EXPERIMENTAL OBSERVATION AND ANALYTICAL MODELLING OF THE RESIN FLOW INSIDE AN OUT-OF-AUTOCLAVE PREPREG

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ABSTRACT: Out-of-autoclave prepregs allow gas removal through dry fibre areas that are infiltrated during cure. The microstructural behavior of such a material was investigated for a chosen processing cycle. First, flat laminates partially processed to different stages were scanned using x-ray micro-tomography to obtain microstructural information at each state and quantify the progressive impregnation. The observed behaviour was then modelled analytically, resulting in the ability to predict it for any cure cycle. The research established a methodology for visualizing a prepreg's evolving microstructure, obtained an understanding of this evolution of an out-of-autoclave prepreg, and developed a model for predicting it for any given cure cycle.

KEYWORDS: prepreg, out of autoclave, microstructure, flow, modeling, micro-ct

INTRODUCTION

A new generation of out-of-autoclave prepregs seeks to deliver performance comparable to autoclaved parts while reducing costs and increasing versatility [1-2]. During vacuum bag processing, the resin pressure available to suppress voids is significantly smaller than in an autoclave, and evacuating entrapped gases becomes critical. To facilitate this, out-of-autoclave prepregs feature initial structures consisting of both impregnated and dry regions; the latter act as air escape paths during the early stages of cure, before being infiltrated by resin [1]. Investigating this initial structure and its evolution with cure is a natural first step in understanding and optimizing the processing of out-of-autoclave prepregs.

The consolidation of fibres impregnated with resin has been previously considered [3-4] and recent research has resulted in generalized models that can address partially impregnated prepregs [5]. Infiltration flows during cure are also at the core of research on liquid molding [6-7]. However, for out-of-autoclave prepregs, there are few experiments on impregnation [8], and there is a need for data and modelling that clarify the key physical phenomena and lead to processing guidelines.

In the present work, flat laminates were processed to different stages of a cure cycle and scanned using x-ray microtomography (micro-ct) to track the microstructural evolution. Then, the experimental data was used as the basis for a model that can predict the impregnation behaviour for any cure temperature cycle.

EXPERIMENTS

Materials and Processing

The out-of-autoclave prepreg was manufactured by the Advanced Composites Group. It consists of 6K carbon fibre tows woven in 5 harness satin fabric and partially impregnated with a 36% by weight MTM45-1 epoxy resin matrix, resulting in an areal weight of 375 g/m^2 [9]. Ten symmetric laminates (Table 1) nominally 10 cm by 10 cm were placed on a flat aluminum tool plate on non-perforated release film. All laminates except 8 and 10 had four plies; the latter had eight due to early trial variations. The number of layers was not expected or found to affect any results. Edge breathing dams made of sealant tape wrapped in fibreglass cloth were placed around the laminate perimeter to allow in-plane air evacuation. A non-perforated release film was placed on top of the laminate to prevent resin bleed, followed by two layers of breather and the vacuum bag. The oven process cycle chosen for characterization consisted of a 60 minute vacuum hold at 25°C, a 2°C/min (±0.5°C/min) ramp to 85°C and a hold at 85°C up to 180 minutes. The bag temperature was recorded with a thermocouple. Laminates were partially processed to different points in the cycle, as shown in Table 1. Once each laminate reached its desired processing stage, it was quickly removed from the oven and placed in a freezer to prevent additional flow.

Table 1 Partially processed laminates and their total processing time.

Laminate	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time [min]	0	15	30	45	60	80	93	104	110	180

X-Ray Microtomography

Two samples (A,B) nominally 19 mm by 19 mm were cut from the center of each laminate and scanned using a Skyscan 1172 micro-ct with no filter at a resolution of 7 µm/pixel, an image size of 4000 x 2096 pixels, a voltage of 64 kV and an intensity of 152 µA. These settings were a compromise between resolvable detail and sample size.

Representative x-ray micrographs are shown in Fig. 1. Laminate 1, Sample A (0 min) shows partially impregnated fibre tows. Laminate 7, Sample B (93 min) and Laminate 9, Sample A (110 min) indicate that tows are progressively impregnated during processing. The tows become invisible because at the chosen resolution, each pixel may contain a portion of a single fibre (diameter of 7 µm, density of 1.7 g/cm^3) and some surrounding resin (density of 1.2 g/cm^3); their comparable responses become "smeared" within the pixel in the final image. The unprocessed samples featured large interply gaps and the largest visible tow area. Both decreased as soon as vacuum was applied. During the room temperature vacuum hold (0 – 60 min), the visible tow area exhibited no consistent decreasing trend. Tow impregnation began once the temperature rose past approximately 60°C (86 min) and continued during the 85°C hold, until 120 min. The extent of impregnation was quantified by choosing a representative micrograph near the center of each sample (to avoid edge effects from sample preparation), measuring the visible areas of eight tows by fitting them to ellipses (see Fig. 1), and averaging these values. Results are shown in Fig. 2 along with cure cycle temperature. Within each sample there was significant variability between the impregnation of different tows (as in [8]) but averaged values were consistent between two samples of the same laminate.

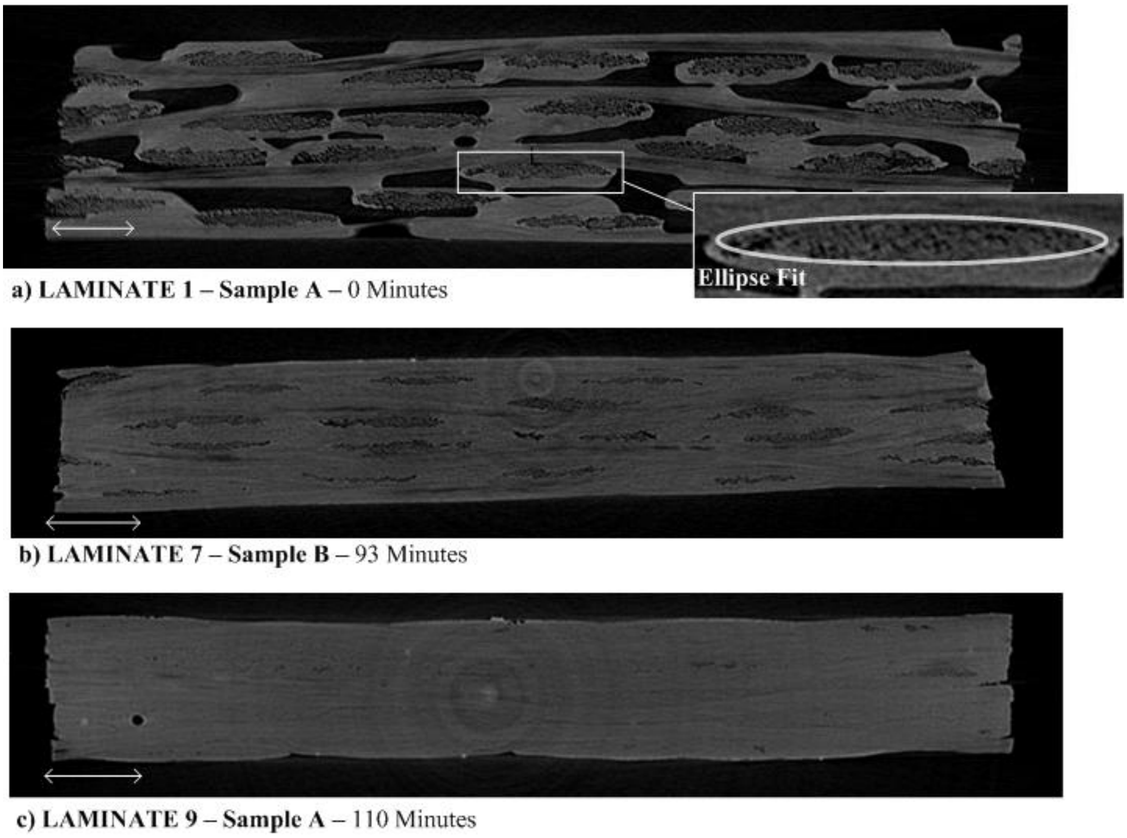

Fig. 1 X-ray micrographs of microstructural evolution (adjusted for visibility).

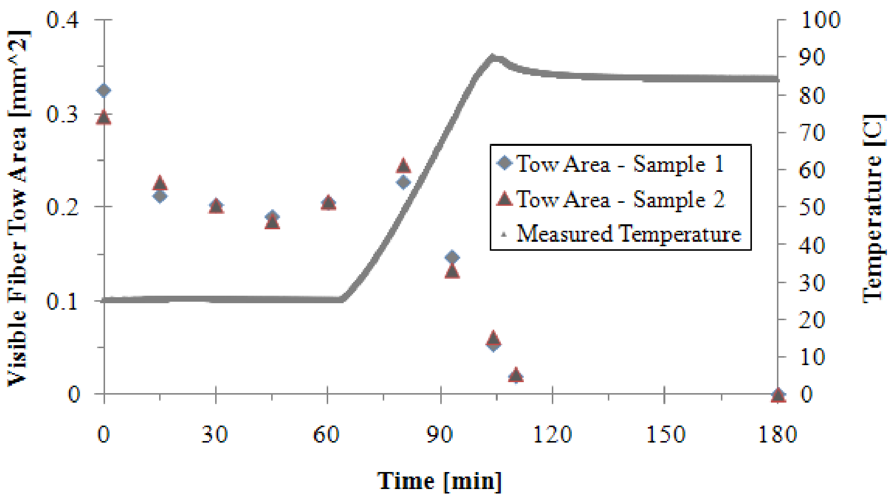

Fig. 2 Visible fibre tow area and cure cycle temperature with processing time.

MODELLING

The micro-ct data describes impregnation for a single cure cycle. In order to better understand the material's processing behaviour, a model was developed to simulate tow impregnation for any cure cycle. Consider a circular fibre tow of total radius R_{tow} infiltrated in the region between R_{tow} and the resin front R_f ($R_f < R_{tow}$), as shown in Fig. 3 a). Assuming axisymmetry and a rigid porous medium, Eqn. 1 for the resin infiltration speed v_f can be obtained from continuity and Darcy's Law similarly to [7].

$$v_f = \frac{dR_f}{dt} = -\frac{K}{\mu(1-V_f)}\left[\frac{1}{R_f}\frac{(P_\infty - P_f)}{\ln(R_{tow}/R_f)}\right] \quad (1)$$

$$\begin{aligned} P_f &= P_{f,0} \; for \; R_f > \alpha R_{tow} \\ &= P_{f,0}\,(\alpha R_{tow})^2/\left(R_f\right)^2 \; for \; R_f < \alpha R_{tow} \end{aligned} \quad (2)$$

P_f is the resin flow front pressure boundary condition due to entrapped air at an initial pressure $P_{f,0}$ being compressed once $R_f < \alpha R_{tow}$ ($\alpha \ll 1$), as in Eqn. 2. Eqns. 1 and 2 can be solved over small time steps for any given cure cycle, provided all parameters remain constant within the step. The model was fit to the micro-ct tow impregnation data. Table 2 provides the constant parameters and their source; the flow-driving pressure P_∞ was used as the fitting constant while the other parameters were obtained from sample inspection, literature or estimates. The resin viscosity at any point in the cure cycle was predicted using a model developed in [10].

Table 2 Fibre tow impregnation model: constant parameters.

Parameter		**Value**	**Source**
Tow Radius	R_{tow}	2.6E-4 m	Initial visible fibre tow area = πR_{tow}^2
Tow Volume Fraction	V_f	0.643	Micrographs of cured laminates
Permeability	K	4.52E-14 m^2	V_f and lubrication model from [7]
Pressure at $r = R_{tow}$	P_∞	10 900 Pa	Fitting constant
Pressure at $r = R_f$	$P_{f,0}$	5065 Pa	Estimated as 5% of atmospheric pressure
Entrapment Factor	α	0.015	Estimated as 1.5% of initial tow size

The model and experimental data are plotted in Fig. 3 b) for the elevated temperature partial processing period (60 – 180 minutes). There is good agreement between experiments and modelling once infiltration starts, but some discrepancy exists in the early stages. The difference is likely explained by resin flowing into interply gaps and large voids before infiltrating the tows, as observed in the x-ray micrographs and in [8].

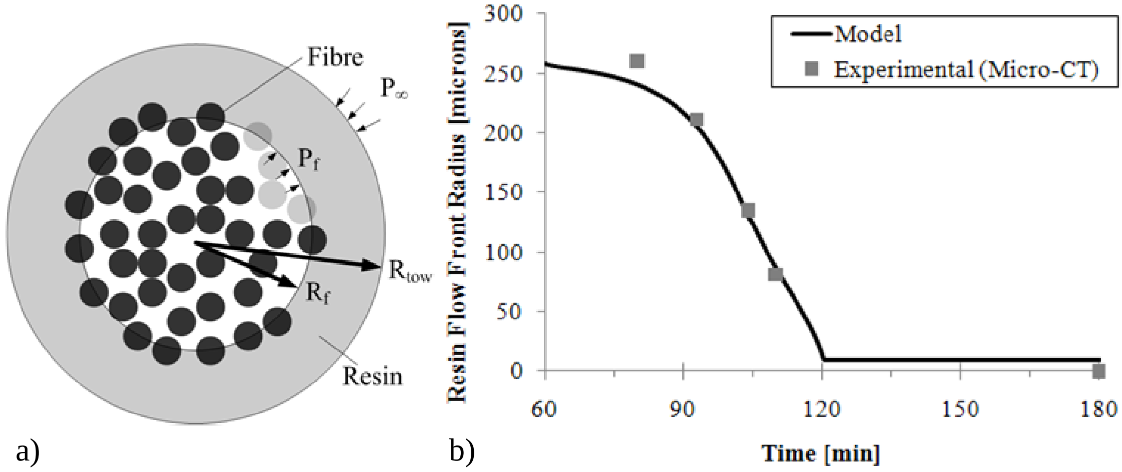

Fig. 3 a) Schematic of fibre tow approximation; b) graph of resin flow front radius versus time from experiments and modeling.

CONCLUSIONS

The present work used experiments and modelling to understand the resin flow behaviour within an out-of-autoclave prepreg. Partially processed laminates were analyzed using a novel x-ray microtomography method to visualize and quantify the extent of impregnation of dry areas. Then, a model was developed and fit to the measured data, to simulate the transverse impregnation of dry fibre tows for any cure cycle. The research demonstrated a new methodology for investigating prepreg microstructures, applied it to an out-of-autoclave prepreg, and developed a model to predict tow impregnation for any cure cycle.

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