Article



Effects of post-infusion dwell on vacuum infusion of thermoset composites toughened by thermoplastic interlaminar veils

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Abstract

The effects of post-infusion dwell on vacuum infusion of thermoset composites toughened by non-woven thermoplastic interlaminar veils were investigated. Permeability measurements and simulation of the resin infusion process demonstrated that the toughening interlayers can effectively act as interlaminar flow distribution media. Local variations in permeability induced non-uniform flow fronts, resulting in high porosity. However, introduction of a low-temperature post-infusion dwell allowed more time for the resin to equilibrate pressure and redistribute during the post-filling stage, achieving full saturation of dry regions. The process parameters of the post-infusion dwell were determined using cure kinetics and viscosity models, while *in situ* process adjustments were implemented using dielectric cure monitoring system in conjunction with resin cure maps. Laminates fabricated with the modified cycle exhibited reduced porosity and greater peak load during impact testing. This work highlights potential advantages of the post-infusion dwell, which can similarly be applied to other vacuum infusion processes requiring a protracted post-filling stage.

Keywords

Cure behavior, porosity, process monitoring, vacuum infusion

Introduction

In this work, we investigate the effects of post-infusion dwell on part quality for vacuum infusion (VI) of thermoset composites toughened by thermoplastic interlaminar veils. Previous studies have demonstrated that insertion of non-woven thermoplastic veils increases impact strength of thermoset composites,1-7 yet can also cause increased porosity because of local variations in preform permeability created by the highly permeable veils.^{2,8} One of these reports speculated that modifying the manufacturing process to provide more time for resin to saturate dry regions may reduce laminate void content,² although no such studies have been reported. To promote impregnation of partially saturated interlaminar regions, a lowtemperature post-infusion dwell was introduced to allow greater time for low-viscosity resin to equilibrate pressure and redistribute during the post-filling stage. The process parameters of the post-infusion dwell were determined using resin cure kinetics and viscosity

model predictions. Dielectric cure monitoring (DCM) system permitted *in situ* measurement of resin ion viscosity, while resin cure maps and ion viscosity model were employed to convert the collected ion viscosity data into useful cure process metrics and to adjust the cure cycle accordingly.

Conventional autoclave cure of prepreg composites is robust, yet involves high capital and operating costs, driving the exploration of faster and more cost-efficient out-of-autoclave processes.^{9,10} A promising alternative

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to autoclave processing is liquid composite molding (LCM), which involves lay-up of a dry fiber preform in a mold, infusion of liquid resin through the preform, and resin cure.^{11–14} Based on tool design, LCM processes are generally divided into two—resin transfer molding (RTM) and VI. The RTM process features a two-part rigid mold for top and bottom tool surfaces, and resin is injected under positive pressure (greater than ambient pressure); in contrast, in the VI process, the top tool surface is replaced by a flexible vacuum bag, and resin is infused using atmospheric pressure differential.^{11,13,15–17}

Using a one-sided rigid mold, the VI process can be employed to produce large, complex, and unitized (without secondary bonding) parts at relatively low cost.^{11,17–20} However, because only atmospheric pressure is applied from vacuum-only consolidation, VI requires use of low-viscosity thermoset resins, which in general are brittle and thus susceptible to impact damage.^{1,8,20,21} Introducing thermoplastic content into thermoset composites through bulk resin modification or interlaminar toughening can mitigate the inherently low toughness of thermosets.^{1,3} Bulk matrix toughening generally is achieved by mixing rubber/thermoplastic liquid or particles into the thermoset resin. Yet this approach is often incompatible with liquid molding because of the associated increase in resin viscosity.¹⁻⁴ In contrast, interlaminar toughening, which includes co-mingled fibers, thermoplastic films, and non-woven fiber veil additions, can potentially impart toughening to weak interlaminar regions of thermoset composites while simultaneously mitigating the processing concerns arising from elevated resin viscosity for LCM manufacturing.^{1,3} Note that the two methods (bulk resin modification and interlaminar toughening) are not necessarily mutually exclusive, but potentially can be used together.

Inserting non-woven thermoplastic veils at interlaminar regions is a cost-effective toughening method that can be applied to any composite manufacturing processes, but is relatively less technologically mature than alternative approaches.¹⁻³ In hand lay-up of dry preforms, the veils can be inserted between reinforcement plies. For automated tape laying (ATL) or automated fiber placement (AFP) processes, the automated lay-up of preforms must be paused intermittently for veil insertion between layers. However, the addition of thermoplastic veils can be automated potentially, because the veils can be produced in tape form and heated to promote tack. When inserted at interlaminar regions, the veils embed into adjacent fabric plies during infusion and consolidation stages, resulting in a fiber bridging effect during interlaminar fracture.^{1,5} This interleaving mechanism can effectively resist delamination and crack propagation by arresting damage growth and localizing damage.^{1,2} Therefore, adding thermoplastic veils of low areal weight to thermoset composites can enhance resistance to impact damage, while increasing Modes I & II interlaminar fracture toughness and compression-after-impact (CAI) strength.^{2,3,5–7} Exhibiting greater permeability than reinforcement fabrics, the veils also can serve as interlaminar flow distribution media, through which resin can flow more rapidly during the infusion process.^{2,8} However, the local variations in preform permeability can induce non-uniform flow fronts and high porosity.^{2,8}

In the VI process, local compaction pressure exerted on the preform varies as the resin flow front progresses during the infusion (from vacuum to atmospheric pressure), and the top flexible vacuum bag surface allows preform thickness to adjust accordingly.^{13,22} The continuously evolving preform deformation and pressure gradient result in a unique processing stage in VI, called "post-filling" stage, which refers to the period between the completion of infusion and resin gelation.^{12,22,23} Once infusion is complete and the resin inlet is closed, the resin pressure field within the part is initially not uniform, and resin continues to flow and redistribute to reach an equilibrium state, impregnating partially saturated regions.^{12,22,23} Therefore, insufficient post-filling time in VI can lead to incomplete saturation of dry areas, leading to greater void content in cured laminates toughened by non-woven thermoplastic veils.

Typically, the manufacturer-recommended cure cycle (MRCC) of high-performance infusion-grade thermoset resin is comprised of a single isothermal dwell,²⁴ during which the resin degree of cure and viscosity rise sharply after a brief reaction induction period.^{12,24} For the epoxy resin used in this study (Hexcel HexFlow[®] RTM6), the MRCC features a single dwell for 120 minutes at 180 °C, and the resin gels fast within 20 minutes at the prescribed temperature.²⁴ However, such a brief post-filling period may not provide sufficient time to complete saturation of partially filled regions, especially for preforms containing multiple plies of thermoplastic veils.

The addition of interlaminar thermoplastic veils enhances impact resistance of thermoset composites, but the relatively high permeability of the veils also introduces disparities in flow rates in the layers and thus can result in unacceptable levels of porosity. To address this issue, we modified the MRCC for the resin (RTM6) by adding a low-temperature post-infusion dwell to provide additional time for the resin to fully saturate dry interlaminar regions before gelation. The process parameters of the post-infusion dwell were initially determined using resin cure kinetics and viscosity models. Resin cure maps (degree of cure and mechanical viscosity isolines) provided correlations between the isoline metrics and ion viscosity, and were employed to identify the gel point during the cure cycle. A DCM system was used to monitor the cure state *in situ*, to adjust the cure cycle, and to validate and refine the cure maps accordingly.

Three different VI process cases were examined, demonstrating that the post-infusion dwell step effectively achieved full saturation and void removal by extending the post-filling stage. Overall, this work describes a potential pathway to address the problem of high porosity commonly encountered in VI processes. The pathway includes a low-temperature post-infusion dwell informed by material characterization, the use of resin cure maps, and *in situ* process diagnostics. The post-infusion dwell can also be extended to other VI process variants that require a protracted postfilling period for full impregnation of dry regions, including those that feature multi-component or complex-shaped preforms.

Experimental

Materials

An aerospace-grade epoxy resin designed for infusion processes was selected and acquired (HexFlow[®] RTM6, Hexcel²⁴). The resin was stored under refrigeration at -18° C to prevent undesired curing of the material before use. For the reinforcement, a plain weave carbon fiber fabric with an areal weight of 193 g/m² and 3000 fiber/tow count was used (part #1530, Fibre Glast²⁵), while for the thermoplastic interlayer, a non-woven PBN-II[®] polyamide (nylon 66) veil with an areal weight of 34 g/m² was supplied by Cerex Advanced Fabrics.²⁶ Before part lay-up, the polyamide veils were dried in a vacuum oven at 90 °C for two hours at 0.1 MPa (absolute pressure) to remove any absorbed moisture during storage.

Cure kinetics & rheology characterization

Modulated differential scanning calorimetry (MDSC). The cure kinetics of the epoxy resin was characterized using DSC (TA Instruments, Q2000) under nitrogen purge ($50 \text{ cm}^3/\text{min}$). Resin samples (8-10 mg) were sealed in aluminum hermetic pans with lids, and exposed to a dynamic ramp at four heating rates (5, 10, 15, and 20 °C/min) to determine the total heat of the cure reaction. The temperature range of the dynamic runs spanned from -50 to 340 °C. Isothermal dwell measurements were conducted at eight temperatures: from 80 to 120 °C in 10 °C increments to monitor advances in degree of cure under filling conditions, and from 120 to 180 °C in 20 °C increments to examine

and model resin cure kinetics. Following the dwell, the samples were heated to 300 °C at 5 °C/min to measure the residual heat of cure. However, during the temperature ramp to 300 °C, the samples exhibited an endothermic annealing peak near the residual cure exotherm due to physical aging of the resin.²⁷ To eliminate this annealing peak, the specimens were quenched rapidly at the endotherm spike to -50° C and then reheated during re-scan (Figure 1), using the method developed by Karkanas et al.²⁷ For the ramping step, a sinusoidal temperature modulation of ± 0.5 °C/min was applied to distinguish reversing and non-reversing heat flow signals. Reversing heat flow depends on heat capacity and the rate of temperature change, while non-reversing heat flow is associated with kinetic components of the reaction.^{28,29} Glass transition can be examined from the reversing heat flow signal because it is a heating rate dependent transition, while the curing reaction is a temperature dependent transition and thus appears in the non-reversing signal.^{28–30}

Rheometry. Viscosity evolution during epoxy resin cure was characterized using a rheometer (TA Instruments, AR2000ex). The resin samples were exposed to an iso-thermal dwell at eight temperatures, analogous to MDSC isothermal dwell measurements. Two different



Figure 1. Heat flow and reversing heat flow profiles during the residual cure of the epoxy resin after isothermal dwell at $140 \degree C$ before and after annealing peak removal.

rheometer geometry fixtures were applied for different dwell temperature ranges. A cone-and-plate geometry fixture (cone angle 2°, cone diameter 40 mm, and truncation 53 um), which provides uniform shear rate and high rheological accuracy, was equipped with a Peltier plate to accurately measure low initial resin viscosity (below 0.1 Pa \cdot s) during the filling condition rheology tests (80–120°C). For high temperature cure condition tests (120-180°C), disposable parallel-plate geometry fixture (diameter 25 mm) and environmental test chamber setup were used instead to measure the wide range of evolving resin viscosity throughout the entire cure span. For the rheology tests, cell temperature was increased from the pre-heating temperature (either 80 or 100 °C) to the dwell temperature at approximately $30 \circ C/min$ (the maximum ramp rate of the rheometer) and held constant for either 3 hours (for filling condition tests to prevent resin full cure) or prescribed durations (for curing condition tests), under oscillatory shear at 1 Hz frequency and 5% strain (within the linear viscoelastic region).

Dielectric analysis (DEA). DEA measurements were conducted using a dielectric cure monitoring system (Netzsch, DEA 288 Epsilon) in frequency intervals from 1 Hz to 1 kHz at four cure temperatures (120, 140, 160, and 180 °C). A dielectric sensor (Netzsch, Mini-IDEX 100/35) with 33 mm^2 sensing area and 100 µm electrode spacing, along with thermocouple, was mounted on the rheometer Peltier plate, which served as the heated tool. The resin sample was placed on the dielectric sensor, and cell temperature was rapidly increased from 80 °C to the dwell temperature at \sim 30 °C/min and held constant until the resin fully cured. Once a sinusoidal excitation voltage is applied to the resin sample, dipoles align with the applied electric field and charge carriers move toward electrodes of opposite charge, resulting in sinusoidal current and phase shift responses, which can be converted into useful dielectric properties such as complex permittivity (ε^*) and ionic conductivity (σ).^{31–33} The reciprocal of σ is called ion viscosity and strongly correlates to mechanical dynamic viscosity (η) .^{33–35}

Permeability measurement & infusion simulation

The permeabilities of plain weave carbon fiber fabric and non-woven polyamide veil were assessed using unsaturated radial flow test. The isotropy of the polyamide veil was unknown prior to testing, and the radial test configuration permitted simultaneous measurement of the two principal permeability values of an anisotropic material.^{36,37} A vacuum-driven constant injection pressure experimental setup (Figure 2) was used, as demonstrated by Pierce et al.^{38,39}



Figure 2. Experimental setup of vacuum-driven constant injection pressure radial flow permeability measurement.

A single-ply sample was placed between a bottom tool plate and an upper polycarbonate caul plate (measured cavity thickness of 1.50 ± 0.05 mm), while breather cloth surrounded the sample periphery to establish an even pressure gradient and to maintain cavity thickness. Instead of epoxy resin, olive oil (Kirkland Signature) was infused as a facsimile resin to allow permeability measurement at ambient temperature, as the viscosity of olive oil at room temperature (measured, $0.07 \,\mathrm{Pa} \cdot \mathrm{s}$) was consistent with that of the pre-heated RTM6 resin ($<0.10 \text{ Pa} \cdot \text{s}$). The test fluid was infused into the center of each sample through 6 mm ID inlet tubing, and the resulting radial flow pattern was recorded using a digital video camera. Through visual image processing, flow front position against flow time data were obtained, which were used to calculate material permeabilities. For each material, the permeability measurements were conducted three times.

With the measured permeability values, resin infusion through a multilayer laminate ($355 \text{ mm} \times 255 \text{ mm}$) comprising 16 plies of plain weave carbon fiber fabric and 7 plies of polyamide veil was simulated using a commercial finite element analysis software (PAM-RTM, ESI Group). For the preform stacking sequence, one layer of veil was inserted after every two layers of carbon fiber fabric. The infusion simulation was performed to demonstrate that the permeability of thermoplastic interlayers was greater than that of carbon fiber fabric, such that the veils effectively functioned as interlaminar flow distribution media.

Part manufacture & quality analysis

Laminate manufacture. Carbon fiber-epoxy laminates $(355 \text{ mm} \times 255 \text{ mm})$ were produced by VI. A reference sample (Case A), consisting of 16 plies of carbon fiber fabric with no veils, was fabricated first, followed by toughened samples (Case B and C), which included the same number of carbon fiber reinforcement plies with

an additional 7 plies of polyamide veils. For Case B and C, one ply of thermoplastic interlayer was inserted after every two plies of carbon fiber fabric. Although the resin sample was already degassed, the resin was still degassed once more prior to infusion for 30 minutes to remove air entrapped during resin transfer. Following the manufacturer process specifications,²⁴ the degassed resin and the mold were pre-heated to 80 and 120 °C respectively. The cure cycle was initiated once infusion completed: Case A and B were cured using the MRCC (120 minutes at 180 °C), while Case C was cured using the modified cure cycle (170 min at 129 °C, followed by 60 min at 180 °C). During Case C sample manufacture, a dielectric sensor (Netzsch, Mini-IDEX 100/35) was embedded inside the vacuum bag to monitor ion viscosity throughout the cure process. The manufactured laminates were cut into test specimens using an abrasive waterjet cutting system.

Porosity analysis & impact testing. For microstructural porosity analysis, laminates were sectioned, ground, and polished. Images of each cross-section were recorded using a digital stereo microscope (VHX-5000, Keyence). Impact resistance of the manufactured laminates (101 mm \times 152 mm) was assessed using a drop tower impact device (9250HV, Instron) in accordance with ASTM D7136.⁴⁰ Four samples for each case were tested at 30 J impact energy level, with a

7.8 kg impactor, a 16 mm diameter hemispherical striker tip, and a pneumatic rebound brake system. The force-time profile was recorded during the impact, and the damage area from impact was measured using a non-destructive testing (NDT) ultrasound system with a 20 MHz transducer (Mistras NDT) and ImageJ software.

Results & discussion

Cure kinetics & rheology characterization

Cure kinetics. Figure 3(a) shows MDSC heat flow data measured during the dynamic ramp tests. The total heat of cure reaction (H_r) was independent of the applied heating rate, and was determined to be 429.9 (± 4.6) J/g, similar to previously reported values.^{27,41} Heat flow data were also obtained during the isothermal dwell tests performed at filling and curing temperatures. Under the assumption that the rate of cure reaction is proportional to the rate of heat flow, the heat flow data can be converted into cure rate data using the following equation:^{42,43}

$$\frac{\mathrm{d}\alpha}{\mathrm{d}t} = \frac{1}{H_r} \left(\frac{\mathrm{d}H}{\mathrm{d}t} \right) \tag{1}$$

where α is the resin degree of cure, *t* is time, and *H* is the heat flow measured from the MDSC. The resin



Figure 3. MDSC data showing heat flow profiles measured during (a) dynamic ramp tests of RTM6 resin, (b) isothermal dwell tests at filling temperatures ($80-120^{\circ}C$), (c) isothermal dwell tests at curing temperatures ($120-180^{\circ}C$), and (d) cure kinetics model fitting results, expressed in terms of resin degree of cure vs. time (experimental: solid line, model-prediction: dotted line).

degree of cure can be calculated by integrating equation (1). At 80 and 90 °C, α advanced negligibly, while at 110 and 120 °C, α evolved slightly faster, and the resin reached full cure toward the end of the 8-hour dwell step (Figure 3(b)). Thus, the resin and mold pre-heating temperatures of 80 and 120 °C were deemed appropriate.²⁴ As expected, the heat flow curves evolved much faster at higher curing temperatures (120-180 °C), with much shorter reaction induction periods (Figure 3(c)). The final degree of cure (α_f) of the resin samples reacted at 120, 140, 160, and 180 °C were 0.83, 0.88, 0.93, and 0.97 respectively.

The MDSC data for resin cure were fit to a phenomenological cure kinetics model developed by Kratz et al.,⁴² which accounts for transitioning from kinetics-controlled to diffusion-controlled cure reaction as α rises above α_{gel} (α at resin gelation), and a faster cure rate present in the low- α region ($\alpha < 0.1$):

$$\frac{\mathrm{d}\alpha}{\mathrm{d}t} = K_1 \alpha^{m_1} (1-\alpha)^{n_1} + \frac{K_2 \alpha^{m_2} (1-\alpha)^{n_2}}{1 + \exp(D(\alpha - (\alpha_{C0} + \alpha_{CT}T)))}$$
(2)

$$K_i = A_i \cdot \exp\left(-\frac{E_{Ai}}{RT}\right)$$
 where $i = 1, 2$ (3)

where K_i is the Arrhenius temperature dependent term, m_1 , m_2 , n_1 , and n_2 are the reaction order-based fitting constants, D is the diffusion constant, α_{C0} is the critical α at absolute zero, α_{CT} accounts for the increase in critical α with temperature, A_i is the pre-exponential factor, E_{Ai} is the activation energy, R is the universal gas constant, and T is the temperature [K].

Figure 3(d) shows both experimental (solid line) and model-predicted (dotted line) degree of cure for reactions conducted at 120, 140, 160 and 180 °C. The model predicted resin degree of cure with high precision, showing only minor fit deviations (from the experimental data) near the onsets of the final plateaus, toward the end of the cure reaction. The values of the cure kinetics model parameters are shown in Table 1. Karkanas et al.²⁷ studied the cure kinetics of epoxy resin (RTM6) and developed a modified autocatalytic cure kinetics model (equation (4)). Predictions from both Kratz and Karkanas models (equations (2) and (4)) resulted in agreement with the experimental results. However, the Kratz model (equation (2)) achieved a slightly closer fit, because the model included more fitting parameters that accounted for the shift from a kineticscontrolled to a diffusion-controlled cure reaction.

$$\frac{d\alpha}{dt} = K_1 (1 - \alpha)^{n_1} + K_2 \alpha^m (1 - \alpha)^{n_2}$$
(4)

Table 1. Values of the cure kinetics model parameters for

 RTM6 epoxy resin.

Parameter	Value	Parameter	Value
A ₁ [s ⁻¹] E _{A1} [J/mol] m ₁ D	1.70×10^{6} 8.22 × 10 ⁴ 0.27 10.65 43.34	$A_2 [s^{-1}] \\ E_{A2} [J/mol] \\ m_2 \\ n_2 \\ \alpha_{C0} \\ \alpha_{CT} [K^{-1}]$	$1.63 \times 10^{4} \\ 5.72 \times 10^{4} \\ 1.15 \\ 1.22 \\ -0.11 \\ 2.23 \times 10^{-3}$

Rheology. Viscosity evolution data were obtained during the isothermal scans conducted at low filling temperatures between 80 and 120 °C, shown in Figure 4(a). The use of a cone-and-plate fixture yielded virtually noise-free rheological measurements, even at low viscosity values (below $0.1 \text{ Pa} \cdot \text{s}$). During the 3-hour isothermal dwell, the resin viscosity curves remained nearly flat at 80 and 90 °C, while increasing notably more at 110 and 120 °C. Figure 4(b) (scatter) shows the viscosity measurements performed at higher curing temperatures (120-180°C). After a brief induction period, resin viscosity evolved in a sigmoidal shape, analogous to the degree of cure profiles presented in Figure 3(d).

A phenomenological viscosity model developed by Khoun et al. was used to fit rheology data:⁴³

$$\eta = \eta_1 + \eta_2 \left(\frac{\alpha_{gel}}{\alpha_{gel} - \alpha}\right)^{A + B\alpha + C\alpha^2} \tag{5}$$

$$\eta_i = A_{\eta i} \cdot \exp\left(-\frac{E_{\eta i}}{RT}\right)$$
 where $i = 1, 2$ (6)

where η is the viscosity, η_1 and η_2 are the Arrhenius dependent viscosity component, α_{gel} is the degree of cure at gelation, A, B, and C are the fitting constants, A_{ni} is the Arrhenius constant, E_{ni} is the viscosity activation energy, and T is the temperature [K]. In Figure 4 (b), the experimental data (scatter) and model fitting results (line) of viscosity evolution at four cure temperatures are shown. The values of the viscosity model parameters are shown in Table 2. Figure 4(c) shows representative storage (G') and loss (G'') modulus profiles measured during resin cure at 160 °C. The gelation point (G' = G''), which marks the phase transition from liquid state (G'' > G') to rubbery state (G' > G''),⁴⁴ was determined to be near η of $10^3 \text{ Pa} \cdot \text{s}$ with α_{gel} of 0.63. Gelation time is an important metric in composites manufacturing, because resin flow ceases at gelation. thus preventing further impregnation.³⁴ The gel times of the resin at different cure temperatures were



Figure 4. Rheology data showing mechanical viscosity profiles of RTM6 resin during (a) isothermal dwell tests at filling temperatures (80–120°C), (b) isothermal dwell tests at curing temperatures (120–180°C) and viscosity model fitting results (experimental: scatter, model-prediction: line), and (c) storage and loss modulus profiles measured during resin cure at 160°C.

$A_{\eta I}$ [Pa · s]	E _{ηI} [J/mol]	A _{$\eta 2$} [Pa · s]	E _{η2} [J/mol]	α _{gel} [–]	A [-]	В [—]	C [-]
$1.54 imes 10^{-5}$	3.10×10^3	$\rm 1.02\times10^{-8}$	$\textbf{4.90}\times\textbf{10}^{4}$	0.63	3.72	5.69×10^{-3}	0.03

Table 2. Values of the mechanical viscosity model parameters for RTM6 epoxy resin.

Table 3. Gelation times of RTM6 epoxy resin at different reaction temperatures.

Temperature [°C]	120	140	160	180
Gelation Time [min]	270.6 (±1.9)	101.5 (±0.6)	47.1 (±1.3)	21.5 (±0.3)

measured three times at each cure temperature and are summarized in Table 3.

Dielectric analysis & resin cure map. Previous studies have explored the use of dielectric cure monitoring of epoxy (RTM6) in various aspects. For example, Kazilas et al. conducted a comprehensive DCM study on the resin, ranging from impedance curve modeling to temperature modulated dielectric analysis.⁴⁵ In related work, Skordos et al. used imaginary impedance maximum to accurately monitor cure reaction progress under both isothermal and dynamic curing conditions.⁴⁶ Finally, Karkanas et al. demonstrated that the inflection point of the conductivity curve can be used to identify the point of resin gelation.⁴⁷ However, the principle of determining gel point from the conductivity inflection point was valid only for selected resin systems (*e.g.* autocatalytic resins, including RTM6), and thus was not suitable for universal application.⁴⁷ In this study, we developed resin cure maps to identify the cure state of interest (*e.g.* gel point) during actual part manufacture and to enable process adjustments *in situ*.

Figure 5(a) shows the ion viscosity data measured during the isothermal dwell tests conducted at curing temperatures (120-180°C). The sigmoidal shape of the ion viscosity curves resembles the profiles for degree of cure and mechanical viscosity, indicating that the three properties are correlated. As the state of cure progresses and mechanical viscosity increases, the degrees of both ion mobility and dipole rotation decrease, leading to a rise in ion viscosity.³³ In addition, similar to mechanical viscosity, which exhibits an Arrhenius temperature dependence, ion viscosity decreases with



Figure 5. (a) DEA data showing ion viscosity profiles of the epoxy resin during isothermal dwell tests at curing temperatures (120–180°C), (b) α -based resin cure map, in which the dashed lines represent degree of cure isolines, and (c) η -based resin cure map, in which the dashed lines are mechanical viscosity isolines.

increasing temperature at a given cure state.⁴⁸ The ion viscosity (IV) depends strongly on temperature, and can be expressed using the following equation:⁴⁸

$$\log(IV) = \log\left(\frac{k}{q^2 n D_0}\right) + \log(T) + \frac{Q}{kT \ln(10)}$$
(7)

where k is Boltzmann's constant, q is the magnitude of electronic charge, n is the free ion concentration, D_0 is the pre-exponential factor for the diffusion coefficient, Q is the activation energy for diffusion, and T is the temperature [K]. In equation (7), all parameters on the right side are independent of temperature, and the equation can be re-written as:

$$\log(IV) = A + \log(T) + \frac{B}{T}$$
(8)

where both coefficients A and B depend on α and thus η , because D_0 and Q vary with degree of cure.⁴⁸ Between the two temperature terms, $\log(T)$ and 1/T, the latter dominates the temperature dependence of ion viscosity, and thus ion viscosity decreases as temperature increases for a given α .

Based on these correlations, resin cure maps (for RTM6), plotting ion viscosity along either α or η isolines as a function of temperature, were constructed by correlating cure kinetics and rheology data to dielectric

 Table 4.
 Values of the ion viscosity model parameters for RTM6 epoxy resin.

α -based cure map		η -based cure map			
α	А	В	η	А	В
0.1 0.2	-1.10 -2.32	$\begin{array}{c} \textbf{2.48}\times\textbf{10}^{3}\\ \textbf{3.10}\times\textbf{10}^{3} \end{array}$	 0	-0.62 -3.49	$\begin{array}{c} 2.70 \times 10^3 \\ 4.10 \times 10^3 \end{array}$
0.3	-3.5 I	$3.73 imes 10^3$	10 ²	-5.71	$5.15 imes 10^3$
0.4	-4.96	$4.48 imes 10^3$	10 ³	-6.79	$5.69 imes 10^3$
0.5	-6.09	$5.13 imes 10^3$	-		
0.6	-6.95	$5.69 imes 10^3$	-		

measurements (Figure 5(b) and (c), scatter). The resin cure map data were fit using the ion viscosity model (equation (8)), and the results are presented as dashed lines in Figure 5(b) and (c). The values of the ion viscosity model parameters are shown in Table 4. Near the gel point, the parameters of the α - and η -based ion viscosity models are similar (at $\alpha_{gel} \sim 0.63$ and $\eta \sim 10^3 \text{ Pa} \cdot \text{s}$). The model predicts ion viscosity at the specified level of α or η as well as temperature. The DCM system permits *in situ* monitoring of resin ion viscosity, and the resin cure maps and ion viscosity data into degree of cure or mechanical viscosity metrics. Thus, during actual part manufacture, the resin cure maps can be employed to identify the gel point (or other cure state of interest) at any given cure temperature for real-time process adjustments. The methodology deployed can be applied to any epoxy resin system, in principle.

Permeability characterization & infusion simulation

Both plain weave carbon fiber fabric and polyamide veil exhibited 2D isotropic flow during radial flow permeability measurements. In each experimental run, a radial flow pattern was recorded (1920×1080 px resolution), which was subsequently processed to yield flow front position against flow time data (Figure 6(a)). The permeability value can be obtained using the following isotropic permeability model for constant injection pressure, which is derived from the Laplace equation



Figure 6. (a) Progressive images of flow fronts captured during the plain weave carbon fiber fabric permeability measurements, and (b) N term (terms in the bracket of equation (9)) plotted against flow time for permeability measurements of plain weave carbon fiber fabric and non-woven polyamide veil.

in polar coordinates, combined with Darcy's law and the continuity equation:^{39,49}

$$K = \left[r_f^2 \left(2\ln\left(\frac{r_f}{r_0}\right) - 1 \right) + r_0^2 \right] \frac{1}{t} \frac{\eta \epsilon}{4\Delta P} = F_i \frac{\eta \epsilon}{4\Delta P} \qquad (9)$$

where K is the permeability $[m^2]$, r_f is the flow front radius, r_0 is the inlet port radius, t is the time, η is the fluid viscosity, ε is the porosity, ΔP is the inlet and outlet pressure difference [Pa]. In the experiment, r_f was measured against t, and the terms in the bracket of equation 9 (N) was plotted against time at different flow front positions (Figure 6(b)). Then, the slope of the resulting linear regression line, F_i , was multiplied by $n\varepsilon/4\Delta P$ to calculate material permeability. The resulting permeability of the polyamide veil, 2.3×10^{-10} $(\pm 2.6 \times 10^{-12})$ m², was an order of magnitude greater than that of the carbon fiber fabric, 2.5×10^{-11} $(\pm 4.6 \times 10^{-13})$ m². The permeability value obtained for 3 K plain weave carbon fiber fabric was similar to a previously reported value.³⁹ The goal of this permeability analysis was to verify that the relative permeability difference between the carbon fiber fabric and the thermoplastic veil was large enough to create nonuniform flow fronts during the infusion process.

Using the measured permeability values, resin infusion through a multilayer preform containing 16 plies of carbon fiber fabric and 7 plies of thermoplastic interlayer was simulated using a commercial software (PAM-RTM). Figure 7 shows the simulated resin filling factor (degree of saturation) gradient during infusion. As expected, the simulation predicted more rapid saturation of the veils than the carbon fiber fabrics, creating non-uniform flow fronts. The infusion simulation demonstrates that the degree of permeability difference between the reinforcement and thermoplastic interlayer is sufficiently large to expect the veils to function as effective interlaminar flow distribution media, as first suggested by Nash et al.²

Cure cycle modification & part manufacture

For thermoset composites toughened by thermoplastic interlaminar veils, results in the previous section demonstrated that the interlayer veils can act as interlaminar flow distribution media. During infusion, resin flows more rapidly through the highly permeable veils than through the reinforcement fabrics and accrues in interlaminar regions, creating resin-rich interlaminar thickness.2,8 regions and increasing inter-ply Moreover, local variations in preform permeability induce distorted flow fronts, resulting in air entrapment and dry spot formation, particularly within fiber tows.^{13,50-52} The formed intra-tow voids tend to migrate to resin-rich regions such as veil interfaces, as formation of voids in resin-rich areas is energetically more favorable than formation of voids within fiber bundles.^{50,53,54} Hence, for laminates containing interply thermoplastic veils, porosity eventually concentrates in resin-rich interlaminar areas.

Because the top mold surface in VI is a flexible vacuum bag, preform thickness can accommodate and adapt to the continuously evolving compaction pressure gradient during infusion and post-filling stages. During the post-filling stage (*i.e.*, the period between infusion completion and resin gelation), the resin pressure field gradually becomes homogeneous, while the compaction load on preform increases.¹² As a result, laminate thickness (in this case, especially the inter-ply thickness, which increased during the infusion stage) decreases, and resin continues to flow and redistribute until reaching an equilibrium state, facilitating impregnation of unsaturated regions including macro-pores.^{2,12,22,23} Insufficient post-filling time can thus result in porosity in interlaminar regions of cured laminates containing thermoplastic interlayers. Nash et al. suggested that increasing resin flow time *may* reduce porosity,² although the hypothesis was not tested.

The rapidly advancing degree of cure and viscosity observed during part production using the MRCC (120 minutes at 180 °C) may not always suffice to fully saturate dry spots during the post-filling stage.



Figure 7. Infusion simulation results for a multilayer laminate containing 16 plies of plain weave carbon fiber fabric and 7 plies of polyamide veils, expressed in filling factor (degree of saturation) gradient.



Figure 8. Profiles of the MRCC (dashed) and the modified cure cycle (solid) showing temperature and predicted profiles of (a) degree of cure and (b) mechanical viscosity, and (c) ion viscosity measured during Case C (modified cure cycle) part manufacture.

The time to attain the equilibrium state of resin is inversely proportional to resin viscosity, while resin gel time limits the time window available for resin redistribution.²² To address this potential problem, an additional post-infusion dwell step at low temperature (129°C) was introduced in the process cycle to prolong the period of low-viscosity prior to gelation. The additional dwell was intended to promote resin flow into partially saturated interlaminar regions that remain after infusion completion. Figure 8(a) and (b) show the temperature profiles of the MRCC (dashed) and modified cure cycle (solid), as well as the degree of cure and viscosity profiles predicted by the cure models. In the modified cure cycle, the cure temperature was raised to 129 °C from the fill temperature (120 °C) and held constant until gelation, followed by a one-hour final cure dwell at 180 °C. The viscosity model predicted resin gelation after 160 minutes of the 129 °C dwell, while the cure kinetics model predicted that both MRCC and modified cycle would yield a final degree of cure (α_f) of 0.97.

The mechanical viscosity model provided an estimate of gel time at the desired cure temperature. However, the actual duration of the post-infusion dwell (in this case, the gel time) was determined using the DCM system in conjunction with the resin cure maps. The DCM system provided in situ measurements of ion viscosity during the cure process, while the cure maps were used to identify specific cure states of interest (in this case, the gel point) and adjust process time in situ. The ion viscosity model and resin cure maps predict that at 129 °C, the ion viscosity reaches 9.95 at the gel point ($\eta \sim 10^3$ Pa · s or $\alpha \sim 0.63$). While producing Case C sample with the modified cure cycle, this ion viscosity value of 9.95 was attained 170 minutes into the post-infusion dwell (Figure 8(c)), which was similar to the mechanical viscosity model prediction of 160 minutes. The slight difference between the predicted and measured gel times ($\sim 10 \text{ minutes}$) was attributed to the difference in sample sizes used for rheology tests and for part manufacture.

The main objective of this study was to determine and demonstrate the effectiveness of a post-infusion dwell in reducing porosity in vacuum-infused laminates containing thermoplastic interlaminar veils. During the post-infusion dwell, the resin remained in the lowviscosity region longer, allowing more time for dry spot saturation throughout the post-filling stage. The addition of the post-infusion dwell can facilitate manufacture of autoclave-quality part using VI at much lower cost. Furthermore, the effectiveness of the postinfusion dwell may not be limited to the specific VI process case discussed here (toughening with nonwoven thermoplastic veil interlayers). In fact, other VI processes may also benefit from longer post-filling times, particularly in cases involving multi-component preforms comprised of different reinforcement materials, and/or three-dimensional preforms featuring complex shapes. In such preforms, local variations in permeability can generate distorted flow fronts, causing air entrapments and dry spots.

Note that the addition of the post-infusion dwell reduced porosity in the toughened laminate at the cost of increased cure cycle time (from 2.5 to 4 hours). However, in practice, the entire process of VI generally consists of multiple stages, including preform fabrication and lay-up, vacuum bagging, resin infusion and cure, and post-cure machining. Thus, the cure cycle itself may represent only a minor fraction of the net manufacturing time, and extending the duration of the cure cycle may have negligible effect on the total process time. Admittedly, further process optimization may trim the duration of the cure cycle, possibly by increasing the post-infusion dwell temperature. In this study, the dwell temperature (129 °C) was selected based on resin gelation data (Table 3) to ensure sufficiently long post-filling duration for dry spot saturation. (At 120 and 140 °C, the resin gelled after 270 and 100 minutes.) In practice, the post-infusion dwell temperature can be adjusted to optimize the cure cycle. An increase in dwell temperature (e.g. 140 °C or higher) can reduce both the minimum viscosity level and the processing time, but only at the cost of accelerating the evolution of resin viscosity and abbreviating the postfilling stage, which can increase porosity. Additional work is required to the explore trade-offs between reducing the overall process time (by decreasing the post-infusion dwell period) at the expense of increasing risk of porosity.

Part quality analysis

Porosity analysis. Cross-sectional images of the vacuuminfused 16-ply laminates are shown in Figure 9. Case A samples contained no thermoplastic interlayers and were nearly void-free. Case B and C samples, which were toughened by 7 plies of thermoplastic veils, showed substantial increases (~0.1 mm per one ply of veil) in interlaminar thickness, with veil fibers embedded between plies. The toughened laminates produced using the MRCC (Case B) exhibited high internal porosity ($\sim 6.5\%$ by volume). As reported elsewhere,² voids were elongated and aligned along fiber directions in inter-ply regions, indicating that the voids were originally located within fiber bundles and later migrated to energetically favorable resin-rich interlaminar areas. High laminate porosity can degrade mechanical properties of cured parts, and thus should be minimized or eliminated.55,56 Case C samples, fabricated using the modified cure cycle, showed a marked decrease in



Figure 9. Cross-sectional micrographs of the vacuum-infused laminates (Case A: containing no veils and manufactured using the MRCC, Case B: toughened by veils and manufactured using the MRCC, and Case C: also toughened by veils but manufactured using the modified cure cycle).



Figure 10. (a) Images and (b) ultrasonic C-scan results of the impacted laminates after drop tower impact tests.

porosity. A few elongated voids still appeared between plies, but these were much narrower and smaller, demonstrating that the introduced post-infusion dwell effectively increased resin redistribution and saturation of partially filled regions during the post-filling stage. The overall void content of Case C remained less than 0.5%. *Impact testing.* The key function of the interlaminar veils was to promote resistance to impact damage, a common concern in structural thermoset composites. Figure 10(a) and (b) show images and C-scan results of the laminates impact tested at 30 J energy level. Visual inspection and C-scan analysis of the impacted laminates demonstrated that Case A, which was not



Figure 11. Force-time profiles of Case A, B, and C laminates measured during 30-J drop tower impact tests.

toughened by the thermoplastic interlayers, exhibited 30 and 50% greater damage area than Cases B and C, respectively.

The force-time history curves obtained during the 30-J impact test were characterized by two distinctive peaks (F_1 and F_{max}), as shown in Figure 11. The first load drop (F_1) was associated with initial damage, specifically with the onset of delamination from indentation and local matrix cracking near the impacted region. The peak force (F_{max}) was ascribed to the maximum tolerable load before extensive global delamination to the laminate.^{57–59} The F_1 values for Case B and C (4790 \pm 120 N) were approximately 9% greater than that of Case A (4410 \pm 120 N), indicating that the toughened samples exhibited greater resistance to initial local impact damage. The F_{max} values of Case A, B, and C were 4890 (±120), 5790 (±190), and 6280 (±90) N respectively, and the toughened laminates also showed greater resistance to global delamination, which arose from extensive matrix cracking. In addition. Case C exhibited much lower void content than Case B (<0.5% vs. 6.5%), resulting in 8% greater peak load during impact.

Conclusions

The use of thermoplastic interlaminar veils in VI preforms increases impact resistance but also induces nonuniform flow fronts, leading to porosity. Hence, the conventional cure cycle was modified to include a low-temperature post-infusion (LTPI) dwell to ensure full saturation of dry interlaminar regions. Process parameters for the LTPI dwell were informed by predictions from models for the cure kinetics and viscosity, and *in situ* process adjustments were implemented using the DCM system in conjunction with the resin cure maps. Laminates produced using the modified process cycle exhibited lower porosity and greater peak load during impact testing.

This study explores the effectiveness of a postinfusion dwell on part quality in vacuum-infused laminates with interlaminar veils. Resin cure maps can be employed to identify key junctures in the physical state of the resin, while the use of DCM permits in situ monitoring of the resin state during VI. Thus, DCM can identify key junctures in resin state, particularly the gel point, which can be used to guide in situ process adjustments. Interlaminar thermoplastic veils serve two functions: (a) enhanced impact resistance and (b) inter-ply flow distribution media. However, the latter function also increases the risk of porosity. The veils introduce local variations in permeability and give rise to non-uniform flow fronts during infusion. To mitigate porosity that can arise from the distorted flow fronts, the LTPI dwell can be introduced to extend the postfilling stage. During the dwell, resin can redistribute and saturate interlaminar regions prior to gelation, reducing porosity in cured laminates.

Overall, this work describes a potential pathway to address high porosity commonly encountered in various VI processes, arising from local variations in preform permeability, disparity in flow rates in different layers, and non-uniform or distorted flow fronts. Composites manufacturing is gradually transitioning from expensive conventional methods such as autoclave processing to simpler and more cost-effective yet relatively less robust (thus far) processes including VI. Introducing a LTPI dwell based on material characterization, resin cure map development, and *in situ* cure monitoring may provide a viable solution toward increasing part quality of vacuum-infused laminates.

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