PROCESSING CONDITIONS AND VOIDS IN OUT OF AUTOCLAVE PREPREGS

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ABSTRACT

Processing conditions and process parameters such as moisture content, debulk time and part length all play a major role in the ability to achieve void free out-of-autoclave (OOA) prepreg laminates. In autoclave prepreg processing, low porosity is readily achieved through the application of external pressure, which compresses the laminate and promotes trapped air and other volatiles to remain in solution in the resin. To achieve low-cost processing, OOA processes dispense with the autoclave, and forgo the benefit of external pressure. Because of this, the success of OOA prepreg processing is dependent on other processing parameters for effective removal of gas and prevention of void formation during cure. This paper presents the results of a series of experiments that attempt to determine the relationship between various process conditions, process parameters and the resulting void content of cured laminates. Parameters considered include part length, the amount of moisture present in the laminate, and the time the part is exposed to vacuum prior to cure. Laminates of differing lengths were exposed to environments of varying humidity and then exposed to vacuum for different times before curing. Porosity in the final parts was determined by microscopy and subsequent image analysis. A distinction is made between voids within the fibre tows and voids within the resin. The study showed a strong influence of time under vacuum, moisture content and part length on resulting porosity. Gas transport through the laminate out to the vacuum system was manifested by in-plane porosity gradients in the laminates.

1. INTRODUCTION

Out-of-autoclave manufacturing processes have the potential to substantially reduce the cost of composite manufacturing; however, they are not currently able to provide the same part quality as autoclave methods. This is primarily due to the formation of voids in OOA prepregs, which are suppressed by the application of pressure in autoclave processes. Void formation occurs during a complex process involving multi-phase materials with time-dependent material properties. Modeling the void formation phenomenon from first principles has typically not been feasible, and so the problem currently lacks a robust scientific basis [1].

This paper discusses a series of experiments designed to investigate the relationship between various process parameters and void formation. Parameters studied include exposure to humidity, vacuum pressure level, part dimensions, and debulk time.

1.1 Objectives

The objective of this research is to establish an understanding of the physical processes relevant to void formation. The experiments discussed here will be used to direct the development of models which could be used to predict the porosity of a part given its processing parameters and conditions. For a given part, this would allow the prediction of the necessary processing conditions to produce a part with acceptable porosity.

The experiments discussed here represent an attempt to establish relationships between porosity and the multidimensional process parameter space. Qualitative relationships between these parameters and part porosity can be seen in the data.

2. EXPERIMENTATION

2.1 Methods

In the experiments conducted, composite panels were produced and their void content was quantified. A variety of process conditions and parameters were used so that their correlation with the porosity of the final part could be determined. The material used in all experiments was ACG 5 harness satin carbon-fibre/epoxy prepreg designated MTM45-1/CF2426A [2]. Although the in-plane dimensions were varied, all experiments used laminates with four plies of this material. The process parameters and conditions varied were part size, vacuum pressure, time under vacuum (debulk time) and pre-preg moisture content.

2.1.1 Porosity Measurements

The porosity of laminates was determined by image analysis of micrographs taken of laminate cross-sections. Images were taken using a Nikon Epiphot 300 optical microscope and analyzed using ImageJ image analysis software. Voids were categorized as either *tow voids*, voids that appear inside fibre bundles, or *non-tow voids*, tows appearing between lamina or entirely within the resin [3]. Porosity due to tow voids (ϕ_{tow}) was quantified as the area of the voids in the tows as a percentage of the total area of the cross section. Porosity due to non-tow voids ($\phi_{non-tow}$) was quantified as the area of all other voids as a percentage of the total area of the cross section. The total porosity (ϕ_t) is simply the sum of these two quantities.

2.1.2 Laminate Preparation and Conditioning

To prepare pre-preg with varying moisture content, uncured pre-preg was placed in containers with a controlled humidity level and allowed adequate time to equilibrate. The containers were partially filled with a medium to control the humidity (see Table 1). The pre-preg sat on racks above this medium.

Table 1. Humidity Regulating Media

Medium	Relative Humidity*
Desiccant	0.1 %
Lab Air	30 % - 50 %
Brine	74.8 %
Water	99.9 %

*Measured using Fisher hygrometer 11-661-18

Control of vacuum pressure was achieved by placing a vacuum regulator between the vacuum bag and the vacuum pump.

2.2 Experiments Conducted

2.2.1 Effect of Humidity and Vacuum Pressure

This experiment was conducted using laminates of dimensions 64 mm by 64 mm. Four laminates at a time were conditioned, one at each of the four humidity levels, and then cured on an aluminum tool. The vacuum bagging arrangement used is shown in Figure 1. This was repeated three times, using vacuum levels of approximately 100 kPa (hard vacuum), 80 kPa, and 60 kPa. The debulk time was 30 minutes for all laminates.

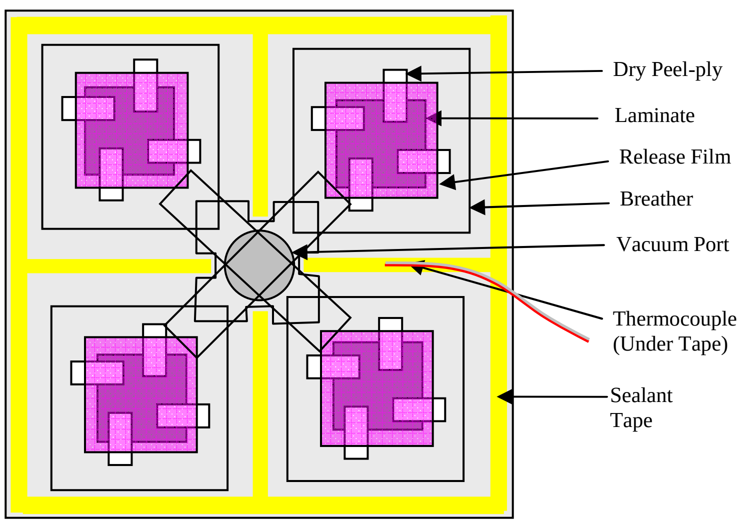

Figure 1. Vacuum bagging arrangement for 64mm laminates

2.2.2 Effect of Humidity, Part Size and Debulk Time

The second experiment used laminates of dimensions 64 mm by 300 mm and 54 mm by 1000 mm. The vacuum bagging arrangement was different from the first experiment in that three of the laminates edges were sealed, and only one edge had access to vacuum, as shown in Figure 2. The purpose of this bag arrangement is to simulate the gas transport in a larger part. By sealing three edges during debulk and cure, gas transport is reduced to an essentially 1D problem, and the distance to the nearest vacuum source is increased across most of the laminate. This was done by placing sealant tape immediately around the edges of the laminate and pressing it into the edge of the laminate. Another strip of sealant tape near the breathing end ensures that the vacuum can only access one end of the part. Debulk time was also varied in this experiment.

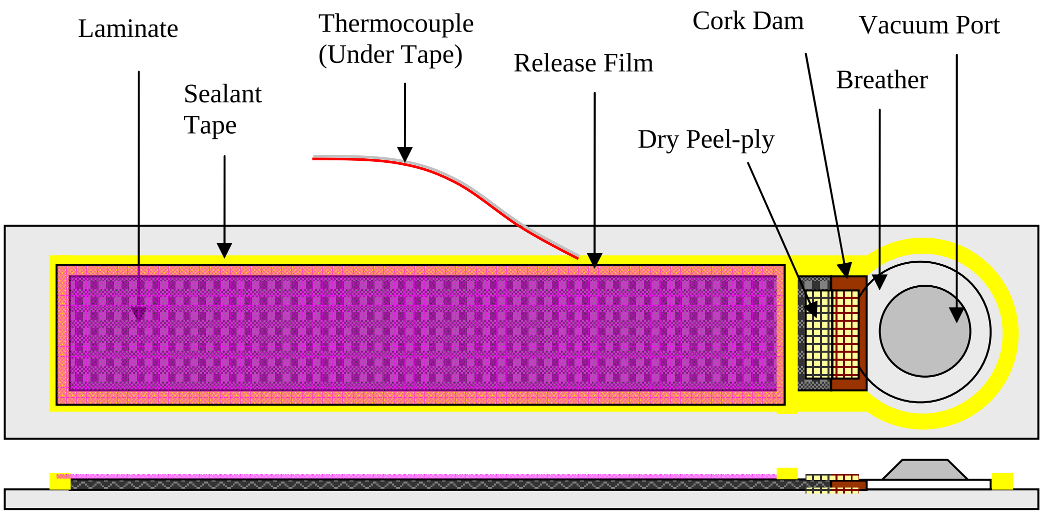

Figure 2. Vacuum bagging arrangement for 300 mm (shown) and 1000 mm laminates

3. RESULTS

3.1 Effect of Humidity and Vacuum Pressure

The twelve 64 mm square laminates were conditioned under varying relative humidity levels, and cured under varying vacuum pressure. All laminates were debulked for 0.5 hours. The laminates were subsequently sectioned and imaged and their porosity was quantified. The total porosity of the laminates is shown in Figure 3.

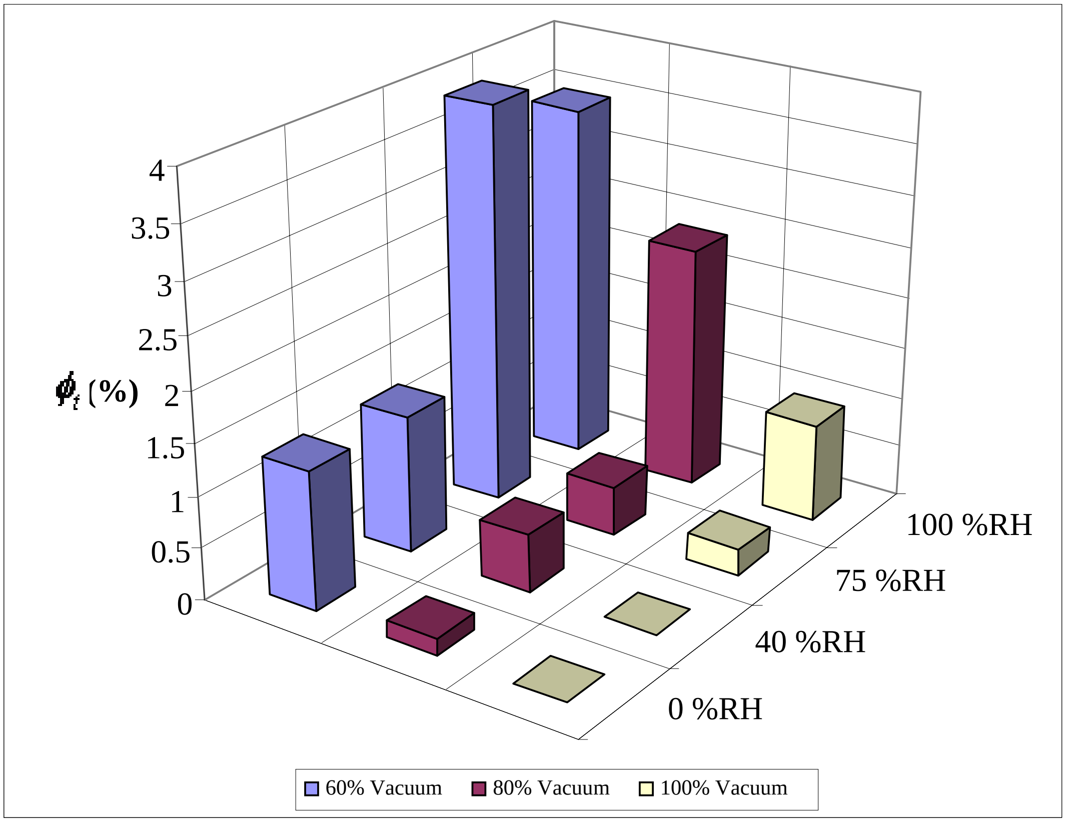

Figure 3: Total porosity of 64 mm laminates

3.2 Effect of Humidity, Part Size and Debulk Time

Four 64 mm by 300 mm laminates were allowed to equilibrate with an environment with relative humidity of either 0 % or 100 %. These laminates were then cured after debulking for either 0.5 hours or 24 hours. Full vacuum pressure was used in all cases. The laminates were then sectioned and imaged and their porosity was quantified. The total porosity of the 300 mm laminates is shown in Figure 4. This experiment was repeated using 64 mm by 1000 mm laminates. These laminates were conditioned in either a 75 % relative humidity environment or left open to the lab air, with a relative humidity of approximately 40 %. These also received either a 0.5 hour or 24 hour debulk before curing. The total porosity of the 1000 mm laminates is shown in Figure 5.

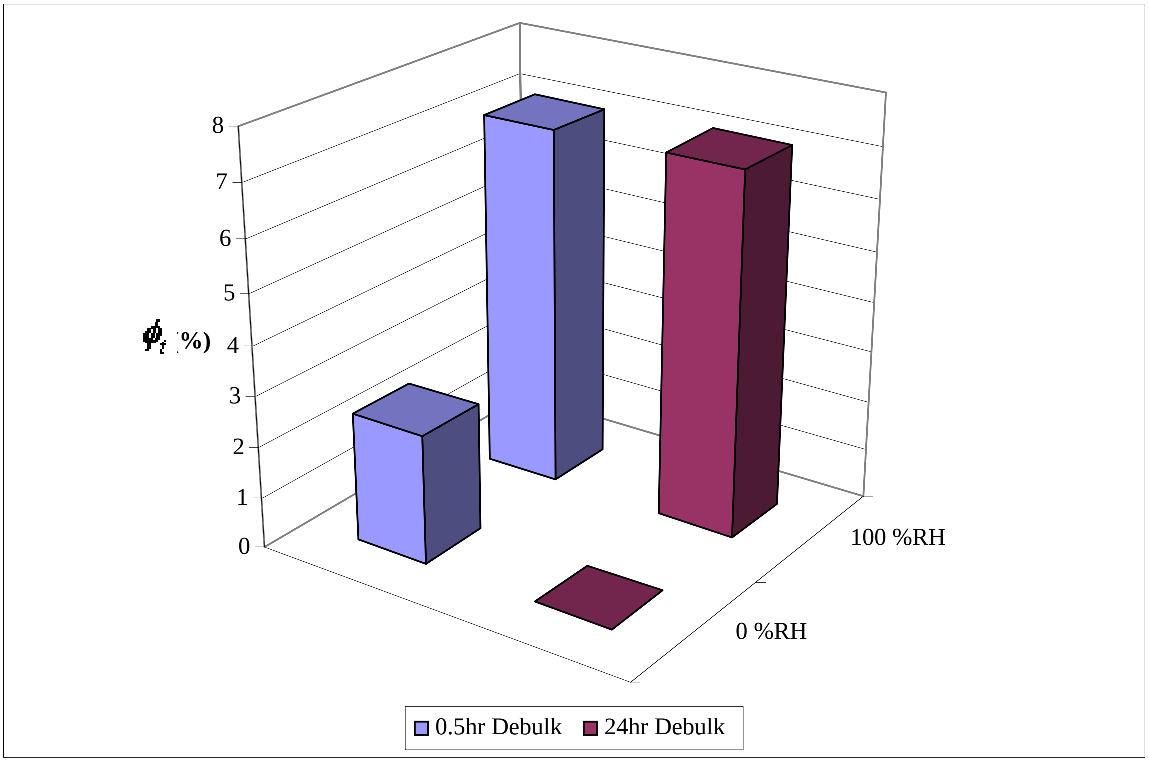

Figure 4: Total porosity of 300 mm laminates

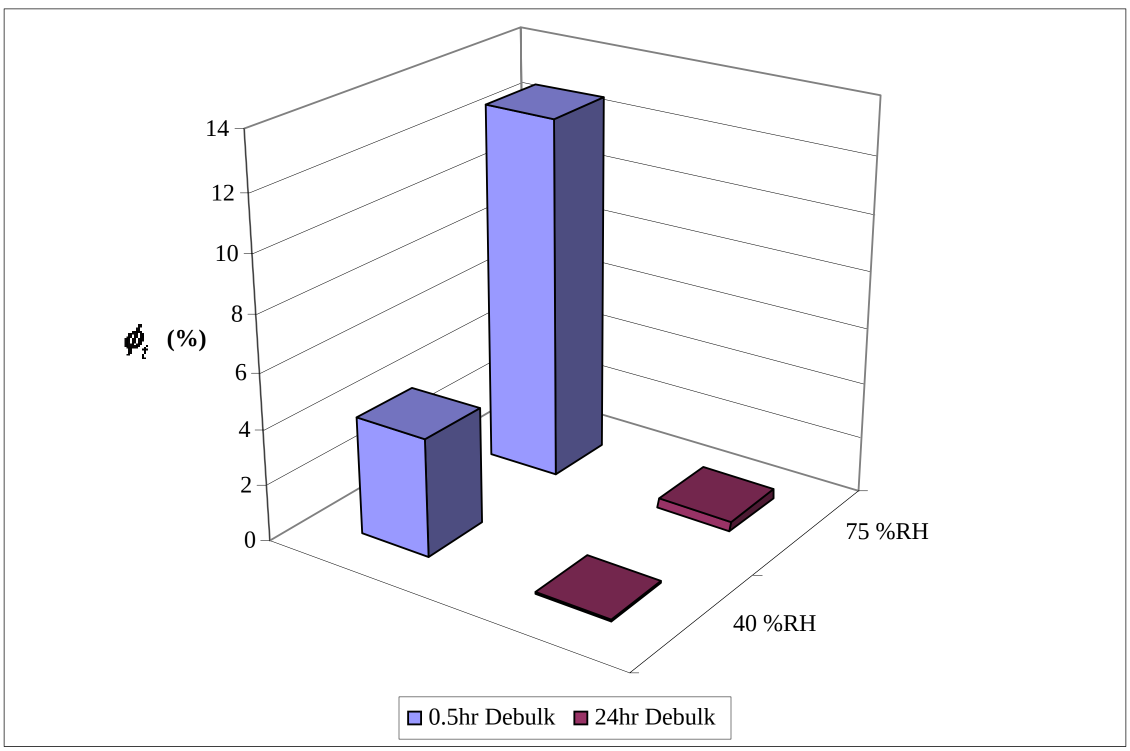

Figure 5: Total porosity of 1000 mm laminates

These laminates were sealed along three edges and could only access the vacuum source through one of their 64 mm edges. Because gas was extracted through one end only, this produced a porosity gradient in some of the cured laminates. The porosity gradients produced in the 300 mm laminates and 1000 mm laminates are shown in Figure 6 and Figure 7 respectively.

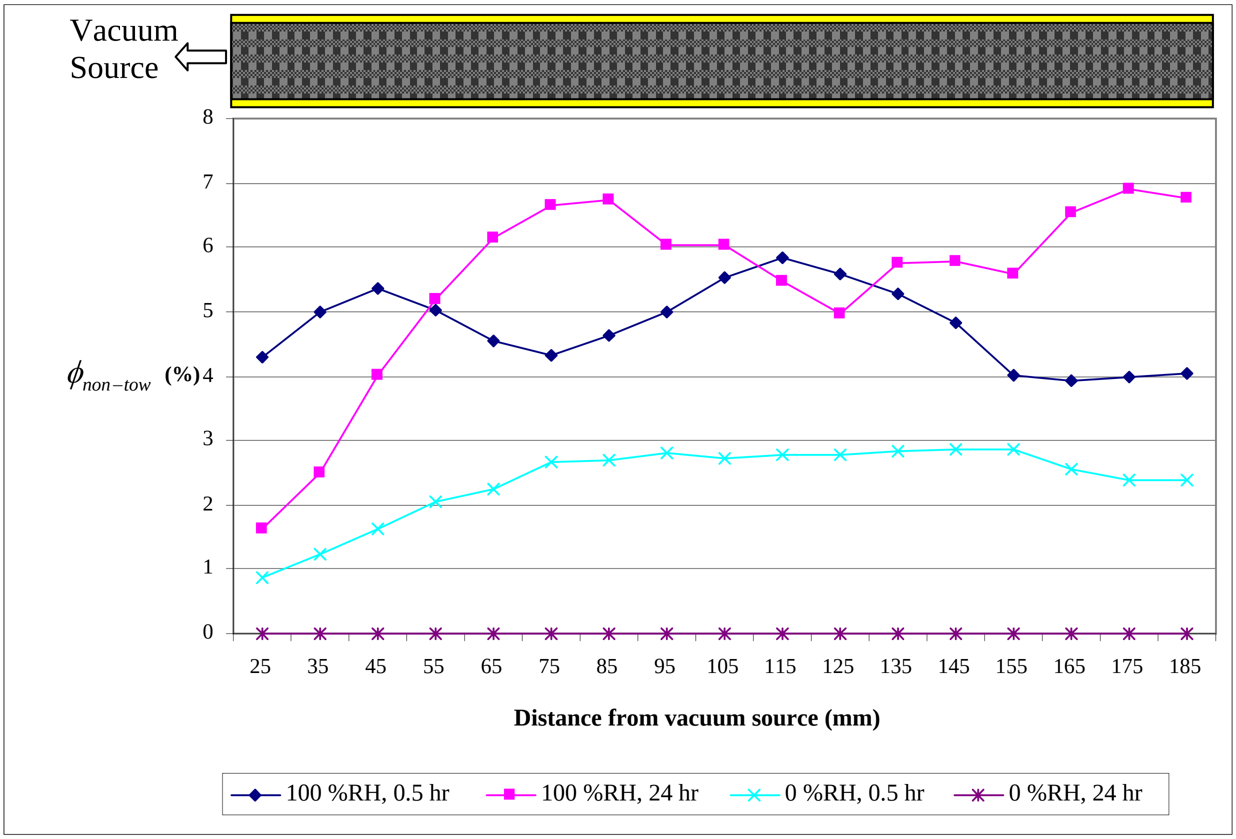

Figure 6: Porosity due to non-tow voids in 300 mm laminates (moving average, 5cm intervals)

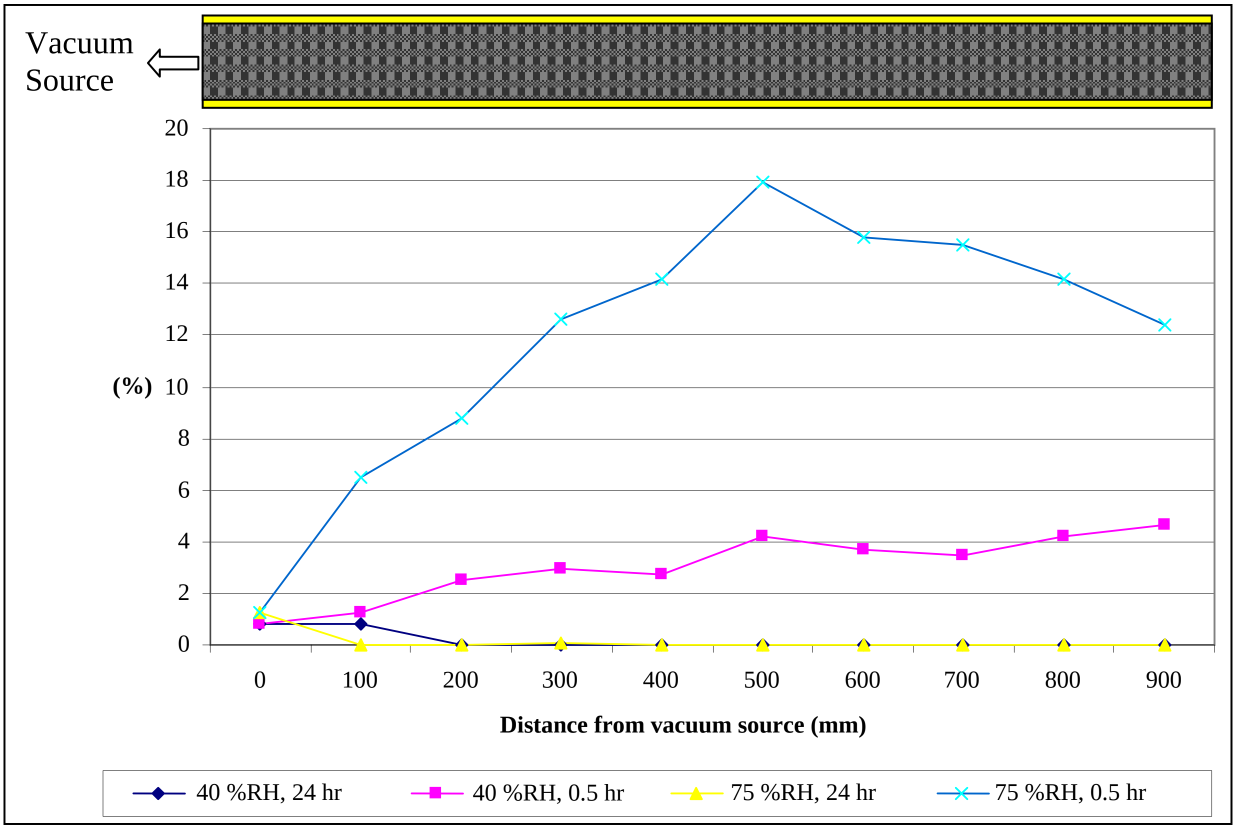

Figure 7: Porosity due to non-tow voids in 1000 mm laminates

4. DISCUSSION

These experiments provide insight into the relationships between porosity and process variables. In general, the relationships shown in Figure 8 hold across the range of the parameters used in the experiments. Larger part size and exposure to higher humidity will produce a part with greater porosity, while higher vacuum levels and longer debulk times will produce a part with lower porosity.

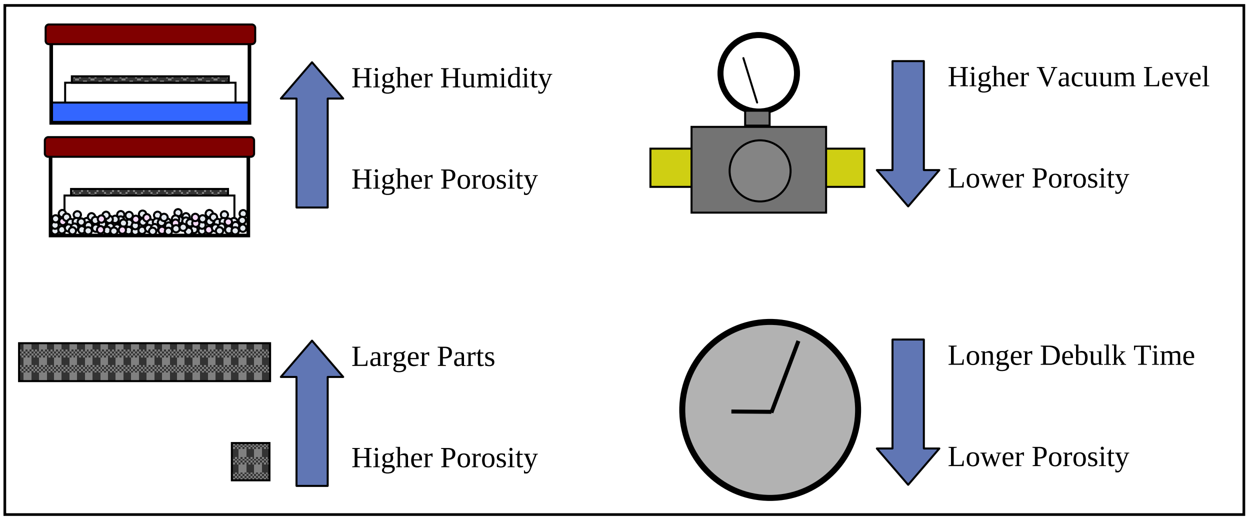

Figure 8: Qualitative relationships between processing parameters and porosity

4.1 Observations

Several interesting features of the data can also be identified. For example, from the experiments with 64 mm square laminates it can be seen that acceptable laminates can be made under very poor processing conditions if the laminates are very small. This observation can be explained by the fact that the time scales of the main gas transport processes involved in the void evolution process, such as Darcy flow, scale quadratically with part length [4,7].

An estimate of the time scales involved can be obtained in the following way. The velocity *v* of a gas molecule moving in one-dimensional Darcy flow is given by

$$v = \frac{K}{\mu}\frac{dp}{dx} \quad [1]$$

where K= gas permeability of the prepreg [m^2], μ =dynamic viscosity [Pa s] and p = pressure [Pa]. Under full vacuum pressure, the pressure gradient between the centre of the laminate and the edge exposed to vacuum can be approximated by

$$\frac{dp}{dx} \approx \frac{100000 Pa}{L} \quad [2]$$

where L is the maximum distance from a point in the laminate to the vacuum source. Typical values of K for this prepreg and μ at ambient conditions are $K = 3 \cdot 10^{-14} m^2$ and $\mu = 2 \cdot 10^{-5} Pa \cdot s$ [5]. This gives a first approximation for the velocity with which a gas molecule moves toward the vacuum source, and therefore allows the time needed to remove a gas molecule from the center of the laminate to be estimated (See Table 2).

Table 2. Estimation of Gas Transport Time Scales

Laminate	**L (mm)**	**v (mm/s)**	**Time to Remove (h:m:s)**
64 mm	32 mm*	5	0:0:07
300 mm	300 mm	0.5	0:10:00
1000 mm	1000 mm	0.15	1:51:00

**Laminate vented on all four sides.*

There are two complicating factors when relating gas transport to porosity. The first one is that as gas is removed from the laminate, the pressure inside the laminate decreases and lowers the velocity of the gas flow. As more and more gas is removed, the process will become slower and slower [4,7]. The second factor is that the model does not account for gas generated inside the laminate, which also will alter the internal gas pressure.

In laminates where moisture has been absorbed into the resin, additional gas will be entering the vacuum channels by desorption from the resin, which dramatically increases the amount of gas that the vacuum system needs to remove. This can be seen in the experimental data presented above. A 24 hour debulk was sufficient to produce a part with very low porosity in all cases except for when the part was exposed to an environment of 100% relative humidity. This supports the theory, suggested by [6], that dissolved moisture is a significant source of voids in OOA prepregs with high moisture content, with trapped air and volatiles making a small or negligible contribution to void formation.

A result due to [4,7] is the following relationship, derived from a numerical solution to a one-dimensional Darcy's law with a gas obeying the ideal gas law, which relates the number of moles of gas remaining in a laminate to nondimensionalized time.

$$\frac{n}{n_0} = \frac{1}{1+1.5\tau} \qquad [3]$$

Here nondimensionalized time τ is defined as

$$\tau = t\frac{Kp_0}{\phi_0 \mu L^2} \qquad [4]$$

where t, p_0 and ϕ_0 are time, pressure and total initial porosity. The simplified one-dimensional model assumes that the gas channels have a constant volume and therefore constant permeability during gas evacuation. However, when the resin is heated up and becomes liquid after debulk, we assume that the gas channels will reduce in size until the internal pressure inside the now closed-off gas channels equals the external pressure of one atmosphere. These assumptions lead to

$$\frac{\phi}{\phi_0} \approx \frac{n}{n_0} = \frac{1}{1+1.5\tau} \qquad [5]$$

For each experimental data point, the nondimensionlized time and nondimensionalized porosity was calculated and compared to the predictions by eq. (5), see Figure 9.

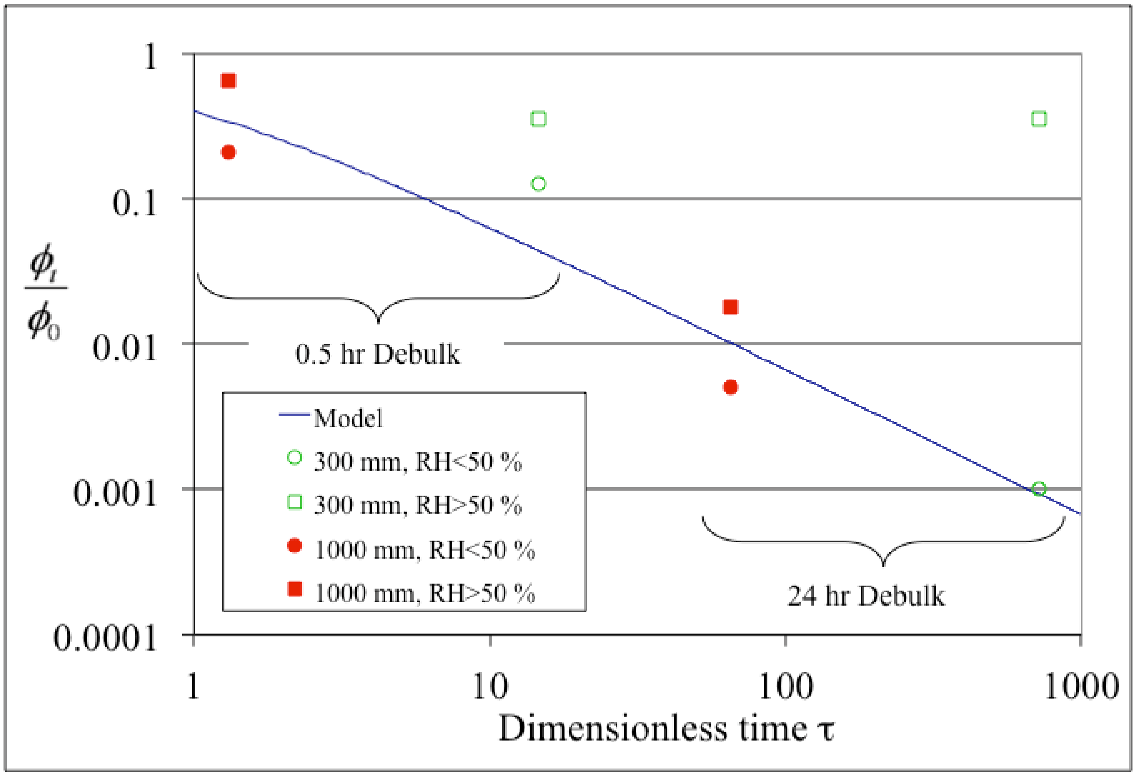

Figure 9: Comparison of Gas Transport Model with Experiments.

The relatively dry laminates (exposed to <50 %RH, shown as circles), agree fairly well with the model, while relatively wet laminates (exposed to >50 %RH, shown as squares) all have much greater porosity than predicted by the simple model. Models that also account for the transport of moisture within the resin are required to predict porosity in parts with high humidity.

It is interesting to note that the shape of the porosity gradients in the 300 mm and 1000 mm parts qualitatively resemble typical solutions to the relevant transport phenomena equations [4,7]. The porosity at the edge of the part exposed to the vacuum source was very low, with the porosity increasing with distance into the part before reaching a plateau. This supports the idea that removal of gas by the vacuum system via in-plane gas transport mechanisms is an important factor in the process of void evolution.

5. CONCLUSION

The evolution of voids in out-of-autoclave laminates is a complex process, which depends on many factors. The experiments presented provide some insight of how porosity depends on four of these factors: part size, exposure to humidity, vacuum level and debulk time. These experiments begin to provide a better understanding of this complex process.

The results of these experiments compares well with the predictions of a simple model within the range of parameters it is expected to. These results will be used to guide the development of more inclusive models of the physical mechanisms involved in the void evolution process, and to suggest strategies for void mitigation. The long-term goal is to develop a quantitative understanding of the relationship between porosity and the relevant process parameters.

6. ACKNOWLEDGEMENTS

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