New Set-Up for Permeability Measurement

Qiang Liu and Richard Parnas

Institute of Materials Science, Univ. of Connecticut, Storrs, CT, 06269 USA:
rparnas@ims.uconn.edu

SUMMARY: The determination of accurate permeability values is critical to process simulations for liquid composite molding. And due to the statistical nature of the permeability[1], set-ups are needed to measure them accurately and quickly. Based on the different available set-ups, especially the one developed by Hoes[1], two new developments will be presented in this paper, one for in-plane permeability and the other for through-thickness permeability measurement. Specifically, a new sensor design is developed for the in-plane set-up to permit the use of electrically conductive reinforcement (carbon fabric), and to check the correctness of the in-plane assumption; and a new design for a through-thickness set-up is offered to permit high-speed data acquisition and reduce the race-tracking error.

KEYWORDS: Permeability; Process simulation; Liquid Composite Molding (LCM); Data Acquisition; Labview; Race-tracking; etc…

I. INTRODUCTION TO HIGH-THROUGHPUT PERMEABILITY MEASUREMENT

Composite materials are used in many kinds of applications. Among composite processing techniques[1, 2], RTM (Resin transfer molding) is one that has recently gained rapid acceptance[3]. RTM allows the molding of large complex shaped composite parts with a good surface finish and little pollution. This process consists of filling a closed mold cavity with reinforcements and injecting a resin through one, or several points. A unique feature of the RTM processing technique is that liquid resin has to flow a long distance to impregnate the dry fibers. The measure for the ease of the resin flow in the fiber preform is the permeability of the preform. Accurate permeability values are extremely important for the resin flow simulation and mold design due to the often encountered problems of non-uniform impregnation, void and dry spot formation[4], lengthy impregnation cycles, etc… With known permeability one can compute flow behavior in large complex molds, the pressure distributions in the mold, the required clamping pressure to hold the mold closed, and the required strength of the mold to retain its shape during the molding operation[5].

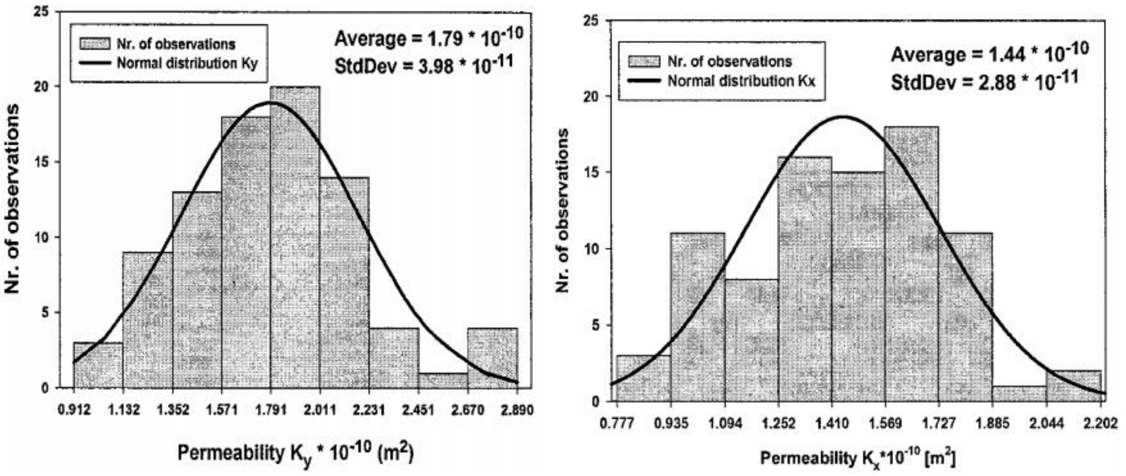
Permeability is a pore-structure parameter which depends only on the pore geometry of the porous media[6] but is complex and even changes during the preform manufacture. Rudd, et al. reviewed the deformation behavior of some mats and fabrics and made fiber architecture predictions, and then they applied models to estimate the in-plane permeabilities of those performs[7]. The usual way of getting permeability values is by experiments. Two kinds of experimental methods for permeability measurement are distinguished: unidirectional flow methods[8-14] and radial flow methods[8, 10, 11, 19-21]. In addition, unidirectional (1D) flow methods can also be distinguished by saturated and unsaturated flow methods.

In the saturated 1D method, experiments are conducted by forcing a test fluid through the whole mold in which the fabric is preplaced and compressed, and measuring the steady-state relationship between the flow and the pressure drop across the length of the mold[8]. Usually a linear relationship is obtained between the steady-state flows of a Newtonian fluid and its pressure drops. And in the unsaturated 1D test, the fluid flows through the dry fiber bed, replacing the air present in the material. Although the 1D test is the most straightforward set-up, there are a number of errors associated with it. The first is the "race tracking", or "edge effect" error, which means the preferential flow of the fluid along the mold walls when there is a small gap between the edges of the preform and the mold wall. According to Neale[15] and Parnas[16], the sensitivity of the 1D test to the edge effect is a function of the mold width. Many have made efforts to minimize the error due to the edge effect in their measurement. Diallo[14] et al. and Binetruy[17] et al. used silicone sealant; Parnas[18] et al. and Lekakou[19] et al. stuck a tape on the material edge. Another disadvantage of the 1D test is that at least three measurements have to be done to fully characterize the in-plane permeability tensor: one for the permeability values in two perpendicular axes and one to derive the angle with respect to the reference axis. In addition, mold deflection problems (especially associated with transparent plastic mold lids) and the problems due to incomplete saturation also exist[7].

The radial test (or 2D test) can only be used in unsaturated flow methods. The set-ups consist of a lower metallic mold-half with an injection hole in the middle, and a transparent top. Inside the mold, the fluid flows through the fabric from the central injection port. One uses constant injection flow rate or injection pressure. The transparent top-half allows one to record the flow front progression by means of a video camera for later derivation of fluid superficial velocity. The main advantage of the 2D test is that it allows the determination of both in-plane permeability components and the angle of them with respect to the reference axes all in one single experiment[1]. So it is much less time-consuming than 1D test. However, the data reduction procedure which converts the pictures of flow front positions to usable digital values is still time-consuming. In addition, the material and the flow front have to be visible throughout the experiment for people to use the camera. But there is always mold deflection problems[11] associated with the transparent plastic top of the mold. To counter the problem the transparent top plate is often covered with a steel frame to enhance bending stiffness[11], while this method reduces the visibility.

To counter the problems associated with the 2D tests, Hoes[22] et al built a new set-up, in which both the top-half and the bottom-half were made of stiff metals which solved the mold deflection problem quite well (see figure 1 below). The bottom-half includes 43 sensors located on straight lines at 0, 22.5, 45, 67.5, 90, 180, 270^0, and the injection hole can be seen in the middle. An epoxy seal provides electrical insulation between the sensor wire and the metal mold plate as well as preventing fluid leakage. This sensor design is limited to electrically nonconductive fabric and electrically conductive fluid. When fluid flows over the sensor, the circuit is closed, providing a signal to the computer indicating the time of arrival of fluid at each sensor location. The whole set-up sits in a Carver Press. The data acquisition and analysis are automated so the permeability is known within a few seconds after the experiment. The connection of the instruments with the computer and the use of the software in this set-up greatly saved time compared to the usual 2D tests.

Example permeability values for Syncoglass R420 woven fabric are shown in figure 1[1] below. From this figure, one can see that permeability is a statistically distributed parameter and can not be characterized by only a few experiments[1].



(a) In the warp[4] direction (b) In the weft[5] direction

Figure 1. Syncoglass R420 permeability distribution at 41.7% fiber volume fraction

2. NEW SENSOR DESIGN FOR IN-PLANE PERMEABILITY MEASUREMENT FOR CARBON FABRIC

The sensor plate used earlier contained 43 sensors in one quadrant. While good enough to illustrate the statistical distribution of permeability data, a sensor plate with sensors in all four quadrants around the injection hole will provide several advantages. Figure 2 shows a new sensor plate with 105 sensors.

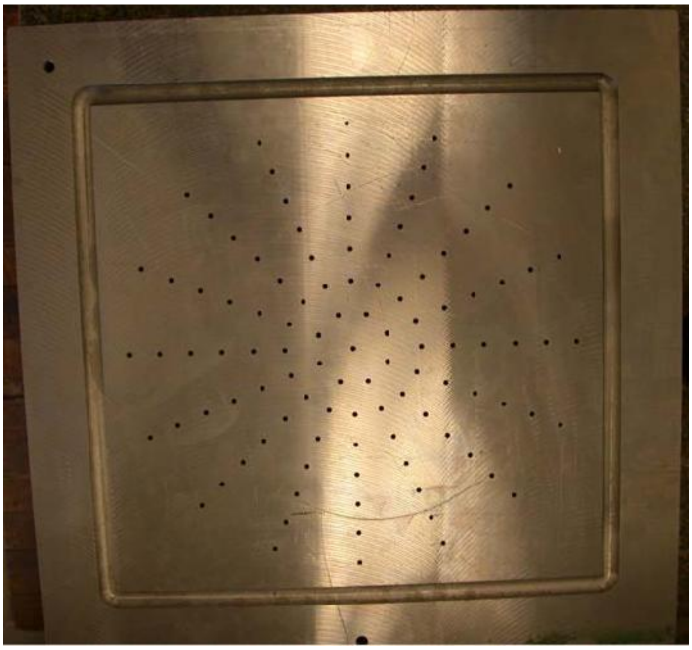


Fig 2. Top Sensor Plate

The new sensor plate also uses a modified sensor, in which the sensor is slightly recessed below the plate level, to prevent compressed electrically conductive fabric from touching and shorting out the sensors (See figure 3 below), while still permitting the injected conductive fluid to produce a response in the computer. In this way one can also measure the in-plane permeability values for electrically conductive fabric such as carbon.

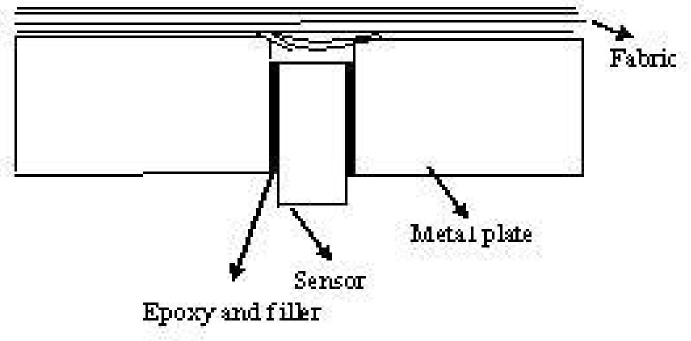
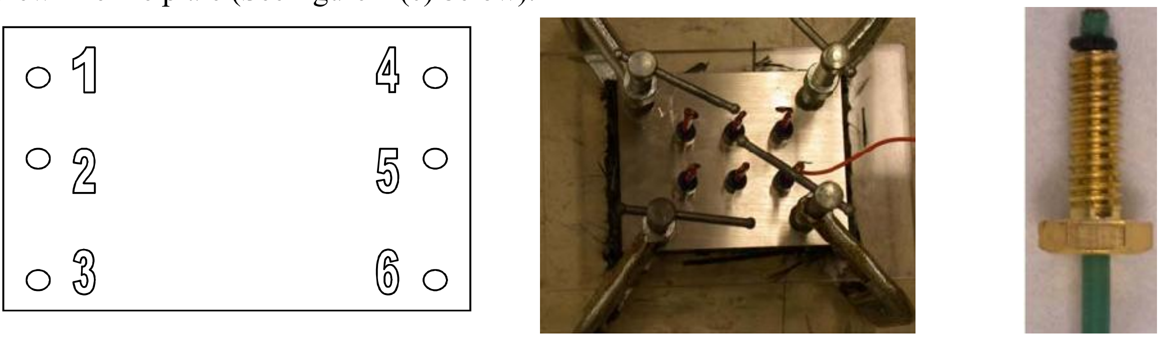


Figure 3. Recessed sensor.

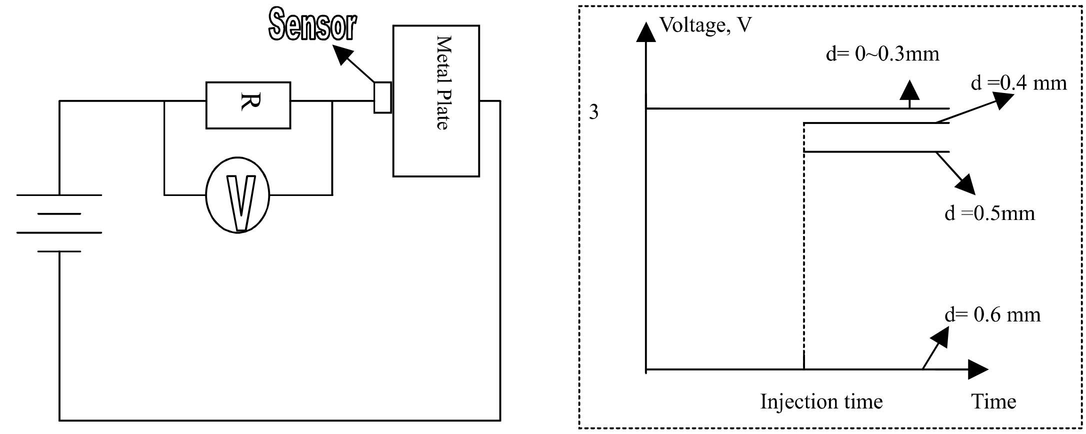
Experiments prove the applicability of this idea. A small rectangular metal plate is used with six sensors inserted in and distributed evenly (See figure 4 below). The sensor is an electrically conductive copper wire with PVC insulation coating. The sensor is inserted through a hollow copper screw. An O-ring seals and fixes the sensor position when compressed by tightening the screw into the plate (See figure 4 (c) below).



(a) Front view (b) Back view (c) Sensor Design

Figure 4. The sensor plate used in our experiment.

Each sensor is recessed a small distance d, which can be quantitatively measured by a Digimatic Depth Gauge (Mitutoyo 700-105). Carbon fabric is placed on the top of the sensor plate, covered with a transparent top plate, and everything is compressed. Next, the voltage across an electrical resistance (in such a circuit as shown in figure 5(a) below) is measured, before and after fluid injection through a central hole drilled through the transparent top. Here the results are shown for one sensor in Figure 5(b) in which the voltage values are shown with the corresponding recess distances d. In figure 5, one can see that d=0.4~0.5 mm is the distance range for good sensor performance. This circuit design also permits sensor I/O through the digital channel on the interface card for high efficiency.

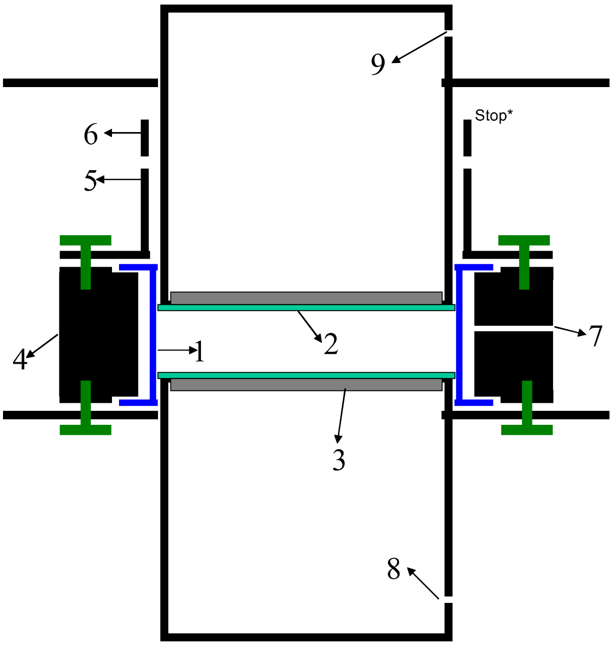


(a) Electrical circuit used in the experiment. (b). Experimental results of one sensor.

Figure 5

3. HIGH SPEED DATA COLLECTION

With this new set-up, the authors plan to measure the statistical distribution of in-plane permeability values for electrically conductive fabric. A number of preforms will be used to collect permeability data at one fiber volume fraction to generate a distribution, and then another set of data can be collected at another fiber volume fraction. A through-the thickness permeability rig is also being developed to rapidly collect necessary to characterize 3-dimensional flow behavior (see figure 6 below).



1. Elastic bladder
2. Filter (Sample filled between the two filters)
3. Metal support
4. Metal frame
5. Guide
6. Stop
7. Air hose
8. Fluid entrance
9. Fluid exit

Figure 6. The set-up being built for through-thickness permeability measurement

An inflatable elastic bladder will seal the edges of the fabric so as to reduce race-tracking errors. Fluid is pumped from bottom to top by a constant flow rate pump and pressure differences are measured by a differential pressure transducer (Dwyer 645-15). Stops of calibrated thickness are used to control the thickness and so the fiber volume fraction of the sample. Then, one can obtain through-the-thickness permeability values at several fiber volume fractions using one sample by replacing the stops.

4. ACKNOWLEDGEMENT

The authors would like to recognize the financial support (grant no. 4000020035) and the discussions with Raymond Boeman and Richard Battiste of Oak Ridge National Laboratory, which allowed us to perform this work.

5. REFERENCES

[1]. Kris Hoes, PhD Thesis, University of Brussels, Brussels, 2003
[2]. Vetrotex – Saint Gobain, Brochure: Memento 2001
[3]. Jingyi Xu and Yulu Ma, The Technology and Applications of Resin Transfer Molding (RTM), China Plastics, 6(1), 9-16 (1992).
[4]. L. Baichen, S. Bickerton and S. G. Advani, Modeling and simulation of resin transfer molding (RTM) – gate control, venting and dry spot prediction, Composites, 27A, (2), 135-141 (1996).
[5] Richard. S. Parnas, Liquid Composite Molding, Carl Hanser Verlag, Munchen, 2000.
[6]. F. A. L. Dullien, Porous Media - Fluid Transport and Pore Structure, Academic Press, INC. 1992
[7]. Kruckenberg, T. and Paton, R., Resin transfer molding for Aerospace Structures, Kluwer Academic Publishers, Dordrecht, The Netherlands, 1998.
[8]. R. S. Parnas and A. J. Salem, A comparison of the unidirectional and radial in-plane flow of fluids through woven composite reinforcements, Polymer Composites, 14(5), 383-394 (1993)
[9]. P. Ferland, D. Guittard and F. Trochu, Concurrent Methods for permeability measurement in resin transfer molding, Polymer Composites, 17(1), 149-158, (1996).
[10]. R. Gauvin, et. al., Permeability measurement and flow simulation through fiber reinforcement, Polymer Composites, 17(1) 34-42, (1996).
[11]. R. B. Gebart and P. LinstrÖm, Measurement of In-plane permeability of fiber reinforcements, Polymer Composites, 17(1) 43-51, (1996).
[12]. T. S. LundstrÖm, et. al., In-plane permeability measurements: a Nordic round-robin study, Composites, 31A(1) 29-43, (2000).
[13]. S. Amico and C. Lekakou, An experimental study of the permeability and capillary pressure in resin-transfer molding, Composites science and Technology, 61, 1945-1959, (2001).
[14]. M. L. Diallo, R. Gauvin and F. Trochu, Key factors affecting the permeability measurement in continuous fiber reinforcements, Proceedings of ICCM, 11, 441-451, (1997).
[15]. G. Neale and W. Nader, Practical significance of Brinkman's extension of Darcy's law: coupled parallel flows within a channel and a bounding porous medium, Canadian Journal of Chemical Engineering, 52, 475-478, (1974)

[16]. R. S. Parnas and Y. Cohen, Coupled parallel flows of power-law fluids in a channel and a bounding porous media, Chemical engineering communications, 53, 3-22, (1987)

[17]. C. Binetruy, B. Hilaire and J. Pabiot, The interaction between flows occurring inside and outside fabric tows during RTM, Composites Science and Technology, 57, 587-596, (1997).

[18]. Parnas, R. S. et al., Permeability Characterization. Part 1: A Proposed Standard Reference Fabric for Permeability, Polymer Composites, 16, (6), 429-445, (1995).

[19]. C. Lekakou, et. al., Measurement Techniques and effects on in-plane permeability of woven cloths in resin transfer molding, Composites, 27A, 401-408, (1996)

[20]. K. L. Adams and L. Rebenfeld, In-plane Flow of Fluids in Fabrics: Structure/Flow Characterization, Textile Research Journal, 57, 647-654, (1987).

[21]. B. N. Greve and S. K. Soh, Directional Permeability Measurement of Fiberglass Reinforcements, SAE Transactions, 99, 331-343, (1990).

[22]. Kris Hoes, et. al., New set-up for measurement of permeability properties of fibrous reinforcements for RTM, Composites, 33A, 959-969, (2002).