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Effective cure cycle development via flow optimization and advanced cure environments

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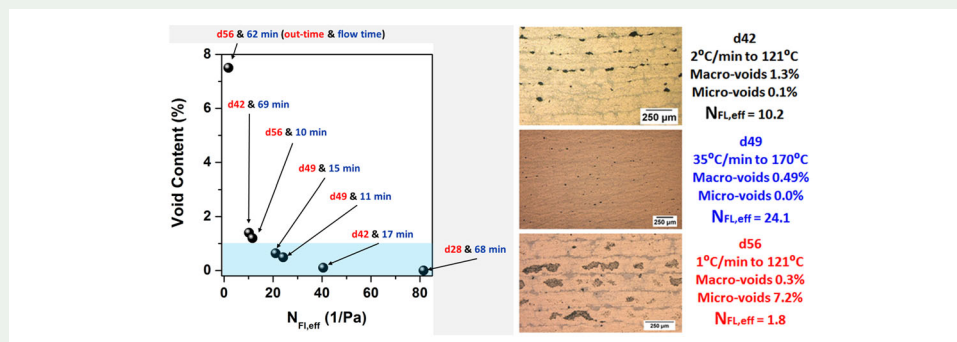
ABSTRACT

Out-time and moisture absorption in prepreps are generally unavoidable in an industrial setting, where prepreg layup can take weeks. The resin cross-linking and viscosity increase that occurs during out-time can reduce resin flow during a specified cure cycle, causing porosity. In this study, a flow-optimized cure cycle was developed, leveraging both flow level and time during out-of-autoclave (OoA) processing. First, predictive cure kinetics and viscosity models were used to model viscosity evolution under selected cure conditions, accounting for initial out-times and humidity conditioning. To quantify resin flow, an 'effective flow number' metric was defined as the integration of inverse viscosity evolution until the resin gelation point. The method described revealed that a rapid heating rate achieved by use of advanced tooling was essential to achieve a high effective flow number. The experimental results showed that the effective flow number is a useful criterion to limit flow-induced defects. The method presented also extends the boundary (by 175%) of the manufacturer's specified out-life for OoA prepreg materials.

KEYWORDS

Prepreg; out-time; out-of-autoclave (OoA); rheology; viscosity

GRAPHICAL ABSTRACT



1. Introduction

In recent decades, out-of-autoclave (OoA) prepreg technology has experienced increasing acceptance as a cost-effective and high-throughput alternative to conventional autoclave cure [1–3]. OoA prepreps are cured using vacuum-bag-only (VBO) processing, where porosity is suppressed by evacuation of entrapped gas (air and volatiles) through dry-fiber regions in a partially impregnated micro-structure. Initially, the dry regions form a permeable vascular network of vacuum channels that allow in-plane gas transfer. During processing, these channels are infiltrated by surrounding resin to form a uniform and, in principle, void-free microstructure. The rate of infiltration and the quality of OoA laminates are

thus strongly influenced by the cure kinetics and viscosity evolution of the infiltrating resin.

During pre-cure operations, the resin state can be affected by environmental factors such as temperature and ambient humidity. Exposure to room temperature conditions for extended periods (or out-time) can advance the degree of cure and increase the viscosity of the resin, factors that may prevent full infiltration of the fiber bed during cure [4–9]. Out-time is particularly relevant to OoA prepreps, which are designed for large, integrated structures that often require weeks to lay up prior to cure. Exposure of OoA prepreg to ambient humidity leads to absorption of moisture, and saturation is reached within days [6]. Absorbed moisture has

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been shown to affect both degree of cure and viscosity by accelerating the resin cross-linking reaction, acting as both catalyst and solvent for the amine-epoxy reaction [10,11].

During cure, the resin degree-of-cure increases as polymerization/cross-linking takes place, leading to changes in thermomechanical properties and influencing the consolidation of the part. To account for this, we have developed accurate process models that comprehensively capture out-time and humidity effects on cure kinetics and viscosity in process conditions in our previous study [4–6]. Given adequate vacuum bagging and vacuum level (948.2 mbar), the viscosity profile most directly affects the lamination process. The viscosity profile is controlled by resin chemistry, prepreg pre-conditions (i.e. out-time and humidity conditioning), and the cure profile. In other words, with given pre-conditions and resin chemistry, the flow window can be modified *via* tailoring of the cure profile.

The limit to which a high-quality part can be manufactured using OoA prepreg is typically defined by the manufacturer's recommended cure cycle (MRCC) and the associated out-life [1]. This is largely due to inhibited resin flow with the advancement of resin cure. Thus, there is a need to create an adaptive cure cycle method that can enhance resin flow and thereby extend the boundaries of prepreg out-life.

The specific objectives of this study were to

1. Develop a quantifiable metric that accounts for both resin flow level and time to control, and characterize resin flow phenomena during cure.
2. Demonstrate that the metric can be accurately simulated and calculated with the previously developed predictive process models.
3. Identify a key variable that indicates the maximum resin flow, and develop a technique to optimize the resin flow.
4. Utilize the method to demonstrate the validity of a flow-optimized cure cycle and to demonstrate that out-life can be reliably extended.

2. Experimental procedure

2.1. Materials and sample conditioning

For this study, we selected a commercial OoA prepreg consisting of a unidirectional carbon fiber tape and a toughened epoxy resin (CYCOM® 5320-1/IM7, Cytec Industries Inc.). The unidirectional fiber bed had an areal weight of 145 g/m². The prepreg resin content was 33% by weight, and the manufacturer's specified out-life was 28 days [12]. All plies were cut to 80 × 80 mm and were subsequently laminated in [0°]₁₂ lay-up sequence with no vacuum

debulk in prior to the aging step. Such condition was chosen to evaluate the out-time effect on intra-ply and inter-ply breathing capability of the prepreg, isolating the tack effect.

All samples were stored in a humidity chamber containing a saturated salt solution, allowing accurate control of equilibrium vapor pressure [13]. Samples were conditioned at relative humidity levels of 60% for 0 to 56 days and tested within 7 days. For laminate processing, each cycle included an initial 12-hour vacuum hold at room temperature to ensure that air was adequately extracted from the laminate. All laminates were cured well beyond gelation (viscosity (η) = 10 kPa·s [14] or at $G' = G''$ [15]) to ensure that complete resin flow was attained from all cure cycles evaluated here.

2.2. Heated tool

A heated tool was designed and assembled in-house to enable heating rates up to 35 °C/min and accurate temperature control between 30 °C and 200 °C. The tool consisted of six independent cells in a 100 × 100 mm matrix. Each cell consisted of a heat sink, four 300 W cartridge heaters, and a cooling fan. Two thermocouples were also embedded under a flat aluminum tool plate connecting all six cells for temperature measurement and control. Heating and cooling of each cell was independently controlled *via* solid state relays (DigiKey DRA1-MCX240D5) by a digital acquisition and control system (National Instruments cRIO) and a virtual instrument (National Instruments LabView). Further details on the equipment can be found from Centea et al. [16].

2.3. Image analysis

The quality of cured laminates (i.e. void area % calculation) was evaluated using light microscopy on polished cross-sections. First, cured laminates were sectioned in the middle and polished on a metallographic polisher (Buehler MetaServ) to 4000 grit. A video microscope (Keyence VHX-600) at 100× and 200× magnification was used to record cross-sectional images of cured laminates. Void area % was measured and calculated using image analysis programs (Photoshop and ImageJ).

3. Model framework

The detailed protocols and results associated with cure kinetics and viscosity modeling accounting for out-time and moisture absorption effects on this prepreg (CYCOM® 5320-1, Cytec Industries, USA) have been described previously [4–6].

3.1. Cure kinetics and viscosity model

The viscosity η of curing epoxy resin is affected by two competing phenomena. Heating the resin increases molecular mobility, thereby decreasing η , but also eventually increases molecular size or degree of cure (α), thus increasing η . To capture these competing phenomena, the following models have been developed in our previous study [4–6], which were shown to capture out-time and moisture absorption effects:

$$\frac{d\alpha}{dt} = K_1 \alpha^{m_1} (1-\alpha)^{n_1} + \frac{K_2 \alpha^{m_2} (1-\alpha)^{n_2}}{1 + \exp(D(\alpha - (\alpha_{c0} + \alpha_{CT} T)))} \quad (1)$$

$$\eta = \eta_1 + \eta_2 \left(\frac{\alpha_1}{\alpha_1 - \alpha} \right)^{A+B\alpha^d+C\alpha^e} \quad (2)$$

The cure kinetic model is developed *via* DSC experiments [17,18]. The model details and parameters can be found in [6]. Note that the η profile is a function of α , dwell temperature, and heating rate. The focus of this study is first to identify the main controlling variables that affect resin impregnation in the fiber bed during cure, given the initial α (α_0) or out-time. The second goal is to use the variables to develop a cure cycle that optimizes resin flow, and thus can limit flow-induced defects.

3.2. Flow model

A squeezing flow geometry has been used in the past to quantify resin flow during lamination [19]. Here, the prepreg layup is sandwiched between stainless steel plates and placed in a load frame (Instron), where a linear variable differential transformer (LVDT) is used to measure the movement of the crossheads. While the plates are heated, a constant force is applied and controlled using the load cell. Assuming that the resin flow can be approximated as Newtonian before it gels, a characteristic flow quantity metric called flow number (N_{FL}) has been defined as follows:

$$N_{FL} = \left(\frac{\rho_p}{\rho_o} \right) \left[1 - \left(\left[\frac{16Fh_o^2}{3\pi R^4} \int_0^{t_{gel}} \eta(t)^{-1} dt \right] + 1 \right)^{-0.5} \right] \cdot 100 \quad (3)$$

where ρ_p is the resin density, ρ_o is the prepreg density, F is the lamination press force, h_o is the initial stack thickness, t_{gel} is the time to resin gelation, and R is the effective radius of the resin. From the equation, integration of inverse η evolution up to t_{gel} , the resin flow stop point, yields a quantifiable metric to characterize resin flow. This metric is defined here as the ‘effective flow number’ or $N_{FL,eff}$:

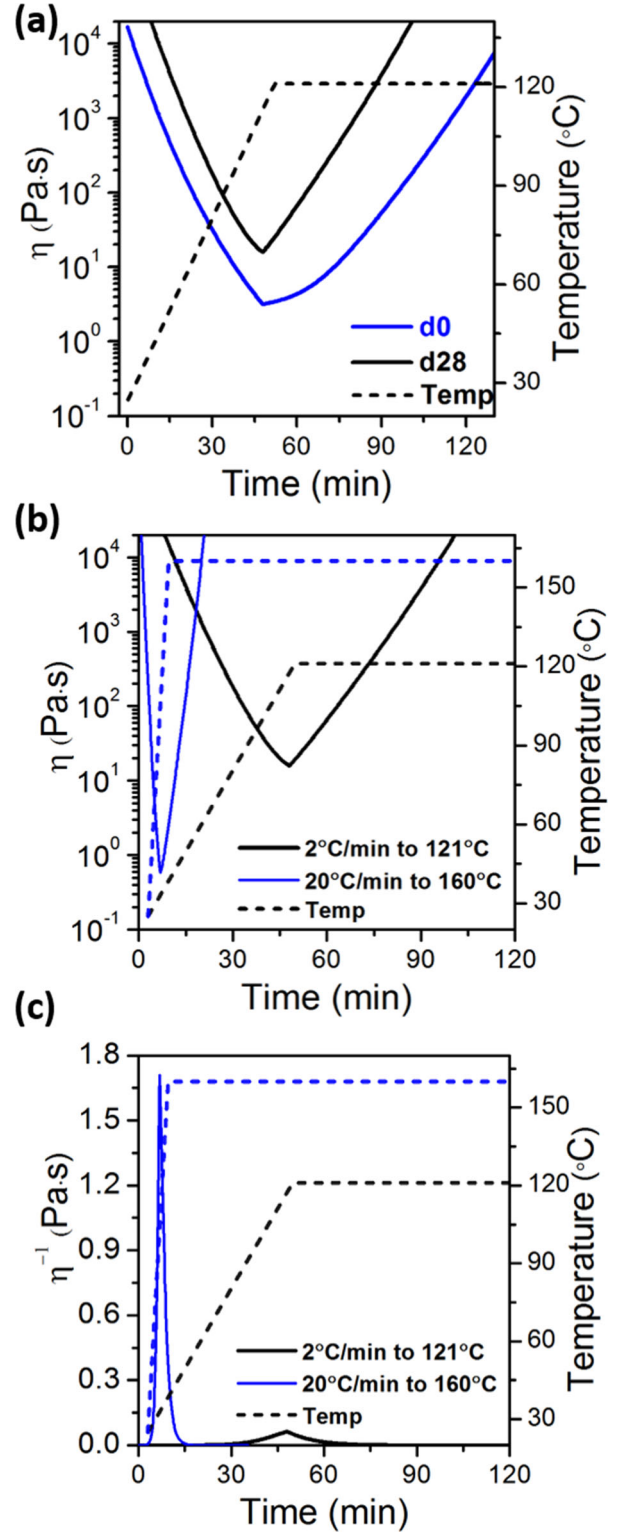


Figure 1. (a) Representative viscosity (η) model prediction for day 0 and day 28 samples cured under 2°C/min to 121°C dwell; and Model prediction of (b) η and (c) η^{-1} evolution for day 28 sample.

$$N_{FL,eff} = \int_0^{t_{gel}} \eta(t)^{-1} dt \quad (4)$$

Thus, greater $N_{FL,eff}$ means more resin flow. Using the predictive model for η developed previously [6], there are two main variables that affect η evolution or resin flow upon laminate cure: heating rate and dwell temperature. The η evolution during

Table 1. Test parameters and results.

Out-time (days)	α_0 (–)	$T_{g,0}$ (°C)	Ramp rate (°C/min)	Dwell temperature (°C)	Flow time (min)	$N_{Fl,eff}$ (1/Pa)	Micro-voids (%)	Macro-voids (%)
0	0	–0.7	2	121	130	542.1	0	0
28	0.12	12.9	2	121	68	81.5	0	0
28	0.12	12.9	20	160	19	281.8	0	0
42	0.21	23.4	2	121	69	10.2	0.1	1.3
42	0.21	23.4	20	160	17	40.5	0	0.3
49	0.27	29.6	25	160	15	21.1	0	0.64
49	0.27	29.6	35	170	11	24.1	0	0.49
56	0.32	36.5	1	121	62	1.8	7.2	0.3
56	0.32	36.5	35	170	10	11.6	0	1.2

cure depends primarily on cure temperature and α , where an increase in temperature leads to a decrease in η . Simultaneously, an increase in temperature also accelerates cure, which increases α . Additionally, a more rapid heating rate leads to a more rapid decrease in η , allowing the resin to reach lower η before the substantial effect of cure kicks in. However, once the resin reaches the cure onset temperature, a rapid cure effect is expected, which will then increase η , thus reducing resin flow time.

4. Results and discussion

4.1. Flow modeling

Representative curves showing η evolution (from model predictions) for heating at 2 °C/min to a dwell temperature of 121 °C are plotted in Figure 1a for samples aged for 0 and 28 (out-life) days. The curves show that initial η (η_0) increases with increasing ambient exposure, which advances resin cure. Thus, for the same cure cycle, less resin flow is expected with longer aging, as shown in the plots. The effect of increasing the ramp rate from 2 to 20 °C/min and dwell temperature from 121 to 160 °C on η evolution for the out-life sample is shown in Figure 1b. Note that increasing ramp rate and dwell temperature reduces the minimum η (η_{min}), thereby allowing more ‘low- η flow’. Conversely, a slower ramp rate combined with a lower dwell temperature causes a greater degree of cure in the course of the temperature ramp step, leading to a decrease in ‘low- η flow’. Thus, in this context, higher ramp rates and dwell temperatures are expected to induce more ‘low- η flow’ by attaining lower η_{min} . Such cure profiles could then increase resin flow for samples with greater out-time, where a greater initial degree of cure (α_0), or η_0 , level is expected compared to the typical MRCC.

However, an increase in ramp rate and dwell temperature both cause large reductions in flow time (defined here as the flow duration between $\eta = 10$ kPa·s to 10 kPa·s, going from the pre-cure state to the gelation point). The gelation point is known to be an iso-conversional or a cure path-independent event, where $\alpha_{gel} = 0.37$ is achieved at the gelation point for this resin (CYCOM® 5320-1).

Both $\eta = 10$ kPa·s [13] and $G' = G''$ [15] are commonly defined as gelation points for aerospace grade epoxy resins, and these have been evaluated. Both conditions resulted in equivalent gelation times (t_{gel}), with no more than 3 min difference from each other. As shown in Figure 1b, changing the cure cycle from 2 °C/min to 20 °C/min and the dwell temperature from 121 °C to 160 °C reduces the flow time by 49 min. Refer to Table 1 for flow time, $N_{Fl,eff}$ and void analysis for the out-time values and cure cycles presented here.

To evaluate the effects of both flow level, which is directly proportional to the inverse viscosity (η^{-1}), and flow time integrally on resin flow, the η^{-1} evolution for day 28 samples are plotted in Figure 1c for (a) heating at 2 °C/min to a dwell at 121 °C and (b) heating at 20 °C/min to a dwell at 160 °C. Integration of η^{-1} curves results in $N_{Fl,eff} = 63.6$ and 281.8 Pa^{–1}, respectively. As the resin cures and progresses toward gelation, a change in resin flow characteristics from Newtonian flow to shear flow will occur [20]. However, the post-gelation portion of the η^{-1} curve integral contributes less than 1% of the total $N_{Fl,eff}$ (i.e. η of 10 kPa·s or greater corresponds to 10^{-4} Pa^{–1}·s^{–1} or less in η^{-1}). Therefore, the entire η^{-1} curve was integrated to compute $N_{Fl,eff}$. The model results predict that low- η flow (or η_{min}) attained by higher ramp rates is more effective in promoting resin flow during fiber impregnation, despite a reduced flow time compared to cure cycles with slower ramp rates.

4.2. Void content analysis

Representative micrographs of uncured laminates aged for 0 and 49 days are shown in Figures 2a and 2b, respectively. Dark regions indicate un-filled voids and lighter regions indicate a resin-filled area. Day 0 and 49 samples were cold-cured using ammonia [21]. An ammonia bath was created in which the low vapor pressure of aqueous-ammonia created an ammonia vapor atmosphere inside the container. Ammonia can readily diffuse throughout the laminate *via* air pathways and react with epoxy resins at room temperature, preventing resin flow [21]. The micrographs show similar partially impregnated

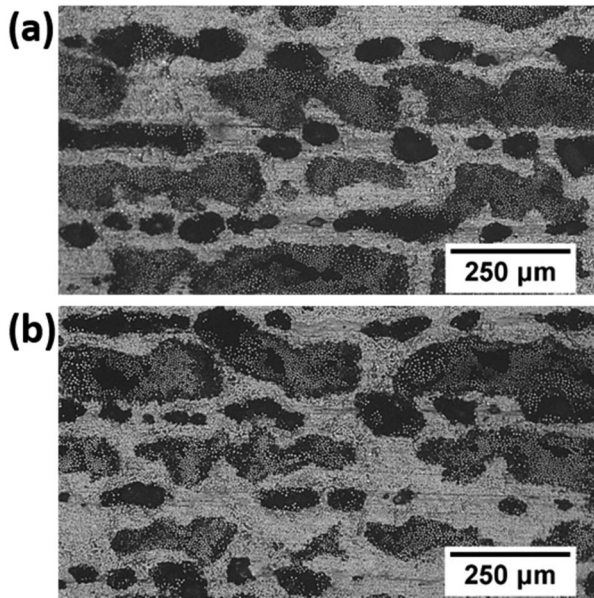


Figure 2. Optical micrograph of cold cured laminates at (a) day 0 and (b) day 49. Dark regions indicate un-filled voids and lighter regions indicate a resin-filled area.

Table 2. Effective flow number ($N_{FL,eff}$) for the out-life (day 28) sample cured under manufacturer's recommended cure cycle.

Cure cycle	$N_{FL,eff}$ (1/Pa)
0.6°C/min to 93°C	23.8
1.7°C/min to 93°C	27.4
0.6°C/min to 121°C	36.5
2.8°C/min to 121°C	71.7

prepreg microstructures at day 0 and day 49, which confirms that there are negligible changes to the unsaturated tow area at room temperature. Thus, the effect of out-time on the initial prepreg structure was judged to be negligible.

Calculated $N_{FL,eff}$ values are summarized in Table 2 for out-life samples (day 28) cured using low- to high- ramp rates and dwell temperatures from the manufacturer recommended cure cycles (MRCCs). One can expect that the lowest $N_{FL,eff}$ value for a given out-life obtained using MRCCs will set the lowest mark to produce a part of acceptable quality (generally <1% voids by volume). In this case, the value of $N_{FL,eff}$ is 23.8 Pa^{-1} , which is obtained from the slowest MRCC of 0.6°C/min to 93°C dwell at the 28-day out-life.

The micrographs shown in Figure 3a and 3b depict laminates cured at day 0 ($\alpha_0 = 0$), and at day 42 ($\alpha_0 = 0.21$) following a MRCC (2°C/min heating to a dwell at 121°C) are, respectively. As expected, complete resin impregnation with no void content is obtained for the Day 0 sample, for which $N_{FL,eff} = 542.1 \text{ Pa}^{-1}$ (see Table 1). In contrast, the Day 42 sample, for which $N_{FL,eff} = 10.2 \text{ Pa}^{-1}$, showed a void content of 1.4% (macro-voids = 1.3% and micro-voids = 0.1%). The term macro-voids as used here designates unconsolidated voids in the resin matrix,

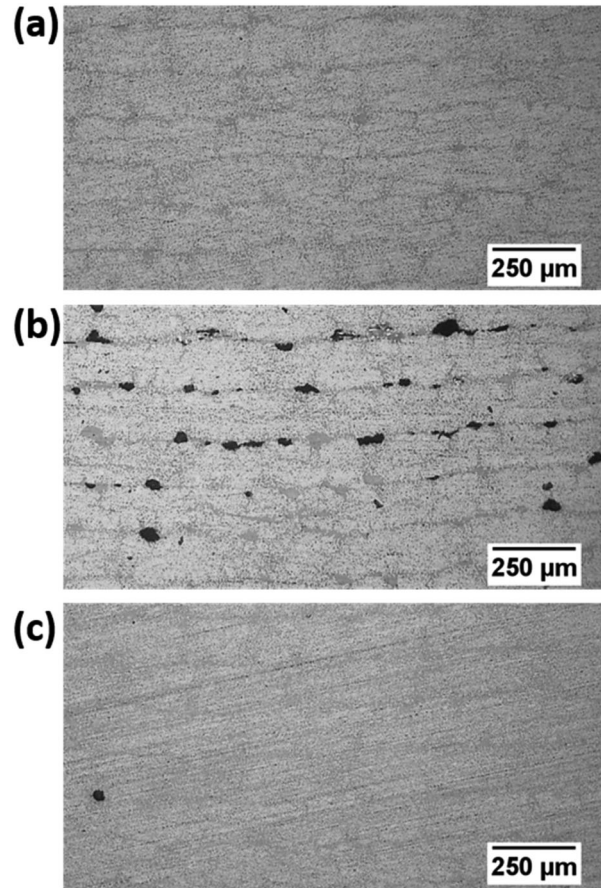


Figure 3. Optical micrograph of laminates cured under 2°C/min to 121°C dwell at (a) day 0 and (b) day 42; and of laminates cured under 20°C/min to 160°C dwell at (c) day 42.

while the term micro-voids designates the un-impregnated fiber tow. Note that in this case, the value of $N_{FL,eff}$ was well below the minimum value required to produce parts of acceptable quality ($N_{FL,eff} = 23.8 \text{ Pa}^{-1}$). Note that the micro-void content (only 0.1%) is expected to be the predominant type of voids in aged samples [1], while the macro-void content (1.3%) was much larger. The void distribution (see Table 1) indicates that most of the fiber tows were impregnated *before* entrapped air and volatiles in the resin were able to migrate to sample edges (or exit *via* intra-ply breathing).

A faster heating rate (20°C/min) and higher dwell (160°C) sharply reduced the void content in the Day 42 sample, as shown in Figure 3c. This cure cycle, for which $N_{FL,eff} = 40.5 \text{ Pa}^{-1}$) resulted in a macro-void content of just 0.1%, and a micro-void content of zero. The flow time calculated for the cure cycle was 17 min, which is 51.7 min less than the flow time for the Day 42 sample processed using the MRCC (2°C/min to 121°C dwell). Recall that that sample resulted in a void content of 1.4%. This result teaches that lower- η flow obtained by a faster heating rate and higher dwell temperature promotes volatile removal and resin infiltration. In other words, $N_{FL,eff}$ which leverages both flow level and

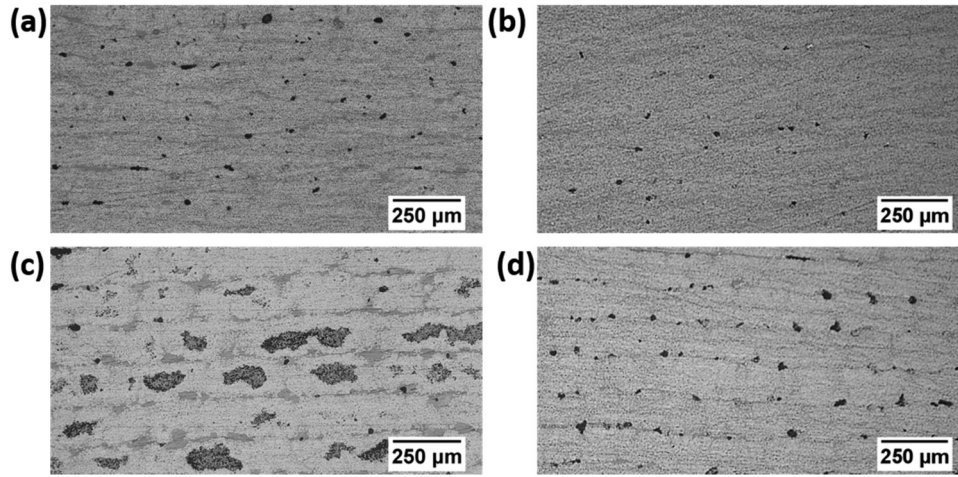


Figure 4. Optical micrograph of laminates cured under (a) 20 °C/min to 160 °C dwell and (b) 35 °C/min to 170 °C dwell at day 49; and of laminates cured under (c) 1 °C/min to 121 °C dwell and (d) 35 °C/min to 170 °C dwell at day 56.

time, is an effective quantitative measure of the total resin flow.

Further evaluation of resin flow enhancement *via* $N_{FL,eff}$ optimization is depicted in Figure 4, showing polished sections of laminates at day 49 ($\alpha_0 = 0.27$) cured using (a) 25 °C/min to a dwell at 160 °C, and (b) 35 °C/min to a dwell at 170 °C. The two cure cycles resulted in values of $N_{FL,eff} = 21.1 \text{ Pa}^{-1}$ and 24.1 Pa^{-1} with micro-void contents of zero, but macro-void contents of 0.64% and 0.49%, respectively. The latter cycle yielded a lower η flow, but a shorter flow time. The results further demonstrate that a greater flow number ($N_{FL,eff}$) yields a lower void content. Optimization of $N_{FL,eff}$ is achieved primarily *via* increasing the heating rate and the dwell temperature, both of which produce flow at lower η . In addition, a 25 °C/min ramp to a dwell at 160 °C on the Day 49 sample resulted in acceptable quality (voids < 1%) with a flow number $N_{FL,eff} < 23.8 \text{ Pa}^{-1}$ - this was assumed to be the minimum $N_{FL,eff}$ required to produce quality parts. The results demonstrate that the criterion used is on the conservative end.

Micrographs in Figure 4 show laminates cured at Day 56 ($\alpha_0 = 0.32$) subjected to (c) a 1 °C/min ramp to a 121 °C dwell, and (d) a 35 °C/min ramp to a 170 °C dwell. At this point, the ambient temperature cure reaction has advanced substantially, such that the criterion of $N_{FL,eff} > 23.8 \text{ Pa}^{-1}$ can no longer be met within the maximum heating rate capacity of the heated tool. That is, given $\alpha_{gel} = 0.37$, α_0 of the Day 56 sample, only has 5% degree-of-cure remaining before the resin reaches gelation (the flow stop point). The sample heated at 1 °C/min to a 121 °C dwell, for which the flow number $N_{FL,eff} = 1.8 \text{ Pa}^{-1}$, resulted in a macro-void content of 0.3% and a micro-void content of 7.2%. Note that compared to samples with higher $N_{FL,eff} > 10 \text{ Pa}^{-1}$, the macro-void content actually decreased, while the micro-void content increased (see Table 1). We can only

speculate that in this case, open micro-voids facilitated removal of macro-voids, as the resin η_{min} was too high to sufficiently infiltrate the fiber beds. In this regard, an in-depth evaluation of micro-void and macro-void formation phenomena, accompanied by process simulations and heated tooling, could yield greater clarity. However, there has been little conclusive evidence of the dependence of laminate performance on the *type* of voids in the laminate. Future efforts to apply $N_{FL,eff}$ optimization to consider prepreg fabric type, degree of impregnation level, and out-time should help to better understand OoA prepreg consolidation phenomena.

The sample heated at 35 °C/min to a dwell at 170 °C resulted in $N_{FL,eff} = 11.6 \text{ Pa}^{-1}$ and yielded a macro-void content of 1.2%. Therefore, in the range of $1.8 \text{ Pa}^{-1} < N_{FL,eff} < 11.6 \text{ Pa}^{-1}$, a transition from macro-void formation to micro-void formation occurs. This finding teaches that adequate flow time is also required for volatiles entrapped in the resin to escape *via* the intra-ply breathing path.

Using thermal analysis (TGA), the volatile content in prepreg aged in 60% relative humidity was measured to be 0.5 wt%, and the primary volatile species in epoxy resin is assumed to be H₂O [22]. Figure 5a plots the evolution of absorbed H₂O (wt%) under different ramp rates. The data is obtained using TGA at 1 atm. Although the H₂O removal rate may be faster under vacuum, the H₂O removal trend is expected to be the same regardless of pressure. Figure 5b shows plots of the H₂O remaining at the gelation point for the ramp rates of Day 0 and Day 49 samples. The plots show that the faster ramp rate needed to produce low-porosity parts, either by attaining lower η_{min} flow or by increasing $N_{FL,eff}$, results in early infiltration of the fiber bed. This early enclosure of tow area or the primary breathing path then entraps volatile species, primarily H₂O. This early infiltration translates into

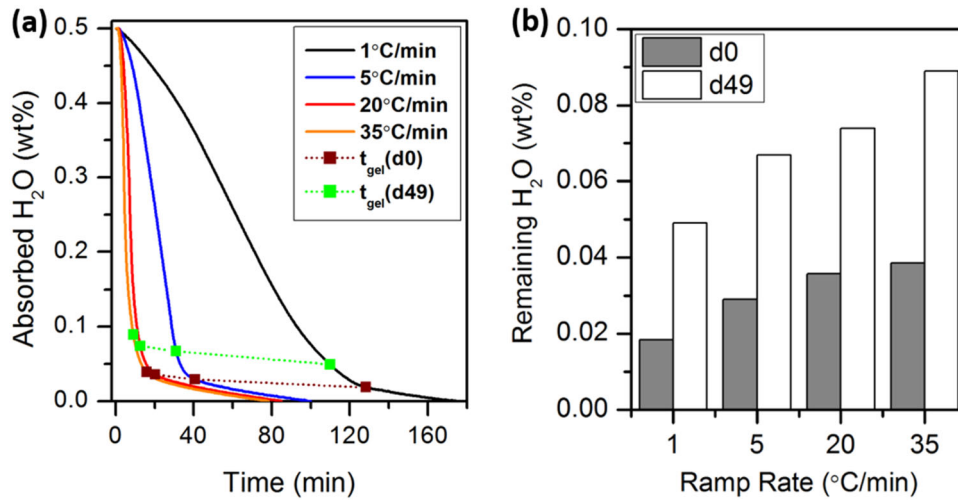


Figure 5. (a) Evolution of absorbed H₂O (wt%) under various ramp rates (b) Remaining H₂O (wt%) of day 0 and 49 samples cured under various ramp rates.

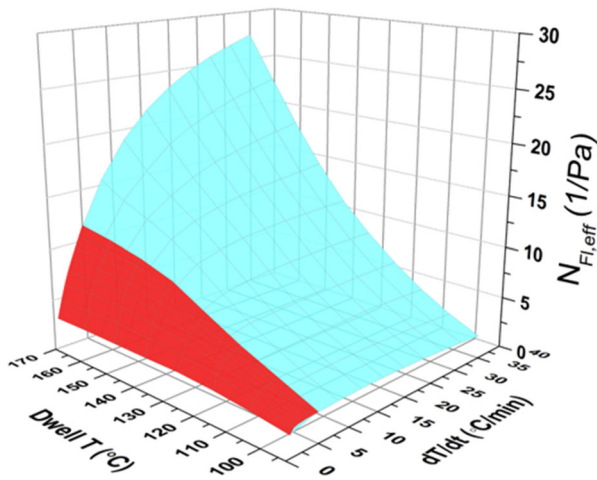


Figure 6. Representative processing map of $N_{FL,eff}$ vs. dwell temperature vs. heating rate at day 49. Red portion indicates the capability of conventional oven (5 °C/min) and teal portion indicates the capability of a heated tool (35 °C/min).

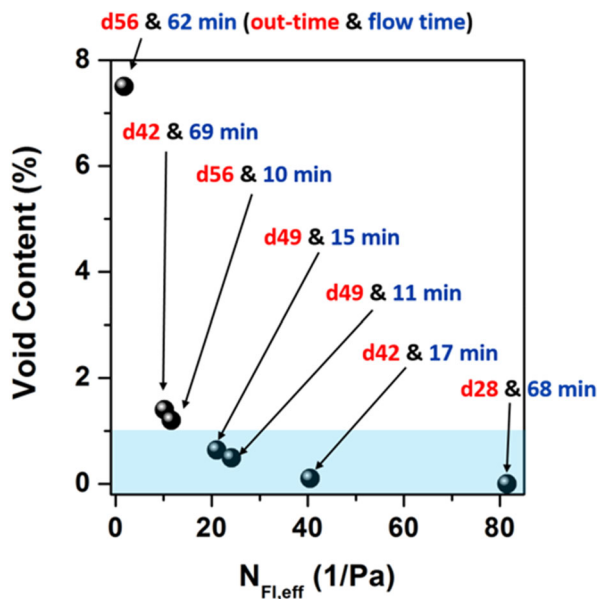


Figure 7. Void content (%) area vs $N_{FL,eff}$ (1/Pa). Teal portion indicates the 1% void content criterion generally used in the aerospace industry.

macro-voids as opposed to micro-voids. However, larger values of $N_{FL,eff}$ consistently produce parts with lower overall void content, illustrating the utility as an effective flow control variable.

Figure 6 shows a 3-axis plot of $N_{FL,eff}$ vs. dwell temperature vs. heating rate for the Day 49 sample. The plot, which can be used as a process map to guide the development of a cure cycle, shows that high $N_{FL,eff}$ can only be achieved *via* a fast ramp rate combined with a high dwell temperature. Assuming a conventional oven with a maximum ramp rate of 5 °C/min, plotted as the red surface in Figure 6, can reach a maximum $N_{FL,eff}$ of only 12. Consequently, heated tooling or other means (heating blankets) capable of higher ramp rates is required to attain higher values of $N_{FL,eff}$, especially prepreg has exceeded the specified out-life, a condition in which reduced flow is encountered using the MRCC. Also, when deploying a flow-enhanced cure cycle, the maximum dwell temperature should not exceed the boiling point and/or degradation temperature of the resin.

Figure 7 plots void content versus $N_{FL,eff}$ for all samples aged to the specified out-life and beyond in this study. The plotted data show that $N_{FL,eff}$, which leverages both flow level and time, is an effective, stand-alone metric to minimize void content in parts. The plot shows that a void content of less than 1% is achieved even on a sample well past the manufacturer's specified out-life (28 days), even up to 49 days. Therefore, provided the degree of cure α_0 of the aged prepreg has a sufficient separation from its α_{gel} , a low-porosity laminate can be produced by adjusting the cure cycle *via* the $N_{FL,eff}$ method described here.

5. Conclusions

We have demonstrated a method to adjust the cure cycle to effectively optimize flow, a method that

leverages both flow level and flow time to limit flow-induced defects. First, laminates were laid up with $[0^\circ]_{12}$ sequence and aged for 0 to 56 days (specified out-life = 28 days). A quantitative flow metric, $N_{Fl,eff}$ (Pa^{-1}) was defined as the integration of inverse viscosity evolution during cure, thus taking into account both flow level and time. Laminates with different out-times and cure cycles were produced, guided by previously developed process models [4–6] that comprehensively capture the effects of out-time and humidity. To enable wide control of $N_{Fl,eff}$ values, a heated tool (developed in-house) capable of heating rates up to $35^\circ\text{C}/\text{min}$ and accurate temperature control between 30°C and 200°C was used. The resulting void contents were then measured.

The results showed that a higher heating rate to a higher dwell temperature led to a more rapid decrease in η , allowing the resin to achieve lower η_{min} before the substantial effect of cure manifest. The same cure cycle also led to a decrease in flow time. $N_{Fl,eff}$ Analysis revealed that η_{min} had a greater influence on the $N_{Fl,eff}$ increase than flow time, thus favoring cure cycles with higher heating rates to higher dwell temperatures, the latter being limited by the resin boiling point and degradation temperature.

Porosity analysis revealed a strong correlation between overall decrease in void content with $N_{Fl,eff}$. Higher macro-void contents were observed relative to micro-void content in samples with high out-time cured using a rapid heating rate. Formation of macro-voids resulting from high heating rates indicates that trapped volatiles require sufficient time to evacuate before closure of intra-ply breathing pathways. In contrast, high out-time samples cured at low heating rates exhibited typical micro-void dominant porosity formation, as the resin η_{min} was too high to sufficiently infiltrate the fiber beds.

Regardless of the type of void formation, the findings indicate that $N_{Fl,eff}$ can be used effectively as a criterion to limit flow-induced defects. Quality parts with less than 1% voids were consistently produced with the set minimum $N_{Fl,eff}$ a value that will depend on the specific prepreg used. The current method potentially allows the use of prepreg materials long after the specified out-life, when assisted by advanced curing environments.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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