COMPACTION OF DRY AND WET FIBROUS MATERIALS DURING INFUSION PROCESSES

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ABSTRACT

The objective of this study was to investigate the response of fibrous materials to compaction, since this response can affect significantly a number of important parameters, e.g. required tooling forces and fill-times, for some resin infusion manufacturing processes. A series of compression tests were carried out on both dry and wet (resin-impregnated) samples, at a number of different compaction speeds and to various final volume fraction levels. The materials were seen to exhibit a significant viscoelastic (stress relaxation) response, which changed according to whether the fibers were dry or wet. A thermomechanical model of fibrous material deformation was developed, incorporating the observed non-linear viscoelastic response, and the wet-dry change in response. The model is appropriate for the simple fibre compaction deformation which occurs during many of the liquid composite molding (LCM) processes. The model gives reasonably good results over a range of fiber volume fractions and compression speeds.

KEY WORDS: Resin Transfer Molding (RTM), Vacuum Assisted RTM (VARTM), Viscoelasticity/Creep Behaviour

1. INTRODUCTION

The Liquid Composite Molding (LCM) processes have become a popular means of manufacturing composite materials and are now used in the aerospace, automotive, infrastructure and military industries. Some of the reasons for their appeal are that they can allow for large and complex geometries and, because of the closed molds, harmful emissions are minimized.

The LCM family of processes includes Resin Transfer Molding (RTM), Injection Compression Molding (I/CM) and the flexible bag processes such as Vacuum Assisted Resin Transfer Molding (VARTM) and Seeman Composite Resin Infusion Molding (SCRIMP). The common features of these processes are that a dry fibrous material (preform) is placed in a mold, the material is compacted under pressure, a resin is injected into the preform, and the resin is allowed to cure (usually after the fibrous reinforcement has been fully saturated). In RTM a rigid mold is closed and the preform compacted to the final required thickness, then held until saturation is complete. In I/CM, the mold is partially closed, the resin is infused, then the mold is compressed to the final part thickness, meanwhile driving the fluid through the remaining dry material. In both the RTM
and I/CM processes, large tooling forces are required for the compression phases. In the flexible bag processes, the upper surface of the preform is covered with a bag, a vacuum is created inside the bag allowing the atmospheric pressure to compact the material, and the resin is finally driven through the material by a pressure differential between the resin inlet and vacuum. Thus the compaction forces are much smaller in the VARTM and SCRIMP processes, and these processes are further distinguished by the continual thickness changes which occur due to the flexibility of the covering bag.

The development of numerical models and simulations of the RTM [1-3], I/CM [4-6] and flexible bag [7-9] processes have helped in manufacture and design, for example by predicting fill times, fluid pressures and optimum placement of fluid injection ports. These models are not yet perfect, particularly in their ability to estimate the tooling forces in RTM and I/CM, and the thickness changes which occur in the flexible bag processes.

There are a number of different elements which are required in any model and simulation of a resin infusion process. One of the most important of these is the relationship between the compaction forces and the fiber volume fraction. This relationship is known to depend in a complex way on the fibre architecture, the number of layers in the preform, preform stacking sequence and the history of loading [10]. Many empirical models have been proposed to capture this response of fibrous materials to compaction, e.g. [11]. Most commonly used in process simulations is a non-linear elastic power law expression, usually given in the form [12]

$$\sigma = A(V_f)^n.$$  \hspace{1cm} (1)

where $\sigma$ is the fibre stress and $V_f$ is the local fibre volume fraction, and $A$ and $n$ are material parameters to be determined through experiment. The parameters in (1) need to be changed according to whether the fibers are dry or wet (saturated), since the stress required to compress saturated fibers is appreciably less than that to compress dry fibers, to a given volume fraction. The power law model captures well the relationship between stress and fibre volume fraction during compaction, both when the fibers are dry and when they are wet [13].

A number of semi-empirical micromechanical models, which assume that the individual fibers bend according to the elastic beam theory, have also been proposed. Some of these lead to power-law expressions similar to (1) [14] whereas others lead to more complicated functions of the volume fraction, with additional material parameters describing the microstructural geometry and fibre bending stiffness [15].

Elastic laws such as equation (1) do not give any information regarding the observed time-dependent response of fibrous materials. The most important manifestation of this viscoelastic behaviour is the significant stress relaxation which occurs, observed by many investigators, e.g. [16-17], and thought to be due to frictional fibre slippage. The stress relaxation response implies that the stress required to hold a fibrous material at a constant volume fraction decreases over time, and this can have consequences for the tooling forces in the RTM and I/CM processes, particularly since the observed stress relaxation often occurs over time scales on the same order as that of a full LCM process. Similarly, the observed thickness fluctuations during the flexible bag processes are due in part to the viscoelastic nature of fibrous materials [18], and an accurate
simulation of these processes requires a time-dependent model of fibre compaction. A number of authors have proposed that an elastic model be employed for the compaction phase and a separate model to capture the relaxation behaviour once compaction has ceased/paused [19-21], usually of the form

\[ \sigma(t) = \sum_{i=1}^{n} a_i e^{-t/t_{\text{R}}} , \]  

(2)

where the \( a_i \), and the relaxation times, \( t_{\text{R}} \), are model constants, though models of this type have yet to be incorporated into full process simulations.

In this study, a single viscoelastic model is proposed, to be used for both the compaction and relaxation phases of an LCM process. First, in the next two sections, an experimental study into the compaction behaviour of a fibrous material is described.

**2. EXPERIMENTAL PROGRAM**

An experimental study has been undertaken into the deformation behavior of a glass-fibre continuous filament mat (CFM, density 2.58 g/cm\(^3\), areal mass 450 g/m\(^2\)), Figure 1(a). The behavior of this material was considered at varying compaction levels, at various compaction speeds, and when both dry and wet (saturated).

**2.1 Experimental Procedure for Dry Material**  
Preform samples were prepared, each consisting of 8 layers cut into 0.2 m squares from a roll of CFM, Figure 1(b). The samples were placed between a set of parallel plates set up in an Instron 1186 testing machine, the upper plate fixed with the cross-head moving up. A constant velocity compressive strain history was applied, and the compaction force applied to the sample was recorded. This constant velocity compaction was then followed by a period in which the sample was held at constant thickness, as shown schematically in Figure 2. The force data recorded during the test was converted to stress by dividing by the preform surface area.

![Figure 1](image)

**Figure 1:** (a) Glass-fibre continuous filament mat (CFM) (b) roll from which layers were cut
Two series of tests were carried out. In series one, samples were compacted to a volume fraction $V_f = 0.35$ at speeds of 0.035, 0.5, 2, 5 and 100 mm per minute. After compaction, the samples were held at the constant thickness for about $5T$ seconds, where $T$ is the time taken for compaction, Figure 2(a). After this time, most of the stress relaxation was judged to have occurred and the stress leveled out towards a constant equilibrium value.

In theory, an infinitely slow compaction would allow for all viscous processes to occur during the compaction phase, with a consequent zero relaxation during the constant holding thickness phase of the experiment. In addition, the rate dependent viscous stresses would be zero during the compaction so that the overall stress required to compact would be relatively low. The first, slow, test was carried out to approach this equilibrium response of the material. In theory, an infinitely fast compaction would not allow time for any viscous effects to occur during compaction, with all these effects occurring during the constant holding thickness phase, resulting in a large stress relaxation. The final, rapid, test was conducted to approach this limiting case. A schematic of the expected response of the material to these limiting compaction speeds is given in Figure 2(b), where time is normalized with respect to $T$.

In the second series of tests, samples were compacted at 2 mm/min to final volume fractions of $V_f = 0.25$ and 0.45 (to complement the test from series one at 2 mm/min to $V_f = 0.35$).

Laser displacement gages were used to detect any elastic deflections in the upper plate. The deflections were recorded and translated into a discrepancy between the measured volume fractions and the true volume fractions. The errors were $\sim 0.5\%$ for the test to $V_f = 0.25$, $\sim 1-2\%$ for the tests to $V_f = 0.35$, and $\sim 3\%$ for the test to $V_f = 0.45$. The measured volume fractions were not adjusted for the discrepancy, the error merely being noted.
2.2 Initialisation of Preforms The initial fibre volume fraction $V_{f0}$ is often required in a mathematical model of fibre compaction (see below). The volume fraction at zero stress is very difficult to determine and it is usual to take $V_{f0}$ to be the fibre volume fraction at some nominal stress level. In this study a value $V_{f0} = 0.1051$ was chosen since this occurred at a stress level (approximately $\sigma = 6\,\text{kPa}$ for the dry samples) which was probably somewhat larger than the preform would have experienced on the uncut roll, it was accurately measurable in the testing machine, and it was deemed low enough not to affect too much the response of the material at the much higher loads encountered during full compaction. The initial volume fraction level was maintained until no further stress relaxation could be detected, typically after about 10-15 minutes, and the compaction phase was then begun.

2.3 Experimental Procedure for Wet Material For the wet experiments, circular preform samples were prepared, each consisting of 8 layers of diameter 265 mm. A hole of diameter 15 mm was cut into the centre of each sample to facilitate fluid injection. The samples were placed in a circular mold attached to the Instron testing machine, Figure 3.

Glucose syrup, with a viscosity of approximately 0.16 Pa.s at 17°C, was used as the test fluid. The fluid was injected into the mold cavity through a central injection gate, using a pressure pot at low pressure ($\sim 1\,\text{bar}$) so that the preform architecture was not disturbed. Once the preform was fully saturated, the injection line valve was shut off to prevent fluid returning to the pot. The fibers were then given some time to settle down.

As in the dry tests, the preforms were next compacted with constant velocity and then held for a time at a constant thickness. Two series were carried out. In series one, samples were compacted to a volume fraction $V_f = 0.35$ at speeds of 0.5, 2, and 5 mm per minute. In series two, samples were compacted at 2 mm/min to final volume fractions of $V_f = 0.25$ and 0.45. A final, dry, sample was compacted in the circular mold, to compare directly with the earlier dry tests. A laser displacement gage was used to measure plate deflection as in the dry tests. In these tests, the
error in the recorded volume fraction was always less than 0.01%, except for the test to \( V_f = 0.45 \), where the error was \( \sim 4.5\% \).

The fluid carries some of the compaction force and this has to be accounted for when evaluating the stress carried by the fibers. First, it is assumed that the total stress applied to the saturated preform, \( \sigma_{\text{total}} \), can be decomposed into a component taken up by the fibers, \( \sigma_{\text{fibre}} \), and a pressure in the fluid, \( p \),

\[
\sigma_{\text{total}} = \sigma_{\text{fibre}} + p \quad (3)
\]

From conservation of fluid mass within the axisymmetric preform [22],

\[
-\frac{1}{r} \frac{d(rq)}{dr} = \frac{\dot{h}}{h} \quad (4)
\]

where \( r \) is measured from the preform centre, \( q \) is the volume average fluid velocity, \( h \) is the preform thickness and \( \dot{h} \) is the rate of change of preform thickness. It is assumed that Darcy’s law governs the fluid flow through the fibrous material,

\[
q = -\frac{k}{\mu} \frac{dp}{dr} \quad (5)
\]

where \( k \) is the permeability. These lead to a differential equation for pressure, subject to the boundary conditions

(1) zero pressure at outer preform boundary, \( p(r_0) = 0 \)

(2) zero radial velocity at inner preform boundary, \( p'(r_i) = 0 \)

where \( r_i \) and \( r_0 \) are, respectively, the inner and outer radii of the preform. The total force carried by the fluid is then found to be

\[
F = 2\pi \int_{r_i}^{r_0} p(r) r dr = p_{r_i} \pi \left[ \frac{1}{2} \left( \frac{r_o^2 - r_i^2}{r_o^2 - r_i^2} - 2r_i^2 \ln \left( r_o / r_i \right) \right) \right] \quad (6)
\]

where \( p_{r_i} \) is the pressure at the inner preform boundary. This central fluid pressure, \( p_{r_i} \), was recorded using a pressure transducer with a range from \(-0.103\) to \(1.965\) MPa gage pressure. The force in (6) was then subtracted from the total compaction force to obtain the force carried by the fibers, and hence the fiber stress.
3. VISCOELASTIC MODEL

A non-linear viscoelastic model is developed in this section, the same model to be used during the compaction and relaxation phases. The model introduced is purely mechanical, not accounting for temperature variations; it is essentially a one-dimensional version of a finite–strain, linear viscoelastic, thermomechanical model (see, for example, [23]), extended to the non-linear regime. First, introduce the free energy function

\[ \Psi(e, \xi_i) = \Psi_w(e) + \sum_{i=1}^{N} \Gamma_i(e, \xi_i) \]  

(7)

Here, \( e \) is the observable strain and \( \xi_i \), are \( i = 1, \ldots, N \) internal kinematic variables, describing the viscous effects in the material. \( \Psi_w(e) \) is the free energy at equilibrium, that is, the energy stored after all viscous effects have terminated, whereas the second term, the so-called configurational free-energy, characterizes the non-equilibrium state.

Any of a number of different strain measures can be used for \( e \) in (7). For example, although the deformations are large, because the problem is one-dimensional one can use the engineering strain \( e = (h_0 - h)/h_0 \), where \( h \) is the current and \( h_0 \) the initial preform thickness. Here, however, as in [24], the strain measure to be employed is the true (logarithmic) strain and \( \dot{e} \) is the rate of deformation. In terms of volume fractions, these are (\( \dot{V}_f \) is the rate of change of \( V_f \))

\[ e = \ln \frac{V_f}{V_{f0}}, \quad \dot{e} = \frac{\dot{V}_f}{V_f} \]  

(8)

The free energy is chosen to be of the form

\[ \Psi(e, \xi_i) = \Psi_w(e) + \sum_{i=1}^{N} \frac{E_i}{n_i + 1} (e - \xi_i)^{n_i+1} \]  

(9)

since this reduces to the linear generalized Maxwell model (a free spring in parallel with \( N \) Maxwell units) when \( n_i = 1 \). By taking \( n_i \geq 1 \) one obtains models incorporating non-linear effects. Following standard thermodynamics arguments, the stress is now obtained through a differentiation:

\[ \sigma = \frac{\partial \Psi(e, \xi_i)}{\partial e} = \frac{\partial \Psi_w(e)}{\partial e} + \sum_{i=1}^{N} E_i (e - \xi_i)^{n_i} \equiv \sigma_w + \sum_{i=1}^{N} q_i \]  

(10)

The total stress is thus the equilibrium stress \( \sigma_w \) together with \( N \) viscous stresses; note that \( q_i \) can be interpreted as the viscous forces/stresses, and \( \xi_i \) the strain, acting in a dashpot attached to a non-linear spring, Figure 4. The thermodynamic force is
which can be seen to be equal to the \( q_i \), so the mechanical dissipation (the rate of working of the internal stresses which lead to an energy loss) is, by definition,

\[
\Phi(e, \xi, \dot{\xi}) = -\frac{\partial \Psi(e, \xi)}{\partial \xi} \dot{\xi} = \sum_{i=1}^{n} f_{i} \dot{\xi}_{i} = \sum_{i=1}^{n} q_{i} \dot{\xi}_{i} \tag{12}
\]

Figure 4: Spring and dashpot interpretation of the viscoelastic model

Take now a viscosity law of the form

\[
q_{i} = \eta_{i} (e_{\text{max}} - e)^{-m} \dot{\xi}_{i} \tag{13}
\]

Here, \( \eta \) is the viscosity, \( m \) is a material parameter, and \( e_{\text{max}} \) is the maximum theoretical strain possible in the material. This implies that the greater the volume fraction, the higher the viscous stress required to impart a given strain-rate. The idea here is that the more tightly packed are the fibers, the less room there is for fibre slippage, and hence viscous effects [24]. Eqns. (12) and (13) imply that \( \Phi = \sum \eta_{i} (e_{\text{max}} - e)^{-m} \dot{\xi}_{i}^{2} \), which is positive as required by the second law.

It remains to write down the evolution (first order differential) equations for the viscous forces, which follow from (11-13):

\[
\dot{q}_{i} = -\frac{n_{i} E_{i}^{1/n_{i}}}{\eta_{i}} \left[ (e_{\text{max}} - e)^{m} q_{i}^{2-1/n_{i}} - \eta_{i} \dot{q}_{i}^{1-1/n_{i}} \right] \tag{14}
\]
4. RESULTS

Figure 5 shows results for compaction of two dry preforms at 2 mm/min to $V_f = 0.35$. The first (solid line) is from the first dry series of tests, involving a square preform; the second (dotted line) is for a circular preform with a circular hole cut out, as for the wet tests. The two preforms were also cut from different rolls. These results indicate the high degree of repeatability of these compaction-type tests on CFM.

Figure 5: stress for compaction to $V_f = 0.35$ at 2 mm/min (square preform: solid line, circular preform with central hole: dashed line)

Figure 6 shows results for compaction of both dry and wet preforms to $V_f = 0.35$ at different compaction speeds (solid lines). These results show that the maximum stress increases with compaction speed and that the stress required to compact the wet preform is up to 40% less than that required for the dry preforms. Also shown are model results, using the 6 parameters given in Table 1, with $N = 1$ in equation (9). These parameters were chosen so as to give good agreement with the 2mm/min experiment, with the accuracy somewhat less at speeds removed from this. It was found that the model for wet compaction could be obtained from the dry model by changing only the equilibrium stress $\sigma_w = E_n e^{n_w}$ (see Eqn. 10) and one other parameter.

Table 1: Model parameters (see Eqns. 10 and 14 with $N = 1$); $\sigma_w$ in Eqn. 10 is given by the power law $E_n e^{n_w}$
Figure 6: Stress as a function of time for CFM preforms compacted to volume fraction $V_f = 0.35$ at different compaction speeds (experiment: solid line, model: dashed line)

Figure 7 shows normalised results; time normalized with respect to $T$ and stress with respect to the maximum stress reached in each test. A feature of these results is that the effect of the different compaction speeds only manifests itself during the relaxation phase.

Figure 7: Normalised stress as a function of normalized time for dry CFM preforms compacted to volume fraction $V_f = 0.35$ at different compaction speeds
Finally, Figure 8 displays results for compaction to different volume fractions. Again, the model captures the viscoelastic features displayed by the fibrous material.

**Figure 8:** Stress as a function of time for CFM preforms compacted at 2 mm/min to different final volume fractions (experiment: solid line, model: dashed line)

5. DISCUSSION

A single model has been used in the above for both compaction and relaxation phases. Further work needs to be done to better relate components of the model to the observed phenomena and the underlying mechanisms which occur, and to modify the model accordingly. For example, some of the mechanisms which govern compaction response are likely to be different to those which govern the relaxation response [10], and features such as those observed in Figure 6 need to be explained and captured. It might be necessary to have some model components which only operate during one of either the compaction or relaxation phases, as in [25], where two different linear viscoelastic models were utilized, one for each phase.

The model used here should better predict the forces arising in RTM and I/CM processes. In flexible bag processes the compaction pressures are very much lower than those which emerged in the experimental study described above, and further tests would have to be carried out at lower load levels to test the viability of the model for these processes. Further, any model of a flexible bag process would have to be robust enough to cope with the continuous time dependent
component thickness changes which occur. This would have to be tested for by incorporating the model in a full simulation of an actual filling process.

An important feature of fibre compaction not addressed in this paper is the permanent deformation which occurs [26]. For example, the response of a fibrous material during one loading and unloading cycle will in general differ from that during subsequent cycles. This permanent change in material behaviour is thought to be due to the nesting of fibers into gaps in the preform structure, and to a permanent increase in aspect ratio of fibre bundles due to stressing [26]. In a recent study [27], it was found that these permanent deformation effects might well dominate over the viscoelastic effects accounted for by the model described in this paper, and that most of the hysteresis effects seen with these materials are due not to viscous mechanisms such as fiber slippage, but due to the permanent changes that take place to the reinforcement architecture. It will be necessary to include these effects in any ideal compaction model.

6. CONCLUSIONS

An experimental study has been carried out into the response of a fibrous material to transverse compression, as occurs in many composite material manufacturing processes, in particular the Liquid Composite Molding (LCM) processes. The experiments highlighted the complex time-dependent response of such reinforcing materials, and the difference in response according to whether the reinforcement is dry or saturated with resin. A non-linear viscoelastic model of material response to load has been developed which accounts for much of the observed phenomena, for the purpose of incorporation in full simulation models of the LCM processes. Further work needs to be done in this area; in particular, an LCM process model should ideally account for the significant permanent change effects which are known to occur to reinforcements during compression.

7. REFERENCES