In situ estimation of through-thickness resin flow using ultrasound

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Abstract: Ultrasonic imaging in the C-scan mode was used to measure the flow rate of an epoxy resin film penetrating through the thickness of a single layer of woven carbon fabric. Assemblies, comprised of a single layer of fabric and film, were vacuum-bagged and ultrasonically scanned in a water tank during impregnation at 70 °C. The permeability of the fabric was calculated using Darcy's law. The results demonstrated that ultrasonic imaging in the C-scan mode is an effective method of measuring z-direction resin flow through a single layer of fabric. Comparison of ultrasonic and microscopy images yielded consistent results and demonstrated the effectiveness of ultrasonic imaging as an in situ process diagnostic for monitoring through-thickness impregnation and flow rates.

Key words: Out-of-autoclave; A. Carbon fibres; D. Ultrasonics; D. Optical microscopy

1. Introduction

1.1. Summary of VBO Processing

Traditional processing of high-strength polymer matrix composite materials requires autoclave consolidation and curing at high pressures and temperatures (pressures of several atmospheres and temperatures above 100 °C). A new class of vacuum-bag-only (VBO) prepregs has been introduced for processing at much lower pressures and temperatures. VBO prepregs are intended to yield

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autoclave-quality parts without the use of an autoclave, and are thus a member of the family of out-of-autoclave (OOA) techniques for composite manufacture. In the VBO process, composite laminates are produced from prepregs by vacuum-bag consolidation followed by curing in an oven at atmospheric pressure. Like resin film infusion (RFI), the VBO prepreg is produced by impregnating dry fabric with a resin film. However, unlike RFI, the VBO prepreg is produced by partial impregnation, leaving vacuum channels for air escape during subsequent consolidation in a vacuum bag. Eliminating autoclave processing simplifies the manufacturing process and greatly reduces operational and capital equipment costs.

One purpose of the autoclave is to apply pressure greater than the vapor pressure of volatile components. Preventing gasses from evolving from the resin during the cure cycle produces a dense structure free of porosity. In RFI and VBO processing, unlike autoclave processing, there is no high pressure to force volatile gasses to remain dissolved in the resin, and the maximum applied pressure is only 1 atm. Therefore, all of the gasses evolved must be removed prior to resin curing, and the cure process cannot produce volatile reaction products. The maximum fiber volume fraction of a composite laminate fabricated from VBO prepregs is limited by the low pressure that is characteristic of the process. Fiber volume fractions up to 65% for unidirectional composites and 52–57% for composites made with fabrics have been achieved [1].

1.2. In situ methods for measuring resin flow

Understanding resin flow and impregnation of the reinforcing medium is critical to the manufacture of consistent composite laminates free of porosity and flow-related defects. Although resin flow measurements are generally not performed in prepreg processes, they are routinely performed in resin transfer molding and related liquid molding processes. For example, fiber optic sensors, used in the transmission or reflection modes and embedded at various locations in the layers of reinforcing

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material, have been used to measure resin flow in resin transfer molding (RTM) processes [2] and [3]. Optical fibers can be fragile, however, and fiber optic sensors are not as sensitive to resin flow inside fiber tows, so alternative approaches are often employed.

Resin flow in RTM processes is often measured by employing a transparent mold, allowing one to visually monitor and record the advance of resin flow fronts as the mold is filled [4], [5], [6], [7] and [8]. In this approach, the flow rate through and around the stack of fabric or plies is measured directly, while the flow rate through the fiber tows is measured indirectly by a non-linear pressure-time profile [4]. The video recording method works well for measuring the bulk flow rate in in-plane RTM processes, in which the mold is initially dry (provided also that the mold is transparent). However, video recording methods are not useful for measuring flow rates in prepregs, where impregnation occurs primarily in the through-thickness direction.

Microstructural analysis of composites produced by RTM has also been used to study impregnation kinetics and to provide a basis for model simulations [9]. This work provided the basis for a permeability model for RTM processes [4], as well as two-dimensional elliptic flow models [10] and [11]. Attempts to measure transverse flow have met with limited success. Wu, Li, and Shenoi demonstrated a simple method of measuring transverse flow in which fibrous material was wound in a coil and resin was injected through the center [12]. By observing the flow front, the flow rate and permeability could be determined. However, if gaps existed between fiber tows, as in most woven fabrics, the rapid flow of resin through the gaps prevented determination of flow rates and permeability. Using a different approach, transverse or through-thickness flow measurements have been performed by pumping fluid through a porous sample at a prescribed rate. Permeability was determined from a linear plot of the pressure drop as a function of flow rate [13] and [14].

An alternative approach to measuring resin flow during impregnation involves the use of ultrasonic imaging, which has been used in the transmission mode to study the flow front of resin in an RTM process [15]. The samples were cured at different stages of the impregnation process and imaged to determine the flow pattern. In other work, ultrasonic transmission with air coupling was used to measure the impregnation and fill rate of the preform in a resin transfer molding process [16]. The ultrasonic transmitter and receiver were aligned on opposite sides of the mold. The mold fill rates determined ultrasonically were equivalent to those determined visually. This ultrasonic transmission method is well-suited to situations in which the transducers cannot be immersed and do not require immersion in a coupling fluid. The ultrasound signal resolution achieved with air coupling is generally inferior to liquid coupling, however [17].

In the present work, we utilize ultrasonic imaging and reflectivity to monitor the impregnation of fabrics in situ (reflectivity is the amplitude of the reflected signal). An assembly consisting of fabric, resin film, and vacuum bag is immersed in a heated water tank while performing C-scan imaging to produce a density map or profile of the scanned material. The method relies on the acoustic contrast provided by scattering from wet and dry regions of the fabric. As the resin flows into the fabric and permeates the fiber tows, the volume of the material system decreases and the density increases [18]. The volume and density of the material stop changing when the resin stops flowing, and this is detected in sequential C-scan images, which cease to change when fiber wetting is complete and resin flow ceases. The procedure provides a non-invasive, in situ inspection method for monitoring resin impregnation of fiber tows.

The capabilities and limitations associated with this technique are considered and analyzed. The primary intent is to demonstrate the utility and limitations of the method for monitoring through-thickness resin flow in RFI, VBO prepregs, and other non-autoclave processes – not to investigate

the effects of process parameters or to detect micro-porosity. (Future work will address the influence of process parameters on resin flow, such as temperature, multiple layers of prepreg, and fiber architecture). The ultrasound data are used to calculate permeability using Darcy's law.

Darcy's law relates the flow rate of a viscous fluid to the permeability of the porous material. The flow rate V (m/s), permeability, K (m2), pressure gradient, ∇P (Pa/m), and viscosity μ (Pa∗s) are related by

$$K = \frac{\mu V}{\nabla P} \qquad (1)$$

Flow into the fiber tows is two-dimensional. The cross-section of the fibers tows is approximately elliptical and the relationship between elliptical and Cartesian coordinates is well established [19] and [20]. A diagram of an elliptical fiber tow is shown in Fig. 1.

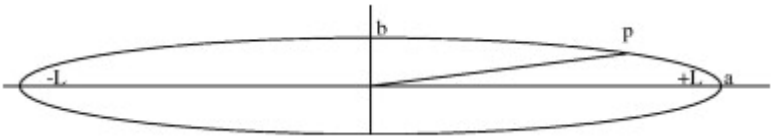

Figure 1: *Diagram of elliptical fiber tow with coordinates.*

The sum of distance from the positive and negative values of the foci (L) to any point (p) on the surface of the ellipse is equal to 2a. The major and minor coordinates of the ellipse (a,b) and their relation to the Cartesian coordinates (x,y) are defined below.

$$a = L\cos h(\xi), \quad b = L\sin h(\xi), \quad x = a\cos(\eta), \text{ and } z = b\sin(\eta)$$

$$\xi = (\overline{+Lp} + \overline{-Lp})/2L = a/L, \eta = (\overline{-Lp} - \overline{+Lp})/2L$$

Values of the elliptical parameters are listed in Table 1.

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Table 1: *Elliptical parameters.*

a (mm)	b (mm)	$\pm L$ (mm)	ξ	H	X (mm)	Z (mm)
0.825	0.075	± 0.822	1.00	0.60	1.05	0.55

2. Experimental

The materials selected for the study included a plain-weave carbon fabric (elastic modulus and tensile strength of fibers = 231 GPa and 3650 MPa) containing 3000 fibers per tow (density = 194 g/m2) and an epoxy resin film (Cycom 5215, density = 102 g/m2). A single layer of resin film on a single ply of woven carbon fabric was used in all trials. A two-step process was employed in which the resin film was first attached to the fabric by lamination, which was followed by impregnation in a vacuum bag assembly. The lamination procedure involved removing the release liner from the resin film, placing the resin film and protective paper layer on top of the fabric, placing the laminate on a Teflon® coated flat surface preheated to 50 °C, applying a pressure of 1517 Pa (15 mbar) for 2 s, rapidly cooling to room temperature, and removing the protective paper. The lamination process was followed by the second step of the process – full impregnation by vacuum bagging at 70 °C. Preliminary experiments were conducted to determine the depth to which the resin film penetrated the carbon fabric during the lamination step (prior to impregnation).

2.1 Lamination

Sections of resin film, 254 mm × 254 mm, were cut from a large roll and lightly pressed onto a piece of plain-weave carbon fabric of equal dimensions at room temperature. Next, lamination was performed using all combinations of the process parameters shown in Table 2. An alternative lamination procedure involved ironing the resin film onto the fabric, although pressure, temperature, and time were not determined during the ironing process.

Table 2: *Lamination process parameters*

P (Pa)	1517	15170	15170
T (°C)	50	60	70
t (s)	2	10	30

Laminated samples were cured, sectioned, and polished prior to microscopic examination to determine the resin penetration depth during lamination. When the resin film was laminated to the fabric at 1517 Pa (15 mbar) and 50 °C for 2 s, resin flow through the fabric and into fiber tows was minimal. These conditions were used for lamination of all samples. In samples thus produced, the resin film was lightly laminated to one side of the 0.254-mm thick carbon fabric.

2.2 Capillary flow measurements

The release liner was removed from a 100 × 100 mm sheet of resin film that was lightly pressed onto a 100 × 100 mm piece of carbon fabric at room temperature. The sample was placed in an oven for 20 min preheated to 70 °C without applied pressure. The samples were cured, sectioned, mounted, polished, and examined. Microscopic examination revealed that the resin did not flow into the fabric, indicating an absence of capillary flow.

2.3 Impregnation

RFI samples were vacuum-bagged, out-gassed, and heated under full vacuum of 100 kPa (1000 mbar) for 3, 9, and 15 min in the water tank at 70 °C. Flow of the resin was halted by immediately removing the samples from the heated water tank, venting the vacuum-bagged samples, and quenching in cold water. The samples were cured, and polished sections were examined by light microscopy.

2.4 Measuring flow

Ultrasonic imaging was performed to measure the extent of resin flow through a single-ply RFI sample during the impregnation process. The experimental configuration consisted of a thin epoxy resin film laminated to a single ply of woven carbon fabric. The fabric rested on an aluminum base plate, and the fabric-film assembly was sealed in a vacuum bag, as shown in Fig. 2.

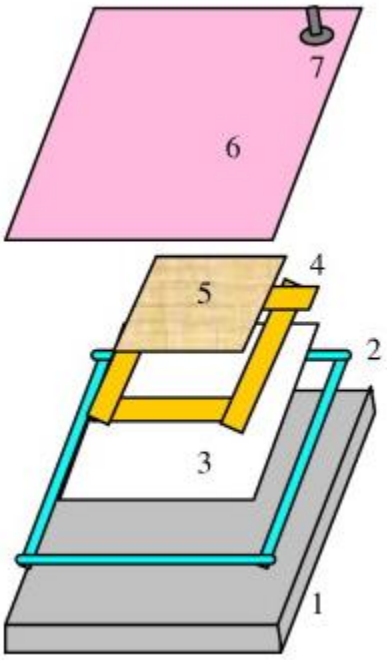

Figure 2: *Diagram of the vacuum bagging process for measuring resin flow by ultrasonic imaging. The assembly consists of: (1) aluminum base plate, (2) sealant tape, (3) Teflon® film, (4) breather cloth, (5) RFI sample with resin film on top of the fabric, (6) vacuum bag film, and (7) vacuum valve.*

The resin film comprised 34% by weight of the fabric-resin system. The assembly was immersed in a heated water tank designed for ultrasonic C-scan imaging, and imaging was performed to monitor the impregnation while vacuum of 100 kPa (100 mbar) was applied to the assembly. A commercial ultrasound system (Physical Acoustics Ultrapac II) was used for the ultrasonic imaging. A 0.5-in. diameter (12.7 mm) 10 MHz focused transducer (focal length 38.1 mm, focal point 1 μm) was used for scanning in the reflected mode. The beam diameter at half a wavelength was 24 μm. The gates were set to record signal between the top of the sample and bottom of the base plate. A constant

signal was produced from the base plate; so that any change in the signal resulted from a change in the sample. The amplitude of the signal was set to 90% of the maximum on the base plate.

First, the assembly was placed in a water tank (60-cm long × 45-cm wide × 15-cm deep) at 20 °C to measure the initial condition. The A-wave scanning parameters for ultrasonic imaging were established on a flat region of the vacuum bagged assembly near the ply. The signal amplitude was plotted against time. A support fixture was used for the vacuum bagged assembly that allowed removal and re-insertion in the water tank at the same position. A 25.4 mm × 25.4 mm area near the center of the ply was scanned in the C-scan mode in one minute at 20 °C to document the initial condition. The assembly was removed from the water tank after the initial condition was recorded.

In preparation for flow measurements, the water tank was heated to the desired temperature and maintained to within ±1 °C. The vacuum-bagged sample was re-inserted into the heated water tank. Flow of the resin through the carbon fabric was measured at 70 °C at three-minute intervals until the C-scan image was unchanged from the previous interval (see below). Measurements of the initial condition at 20 °C and flow of the resin at 70 °C were repeated on three samples. A constant pressure of 100 kPa was applied to the vacuum-bagged plies during the flow measurements using a mechanical pump. A thermocouple was embedded under the vacuum-bagged sample. The samples reached 70 °C in approximately 40 s after immersion into the heated water tank and the temperature remained constant during the flow measurements.

The amplitude of the reflected signal was monitored and plotted as a function of time during impregnation. The cessation of resin flow was determined by comparing successive scans, and when the reflectivity of the last two consecutive scans changed by less than one percent, resin flow was taken to be complete. Permeability was then calculated from Darcy's law, the general form of which is given by

$$K = \frac{\mu V}{\nabla P} \quad (1)$$

The pressure gradient ∇P, in two dimensions is approximated as ΔP/x + ΔP/z [11] and [14].

The C-scan images are affected by multiple factors, including density and thickness variations of the fabric, porosity, temperature, and the gain setting. For example, the amplitude of the signal decreases as the temperature increases. Once a constant temperature is reached, the gain must be increased to an appropriate setting. If the gain is not set appropriately, then the gradual change in the ultrasound reflectivity caused by permeating resin will not be detected. Note that the amount of porosity at any given time is not determined from the C-scans, nor was that the intent of this investigation. Instead, the purpose was to measure average flow rates by determining when the resin flow ceased.

3. Results and discussion

Representative C-scan images recorded at 70 °C are shown in Fig. 3. Red indicates a reflected signal intensity of approximately 10%, yellow: approximately 50%, and dark green: approximately 90%. Intermediate colors between red and yellow indicate reflected intensities between 10% and 50%, whereas intermediate colors between yellow and dark green indicate reflected intensities between 50% and 90%. The uniformly spaced green regions of the initial scans correspond to inter-tow gaps in the woven fabric. The resin flows relatively quickly between tows and fills these gaps (macro-flow), then begins permeating the fiber tows, saturating the tow edges first, where the tows are thinnest (the tows are oblate ellipses in cross-section). After penetrating through the inter-tow gaps, the resin flows simultaneously from the top and bottom of the fabric.

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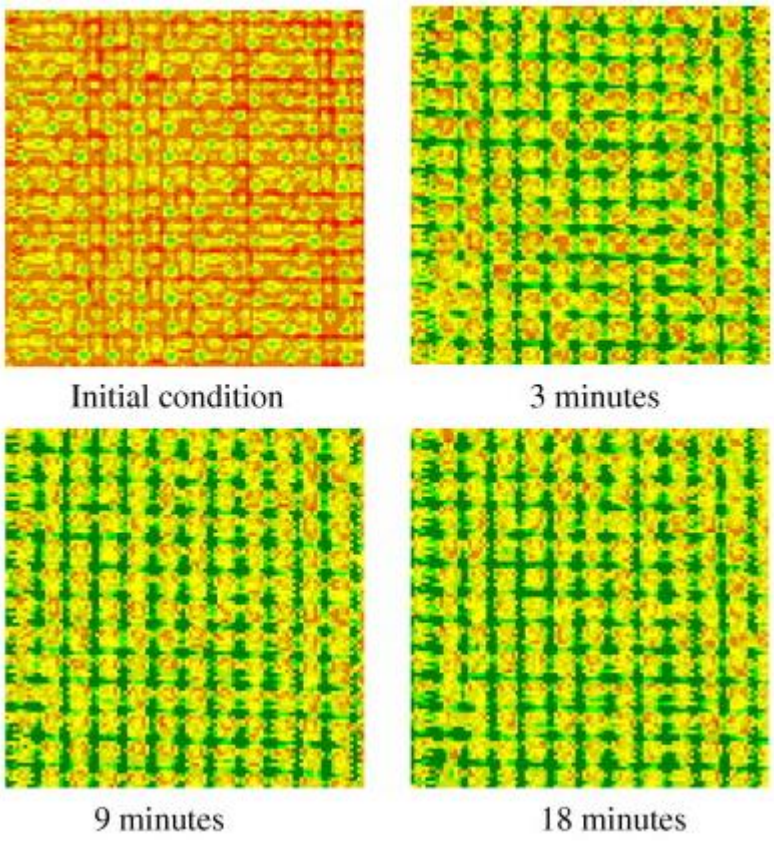

Figure 3: *C-scan images at 70 °C of a vacuum bagged ply.*

The initial condition of Fig. 3 shows that the fabric is not entirely uniform. Thicker regions appear red, thinner regions yellow, and gaps between the fiber tows appear light green. The individual fiber tows are naturally thickest at the centers and thinnest at the edges. The non-uniformity of thickness and density of the fabric is apparent throughout the series of scans. Thicker regions absorb more of the ultrasonic signal than thinner regions. Therefore, one should not expect the C-scan of a fabric fully impregnated with resin to appear dark green. In addition, regions of higher density reflect more of the signal than regions of lower density. Thus, C-scan images provide a qualitative measure of resin flow and determination of when resin permeation ceases.

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Flow at 70 °C: Reflectivity measurements at 70 °C show that the resin stopped flowing after 15 min in samples 1 and 3 and after 18 min in sample 2, yielding an average flow time of 16 min (±1.73 min). Fig. 4 shows plots of normalized reflectivity versus time for the three samples. The greatest change in reflectivity takes place during the initial few minutes, when inter-tow "macro" flow occurs. Subsequently, the resin penetrates the tows ("micro" flow), resulting in gradual increases in ultra-sound reflectivity. Impregnation was considered complete when the reflectivity in sequential scans varied by less than 1%.

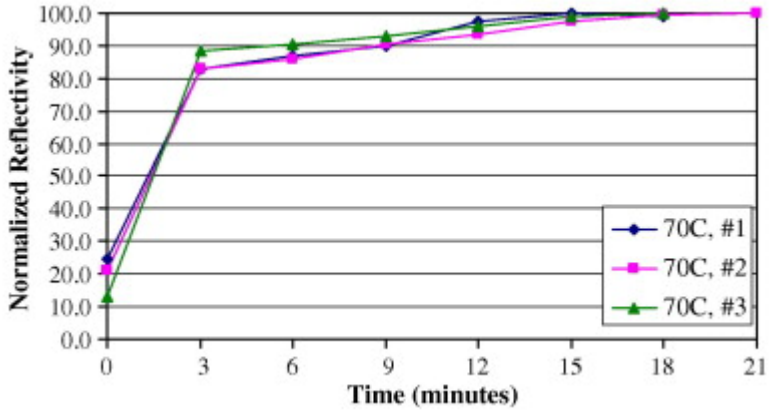

Figure 4: *Normalized reflected signal as a function of time at 70°C.*

The three profiles are similar, with only minor variations. Variability of the flow rate is attributed to non-uniform distribution of the fibers in the fabric, a consequence of fabric distortion during RFI sample fabrication. The standard deviation of the flow time is approximately 11% of the mean value. Based on the 95% confidence interval for a sample size of 3 (View the MathML source ± 4.303sm), the resin should completely fill the fabric within 16 ± 4 min. Achieving full impregnation of the fabric in the shorter time interval is unlikely because complete permeation of the fabric was not observed in less than 15 min. Furthermore, the resin viscosity increases with time, albeit quite

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slowly. (The gel time is 5–6 h at 70 °C, so the change in viscosity in the first 20 min was assumed to be negligible.)

Flow rates measured from the reflectivity data are summarized in Table 3. Permeability values for the fiber tows calculated from Darcy's law are also summarized in Table 3. Note that these values are not necessarily equivalent to bulk permeability values. Bulk permeability cannot be determined for a single layer, because the Teflon® film on the bottom of the carbon fabric provides a flow resistance different from an adjacent ply of carbon fabric. However, bulk permeability in laminates made from prepregs is less relevant than tow permeability, primarily because the region surrounding the prepregs is saturated with resin when the laminate is laid up and fiber nesting reduces the likelihood of contiguous through-plane porosity. While bulk permeability is somewhat important for air removal through resin bleeding, air is normally removed from the laminate by application of vacuum during vacuum bagging. Thus, vacuum-bag processing of prepregs relies on removing all of the air and vapor from the laminate before the vacuum channels are sealed off by flowing resin.

Table 3: *Flow properties at 70 °C determined by C-scan. Viscosity at 70 °C = 25.1 Pa∗s, ΔP = 100000 Pa.*

Property	#1	#2	#3	$\bar{x}$	σ
Flow time (min)	18	15	15	16	1.73
V_x (10^{-7} m/s)	9.72	11.7	11.7	11.0	1.14
V_z (10^{-7} m/s)	5.09	6.11	6.11	5.77	0.60
K_x (10^{-13} m^2)	2.56	3.08	3.08	2.91	0.30
K_z (10^{-14} m^2)	7.03	8.43	8.43	7.96	0.81

Figs. 5a and b show polished sections of vacuum-bagged samples removed from the 70 °C water bath after 3 and 9 minutes. The original position of the resin film was on one side (top) of the fabric. Dark areas between the fibers indicate porosity. The images show that the porosity is predominantly in the central regions of the tows, indicating that the resin flowed from the sides as well as from the

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top. The resin flowed into the tows from the top, but simultaneously, the resin flowed through inter-tow gaps, and along the opposite (lower) side of the fabric, encapsulating the tows.

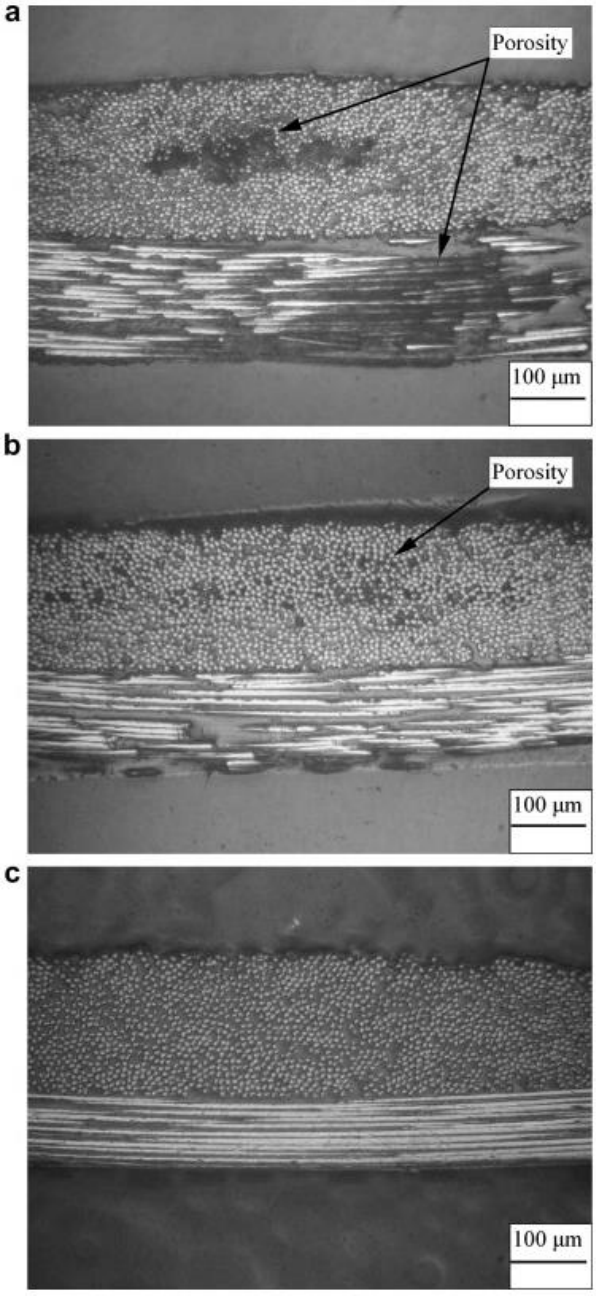

Figure 5: *(a) Full vacuum for 3 min at 70 °C. (b) Full vacuum for 9 min at 70 °C. (c) Full vacuum for 15 min at 70 °C.*

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Microscopic observations revealed that some of the fiber tows were fully impregnated with resin after 3 min at 70 °C. A fully impregnated fabric representative of vacuum-bagged samples removed from the 70 °C water bath after 15 min is shown in Fig. 5c. The number of fiber tows fully impregnated with resin gradually increased with time at both temperatures. As noted above, the resin completely impregnated the fabric after 16 min at 70 °C. Tow permeability values in the x and z directions were 2.91 × 10−13 and 7.96 × 10−14 m2 at 70 °C, and the average was 1.53 × 10−13 m2. The data are summarized in Table 3.

Comparison of the microscopy with the C-scan images revealed qualitative agreement between the two imaging modes. Both image sets revealed that the resin gradually flowed into the fiber tows until flow ceased after 16 min at 70 °C. Furthermore, both image sets also show that the fiber distribution in the fabric was not uniform. However, the two imaging techniques are complementary rather than redundant. C-scan imaging provides a non-destructive in situ method of monitoring resin permeation and determining when resin flow ceases. The microscopy images, on the other hand, furnish insight into the patterns of resin flow within and between tows. While the resolution is superior to the ultrasound images, these images are effectively “post mortem”.

The values of tow permeability for the woven carbon fabric determined in this work are similar to values reported previously. For example, the tow permeability values, Kz and Kx, for the plain woven fabric measured here were 7.96 × 10−14 and 2.91 × 10−13 m2, while values determined by model-based calculations were 3.25 × 10−14 m2 and 2.47 × 10−13 m2 [21]. The porosity of the fabric used for the model-based calculations was 21.6%, whereas the porosity of the fabric used in this study was 33%. Porosity of the fabric is the void space between the fibers before impregnation by resin. Material with a greater porosity should have a greater permeability.

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The similarity in measured and reported values of permeability is encouraging, yet it may also be fortuitous, given the significant differences between the two studies. Accurate determination, whether by measurement or calculation, presents a challenge. For these reasons, experimental process diagnostics in general, and in situ ultrasonic imaging in particular, are useful and necessary tools for measuring permeability, understanding impregnation, and validating model predictions.

4. Conclusions

Ultrasonic imaging and reflectivity in the C-scan mode was used to measure the flow rate of epoxy resin through a single layer of plain-woven carbon fabric. The ultrasonic reflectivity signals were used to determine when resin flow through the fabric ceased, while microscopy observations provided insight and details of how the resin flowed through the fabric. The microscopy images clearly revealed that the resin did not flow uniformly through the fabric from top to bottom. The information in the C-scans was consistent with the microscopy images, and the measured permeability value was consistent with permeability values for woven carbon fabric determined by other methods.

We have demonstrated that conventional C-scan ultrasonic imaging and reflectivity can be used to monitor through-thickness resin flow in fiber arrays. In situ measurement of resin impregnation and consolidation in thicker, multi-layered laminates should be possible with the methods described in the present work. Furthermore, the method can be used to determine the effects of different process parameters on fabric impregnation and to better understand the processing science underlying this new class of low-pressure, VBO prepregs. The method can also be applied to monitor impregnation and consolidation during the production of large, flat composite parts, provided they are

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impregnated, consolidated, and cured at temperatures below the maximum service temperature of the transducer.

Ultrasound imaging, together with ultrasound reflectivity, is especially useful as a process diagnostic for composite VBO prepregs, primarily because these materials are amenable to low-pressure vacuum-bag processing and do not require high-pressure autoclaves. Employing this tool for process monitoring may reduce defect rates and eliminate the need for final NDE inspection after parts are cured, further reducing production time and cost.

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