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RESIN FLOW IN A RESIN FILM INFUSION PROCESS

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SUMMARY: The resin film infusion (RFI) process is a liquid moulding technique which is being used to manufacture advanced composite structures which are cost-effective and of high quality. To successfully manufacture RFI parts, it is desirable to understand and be able to predict the flow behaviour of the resin during the cure. This paper reports some results obtained from a research program conducted at the CRC-ACS focussing on resin flow in a RFI process. The flow performance of different resin films, including Hexcel M18 and RTM6, ACG XHTM45 and Cytec 5250-4, were assessed from dynamic viscosity profile measurements and through a standardised T-panel flow test. A simplified analysis, based on Darcy's law, was used to adequately predict flow heights using the dynamic viscosity profile (from which a flow factor is calculated), fibre volume fraction and applied pressure.

KEYWORDS: Flow Prediction, Resin Film Infusion, Viscosity, Flow Factor

INTRODUCTION

Manufacturing cost plays a key role in the application of advanced composite materials in the aerospace industry. The current manufacturing method is still dominated by the hand lay-up of resin-preimpregnated reinforcement (prepreg) which is later cured in an autoclave under high temperature and pressure. Alternative manufacturing methods being investigated include liquid moulding techniques such as resin transfer moulding (RTM) and resin film infusion (RFI). In the RFI process, a resin film and dry reinforcement is laid into a tool and bagged up as in the case of traditional prepreg manufacture. The lay-up is then placed into an autoclave where temperature and pressure are used to enable the resin to flow and infiltrate the reinforcement.

At the CRC-ACS, a number of research programs have been conducted where RFI has been used to manufacture advanced composite structures [1-4]. These include aileron skins, swaged wing ribs, "T"-section stringers and 3-bay aft boxes. However, to successfully manufacture such parts, it is desirable to understand and be able to predict the flow behaviour of the resin during the cure. To do so, a simple technique was developed at the CRC-ACS [5] which is based on a one-dimensional model of Darcy's Law and the dynamic viscosity curve of a given resin. To validate the model, a standardised T-stiffened panel experiment was used from which flow heights can be measured. To further substantiate the model and characterise the flow performance for a range of resins, further testing and analysis was undertaken. The findings from this work are presented in this paper.

EXPERIMENT

Flow Test

A T-panel flow test was developed, as shown in Fig. 1, to assess the performance of different resins in the reinforcement. In the set-up resin film is placed beneath the reinforcement, which comprises of two angle preforms that are placed back-to-back to form a T-panel. Steel angles are bolted together to encase the preform with aluminium spacers placed between the steel angles to control the web thickness. Sealant tape is used at the edges of the preform to prevent leakage. The nominal web thickness used in the test was 3.5 mm which gives a fibre volume fraction of 59% for the preform used. The assembly is sealed with a sealant tape and bagged under a nylon vacuum bag. The assembly is then cured in an autoclave to a specified cure cycle. Flow heights can then be measured from the top of the skin to the edge of the flow front.

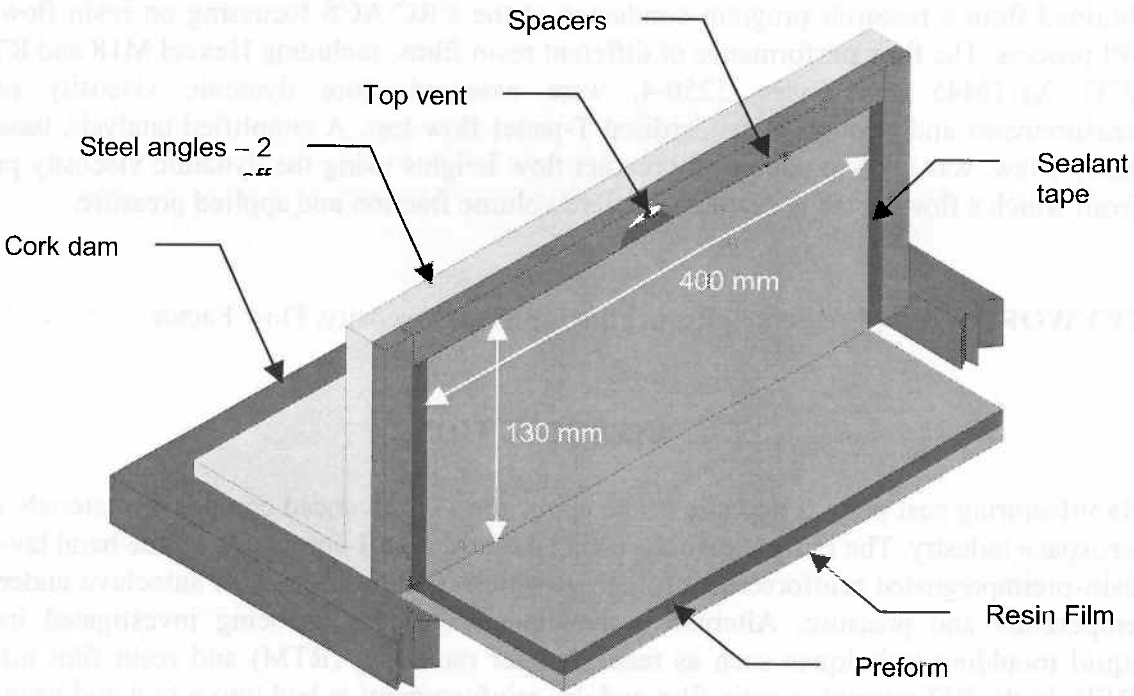

Figure 1 A basic lay-up for a T panel

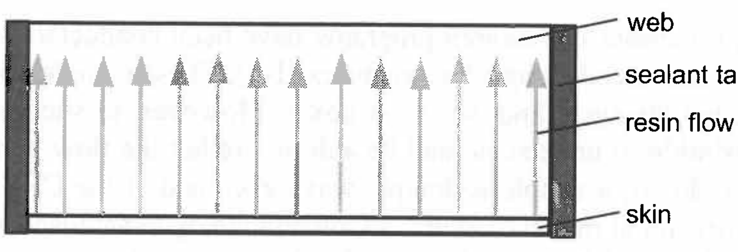

Figure 2 Resin flow pattern in the T Panel test

Dynamic Viscosity Test

To obtain viscosity data, dynamic viscosity profile (DVP) testing was performed by the CSIRO Molecular Science Group, in Melbourne. The instrument used was a parallel plate Control Stress Rheometer (model CSR10) from Bohlin Instruments. Testing was undertaken at a frequency of 10 Hz for a range of temperature profiles.

Materials

Four different resin systems were used in the testing: M18 and RTM6 from Hexcel, XHTM45 from the Advanced Composites Group and 5250-4 from Cytec-Fiberite. The first three resins are epoxies while 5250-4 is a bismalimide. All the resins came in film form, apart from RTM6 which was 'staged' into a film. The staging involves heating the liquid resin at 160°C for a predetermined length of time. To quantify the staging, the degree of cure and rheological properties were determined by a DSC test and Rheometer, respectively.

The angle preform consists of 5 layers of G926 5 harness satin, 370g/m^2 carbon fabric from Hexcel. All test panels were oriented with the fabric 90° (weft) direction in the flow direction up the stiffener.

Test Matrices

The majority of flow tests and DVP measurements were performed on the M18 resin system. M18 has been used on a number of demonstrator products at the CRC-ACS and can be considered as a baseline for this work. A summary of the flow tests undertaken in this work is shown in Table 1, indicating the number of tests and cure cycle used. As can be seen, two cure cycles were used. The standard cure cycle consists of a nominal heating rate of 2°C/min and a 2 hour cure at 177°C while pressure of 630 kPa and vacuum of -90 kPa is maintained throughout. The dwell cure, which is used only for 5250-4, is the same as the standard cure cycle except a temperature dwell of 30 minutes at 140°C is introduced, as per the requirements of the resin system.

A number of DVP tests were performed on the resin systems to look into the influence process parameters have on the flow performance of the resin. The parameters included heating rate, temperature dwell, resin ageing at room temperature and level of staging (for RTM6). A summary of the test matrix is given in Table 2. All tests were started at 50°C and were stopped after the 30 minute dwell at 177°C.

Table 1 Flow height test matrix

Resin	No. of Tests	Cure Cycle
M18	7	Standard
RTM6	1	Standard
XHTM45	2	Standard
5250-4	2[1]	Dwell

[1] Stiffener height of 180 mm used in second test.

Table 2 DVP test matrix

Resin	Sample ID	No. of Samples	Heating Rate (°C/min)	Dwell Cycle	RT[1] Ageing (days)
M18	M18/1	2	1	177°C for 30 min	0
	M18/2	2	2	177°C for 30 min	0
	M18/5	1	5	177°C for 30 min	0
	M18/10	1	10	177°C for 30 min	0
	M18/28	1	28	177°C for 30 min	0
	M18/D140	1	1	140°C for 30 min + 177°C for 30 min	0
	M18/D160	1	2	160°C for 30 min + 177°C for 30 min	0
	M18/A7	1	1	177°C for 30 min	7
	M18/A14	1	1	177°C for 30 min	14
XHTM45	XHTM45/1	1	1	177°C for 30 min	0
RTM6 – no staging	RTM6/NS	1	2	Straight to 200°C – no dwell	0
RTM6 – staging 5 min @ 160°C	RTM6/S5	1	2	Straight to 200°C – no dwell	0
RTM6 – staging 10 min @ 160°C	RTM6/S10	1	2	Straight to 200°C – no dwell	0
5250-4	5250-4/1	2	1	177°C for 30 min	0
	5250-4/2	1	2	177°C for 30 min	0

[1] Sample aged in a temperature controlled lab – RT = 21°C

FLOW ANALYSIS

Model

A simple technique was developed at the CRC-ACS to predict the flow in the T-panel flow test. The technique, which has been described previously [5], is based on the one-dimensional model depicted in Fig. 3. As can be seen, the model ignores the width of the skin laminate and considers one-dimensional flow through two regions of different permeability. These regions represent the through-thickness flow through the skin, which is assigned a permeability K_z and a distance x_{TT}, and the in-plane flow through the stiffener, which is assigned a permeability K_x and a distance x. It is also assumed that there is no pressure drop in the resin, which is at pressure P, and that the resin behaves as a newtonian fluid.

The governing equation is based on Darcy's Law which may be represented as follows:

$$\frac{dx}{dt} = \frac{KP}{(1 - V_f)x\eta(t)} \tag{1}$$

where x is the flow distance, dx/dt is the flow velocity, K is the permeability in the flow direction, P is the pressure, V_f is the fibre volume fraction and $\eta(t)$ is the resin viscosity defined by the dynamic viscosity curve.

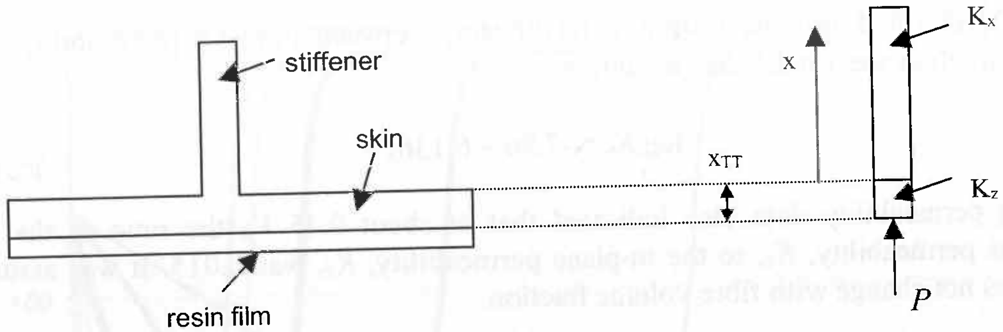

Figure 3 One-dimensional flow model

If we assume the initial condition is at: t=0, x=0, the solution for $\eta(t)$ as a function of time is:

$$x^2 = \frac{2KP}{(1-V_f)}\int_0^t \frac{1}{\eta(t)}dt \tag{2}$$

where we call $\int_0^t \frac{1}{\eta(t)}dt$ the Flow Integral.

Since the flow distance up the stiffener is proportional to the square root of the flow integral, we can define a Flow Factor, F, which is directly proportional to the flow height:

$$F = \sqrt{\int_0^t \frac{1}{\eta(t)}dt} \tag{3}$$

The Flow Factor is calculated from the dynamic viscosity data by numerical integration. It can be seen that the flow factor is inversely related to the area under the viscosity curve. The smaller the area, larger the flow factor and further the resin will flow.

Eqn. 3 can be used to predict the flow height up the stiffener if the permeability is known. However, resin flow is both through-thickness of the skin as well as in-plane in the stiffener as shown in Fig. 3. To make the flow length easier to calculate, the through-thickness section can be represented by an "equivalent length" of in-plane flow. A scale factor is required to determine the equivalent length, x_{EL}. The scale factor used is:

$$\frac{K_z}{K_x} = A \quad \text{and} \quad \frac{x_{EL}}{x_{TT}} = \sqrt{\frac{1}{A}} \tag{4}$$

To predict the actual flow length up the stiffener (referenced from the top of the skin), x, the equivalent length must be deducted from the total predicted in-plane flow length as follows:

$$x = \sqrt{\frac{2K_xP}{(1-V_f)}\int_0^t \frac{1}{\eta(t)}dt} - x_{EL} \tag{5}$$

Permeability

Permeability values for the G926 carbon reinforcement were obtained from two-dimensional radial flow tests performed at the CRC-ACS. The advancing flow front technique was used where the permeability is determined by measuring the velocity of the advancing resin front in the dry preform. From the testing, a relationship between in-plane permeability and fibre volume fraction was established as follows:

$$\log K_x = -7.96 - 6.136V_f \tag{6}$$

Existing permeability data also indicated that at about 0.45 V_f, the ratio of the through-thickness permeability, K_z, to the in-plane permeability, K_x, was 0.015. It was assumed this ratio does not change with fibre volume fraction.

RESULTS AND DISCUSSION

Flow Factors

Results from the DVP tests, which include the minimum viscosity and Flow Factor, have been summarised in Table 3. Some of the profiles are also shown in Figures 4 and 5. It should also be noted that the resins tested behaved as newtonian fluids, as is assumed in the flow model. This was verified through trial DVP runs which were performed at different shear rates.

Looking at the results for M18 it can be seen that increasing the heating rate from 1 to 28°C/min generally decreases the minimum viscosity of the resin. This is attributed to the occurrence of two opposing phenomena. A faster temperature rise increases the reaction rate but also lowers the viscosity of the resin more quickly, which happens to be the more dominant effect [6], thus lowering the viscosity. Although increasing the heat-up rate decreases the minimum viscosity, this did not translate to an increasing Flow Factor. This may be explained by the fact that a faster heating rate also causes the resin gel time, which can be considered as the flow time, to be reduced thus negating the gain of a lower viscosity.

Table 3 Results from DVP tests showing minimum viscosity and calculated flow factors

Resin	Sample ID	Min. Viscosity (Pa.s)	Flow Factor ($Pa^{-1/2}$)
M18	M18/1	2.852	30.96
	M18/2	1.535	31.86
	M18/5	0.968	31.64
	M18/10	0.831	30.09
	M18/28	0.951	30.65
	M18/D140	3.094	30.81
	M18/D160	1.604	31.55
	M18/A7	4.500	24.48
	M18/A14	4.841	23.67
XHTM45	XHTM45/1	3.717	28.37
RTM6 – no staging	RTM6/NS	0.740	>69.3[1]
RTM6 – staging 5 min @ 160°C	RTM6/S5	0.804	56.71
RTM6 – staging 10 min @ 160°C	RTM6/S10	3.474	24.26
5250-4	5250-4/1	0.476	108.8
	5250-4/2	0.479	97.0

[1] Initial profile was not recorded therefore values are slightly less than should be.

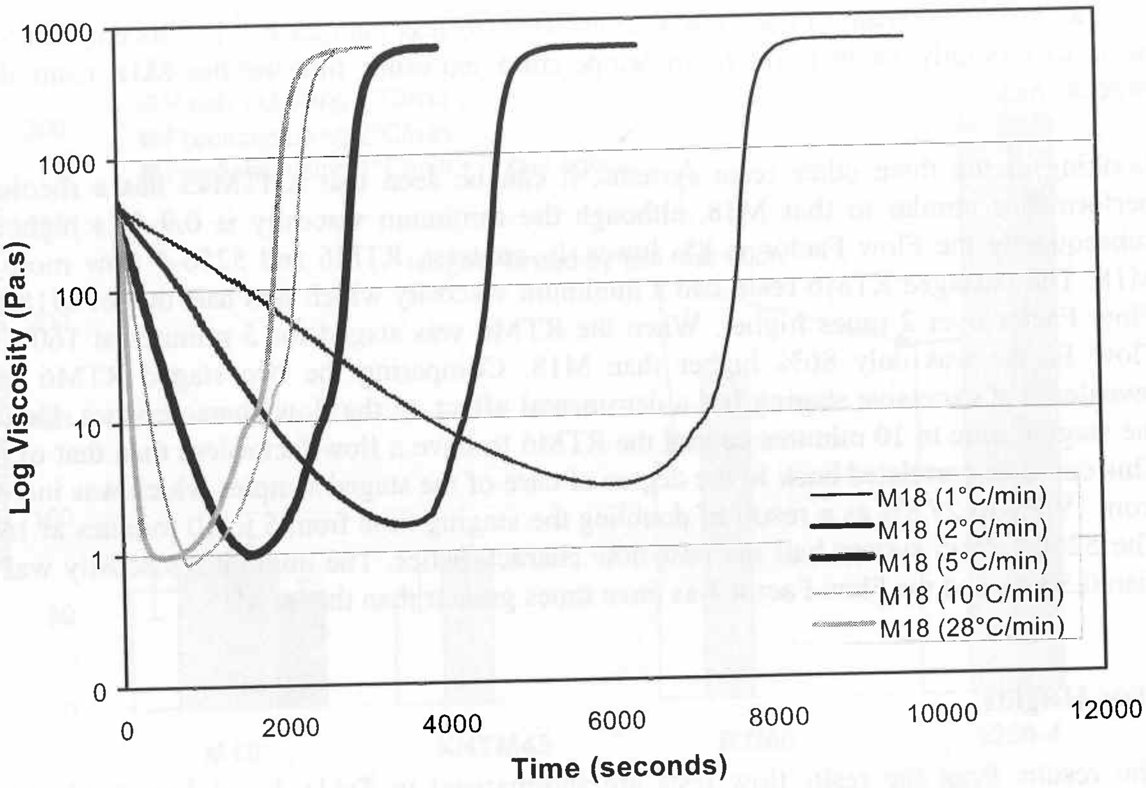

Figure 4 Influence of DVP with heating rate (for M18 resin)

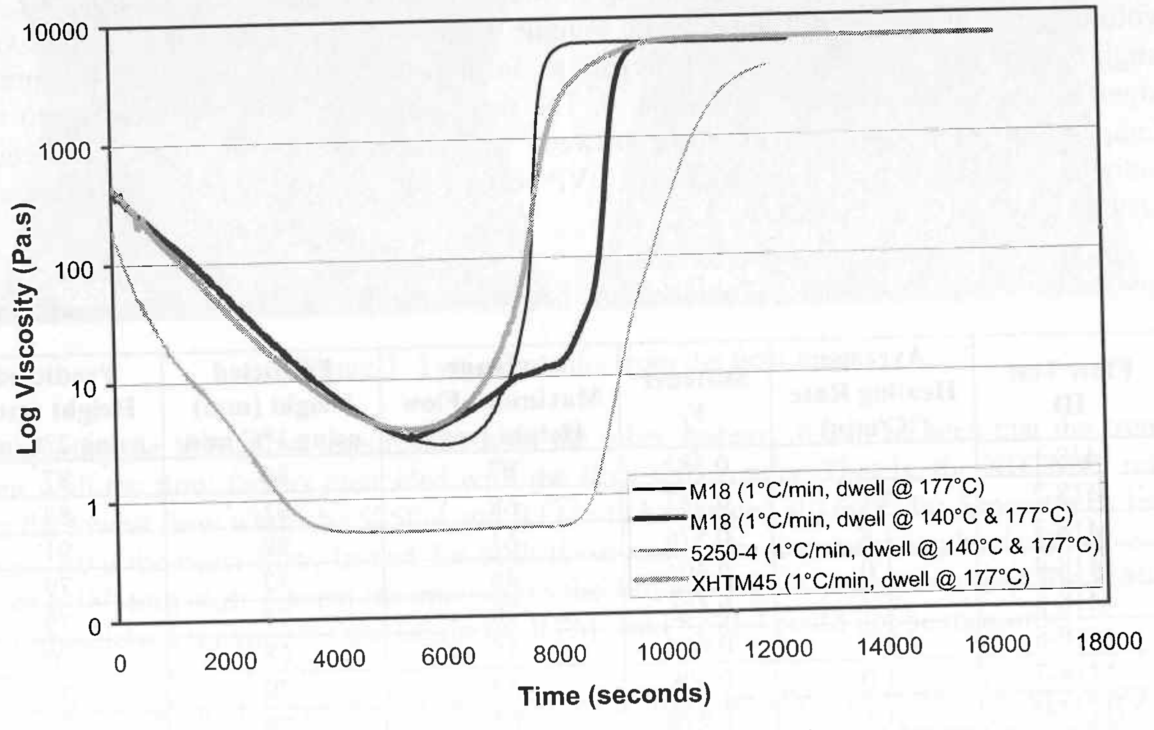

Figure 5 Comparison of DVP for various resin systems

Introducing a temperature dwell into the M18 temperature profile also had little influence on the Flow Factor. Although the processing window is increased by introducing a dwell (see Fig. 5), the minimum viscosity also increases which translated to no gain in the Flow Factor. Looking at the results from the aged samples of the M18 resin, it can be seen that ageing for 7 and 14 days causes a significant increase in the minimum viscosity (2.85 to over 4.5 Pa.s) as well as a 20% decrease in the Flow Factor (30.96 to less than 25 Pa$^{-1/2}$). This emphasises the need to carefully control the room temperature exposure time of the M18 resin during processing.

Looking at the three other resin systems, it can be seen that XHTM45 has a rheological performance similar to that M18, although the minimum viscosity is 0.9 Pa.s higher and subsequently the Flow Factor is 8% lower. In contrast, RTM6 and 5250-4 flow more than M18. The unstaged RTM6 resin had a minimum viscosity which was half that of M18 and a Flow Factor over 2 times higher. When the RTM6 was staged for 5 minutes at 160°C, the Flow Factor was only 86% higher than M18. Comparing the two staged RTM6 results revealed that excessive staging has a detrimental affect on the flow characteristics. Doubling the staging time to 10 minutes caused the RTM6 to have a flow factor less than that of M18. This could be correlated back to the degree of cure of the staged samples which was increased from 19.7% to 27.8% as a result of doubling the staging time from 5 to 10 minutes at 160°C. The 5250-4 resin system had the best flow characteristics. The minimum viscosity was less than 0.5 Pa.s and the Flow Factor was three times greater than that of M18.

Flow Heights

The results from the resin flow tests are summarised in Table 4 and Fig. 6, along with predictions using the resin flow model. Examples of test panels are shown in Fig. 7. As can be seen from the test results, the flow heights achieved with the M18 system ranged between 45 mm and 87 mm, with the scatter being attributed to differences in tool heat-up rate, fibre volume fraction and resin ageing. Fibre volume fraction in the web of the T-panel varied slightly between tests which is sufficient to influence the permeability of the preform significantly, as can be seen from Eqn. 6. The time which the resin was exposed to room temperature prior to cure also varied between tests because of difficulties in scheduling autoclave cures. As was seen from the DVP tests, resin ageing at room temperature can change the flow factor by over 20%.

Table 4 Flow height test results

Flow Test ID	Average Heating Rate (°C/min)	Stiffener V_f	Average Maximum Flow Height (mm)	Predicted Height (mm) using 1°C/min	Predicted Height (mm) using 2°C/min
M18-1	-	0.582	87	84	87
M18-2	1.1	0.587	68	81	84
M18-3	1.8	0.576	61	88	91
M18-4	1.0	0.596	46	77	79
M18-5	1.4	0.596	70	77	79
M18-6	1.0	0.614	45	68	70
M18-7	1.0	0.608	53	70	73
XHTM45-1	1.1	0.576	65	79	-
XHTM45-2	1.8	0.596	50	69	-
RTM6	1.0	0.582	>127[1]	-	166
5250-4	1.0	0.582	>180[1]	332	295

[1] Maximum height restricted by height of test sample

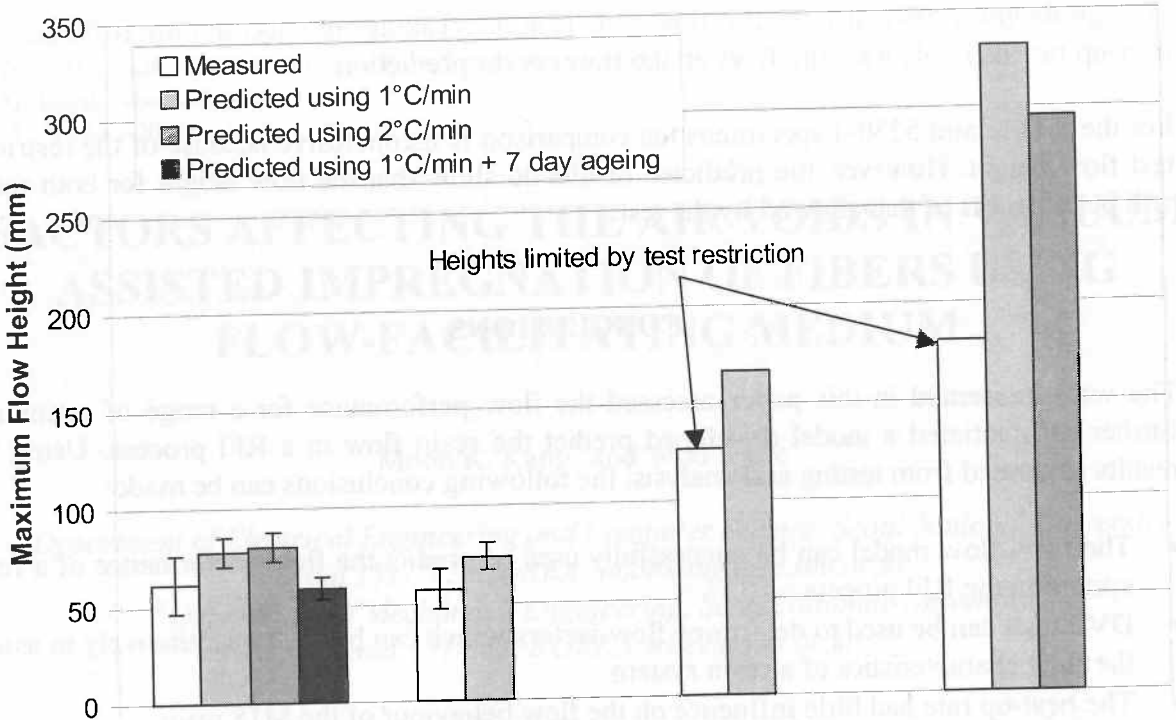

Figure 6 Comparison between measured and predicted flow heights

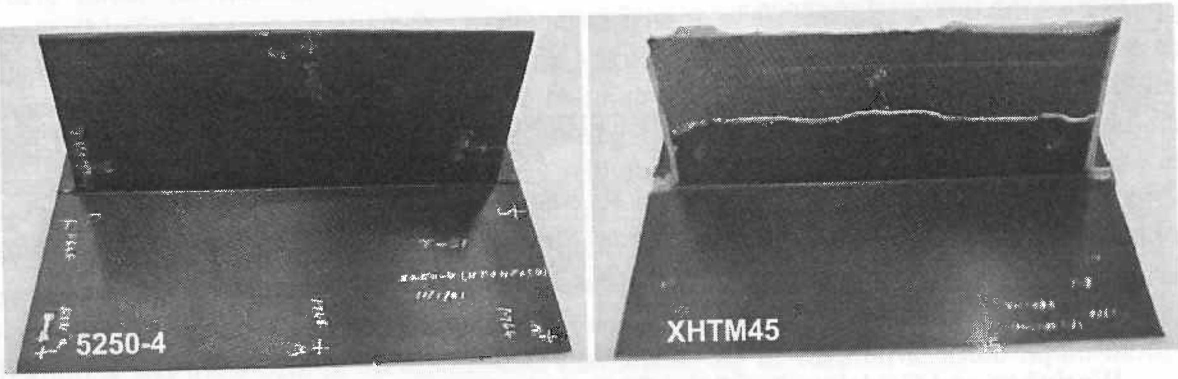

Figure 7 Typical results from the flow tests

Comparing the M18 flow tests results with the other systems, it can be seen that the trends seen with the flow factors coincided with the flow tests results. That is, the XHTM45 resin has the lowest flow while the 5250-4 and RTM6 (when staged at 160°C for 5 minutes or less) resins have the most flow. In fact for both these resins, the T-panel completely filled, even when a 180 mm high T-panel (as opposed to the 130 mm high T-panel) was used for 5250-4. Unfortunately, a maximum flow height for RTM6 and 5250-4 could not be measured.

Using the flow model, the flow factors obtained from the DVP tests and the permeability relationship given in Eqn. 6, maximum flow height predictions were calculated and compared with the test data as shown in Table 4 and Fig. 6. For the M18 and XHTM45 resin systems, the tendency was for the predictions to overestimate the measured flow heights by up to 30%. However when possible resin ageing is taken into account, the difference between the predicted and measured flow height is considerably less. If it is assumed that the resin has aged by up to 7 days at room temperature, as may have been the case with some samples dues to difficulties in scheduling autoclave cures, than the predicted flow height is within 5% of the average height measured from the flow tests (Fig. 6). Taking into account the differences in heat-up rate had only a small effect on the flow height prediction.

For the RTM6 and 5250-4 specimens the comparison is inconclusive because of the restricted test flow height. However, the predicted results do show that the flow height for both resins will be in excess of that allowed by the test.

CONCLUSIONS

The work presented in this paper assessed the flow performance for a range of resins and further substantiated a model developed predict the resin flow in a RFI process. Using the results generated from testing and analysis, the following conclusions can be made:

- The resin flow model can be successfully used to predict the flow performance of a resin system in the RFI process.
- DVP tests can be used to determine flow factors which can be used quantitatively to assess the flow characteristics of a resin system.
- The heat-up rate had little influence on the flow behaviour of the M18 resin.
- Resin ageing at room temperature has a detrimental affect on the flow behaviour of M18. Subsequently, care should be taken to minimise the exposure time of the resin to room temperature. Similarly, excessive staging of a resin such as RTM6 will also severely reduce the flow performance.

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