A PERMEABILITY MEASUREMENT METHOD DEDICATED TO A COMPOSITE PROCESS FAMILY

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SUMMARY: During the last 30 years, Liquid Composite Molding (LCM) technologies have been intensively used to manufacture structural composite parts in a wide range of aeronautical applications. The development and optimization of these technologies as well as the associated numerical simulation require knowledge of physical properties of the fibrous material, called preform. The resistance to the resin flow is defined by the permeability tensor of the preform in the model of Darcy's law. Considering the different industrial LCM processes, a complete identification of the permeability tensor is necessary. Material characterization for industrial applications requires using reliable, reproducible and realistic procedures. With this goal in view, different protocols of permeability measurements have been set up for several years by EADS Innovation works in collaboration with academic partners. A method of permeability measurement was developed in collaboration with École Polytechnique of Montreal in order to identify the unsaturated in-plane permeability. This approach includes a built-in correlation with Darcy's law, allows an estimation of both experimental and numerical errors and has been standardized within EADS with specific automation. In the same context, a through-thickness permeability measurement has been developed in collaboration with University of Le Havre. This system identifies saturated through-thickness permeability for a large range of fiber content. Finally, a specific protocol dedicated to the identification of a various and complete set of permeability values for LCM processes including a double porous medium was developed in collaboration with École Polytechnique of Montreal. This approach consists of characterizing the in-plane and through-thickness unsaturated permeability tensor necessary for this family of processes. Numerical solutions were developed and applied to industrial parts. This clearly demonstrated how these different tasks highly contribute to the integration of composite process simulation in the development of aeronautical structural composites.

KEYWORDS: permeability measurement, double scale porous medium, identification

INTRODUCTION

For 30 years EADS has developed composite structures using mainly conventional prepreg technologies. Although the cost of composite structures has been dramatically reduced it remains a little bit more expensive than metallic structures. Manufacturers are looking for cheap way of manufacturing composite structures. Liquid Composite Molding (LCM) technologies seem promising for cost reduction of aircrafts structures. LCM technologies have not to be considered as an alternative to prepreg technologies, but as a way produce cost efficient composite structures and to increase the potential of applications. Theses technologies provide the opportunity of "low cost" composite parts: low material costs and reduced manufacturing effort compared to other technologies (reduced lay up effort, out of autoclave curing). LCM technologies allow an integral design for composite structures (preform technology) with out-of plane loading capabilities and a suitable for complex parts (e.g. high draping properties of dry textiles). The development and optimization of these technologies as well as the associated numerical simulation require knowledge of physical properties of the fibrous material, called preform. The resistance to the resin flow is defined by the permeability tensor of the preform in the model of Darcy's law. Considering the industrial different LCM processes and the targeted fiber content of aeronautical composite parts, a complete identification of the permeability tensor is necessary.

Characterization of permeability is still an open debate: saturated or unsaturated method, accuracy of identification procedure, dual scale flow issues, etc. Furthermore, the industrial context requires the characterizations procedures to be reliable, reproducible and realistic. Moreover, the LCM technologies used by EADS Business Units change depending on the part targeted (geometry, thickness, fiber content, etc.). Therefore different types of resin flows take place during the injection stage. With this goal in view, different protocols of permeability measurements have been set up for several years by EADS Innovation works in collaboration with several academic partners, in order to be meet our requirements. This paper describes the various methods selected and discusses the reasons of these choices. Finally, examples of application are presented for industrial composite structures.

IN-PLANE PERMEABILITY

A reference method of in-plane permeability measurement has been developed in collaboration with École Polytechnique of Montréal. The main advantage consists in a standard measurement procedure of in-plane unidirectional permeability with fully automated instrumentation (see Fig. 1). This concurrent method includes a built-in correlation with Darcy's law and allows an estimation of experimental and numerical errors.

Fig. 1 View of global measurement system of the in-plane permeability bench.

The displacement of a Newtonian fluid through a porous medium can be predicted by the Darcy's law [1]. This law enables estimating the average superficial fluid velocity $\mathbf{v}$ from the pressure gradient ∇P via the following relationship:

$$\mathbf{v} = -\frac{[\mathbf{K}]}{\phi \, \mu} \nabla P \tag{1}$$

where $[\mathbf{K}]$ is the permeability tensor of the porous medium, μ the dynamic viscosity of the resin and ϕ denotes the porosity of the porous medium. In the unidirectional case, we can write:

$$K = V_i . \mu . \frac{\Delta L}{\Delta P} \tag{2}$$

with V_i, instantaneous velocity of flow, and $\dfrac{\Delta L}{\Delta P}$ is the linear pressure drop.

The principle of the measurement consists in using Darcy's law in order to calculate the permeability value. Also, we must:
- follow during the infiltration, the flow front through the fibrous medium,
- measure the pressure gradient present in the reinforcements.

The developed protocol takes into account these principles by integration of:
- optical fibers in order to detect the resin position,
- pressure sensors in order to measure the pressure gradient,
- temperature sensors inside the mold cavity in order to calculate the fluid viscosity,
- automatic treatment of measured data in order to estimate the permeability value
- GUI (see Fig. 2) in order to follow in real time on a screen the flow front evolution and parameters process (pressure injection as a function of time, for example),
- report obtained by computer including results sheet (permeability value, uncertainty) and other recorded information during tests.
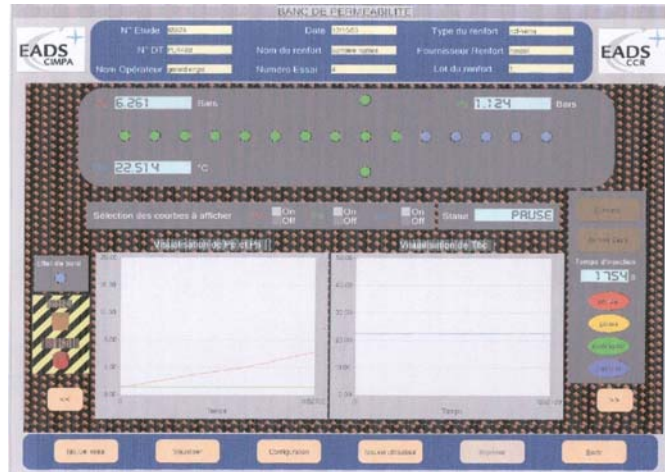
Fig. 2 Screenshot of GUI.

Three different methods have been developed in order to process data of tests and give three different estimations of permeability value [2]. The unidirectional method in opposition of the radial flows method has been selected. The last approach is attractive for non isotropic permeability materials (fabrics for example) because the two in-plane principal permeabilities could possibly be evaluated in a single experiment. However, some difficulties have been shown [3] concerning the complex data acquisition of a central injection (record of an elliptic flow front with sensors, fit the experimental front position to a perfect ellipse) and the influence to the radius of the injection port during the identification of the permeability value (difficulties of comparison of the experimentally observed wetted areas with an analytical solution of Darcy equation in cylindrical coordinates).

The selected measurement consists in a transitory injection at ambient temperature of a preform identical to the one used for the industrial part in term of preparation. This protocol allows possibilities following the flow front through the instrumented transparent mold. Finally, the different choices are lead to be close to the real injection of the structural part in term of injection conditions, preform characteristics.

The processes targeted by this permeability measurement protocol are the Resin Transfer Molding (RTM) process including rigid mold and process inducing constant fiber content during the injection. This family of LCM technologies represents the mayor part of the injected aeronautic composite parts manufactured for EADS products, in order to obtain data about the injectability of the preform. A dedicated numerical approach has been developed in parallel of this protocol and allows having some information on the filling stage of the part [4].

THROUGH-THICKNESS PERMEABILITY

From a few years, some through-thickness permeability measurements have been developed [5-6]. EADS France IW proposes in collaboration with the University of Le Havre a new continuous saturated through-thickness permeability characterization technique (see Fig 3).
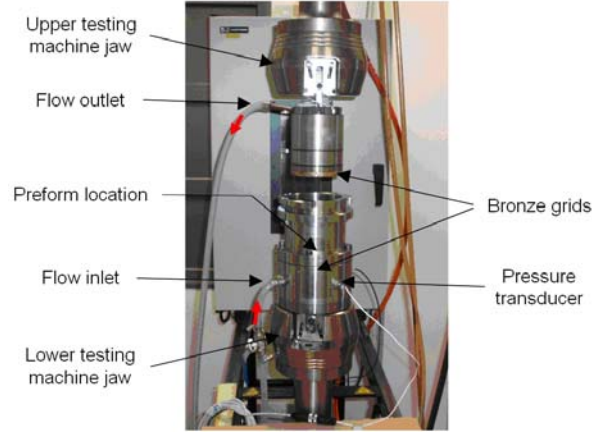
Fig. 3 View of global measurement system of through-thickness permeability.

This apparatus consists in a cylindrical chamber within a guided piston induces the exact fiber compaction in the transverse direction. The device is introduced into a universal testing machine to control the displacement of the piston and the force applied to the fibrous medium. The fluid is directed to the transverse direction by perforated bronze grids. A pressure transducer is placed below the lower grid. Test samples of fibrous preforms are placed between the two grids. An other device is placed on an other universal testing machine in order to apply a controlled flow rate of fluid on the fibrous reinforcement. When a Newtonian test fluid is injected at a constant flow rate through the fibrous reinforcement, an evolution of pressure is measured by the pressure sensor. The transverse permeability Kz is calculated using Darcy's law:

$$K_Z = \frac{\mu\,h}{A\,\Delta P}Q \qquad (3)$$

where μ is the dynamic viscosity of the test fluid, h and A are respectively the thickness and the area of the preform, Q is the imposed flow rate and ΔP is the difference of pressure induced by the constant flow rate of test fluid through the reinforcement. Finally, the through-thickness permeability is evaluated in a continuous way as a function of fiber content through the controlled compression of the porous medium (see Fig 4).
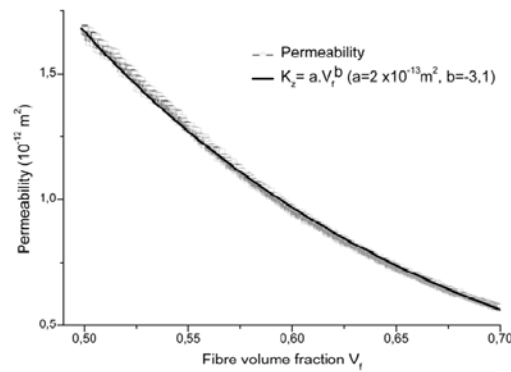

Fig. 4 Example of results of through-thickness permeability method.

The saturated permeability obtained gives important information on the through-thickness injectability of the fibrous preform. The processes targeted by this permeability measurement

protocol are the ones that create a non constant fiber content during the infusion. This family of LCM technologies represents a new part of the injected aeronautic composite parts manufactured for EADS products (large composite parts with high fiber content). A dedicated numerical approach has been developed in parallel of this protocol and allows having some information on the filling stage of the part [7].

DOUBLE POROUS MEDIUM PERMEABILITY

A permeability measurement specifically dedicated to process including double porous medium has been developed in relation with École Polytechnique of Montréal. The infiltration of the fluid through different porous medium can be represented by different flows (see Fig 5).
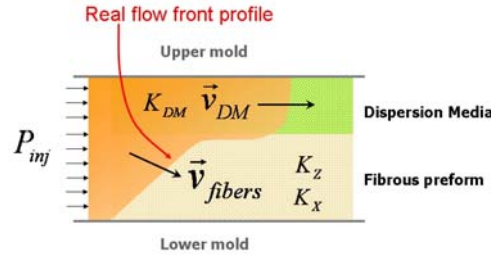

Fig. 5 Definition of flows in a fibrous reinforcement with distribution medium (DM).

For example in the dispersion medium RTM (DM-RTM) process, a highly permeable dispersion medium is added above the fibrous preform to facilitate resin impregnation of the laminate. Important differences between the permeability of the dispersion media and the laminate (more than 1,000 times) induce a complex three-dimensional resin flow. Due to the higher permeability of the dispersion medium, resin initially flows through it forcing a through-thickness impregnation of the low permeability laminate [8]. These resin flows can be estimated by applying Darcy's law with a pressure gradient ∇P_{DM} in the DM, ∇P_{fibers} through the laminate:

$$Q^{DM} = -\frac{[\mathbf{K}_{DM}]}{\phi_{DM}\ \mu\ A}\nabla P_{DM} - Q_t \quad \text{and} \quad Q^{fibers} = -\frac{[\mathbf{K}_{fibers}]}{\phi_{fibers}\ \mu\ A}\nabla P_{fibers} + Q_t \tag{4}$$

where Q^{DM} is the flow rate through the dispersion medium, Q^{fibers} the flow rate in the laminate, Q_t the flow rate between the dispersion medium and the laminate, A the in-plane area of the unit cell, μ is the dynamic viscosity of the test fluid, ϕ_{fibers}, ϕ_{Dm} and $[K_{fibers}]$, $[K_{DM}]$, respectively the fiber content and the permeability tensor of the laminate and the dispersion medium. For thin laminates, the through-thickness flow Q_t can be estimated by applying Darcy's law between the DM and the laminate:

$$Q_t = \frac{\mathbf{K}_{fibers}^T}{\phi_{fibers}\ \mu\ A}(P^{DM} - P^{fibers}) \tag{5}$$

where $\mathbf{K}_{fibers}^T$ is the transverse through-thickness permeability of the laminate, P^{DM} the pressure in the dispersion medium, and P^{fibers} the pressure in the laminate. One conceivable way to

identify experimentally these properties consists of characterizing each one separately: one measurement for DM (in-plane permeability) and two other for fibrous perform (in-plane and through-thickness permeability). Nevertheless, to obtain accurate though-thickness permeability of a high fiber content preform and in-plane permeability of DM is still on debate. Secondly, separated measurements for both porous media does not take into account the coupling between the in-plane permeability of the DM and the fiber content of the preform. The new method developed maintains the in-plane unidirectional permeability measurement method previously presented, but takes into account the intimate link between the two layers of porous media. As illustrated in Fig 6, the new approach proposed allows a coupled characterization of the permeability of a fibrous reinforcement in presence of a distribution medium (DM).
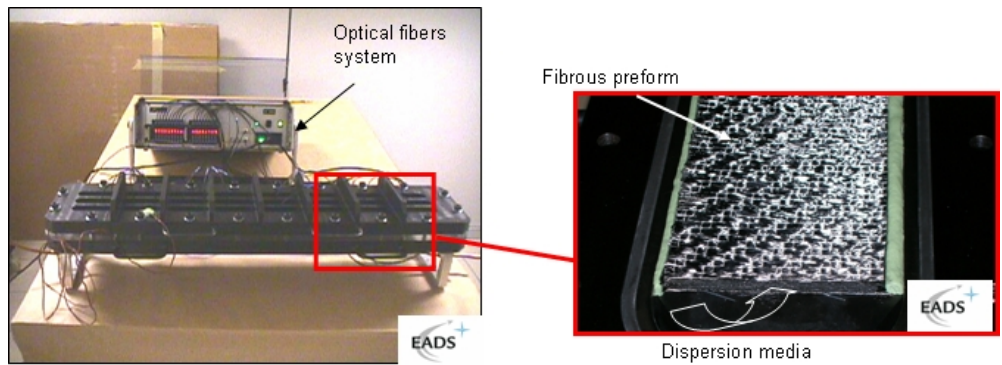

Fig. 6 Views of the experimental test bench and carbon fiber sample.

In the end, we obtain directly the necessary permeability values through only one flow filling of the double porous medium. Specific software has been developed in order to identify the permeability from experimental data (see Fig 7).
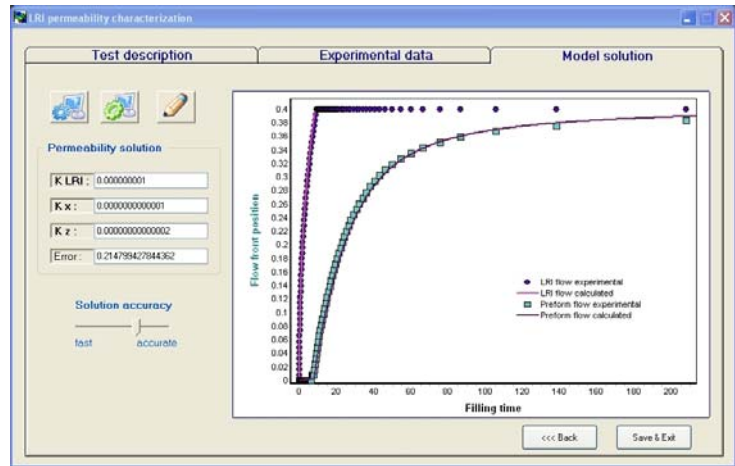

Fig. 7 Data processing window: identification of DM and preform permeability.

The targeted processes by this permeability measurement protocol are processes inducing infiltration of a double porous medium at constant fiber content like Liquid Resin Infusion process. Two finite elements approaches are proposed in parallel of this experimental method to iteratively evaluate the complex resin flow in the process and allow having some information on the filling stage of the part [9].

CONCLUSIONS

In this paper, three different methodology of permeability measurement are proposed. Each developed method has been set up with identified measurement protocol and associated numerical solutions. These different tasks highly contribute to the integration of composite process simulation in the development of aeronautical structural composite parts. The choice of an adequate protocol of permeability measurement depends on one hand of the targeted process and associated structural part, and an other hand of difficulties to set up experimentally a reliable, reproducible and realistic measurement protocol. We hope this integrated approach could contribute to the definition of permeability measurement normalization.

REFERENCES

1.   H. Darcy, "Les fontaines publiques de la ville de Dijon", *Dalmont,* Paris, 1856.

2.   P. Ferland, D. Guittard, F. Trochu, "Concurrent Method for Permeability Measurement in Resin Transfer Molding", *Polymer Composites,* Feb 1996, vol 17, pp 149-158.

3.   R. Gauvin, F. Trochu, Y. Lemenn, M.L Diallo, "Permeability Measurement and Flow Simulation through Fiber Reinforcement", Polymer Composites, 17(1), p. 34-42.

4.   F. Trochu, R. Gauvin, D-M. Gao, "Numerical Analysis of the Resin Transfer Molding Process by the Finite Element Method", *Advances in Polymer Technology,* Vol. 12, no. 4, 1993, pp. 329.

5.   S. Comas-cardona, C. Binetry, P. Krawczak, "Unidirectional Compression of Fiber Reinforcements, Part 2: A continuous Permeability Tensor Measurement", *Composites Science and Technology,* Vol. 67, March 2007, pp 638-645.

6.   S. Drapier, J. Monatte, O. Elbouazzaoui, P. Henrat, "Characterization of Transient Through-Thickness Permeability of Non Crimp New Concept (Multiaxial Fabrics)", *Composites. Part A, Applied Science and Manufacturing ,* 2005, Vol. 36, no. 7, pp. 877-892.

7.   T. Ouahbi, A. Saouab, J. Bréard, P. Ouagne, S. Chatel, "Modelling of Hydro-Mechanical Coupling in Infusion Processes", *Composite Part A, Applied Science and Manufacturing,* Vol. 38, July 2007, pp 1646-1654.

8.   V.M.A. Calado, S.G. Advani, "Effective Average Permeability of Multi-Layer Preforms in Resin Transfer Molding", *Composites Science and Technology,* Vol. 56, no. 5, 1996, pp. 519.

9.   E. Ruiz, S. Chatel, F. Trochu, "A Simplified Numerical Approach to Simulate Resin Transfer Molding with a Dispersion Medium", *8th International Conference on Flow Processes in Composite Materials,* page 331-340, July 2006.