

# IN-PLANE PERMEABILITY TENSOR IDENTIFICATION USING AN INVERSE METHOD

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**SUMMARY:** The simulation of a resin flow through a porous medium by FE-models has become a very important aspect for the design of a high-performance RTM produced composite part. The key parameters to perform RTM flow simulations are the permeability values of the fiber reinforcement. The measurement of this material parameter is still not standardized and thus many different set-ups have been proposed. This paper presents an inverse method, or a so-called mixed numerical/experimental technique, for the identification of the permeability values. In this iterative inverse technique an experimental observation on a highly automated central injection rig, called "PIERS set-up" (Permeability Identification using Electrical Resistance Sensors), is compared with a computed observation using a numerical model that simulates exactly the same experiment. In this model the permeability values will appear as parameters which will be iteratively tuned in such a way that the computed observation matches the experiment. The inverse method combined with the PIERS set-up allows a fast and accurate identification of the permeabilities. Moreover, this article presents a solid test specimen, produced with a stereolithography technique, which can be used as a reference sample for calibration of test rigs and for comparison of results from different test rigs.

**KEYWORDS:** permeability, fabrics, textile, Resin Transfer Molding (RTM), numerical analysis

## INTRODUCTION

In today's market an increasing demand for high-quality fiber reinforced polymers (FRP's) can be noticed. Applications range from automobile, sports, marine, heavy-duty machinery to household applications. Interest in Resin Transfer Molding (RTM) as a production technique has risen significantly over the past few years because of higher demands on finish quality, production volumes, cycle times and last but not least, environmental implications.

In RTM the fiber reinforcement is initially dry and is often assembled outside the mold in the shape of the finished part. The fiber assembly, called a preform, is then placed in the mold cavity and impregnated with the liquid resin which polymerizes to yield a rigid composite. Probably the most critical component in RTM is the mold. It represents a large part of the capital investment of the production line and determines the final shape and quality of the manufactured part. In the past, the positions of resin inlets and air vents to ensure a correct filling of the mold were simply chosen by the designer, based on his experience. The mold was next manufactured, an injection was performed and the result evaluated. If there were defects like incomplete filling, the position of the gates and vents was adapted by trial and error and a new test-run was performed. This trial-and-error method was not only very time consuming, it was also expensive in both labor and material cost. In complex modern molds the flow pattern of the resin can no longer be predicted from basic rules-of-thumb and experience. Therefore flow simulation software has been developed to assist the designer in the development of a new mold design [1, 2]. Through injection simulations performed on a computer, possible problem areas in the design can be identified and the mold design can be adapted appropriately, even long before the mold is actually made [3, 4].

However, the simulation software needs reliable input data to correctly predict the flow front evolution as a function of time, in particular the permeability values of the reinforcement. The permeability of a porous medium may be described as “a measure of the ease with which a fluid flows through the reinforcement” [5] or “the property that defines how easy it is for a fluid to penetrate a porous medium” [6].

It has to be noted that it is a material parameter that is linked to the geometry of the material. This article describes a new method for the identification of the permeability tensor using registered experimental data. The proposed method is a so-called mixed numerical/experimental technique (MNET) for material property identification. The principle of a mixed numerical/experimental technique is to compare an experimental observation on a test set-up with a computed observation using a numerical simulation of the same set-up. The permeability parameters in the numerical model are the unknown material properties that the method aims to identify. These parameters in the numerical model will be tuned in such a way that the computed observation matches the experiment.

In the present work a highly automated central injection rig, called “PIERS set-up”, is used for obtaining the required experimental data. This PIERS (Permeability Identification using Electrical Resistance Sensors) set-up enables a very fast and automated registration of the flow front evolution vs. time [7].

As previous applications of the MNET, one can mention those presented by Sol [8] where the method was applied for anisotropic thin plate rigidities and a non-destructive procedure (Resonalyser) for stiffness identification of orthotropic materials. De Visscher [9] implemented a new feature in the Resonalyser: the identification of damping properties of thin orthotropic plates.

Furthermore, this paper presents a solid test specimen, produced with a stereolithography technique, which can be used as a reference sample for calibration of test rigs and for comparison of results from different test rigs. Since the structure consists of unit cells, the permeability values can also be easily estimated by means of numerical flow simulation software. Hence, this simulation software can be experimentally validated.

## MIXED NUMERICAL/EXPERIMENTAL TECHNIQUE (MNET) FOR PERMEABILITY IDENTIFICATION

A summary on the mixed numerical/experimental technique is given by the following flow chart (Fig. 1):

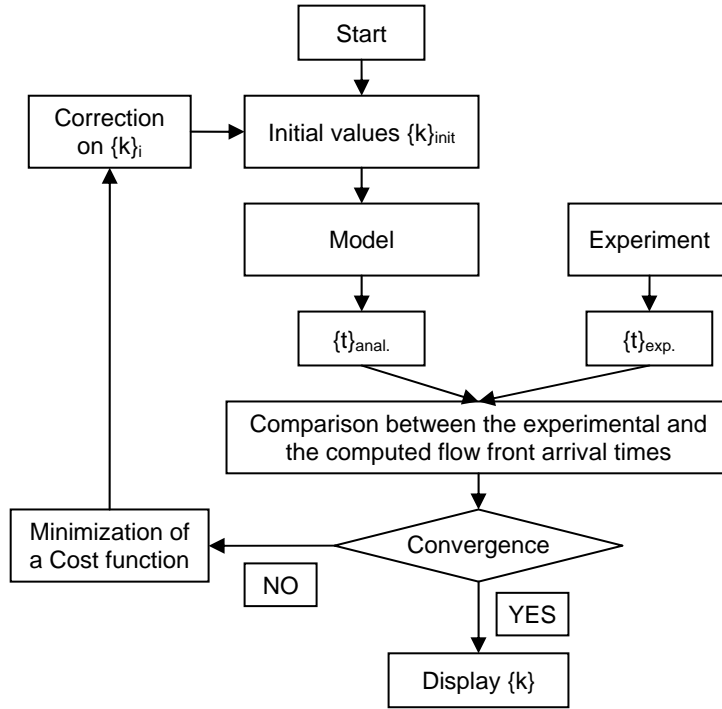


Fig.1 Schematics of the inverse method.

In a two-dimensional central injection experiment the flow front arrival times  $\{t\}_{exp}$  are recorded in well-known positions. On the other hand, there is a mathematical model that simulates exactly the same experiment. In this model the permeability values will appear as parameters and the results  $\{t\}_{anal.}$  are compared with those of the actual experiment  $\{t\}_{exp.}$ . In general there will be a discrepancy between the two results. The parameters of the mathematical model are then updated and the simulation is run once more. The results are again compared with the measurements. If the agreement is not yet sufficient, the model parameters are updated again and the simulation is repeated. This iterative solving process is continued until satisfying agreement between experiment and simulation is reached. The parameter values that were used in the last simulation are the permeability values  $\{k\}$  of the studied reinforcement. The different aspects of this approach are discussed in the following paragraphs.

### Experimental Part (PIERS set-up)

The experimental part of the proposed MNET is represented by the PIERS set-up. This set-up (Fig. 2) was described in detail in a paper of Kris Hoes et al. [10] and in his Ph.D. thesis [7]. In this paragraph, a brief description and some additional enhancements will be given. As shown in Fig. 2, the mold cavity in the set-up consists of 2 so-called sensor plates (300 mm x 300 mm).

These steel plates each hold 60 electrical sensors which are positioned on radial lines, every 30°. Each radial line has 5 sensors at 25, 55, 85, 115 and 145 mm from the center of the plate (Fig. 3).

The sensor plates are built into a hydraulic press which is operated by an electro-hydraulic unit that lifts the lower sensor plate and spacers (Fig. 4) until the spacers touch the upper sensor plate. The 4 spacing rods are specifically developed to assure the correct cavity thickness between the 2 sensor plates for the placement of the reinforcement. After placing the reinforcement and closing the mold, the test fluid can be injected. This is done centrally in the reinforcement through a hole of  $\varnothing$  6mm in the middle of the lower sensor plate. While the flow front propagates through the reinforcement, the fluid flow will make contact with the electrical sensors. Since an electrical conductive fluid (corn syrup) is used, the wetting of these DC-resistance sensors will change their (theoretically) infinite electrical resistance value into a finite value. Such a variation is registered by a PC equipped with a data acquisition system and hence an arrival time for the sensor can be stored.



Fig. 2 PIERS set-up.



Fig. 3: Sensor plate.

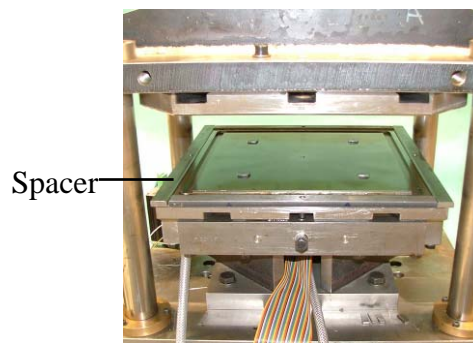


Fig. 4 Set-up with the spacers.

## The Model

As shown in Fig. 1, the computed flow front arrival times  $\{t\}_{anal.}$  are compared with the experimentally measured times  $\{t\}_{exp.}$ , through which the permeability tensor can be adjusted step by step until  $\{t\}_{anal.}$  and  $\{t\}_{exp.}$  agree. This iterative procedure requests a mold filling simulation each time the permeabilities are modified. The number of simulations depends on the initial values of the permeabilities and on the number of steps that are necessary to bring these values close to the final solution. In order to have a fast permeability identification procedure, the computational time required by the model should be as low as possible.

A model that gives accurate flow front arrival times has been developed for the implementation in the MNET and is described in detail in a number of papers of the authors [11, 12]. It has been compared with an analytical solution for an injection into isotropic material. The small relative errors demonstrated that the model gives the same accurate flow front arrival times as the analytical solution.

### Optimization Algorithm

Starting from an initial value, a better set of parameters is generated, and this process is repeated until it is decided that the process has converged (Fig. 1). So there are three basic steps in the search routine which will be discussed briefly hereafter.

#### *Selection of the starting values*

Before beginning the optimization procedure it is necessary to generate starting values. In practice the convergence region of most optimization methods is limited; if the starting values are selected outside this region, the method will diverge. Even if there is convergence, the final result can depend upon the starting values if the cost function has local minima.

The initial permeability is calculated by an analytical solution of the injection into an orthotropic medium as proposed first by Adams & Rebenfeld (A&R) [13, 14].

#### *Generation of an improved set of parameters*

The generation of the improved set of parameters is the kernel of the optimization algorithm. Several different techniques are possible. Here, the method of Levenberg-Marquardt is used. This is a combination of the Gauss-Newton method and the gradient method. New parameter updates are generated using the formula

$$\{\delta\}^{(i)} = \left( [J]^{(i)T} [J]^{(i)} + \lambda [I] \right)^{-1} [J]^{(i)T} (\{T\}^{\text{exp}} - \{T\}^{(j)}), \quad (1)$$

with which the updated permeability vector can be calculated using the formula:

$$\{\mathcal{K}\}^{(j+1)} = \{\mathcal{K}\}^{(j)} + \{\delta\}^{(j)} \quad (2)$$

where:

$\{\mathcal{K}\}$  is a column with the 3 elements of the symmetrical 2D permeability tensor;  $\{\mathcal{K}\} = \begin{bmatrix} K_{xx} \\ K_{xy} \\ K_{yy} \end{bmatrix}$ ,

$[I]$  is the identity matrix,

$\lambda$  is a damping parameter,

$\{T\}^{\text{exp}}$  is the column containing the flow front arrival times at the sensors of the PIERS set-up,

$\{T\}^j$  is the column with the computed flow front arrival times for the same points as  $\{T\}^{\text{exp}}$ ,

[J] is the Jacobian matrix;  $[J] = \begin{bmatrix} \frac{\partial T_1}{\partial K_{xx}} & \frac{\partial T_1}{\partial K_{xy}} & \frac{\partial T_1}{\partial K_{yy}} \\ \vdots & \vdots & \vdots \\ \frac{\partial T_n}{\partial K_{xx}} & \frac{\partial T_n}{\partial K_{xy}} & \frac{\partial T_n}{\partial K_{yy}} \end{bmatrix}$ ,

and n denotes the number of activated sensors and  $T_i$  the arrival time of the sensor i (with:  $i = 1, \dots, n$ ).

The evaluation of this Jacobian matrix, represents a delicate problem. The partial derivatives of the numerical model with respect to the permeabilities ( $\frac{\partial T}{\partial K_{xx}}, \frac{\partial T}{\partial K_{xy}}, \frac{\partial T}{\partial K_{yy}}$ ) can be approximated using backward, central or forward differences. The forward and backward finite difference approaches require less additional FE-analyses than the central differences approach because the response  $T_i(K)$  is anyhow known. Backward differences will be used in this procedure. So, the sensitivity coefficients, for a sensor i, are defined by:

$$\frac{\partial T_i(K)}{\partial K_j} \cong \frac{T_i(K) - T_i(K, K_j - \Delta K_j)}{\Delta K_j} \quad (3)$$

Note that the Jacobian matrix needs to be calculated in every iteration step of the MNET. These calculations are not prohibitively time consuming due to the speed of the numerical model and the limited number of parameters. Furthermore, it appeared to be advisable to systematically normalize the Jacobian matrix.

During the calculation of the solutions of equation (1), numerical problems can appear. These can be strongly reduced by using stable numerical algorithms. Among the many possible choices, here the singular value decomposition (SVD) concept is used.

### *The stop criterion*

The iteration loop of the optimization method is stopped at the moment that the stop criterion is met. There are a number of ways of choosing this criterion. The stop criterion programmed in the presented optimization algorithm is based on the evaluation of the cost function. More specifically, the iteration loop is stopped when there is no longer a significant decrease of the least-squares cost function,  $V$ , with further iteration.

## **A STEREOLITHOGRAPHY (SL) REFERENCE MATERIAL**

Permeability values often cause problems because there are no standardized methods for permeability measurements. Moreover, the measurements are very sensitive to various factors and large scatter in the identified permeability values is usually observed [7]. The latter is not surprising since the permeability is mainly dominated by the geometry of the porous medium. Possible sources of scatter on obtained permeability values are:

- uncontrolled nesting of layers during stacking and compression of multi-layered samples,
- deformation of the fiber texture during sample preparation (shearing),
- existence of micro-flows, also called intra-tow flows, within yarns,

- material heterogeneity due to manufacturing,
- random experimental errors (pressure, temperature and time measurements).

In practice these sources of scatter will act simultaneously, rendering clear distinction between the possible contributions difficult. A major problem in comparing different permeability identification methods has always been the fact that there exists no reliable reference material on which a comparative permeability measurement can be performed. Furthermore, the scatter present in the measurement results makes it difficult to experimentally validate numerical predictions.

Therefore, a solid test specimen that can be used as a reference sample (Fig. 5) has recently been developed by Morren et al. [15] and was produced with a stereolithography (SL) production technique. The additive nature of the stereolithography process allows the production of a structure with specific and complex internal features so that a kind of “artificial” reinforcement can be created in which the propagating fluid is persistently obligated to curve. Consequently, the flow in conventional textiles can be reproduced. Moreover, this concept counters the above mentioned scatter sources so that fixed permeability values are created. Since the permeability properties of the SL specimen do not vary from test to test, an excellent repeatability of the experiments is obtained and any measured difference must be attributed to the set-up and data processing. Consequently, this SL specimen can be used as a reference sample for calibration of test rigs and for comparison of results from different permeability identification methods.

Additionally, the SL sample has a simple and geometrically correctly known unit cell (Fig. 6 & 7). This allows a correct import of the geometry into numerical flow simulation software (such as FlowTex [16, 17]) for the numerical prediction of the permeability. Hence, the SL specimen allows experimental validation of the flow simulation software. A SL specimen was produced for the 2D central injection rig, called “PIERS set-up”. This specimen could be used for 1D experiments as well and the structure can also be easily adapted for through the thickness experiments and possible extra requirements of the used test rig.

Generally, one may conclude that the dimensions are sufficiently accurate and the deviation on the results is acceptably small. A tolerance of 0.1 mm can be easily obtained on all the dimensions. Furthermore, this accuracy was obtained on subsequent produced specimens from which can be concluded that the stereolithography manufacturing process is sufficiently repeatable and allows a very controllable and precise geometry. The newly designed resin additionally allows a rigid structure, with a very good surface quality and a wear resistance, which can be cleaned with water.

Finally, a project was initiated to perform a round-robin study between different research institutes. SL specimens will be sent to the participating partners such that results of permeability measurements can be compared on an objective basis.

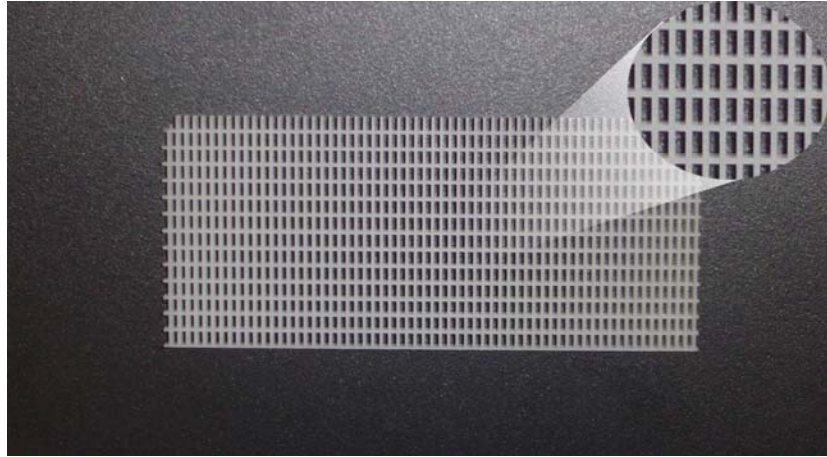


Fig. 5 Stereo-lithographic test specimen.

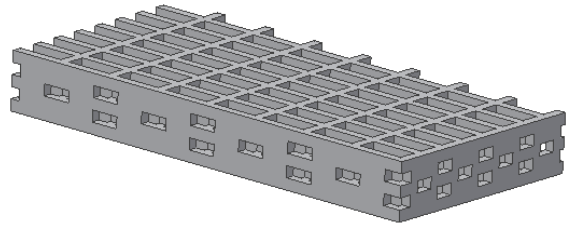
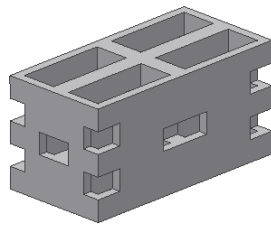


Fig. 6 Unit cell with outer dimensions 6 x 3 x 3 mm. Fig. 7 CAD drawing of 16 joined unit cells.

## CONCLUSION

A mixed numerical/experimental technique for the identification of the in-plane permeability tensor is proposed. The technique is based on an existing central injection rig and an inverse method, adjusting the parameters in a model of the experiment to optimize the agreement between measured and calculated flow front arrival times. A least-squares formulation of the difference between the experimental and the numerical flow front arrival times is used, along with a Levenberg-Marquardt optimization algorithm. The calculations are not prohibitively time consuming and the PIERS set-up can perform the measurement of the flow front arrival times ten times faster than a traditional procedure using a mold with transparent upper half. Moreover, a solid SL specimen has been presented that can be used as a reference sample for calibration of test rigs and for comparison of results from different test rigs. Since its structure consists of unit cells, the permeability values can also be easily estimated by means of numerical flow simulation software. Hence, this simulation software can be experimentally validated.



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