that some measurement points escape from the general rule, in the WF for example, a difference of 1% in FVC is noticed at 2 bar of injection pressure and 53% nominal FVC. A considerable variation of 20% in permeability is also noted in the NCF at 52% nominal FVC for the different pressures.

The reason for the smaller averaged variation of the random mat material compared to the WF and the NCF can be assumed to be the lower compressibility, which is the result of much higher stiffness compared to the other investigated textiles, as observed in Figure 6. Therefore, the hydrodynamic compaction due to the injection pressure is also decreased. Furthermore, the differences between experiment and simulation are quite small over the whole measurement range. The deviation between simulations and experiments can partly be traced back to the variation coefficient of the textiles and the resulting variation coefficient of the experiments. It is clear that the compaction behavior of a single textile layer is not comparable with the compaction behavior of a fiber stack due to nesting effects.

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between the layers. Also, during permeability measurements the fluid flows in out-of-plane direction, while during compaction measurements the fluid is pushed out in in-plane direction, which can cause the sample to widen, which is not possible during permeability measurement due to the surrounding squeezing edge.

5. Conclusion

In this study, a numerical model calculating saturated out-of-plane permeability, while taking into account flow induced deformation, was implemented. The model describes fluid flow through porous and deformable media. The input parameters are based on quasi-static wet compaction measurements and permeability measurements at low pressure difference (where no hydrodynamic compaction is expected). In order to validate the simulation, numerical results were compared with experimental results using an out-of-plane permeability measurement system which allows a simultaneous online monitoring of permeability and compaction in out-of-plane direction. The three textile architectures tested undergo the same influence of injection pressure. However the extent of this influence varies with the fabrics due to the difference in compaction properties. The comparison shows a good agreement between experiments and simulation, where small variations are noted especially at low pressure differences and high nominal FVC, slightly bigger deviations take place in lower FVC and high pressures. Therefore the model can be considered valid and suitable for the prediction of permeability and hydrodynamic compaction.

The current setup and simulation form the basis for the development of a novel measurement system, and a more thorough numerical model, which will allow monitoring of the relevant effects taking place during unsaturated out-of-plane impregnation, including single layer displacement within the stack.

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6. References