Flow Characterisation for Partially Impregnated Prepregs

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ABSTRACT

The flow of resin in ZPREG - a partially impregnated prepreg is considered as the material is heated under a vacuum. A simple 1-D flow model is developed to predict the advance of the flow front with time. Results from the model are compared with experimental data obtained from video capturing images of the advance of the flow front with time. The model is reasonably successful in predicting the flow behaviour, but suggests that the resin advances less quickly than the experimental data. This may be due in part to the enhanced flow rate of a pigment used to colour the resin.

1. INTRODUCTION

Achieving full impregnation for composite parts with thermosetting matrices is often thought to require the use of an autoclave. However, the development of prepregs which can be processed using vacuum consolidation has opened the thermosetting market to manufacturers without specialist facilities and has also facilitated the production of large structures (Steel, 2004). A recent development in this field is the production of partially impregnated prepregs, such as ZPREG from the Advanced Composites Group and SPRINT manufactured by SP SYSTEMS. In the former case, the resin is applied to the reinforcement in a series of parallel stripes, and in the latter case, the resin takes the form of layers of film. Partially impregnated prepregs offer the convenience of pre-impregnated material together with some of the formability associated with dry fabric. In addition, these materials can be used to vacuum form components with very low void content, since the selective application of resin allows the air to be evacuated through well-defined air paths. However, to maintain quality standards, careful consideration has to be given to the flow of the resin through the structure of the reinforcement during consolidation. If the resin cures before the structure has been fully wetted, then the resulting component may be brittle and fail at low levels of applied load due to the presence of large voids. Conversely, if the resin is slow to cure, then the processing cycle will be lengthy, and production costs will rise.

Achieving the correct balance between obtaining full impregnation and keeping cycle times to a minimum depends on many factors. These include: matrix rheology, reinforcement structure and lay-up, complexity of the component, processing parameters such as temperature and pressure, and the initial distribution of the resin.

In this paper, partially impregnated glass/epoxy prepregs supplied by the Advanced Composites Group are characterised by considering the rheology of the matrix and by vacuum forming laminates under a range of processing conditions. The resin is pigmented, which allows the advance of the resin flow fronts to be monitored by video. In addition, a simple Darcy flow model is developed which predicts flow front advance and degree of impregnation.

2. MANUFACTURING AND TESTING OF ZPREG LAMINATES

Laminates were manufactured from the ZPREG material using a vacuum consolidation technique. The material is a 2 x 2 twill woven glass/epoxy prepreg, with a final fibre volume fraction of 56%. Different processing schemes were utilised and mechanical testing and void content measurement was performed on the resultant laminates.

A 750W silicone rubber embedded filament heating mat with a programmable temperature controller was used underneath a glass moulding surface to provide an elevated temperature environment for processing. To verify the temperature indicated by the temperature controller, a thermocouple was placed between the glass mould and the material, and images of the upper surface were taken with a thermal camera. The difference between the temperature controller reading and the thermocouple was found to be negligible. Images from the thermal imaging camera indicated the presence of a through-thickness temperature gradient of about 10%. This is not thought to be large enough to affect the manufacturing process significantly.

To produce laminates, layers of ZPREG were cut and stacked onto the glass moulding surface. Release film was placed over the stack of material such that it extended onto the glass by at least 25mm beyond the edges of the fabric. Since photographic techniques were used to observe the flow progression during moulding, breather cloth was placed around the edges of the fabric, producing a ‘frame’ such that the vacuum was distributed around the laminate edges while observation of the top surface was still possible. Tacky tape was placed around the material and vacuum bagging film was used to cover the assembly. A vacuum gauge was connected using a through-bag connector to measure the vacuum pressure inside the bag. The reading from this gauge was compared with that from the gauge mounted on the vacuum pump manifold and good agreement was observed. The complete moulding set up is shown in Figure 1.

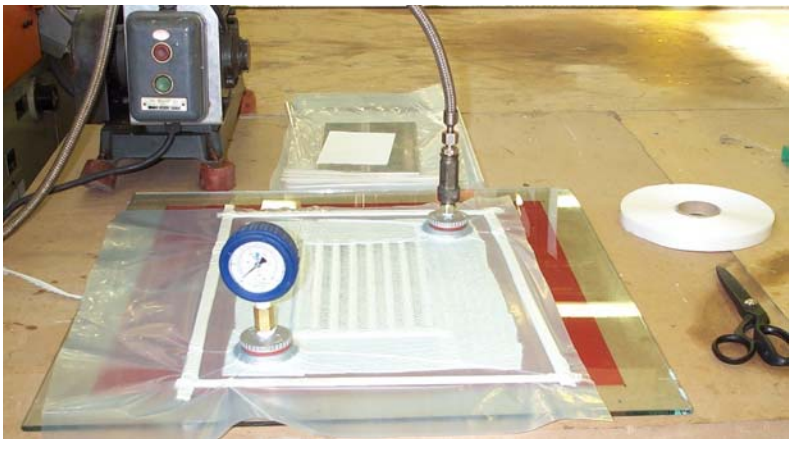

Figure 1 Moulding set up showing in-bag vacuum gauge.

To produce laminates for this study, a heating cycle was started with the programmable thermal controller, and a vacuum was subsequently applied at the required level. Three different heating cycles were used; these are shown in Table 1. In order to examine the effect of vacuum level, selected laminates were produced using a reduced vacuum. All other laminates were produced using the maximum vacuum level which could be obtained. This was typically around 985mbar. Details of the laminate production conditions are given in Table 2.

Flow behaviour was observed by mounting a digital video camera above the set-up and the information recorded was used for flow characterisation using the image analysis technique described in Section 3.

Table 1 Heating cycles used for laminate production

Cycle designation	Type	Description
A(n)	Isothermal	Material heated to target temperature (n) before applying vacuum
B	Single ramp	Temperature raised from ambient to 100°C at 3°C/min then held at 100°C for 2 hours
C	Double ramp	Temperature raised from ambient to 80°C at 2°C/min, held at 80°C for 30 minutes then raised to 120°C at 2°C/min and held at 120°C for a further 30 minutes

Table 2 Summary of laminate production conditions

Laminate designation	Material	Number of layers	Lay-up type†	Heating cycle	Vacuum level (mbar)
RG01	ZPREG264FRB/GF1000/GF1000	1	n/a	C	max
RG02	"	2	S	C	max
RG03	"	2	O	C	max
RG04	"	2	P	C	max
RG05	"	1	n/a	A(65)	max
RG06	"	1	n/a	A(80)	max
RG07	"	1	n/a	A(100)	max
JC02	"	2	S	B	900
JC03	ZPREG264B/GF1100/GF1100	4	S	B	950
JC04	"	4	S	B	900
JC05	"	4	S	B	800
JC06	"	4	S	B	850
JC07	"	4	S	C	800

† - Lay-up types are shown in Figure 2.

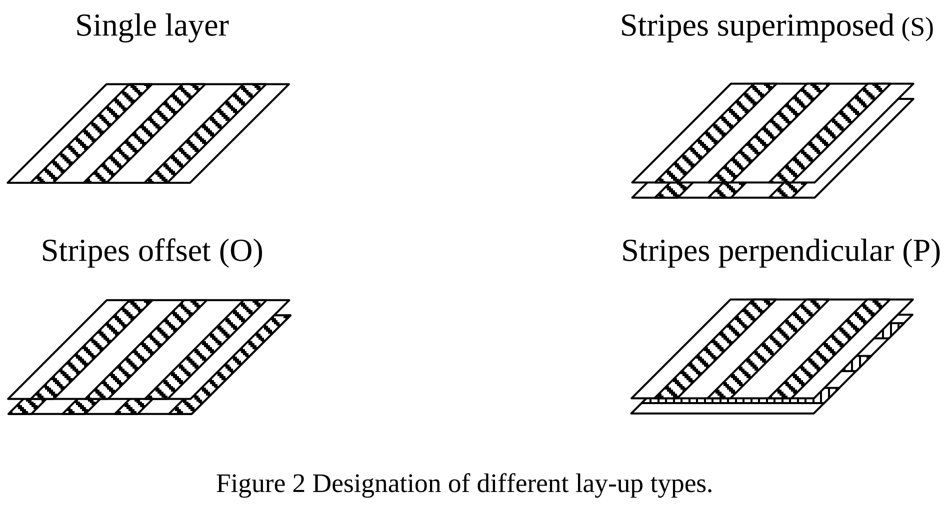

Figure 2 Designation of different lay-up types.

3. FLOW CHARACTERISATION

As stated in the previous section, photographic recordings were made during the production of each laminate. For those laminates designated RG, digital still camera photography was used at regular intervals, while for laminates designated JC, digital video photography was used. Frames were taken from the video footage at one minute intervals for image analysis. The analysis procedure is described in this section.

A typical photograph obtained from the camera is shown in Figure 3. This is a full colour image, and extends beyond the edges of the material. The first steps in the analysis procedure were to crop the image to size and convert it to greyscale. These operations were carried out using commercial image manipulation software (Paint Shop Pro). The area to be cropped is indicated in by a dashed rectangle. It can be seen that this runs between the centres of two dry stripes, and includes three complete resin stripes. The same area was analysed from each image pertaining to a single sample in order to obtain consistent data.

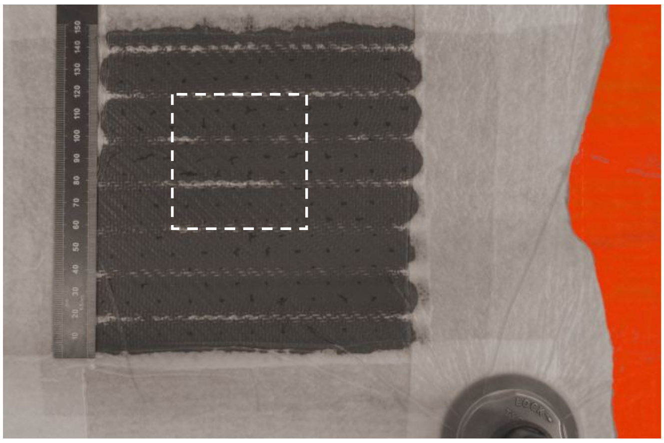

Figure 3 Typical image obtained from overhead camera. Dashed rectangle indicates portion to be used for image analysis to determine flow progression.

The cropped area indicated by the dashed rectangle was imported into an image analysis software package, ImageTool (provided free of charge by the University of Texas Health Science Centre in San Antonio). A threshold was applied such that pixels above a certain grey level became white, while those below it became black, generating a binary image. The threshold level was chosen by examining the grey level frequency histogram for the image and choosing a value between the two modes of the distribution. The black and white pixels were counted using the software and this data (i.e. the proportion of material which had been infiltrated by resin) was plotted as a function of time. Results are presented in Section 4.

4. EFFECT OF TEMPERATURE ON ISOTHERMAL FLOW BEHAVIOUR

Laminates were produced using isothermal conditions by allowing the heated mould surface to reach its target temperature before applying a vacuum. Flow behaviour was observed using a digital camera mounted above the mould, and analysed using the image analysis techniques described in Section 3. The effect of the temperature on the rate of flow progression can be seen from the data shown in Figure 4.

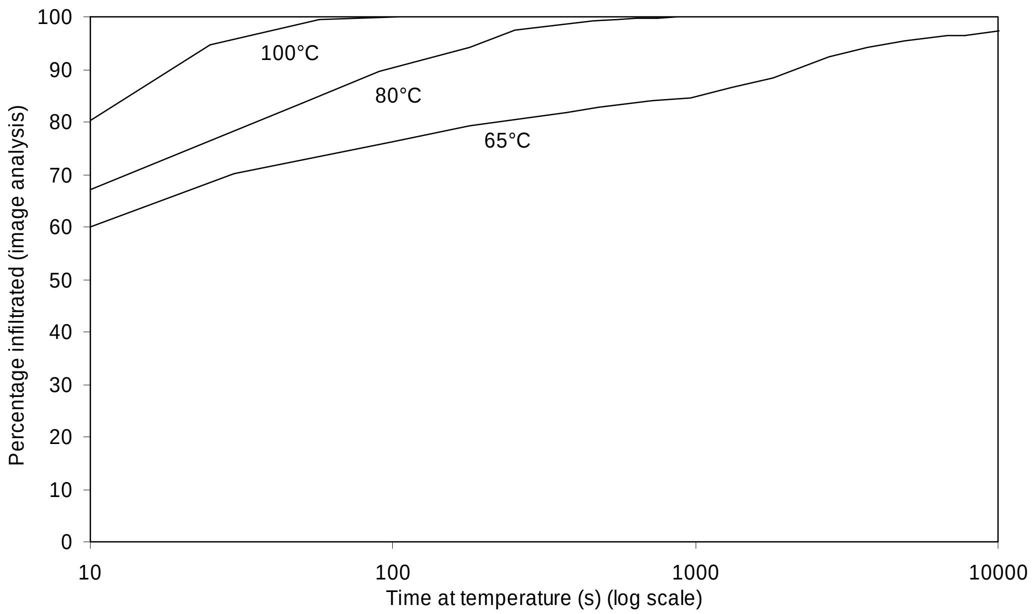

Figure 4 The effect of temperature on isothermal flow behaviour.

From the data presented, it can be seen that some flow occurs during the heating cycle before the vacuum is applied. This is thought to be due to capillary pressure drawing the resin along the tows as the resin viscosity reduces with increasing temperature. The results in Figure 4 show the significance of temperature on processing properties; it is clear that cycle times are greatly extended if a moulding temperature of 65°C is used.

It should be noted, however, that higher temperatures also result in faster cure times; hence under certain conditions, e.g. a lower vacuum, the resin may not fully infiltrate the fibre at higher temperatures before curing causes the viscosity to increase. Some insight into the nature of these mechanisms can be obtained by examining the resin viscosity-time history at different temperatures, supplied by the manufacturer. This is shown in Figure 5.

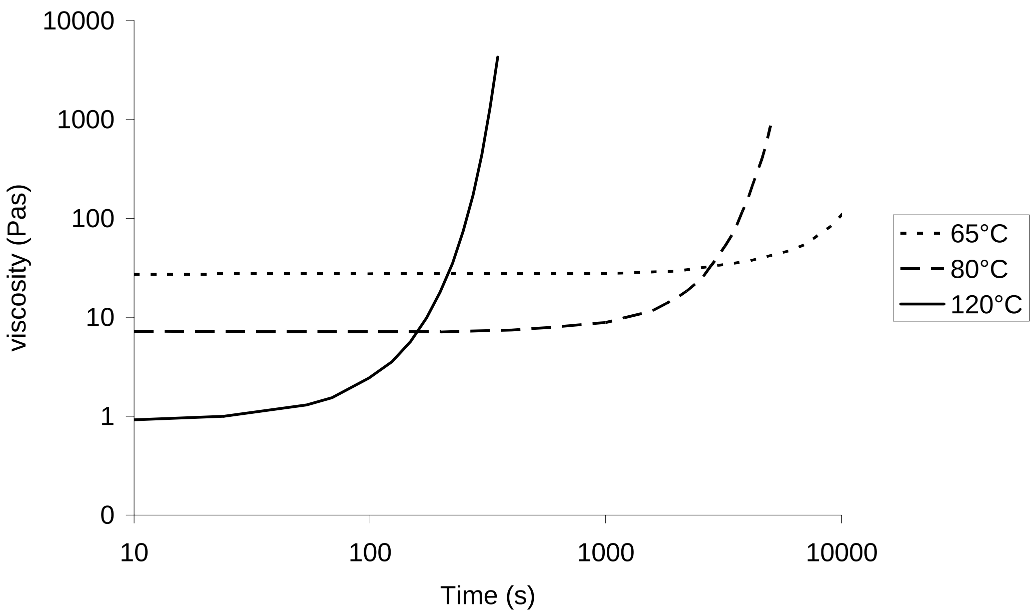

Figure 5 The viscosity-time history of VTM-264 resin at different temperatures (manufacturers data).

It can be seen from Figure 5 that while the resin viscosity at 120°C is initially considerably lower than at 65°C, the curing process occurs so rapidly that the viscosity is higher than at 65°C after only 200 seconds.

5. EFFECT OF VACUUM LEVEL ON NON-ISOTHERMAL FLOW BEHAVIOUR

Plaques designated JC03 to JC06 were produced using identical processing conditions with the exception of the vacuum level. Temperature was increased from ambient to 100°C at a rate of 3°C/min and held at 100°C for 2 hours (cycle B). Flow behaviour was observed and quantified using the techniques described in Section 3. Results are presented in Figure 6.

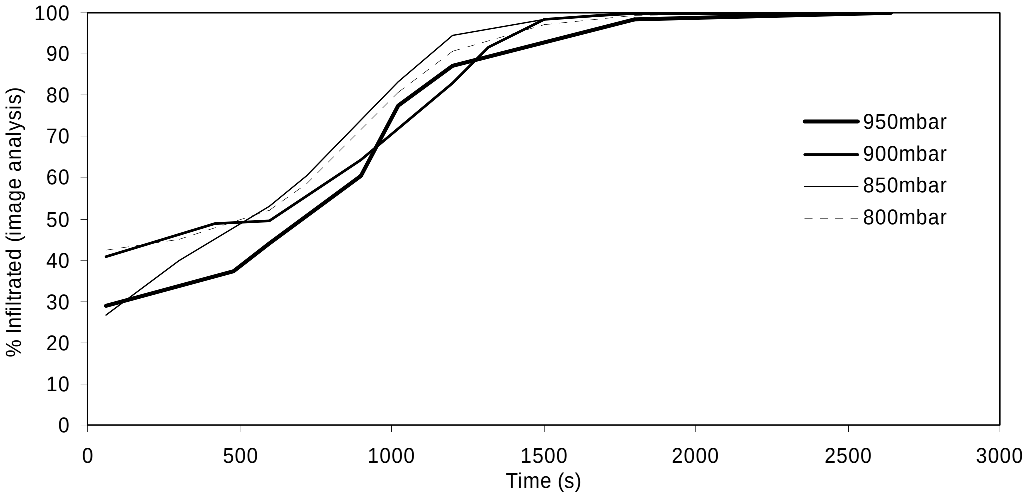

Figure 6 The effect of vacuum level on flow behaviour during processing of ZPREG laminates.

While all laminates appeared to take approximately the same time to consolidate fully, the laminate produced at 950mbar exhibits a more distinctively shaped consolidation-time curve than the other laminates. No significant conclusions on the effect of vacuum pressure on processing behaviour may be drawn from the data available at present.

It should also be noted that the pigment in the resin did not appear to flow at the same rate as the resin. If a single layered laminate is held in front of a lamp, the striped pattern is clearly visible. Narrow pale stripes can also be seen when examining the laminate surface under normal lighting conditions with the naked eye, although this effect is more prominent in laminates produced at lower vacuum levels.

Laminate production using vacuum levels below 800mbar was not attempted since it was observed that fully homogenised consolidation did not occur at 800mbar using this heating cycle. Open voids were observed in the resin at the mould surface of the cured laminates manufactured at both 800mbar and 850mbar. Such voids were not observed in laminates produced with a vacuum pressure of 900mbar or greater. This suggests either that higher vacuum levels are required for production, or that an alternative heating cycle may be used to provide an optimum period of low resin viscosity.

Flow behaviour during production of laminates with an alternative double ramp heating cycle (cycle C - temperature raised from ambient to 80°C at 2°C/min, held at 80°C for 30 minutes then raised to 120°C at 2°C/min and held at 120°C for a further 30 minutes) was also analysed; results can be seen in Figure 7. It can be seen from the data presented, that cycle B offers a faster infiltration time, although it is not possible to determine the onset of cure from this data, and hence it is not possible to determine whether a potential reduction in cycle time may be achieved. A potential route to reducing cycle time suggested by the data in Figure 7 may be to increase temperature to 100°C at 3°C/min, hold for 15 minutes, then increase to 120°C at 3°C/min to increase the curing rate.

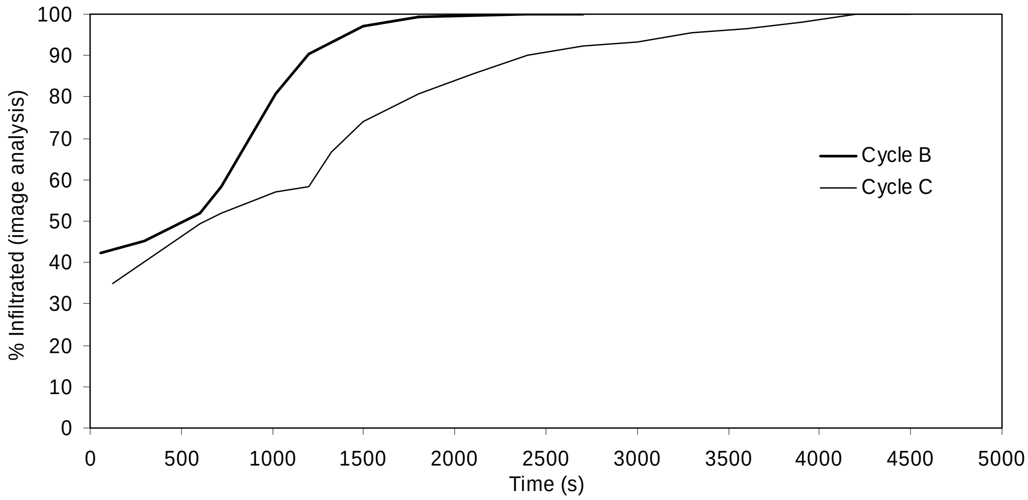

Figure 7 The effect of heating cycle on flow behaviour during production of ZPREG laminates. JC05 was manufactured using heating cycle B, while JC07 used heating cycle C (defined in Table 1).

6. FLOW MODELLING

In this section, a simple model to describe the flow of resin through partially impregnated laminates is developed and applied to ZPREG laminates.

In-plane flow through a porous medium can be described using Darcy's law. For one-dimensional flow in the x-direction can be written as:

$$u_x = -\frac{K}{\mu}\frac{dP}{dx} \tag{1}$$

where K is the permeability, μ is the fluid viscosity (which is a function of time – see Figure 5), and P is the fluid pressure. The permeability can be modelled using the Kozeny-Carman equation:

$$K = k\frac{(1-v_f)^3}{v_f^2} \tag{2}$$

Here, the Kozeny constant, k, was taken as 1.73 x 10^{-12} to fit flow data obtained at 65°C. For many industrial processes such as resin transfer moulding, it is often assumed that the pressure gradient is linear. However, it has been shown that this simplification can lead to inaccurate results Correia (2004). For partially impregnated composites, where there is no further supply of resin, the pressure at the flow front, P_x, can be written as:

$$P_x = P_a - P_f + P_c \tag{3}$$

where P_a is atmospheric pressure, P_f is the pressure due to the fibre bed, and P_c is the capillary pressure. For 1-D flow,

$$P_x = \frac{\mu}{2K}\frac{\dot{v}_f}{v_f}(x_f^2 - x^2) \tag{4}$$

where x_f is half the current resin band width. The fibre volume fraction,

$$v_f = \frac{\rho_a}{\rho_f h} \tag{5}$$

where ρ_a is the superficial density, ρ_f is the fibre density, and h is the current height of the filled region. Hence,

$$\dot{v}_f = -\frac{\rho_a}{\rho_f h^2}\frac{dh}{dt} \tag{6}$$

and so

$$\frac{\dot{v}_f}{v_f} = -\frac{1}{h}\frac{dh}{dt} \tag{7}$$

Hence, Equation 4 can be written as:

$$P_x = -\frac{\mu}{2Kh}\frac{dh}{dt}(x_f^2 - x^2) \tag{8}$$

A similar expression was derived for radial flow by Saunders (1997) based on the earlier work of Gutowski (1987). Note that the negative sign indicates that the thickness of the composite reduces with time as the resin spreads. The total resin pressure can be obtained by integration over the filled region and by dividing by the projected area. Hence Equation 8 becomes:

$$P_x = \frac{\mu x_f^2}{2Kh}\frac{dh}{dt} \tag{9}$$

Rearranging for one time step, Δt, the change in height becomes:

$$\Delta h = P_x \frac{3Kh}{\mu x_f^2}\Delta t \tag{10}$$

To advance the flow front, consider the conservation of resin volume:

$$hx_f(1 - v_f) = C \tag{11}$$

where C is a constant, which can be calculated from the final volume fraction.

According to Robitaille (1998), the pressure due to the fibre bed can be approximated by a power law model,

$$P_f = \left(\frac{v_f}{v_{f0}}\right)^{1/B} \tag{12}$$

where v_f is the fibre volume fraction, v_{f0} is the initial fibre volume fraction, and B is the stiffening index. Based on a large set of experimental data for both aligned and random reinforcements, Correia (2004) suggests that

$$B = k_1 \ln v_{f0} + k_2 \tag{13}$$

where $k_1 = -0.0657$ and $k_2 = -0.0274$

The capillary pressure can be written as:

$$P_c = \frac{4\phi v_f \cos\theta}{d(1 - v_f)} \tag{14}$$

where ϕ is the surface tension, θ is the contact angle, and d is the fibre diameter (Amico 2001). The height, h, of the fabric changes during the impregnation due to the flow of resin, and can be written as:

$$h = \frac{s_d}{\rho v_f} \tag{15}$$

where s_d is the areal density of the fabric and ρ is the density of the resin. By assuming that the volume of resin remains constant, the advance of the flow front can be computed from the fabric height.

The model was implemented in a spreadsheet using material data for ZPREG. The viscosity data was taken as a function of time, and the initial position of the flow front was taken from the video images after the heating phase. It was noted that the resin spread during the heating phase (before the vacuum was applied). This could, in principal, be modelled by capillary flow, but would require more detailed viscosity data. The Kozeny constant, k, was taken as 1.73×10^{-12} for all simulations.

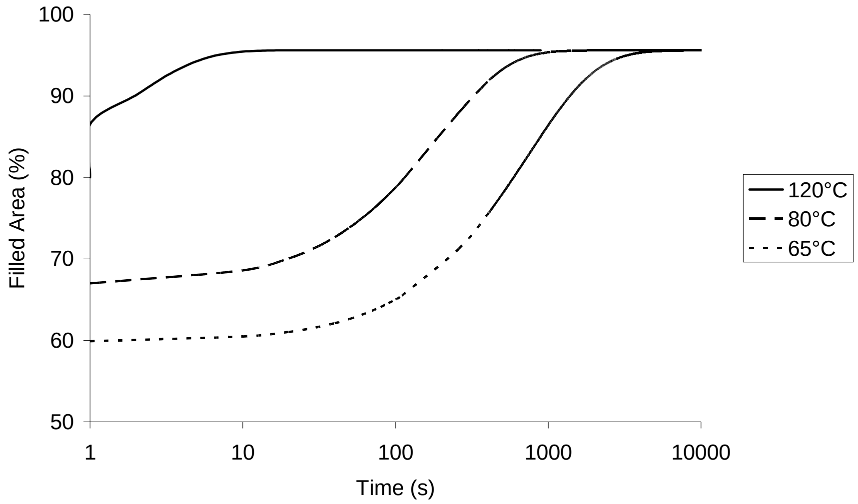

Figure 8 Numerical modelling of the advance of the flow front at three temperatures.

Results of applying the model to isothermal data are shown in Figure 8. Finally, the results from numerical simulations are compared to experimental data in Figure 9. There is reasonable agreement, particularly in the shape of the curves, but at 80°C, the numerical simulations predict that the flow front advances slower than the experimental data suggests. This may be due to the difference in flow behaviour of the pigment and resin – the pigment flows more rapidly, and the video images may suggest that the flow front position is further advanced than the actual position of the resin.

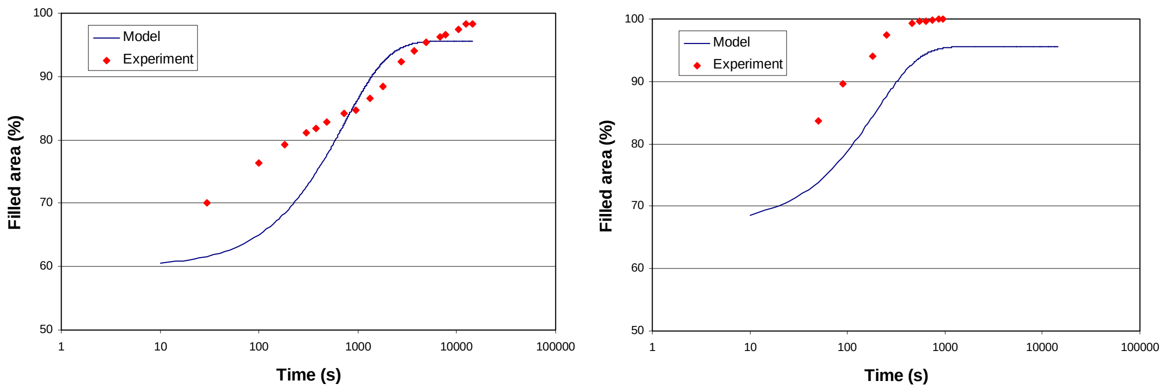

Figure 9a,b Experimental data compared to the model based on Darcy flow. Processing conditions: a) temperature = 65°C, vacuum pressure = 950mbar, b) temperature = 80°C, vacuum pressure = 950mbar.

7. CONCLUSIONS

Laminates were manufactured from the ZPREG material system using various combinations of processing parameters. Layers were oriented differently with respect to one another in order to investigate the effect of lay-up on void content in the resultant laminates. No significant change in void content was observed for different lay-ups, although it was noted that a laminate produced under the same conditions from a single layer of the material system had a lower void content.

Higher temperatures were observed to lead to faster flow front advance, e.g. full impregnation was observed to take approximately 100s at 100°C, compared with over 10,000s at 65°C. It must be noted, however, that higher temperatures lead to faster cure times, and if the temperature is too high the resin may cure before the reinforcement is fully impregnated.

Vacuum pressure was not observed to have a significant effect on flow behaviour determined using overhead photography and image analysis, although the laminate produced using 950mbar vacuum pressure was observed to have a higher maximum flow rate than those produced at lower pressures.

Open voids were observed in the resin next to the mould surface in laminates produced with a lower vacuum pressure (850mbar or less), giving rise to a poor surface finish. These were not evident in laminates produced at 900mbar.

The 1-D flow model predicts the advance of the resin with reasonable success. The difference between predictions and experimental data may be due, in part, to the more rapid flow of the coloured pigment.

ACKNOWLEDGEMENTS

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