Recent Developments and Open Problems in Composites Materials Manufacturing

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Introduction

Resin injection molding is the most widely used technique to produce composites. It consists in injecting a polymeric melt in a porous material, usually called solid preform. The solid preform is placed in a mould and the liquid constituent is injected through it. As far as the liquid front advances and impregnates the preform, it displaces the air that outflows from the mould through suitably located air vents. When the liquid constituent has solidified or is completely polymerized, the mould is opened and the composite materials is available for subsequent finishing operations. The structure of the preform can strongly differ from case to case: it can exhibit a sponge–like structures, or a knitted one, it can be made of fibers, bundles, or mats, and so on. In all these cases the infiltration process can be schematized as the flow of a liquid through a deformable porous material.

The role played by the modeling in such an industrial context is becoming more and more strategic in order to produce high–standard and competitive materials. In fact, by means of computer simulations based on reliable mathematical models of the process, it is hoped to design in advance the best procedures to be pursued, thus reducing expensive experimental testing programs.

The aims of this paper is to present some recent results in the mathematical modelling of processes like resin injection molding and to bring some open problems to the attention of the reader.

1 Mathematical Modelling

Modelling the behaviour of a porous solid and of the fluid (or fluids) that permeate it can be done using several methods that essentially differ for the spatial scale that we are interested in [18]. We look at the dynamics of the processes at the *macroscale*: we neglect the decription of the flowfield of the resin at the spatial size of the pores by assuming that at any point of the material liquid and solid phase are co-present. This assumption of co-occupancy leads to the concept of volume fraction: at each point of the material there is a fraction of volume ϕ_s occupied by the fluid and a fraction ϕ_ℓ occupied by the solid. Then we write down a system of balance equations, formally very similar to the microscopic ones, that are supposed to be able to reproduce the large scale dynamics. Although the behavior at the microscale is disregarded in detail, the characteristics of the components *in bulk* are necessary to provide suitable functions (permeability, porosity,...) that enable the link between micro and macro.

The equations describing the macroscopic field can be obtained by few different approaches. In particular, the ensemble average approach and mixture theory are used and directly compared in [10]. Having in mind resin injection molding processes, the following set of equations can be written

- Mass balance equations

$$\frac{\partial \phi_s}{\partial t} + \nabla \cdot (\phi_s \mathbf{v}_s) = 0\,, \tag{1}$$

$$\frac{\partial \phi_\ell}{\partial t} + \nabla \cdot (\phi_\ell \mathbf{v}_\ell) = 0\,, \tag{2}$$

  where ϕ_s and $\mathbf{v}_s$ (resp. ϕ_ℓ and $\mathbf{v}_\ell$) are the volume ratio and the velocity of the solid (resp. liquid) constituent. The *saturation assumption* implies the geometrical constraint

$$\phi_s + \phi_\ell = 1\,. \tag{3}$$

- Momentum balance equations

$$\rho_s \phi_s \left(\frac{\partial \mathbf{v}_s}{\partial t} + \mathbf{v}_s \cdot \nabla \mathbf{v}_s \right) = \nabla \cdot \mathbf{T}_s + \mathbf{m}_s \,, \tag{4}$$

$$\rho_\ell \phi_\ell \left(\frac{\partial \mathbf{v}_\ell}{\partial t} + \mathbf{v}_\ell \cdot \nabla \mathbf{v}_\ell \right) = \nabla \cdot \mathbf{T}_\ell + \mathbf{m}_\ell \,, \tag{5}$$

where ρ_s and $\mathbf{T}_s$ (resp. ρ_ℓ and $\mathbf{T}_\ell$) are the bulk density and the partial stress of the solid (resp. liquid) constituent. The interaction forces $\mathbf{m}_s$ and $\mathbf{m}_\ell$ correspond to momentum exchange between the constituents; they are not independent, but satisfy the action-reaction principle:

$$\mathbf{m}_s + \mathbf{m}_\ell = 0.$$

- Energy balance equations

$$\rho_s \phi_s \left(\frac{\partial \epsilon_s}{\partial t} + \mathbf{v}_s \cdot \nabla \epsilon_s \right) = \mathbf{T}_s : \nabla \mathbf{v}_s - \nabla \cdot \mathbf{j}_s + \rho_s \phi_s r_s + e_s \,, \tag{6}$$

$$\rho_\ell \phi_\ell \left(\frac{\partial \epsilon_\ell}{\partial t} + \mathbf{v}_\ell \cdot \nabla \epsilon_\ell \right) = \mathbf{T}_\ell : \nabla \mathbf{v}_\ell - \nabla \cdot \mathbf{j}_\ell + \rho_\ell \phi_\ell r_\ell + e_\ell \,, \tag{7}$$

where ϵ_s, $\mathbf{j}_s$ and r_s (resp. ϵ_ℓ, $\mathbf{j}_\ell$ and r_ℓ) are the internal energy, the heat flux and the heat supply for the solid (resp. liquid) constituent. The quantities e_s and e_ℓ, called *energy supply densities*, are related to the energy exchange between the constituents across the interface separating them and satisfy the following relation

$$e_s + e_\ell + \mathbf{v}_s \cdot \mathbf{m}_s + \mathbf{v}_\ell \cdot \mathbf{m}_\ell = 0 \,.$$

- Resin conversion equation

$$\frac{\partial \delta_c}{\partial t} + \mathbf{v}_\ell \cdot \nabla \delta_c = f_c \left(\delta_c, \theta \right) \,, \tag{8}$$

where $\delta_c = \mathcal{H}/\mathcal{H}_c \in [0,\, 1]$ is the *degree of cure* (or *resin conversion*) defined as the ratio of the amount of heat released by the curing exothermic reaction $\mathcal{H}(\mathbf{x}, t)$ over the total heat of reaction $\mathcal{H}_c$. The chemical reaction is described by the experimentally measured function f_c.

The equations above apply to any kind of bicomponent mixture of incompressible constituents (with a polymerizing fluid). Taking into account that the resin-preform mixture is a deformable porous medium, the following assumptions can be made:

A1. Negligible inertia effects: the stress field and the internal forces nearly balance in the momentum equations. This assumptions actually does not apply when a pressure gap is abruptly applied to start up the infiltration process; in such a case, inertial effects can play a role for a short time. This issue is discussed in detail in [1] and [2].

A2. Negligible surface tension and capillary effects.

A3. Slow liquid flow in the porous medium.

A4. The saturation relation (3) holds. This calls for a Liu-Lagrange multiplier in the constitutive equation that accomodates in order to satisfy the saturation constraint. It can be shown by thermodynamics arguments that this multiplier P contributes to the partial stress fields proportionally to the volume fraction. Then one can write

$$\begin{aligned}\mathbf{T}_\ell &= -\phi_\ell P\mathbf{I} + \mathbf{T}'_\ell\,, &(9)\\ \mathbf{T}_s &= -\phi_s P\mathbf{I} + \mathbf{T}'_s\,, &(10)\\ \mathbf{m}_\ell &= P\nabla\phi_\ell + \mathbf{m}'_\ell\,, &(11)\\ \mathbf{m}_s &= P\nabla\phi_s + \mathbf{m}'_s\,, &(12)\end{aligned}$$

where $\mathbf{T}'_\ell, \mathbf{T}'_s$ are called excess stress and P is the pressure of the mixture. Furthermore, from (3),

$$\nabla\cdot(\phi_s\mathbf{v}_s + \phi_\ell\mathbf{v}_\ell) = 0\,, \tag{13}$$

A5. The excess stress of the liquid phase is negligibible, i.e. the internal forces account for all the viscous interactions at the macroscale.

A6. The excess interaction force between the solid and the liquid is proportional to the velocity difference, i.e.

$$\mathbf{m}'_\ell = -\mathbf{M}\,(\mathbf{v}_\ell - \mathbf{v}_s)\,. \tag{14}$$

where $\mathbf{M}$ is an invertible tensor depending on the physical characteristics of the porous solid.

A7. All the constituents have locally the same temperature. This assumption is definetely correct for those manufacturing processes of composite materials under consideration in this paper. In fact, in such processes the fibers of the solid preform are so tiny ($< \; 10^{-4}\, mm$) that they quickly adjust their temperature to the one of the infiltrating liquid, so that one can assume that locally the two constituents have the same temperature θ.

A8. The thermal expansion of the solid and liquid can be neglected. However, it is to be mentioned that in some cases thermal expansion can be important. For instance, when the metal melt, infiltrating a zirconium foam, solidifies it shrinks considerably and can break some bridges of the preform which is quite fragile.

Assumptions A1–A6 simplify substantially the momentum balance equations since they allow to write

$$\mathbf{v}_\ell - \mathbf{v}_s = -\frac{\mathbf{K}}{\mu\,\phi_\ell}\nabla P\,, \tag{15}$$

$$\nabla P - \nabla\cdot\mathbf{T}'_m = 0\,, \tag{16}$$

where $\mathbf{T}'_m$ is the mixture excess stress, μ is the dynamic viscosity of the liquid which depends, in general, on the degree of cure δ_c and on the temperature θ, $\mathbf{K}$ is the so–called *permeability tensor* defined as $\mathbf{K} =: \mu\,\phi_\ell^2\,\mathbf{M}^{-1}$, which, for saturated deformable porous media depends on the deformation gradient of the solid constituent, i.e. $\mathbf{K} = \mathbf{K}\left(\mathbf{F}_s\right)$. Equation (15) is known as *Darcy's law* for a deformable porous medium.

2 Some analytical and numerical results

The equations above have been used in a studied in a number of papers, from both an analitical and a numerical point of view.

The equations driving one–dimensional isothermal infiltration are simple enough that few exact results can be obtained. Generally speaking, the infiltration process can be driven by a pressure gap applied at the boundaries of the solid preform or by assuming that the fluid enters the matrix at a known velocity. When considering the pressure–driven flow, the void ratio in the dry region instantaneously reaches the value necessary to balance the applied pressure jump. Billi and Farina [6] have shown that equations read as a a Stefan–like problem, which can be shown to have a unique self–similar solution, with interface position growing in time as $t^{1/2}$. For velocity–driven flow, the equations can be partially integrated, and the final problem reads as a nonlinear parabolic equation with non-standard boundary conditions [3].

Some more care is needed in handling the energy equation. However, applying assumptions A7-A8 the problem can be greatly simplified. Referring to [11], one can write the following heat equation for the mixture

$$\rho_m C_m \frac{\partial \theta}{\partial t} + \left(\rho_s \phi_s \widetilde{C}_s \mathbf{v}_s + \rho_\ell \phi_\ell \widetilde{C}_\ell \mathbf{v}_\ell\right) \cdot \nabla \theta = \tag{17}$$
$$\nabla \cdot (\mathbf{k}_m \nabla \theta) + \mathbf{T}'_m : \nabla \mathbf{v}_s + \frac{1}{\mu} \nabla P \cdot \mathbf{K} \nabla P + \phi_\ell H_c f_c (\delta_c, \theta) \ ,$$

where $\widetilde{C}_s$ and $\widetilde{C}_l$ are respectively the heat capacities of the solid and liquid constituents and

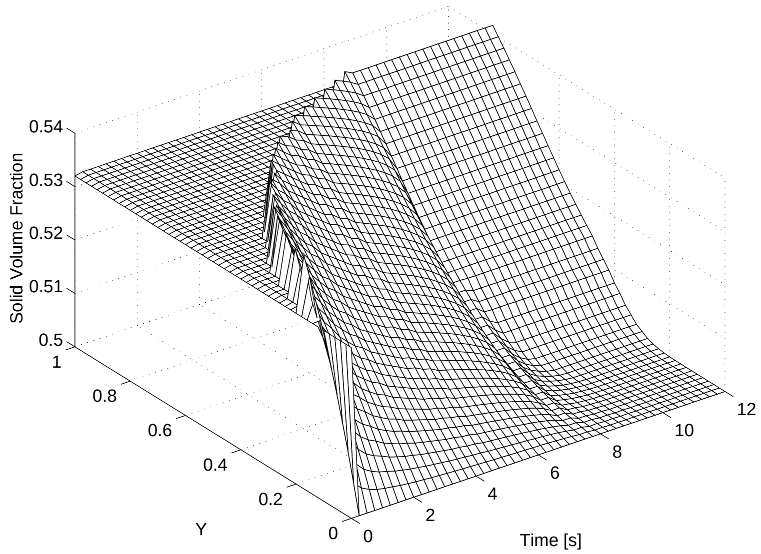
$$C_m =: \frac{\rho_s \phi_s \widetilde{C}_s + \rho_\ell \phi_\ell \widetilde{C}_\ell}{\rho_m} \qquad \rho_m =: \rho_s \phi_s + \rho_\ell \phi_\ell. \tag{18}$$

Summarizing the model to describe resin injection processes is given by Equations (1), (8), (15), (16), (18) with the constraint (3) and (13).

In Figure 1 we present the results of one–dimensional simulations obtained with the model above using an elastic constitutive equation for the stress field. The volume ratio is plotted versus time and the dimensionless Lagrangian coordinate $Y = \xi/L \in [0, 1]$.

The simulation in Figure (1a) corresponds to an unsuccessful industrial process in which resin cures before filling up the preform. The constant injection pressure of $0.1\,MPa$ determines a compression of the solid preform up to 94% of its initial length. In addition, this compression is non-uniform, with volume ratio ranging between $\phi_s = 0.5$ near the injection port and $\phi_s = 0.542$ near the infiltration front. About 8 seconds later the infiltration stops because the resin is gelling near the infiltration front. The chemical reaction is completed after about 50 seconds. By looking at the other state variables, it is possible to identify an initial time interval during which the mechanical aspects are dominant (say, up to 10 seconds). Then curing becomes the driving mechanism until that a final heat conduction–dominated stage occurs (say, from 40 seconds on). This rough distinction between an

(a)



(b)

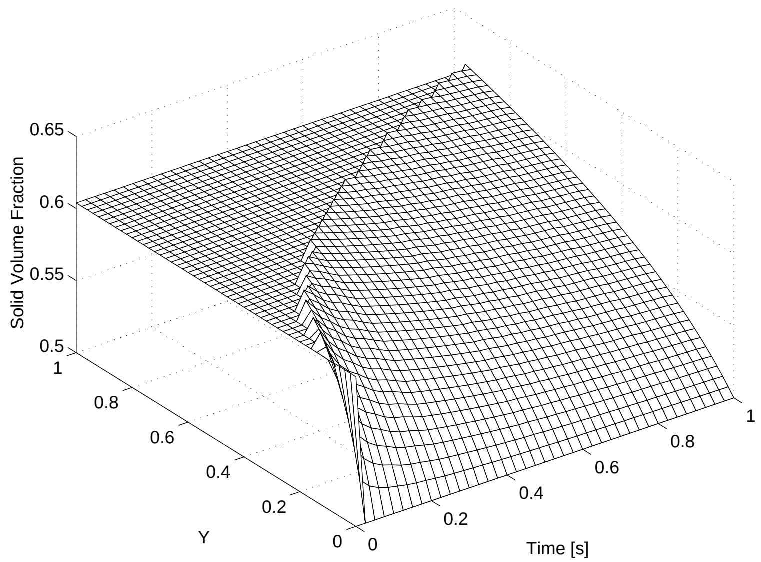


Figure 1: *Infiltration generated by a pressure drop of* $\Delta P = 0.1\ MPa$ *(a) and of* $\Delta P = 1\ MPa$ *(b).*

infiltration period and a temperature cycle, which is often used in describing some composites manufacturing processes, is not possible in other cases (metal matrix composites) and when fast reacting resins are used.

The simulation of Figure (1b) corresponds to a successful process which is achieved applying an injection pressure much larger than in the previous case (1 MPa). As a counterpart, the pressure gap used to avoid that the resin gel before infiltration is complete leads to a stronger compression of the preform. In fact, the preform compresses up to 82.5% of its original length, the volume ratio ranges between $\phi_s = 0.5$ (near the injection port) and $\phi_s = 0.616$ (near the infiltration front). Infiltration is however successfully completed in 0.97 seconds.

From a practical viewpoint, analyzing these simulations one could infer the following conclusions for the specific one–dimensional set up under consideration:

i) The smallest infiltration pressure sufficient to fill the preform is slightly larger that $0.1\, MPa$.

ii) The infiltration time is about $8\, s$.

iii) After infilitration the mould can be heated to speed up the curing reaction.

iv) If the preform is not pre–compressed in the mould, a gap will arise near the injection port. This may cause race–tracking (see next section) as sketched in Figure 3. It could be inferred that it is convenient to put an air vent along the infiltration axis and to pre–compress the preform with a pressure of say $0.2\, MPa$.

v) The final product will have a higher volume ratio near the air vents.

On the basis of this type of simulations one can produce moldability diagrams as the one shown in Figure 2, (see the caption). They give a glance of the range of parameters that can be used to produce successful manufacturing processes, of the inhomogeneous characteristics of the produced composite material, and of the time needed (and possibly of other quantities of interest, i.e. the maximum degree of cure achieved when the infiltration is completed). The window of parameters which can be used is usually upperly bounded by a maximum pressure which should not be overcome to avoid damages of the preform. Moreover a maximum temperature should not be overcome to avoid degradation.

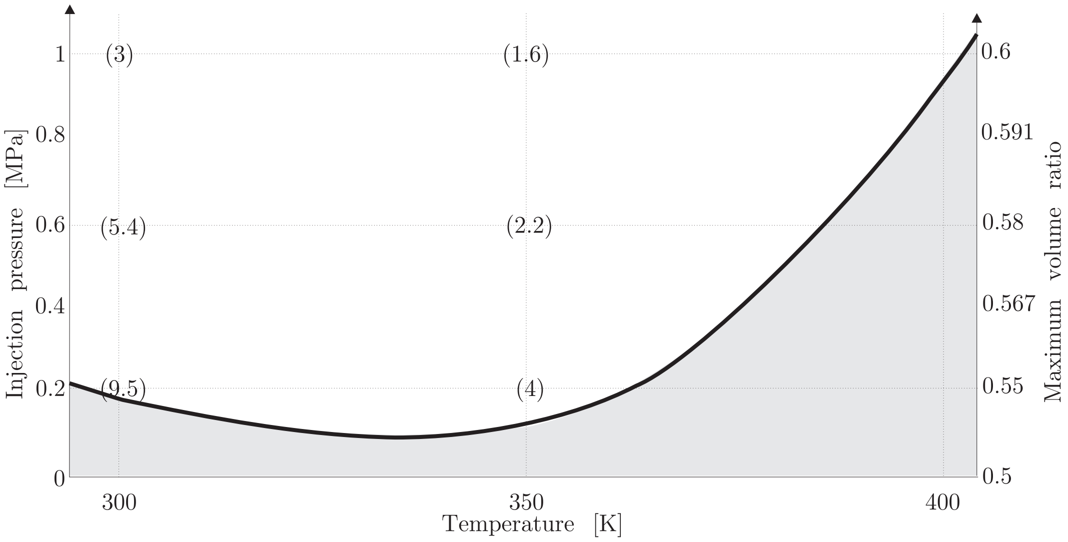


Figure 2: *Moldability diagram. Minimum injection pressure to achieve full infiltration. On the right the maximum volume fraction achieved near the injection port is indicated. The minimum is always 0.5 and is achieved near the injection port. The numbers in parentheses refer to the time needed to complete the infiltration process.*

3 Open Problems

Looking through the technological literature the reader can realize that there is a need to develop mathematical models for composites manufacturing processes and obtaining qualitative results which can help in identifying the process parameters and in improving the processes themselves.

Even though some issues have been clarified, they are actually a very early step toward a complete understanding of injection moulding. Many questions are still open and many areas deserve to be explored. In particular, we will point out some possible developments which should be carried out regarding the modelling aspects, being aware of the necessity of developing or founding the research in this field on experimental evidences which are still very scarce and mainly directed toward one dimensional elastic set–up.

The first problem which has to be solved before dealing with the most general three-dimensional simulations is the definition of the boundary conditions.

Even the meaning of boundary of a porous medium is not too clear. It is true that from the macroscopic view point one has an idea of where the border of a sponge is. It is however true that the matter becomes a bit uncertain as we zoom in as shown in Fig.(4). Even for the ideal perfectly ordered material in Fig.(4b) it is not clear whether the boundary is the "plane" that barely touches all the aligned fibers or should penetrate to some extent.

In the case of ideal perfectly ordered material, homogenization methods can hopefully give an answer, but in the case of Fig.(4a) we are still left with the problem of approximating a strange holed surface with a smooth boundary.

Figure 4 also allows to point out other crucial issues. First of all one may notice that the area occupied by the solid depends on how the "boundary" is defined. One then encounters some difficulties in defining a surface area fraction, which, of course, is not said to be equal to the volume fraction. This is particularly important as several authors assert that boundary conditions have to be based on surface quantities and therefore on surface area fractions. Fortunately, in composite materials and in most applications it is impossible to have perfectly aligned fibers, and the alignment will differ from specimen to specimen, so in the ensemble average framework we think it is plausible to assume that the surface area fraction and volume fraction are equal.

Another unsolved problem which is also related to the definition of a surface and to the difference between surface area fraction and volume fraction

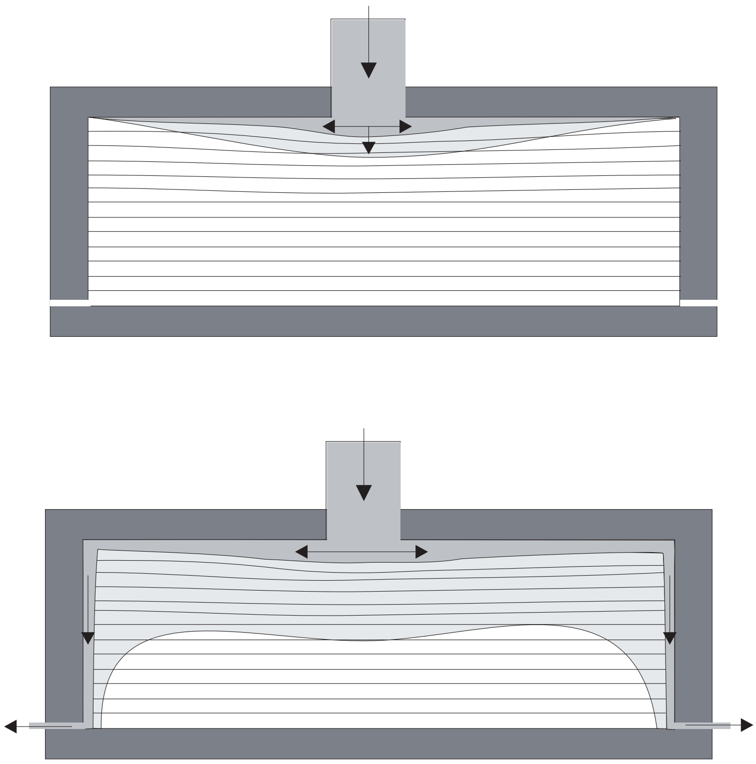
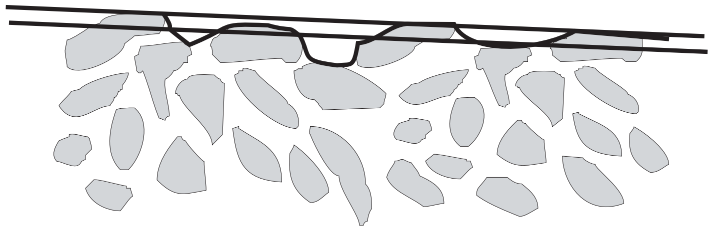


Figure 3: *The racetracking phenomenon.*

(a)



(b)

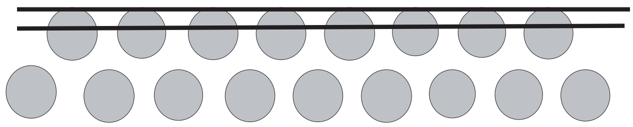


Figure 4: *Possible locations of the ideal edge of a porous material.*

is the transfer of loads. For instance, if a force is applied to a rigid boundary touching the border of the sponge, how does such a force transfer to the porous solid and to the liquid permeating it? Several methods have been suggested (e.g. continuity of chemical potential, saturation condition, mechanical splitting of traction [16], [20], [22], [23], [25]) but their results have still to be validated experimentally while a general theory is still missing.

Finally, it is useful to recall that in continuum mechanics the no–slip condition between, say, a fluid and a solid wall is only a successful empirical law which is valid if not pushed to the limit (rarefied gas, viscoelastic liquids, and so on). When dealing with flow over a rigid porous materials this boundary condition is usually replaced by the Beavers–Joseph's condition [5]. This is again a condition originally deduced from experimental evidences and then confirmed by several arguments [24] and lately also by homogenization methods [15] (see also Section 3.5 of [14]). Nothing is known, however, in the case of deformable matrix or when the flow is impinging the porous material.

Once the boundary condition issue is solved, it would then be possible to study one of the most relevant problems leading to industrially unsuccessful processes, the race–tracking problem [7], [12], i.e. the resin flow between the moulds walls and the preform (see Figure 3). In fact, in liquid composite moulding it is difficult to cut precisely the preform to the exact shape of the mould. Sometimes a gap exists between the preform and the mould edge. This gap, usually small (1 or 2 mm), can create a preferential flow path, giving rise to a non–uniform impregnation of the porous preform and possibly to the formation of dry spots. In some cases these preferential channels are opened by the flow itself which displaces the preform [13] [26]. In this way the liquid finds a more comfortable way to reach the air vents than the one passing through the porous preform, preventing the full infiltration. In order to foresee such phenomena one has to build a more detailed model which, for instance, the Stokes equation are coupled with a deformable porous mediumflow model. Again, this coupling requires a careful treatment of the interface conditions at the preform border when the impinging flow is not perpendicular to the border of the preform.

Particular attention should also be paid to the formulation of the constitutive equations of the solid preform. Experiments are to be addressed to determine the mechanical behaviour of solids so strongly anisotropic and heterogeneous and their typical parameters, e.g. elastic constants, retardation time, relaxation time, convective parameters and so on. The use of homogenization methods can be very useful in this field. The whole modelling suffers from the lack of precise measurements.

Even when the anisotropic and heterogeneous characteristics of the preform are undirectly known from the measurements on the permeability tensor (see [21] for a review), there are phenomena involving the coupling between the flow among the plies or the tows and inside them which are unresolved. This requires the study between micro and macro flow which again involves boundary conditions issues (see Figure 5). However, it would be desirable to deduce a macroscopic model able to evaluate, for instance, the amount of air trapped in the fiber tows.

While infiltrating in metal matrix composites the liquid melt solidifies, thus thickening the colder preform walls. In this way the space available for the metal flow narrows, thus decreasing the permeability. Eventually the flow can be stopped. This problem has only been addressed in [17], [19], [27] but deserves more attention.

Other processes for the production of composite materials which deserve to be studied are the so-called compression moulding and autoclave moulding. In such processes a number of fibers is preimpregnated ("prepreg") with a certain quantity of liquid matrix (for example epoxy resin which might be partially cured to facilitate handling), distributed in piles in a one–directional or multi–directional fashion and then placed in a possibly porous mould. The mixture of solid and liquid is then heated and compressed. The compression, operated by a piston, increases the solid volume fraction and produces a flow

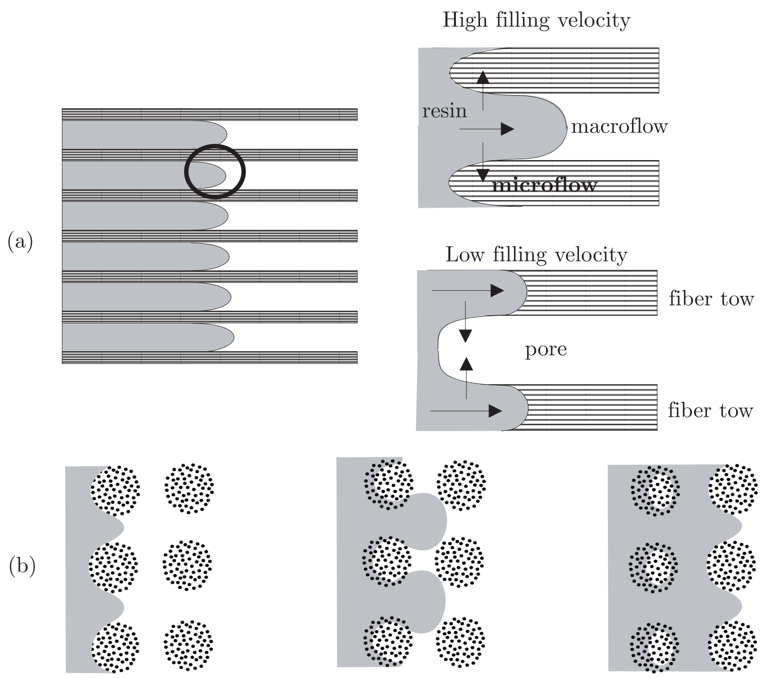


Figure 5: *Schematization of the interaction between micro and macro flow for a wetting resin. (a) Flow along the fiber tows. (b) Possible mechanisms of air entrapment for fast flow perpendicular to fiber tows.*

in a deformable porous medium squeezing the exceeding liquid out of the pile. The whole process is rather complex since it has to be fast enough to reduce fabrication costs and control resin cure and, at the same time, has to be accurate to avoid generating damages of the solid preform. In [8] a model for the 1D isothermal process has been developed. It could be very interesting to improve such a model considering a more realistic 3D geometry and including thermal effects.

Finally, there are many problems which could be studied from a qualitative viewpoint. Actually very few of them have been studied in some detail. The results obtained so far are reviewed in [9] where more open problems of practical interest are identified.

This short list of possible future developments of the research is not exhaustive, not only for obvious reasons of conciseness, but also because these studies are more at the "beginning than at the conclusion". This area is developing fast and is an incredible source of interesting mathematical problems.

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