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Lab-based *in-situ* micro-CT observation of gaps in prepreg laminates during consolidation and cure

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Abstract

An experimental technique was developed to obtain 3D microstructural information on two typical lay-up features that often appear in automated fibre placement (AFP) or automated tape laying (ATL). Parallel-ply gaps between adjacent courses and ply-drop gaps needed to change the thickness of composite laminates were studied. Using a lab-based CT scanner, the different squeezing and bleeding mechanisms were observed while the manufacturing process was occurring. The initially large gaps were closed by fibre and/or resin flow, with the parallel-ply gap consolidating faster and reaching a lower porosity level than the ply-drop gap. However, after reaching a minimum porosity, previously consolidated voids reappeared and remained locked into the final microstructure. The in-situ CT data was helpful to identify opportunities for future process model developments. Overall, lab-based in-situ micro-CT was proven to be an effective and accessible technique to study how composite features evolve through the manufacturing process.

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1 Introduction

Composite parts are designed to exacting standards, with lay-up guidelines often specifying ply positional accuracy of less than 0.5mm. Parallel gaps wider than 1mm are often found in laminates made by hand lay-up and gaps wider than 2mm are common in automated deposition processes, such as automated fibre placement (AFP) and automated tape laying (ATL). Complex surfaces can be particularly problematic. Gaps are often a result of design decisions to lay unidirectional tape over curved surfaces or to deposit plies along a geometry that will create variable stiffness composites [1]. The gaps required during deposition can have a negative structural impact unless their width reduces during ply consolidation and subsequent part curing.

Ply fibre squeezing, where the fibres and resin move together, and resin bleeding, where resin flows relative to fibre, are possible during consolidation and curing of parallel gaps. However, resin and fibre flow are restricted where plies end, such as at ply-drops found in laminate thickness changes due to tapering and pad-up regions. The gaps introduced at ply terminations are expected to be filled by a combination of resin bleeding flow and bending of adjacent plies above and below the gap. Resulting resin rich regions and fibre waviness are often classified as defects [2] and have been shown to reduce the structural properties of the final composite product [3]. Better techniques to describe how features introduced during ply deposition evolve through manufacture are needed to inform design specifications and manufacturing choices, particularly with respect to defining defect tolerances.

Composite process modelling offers the possibility to test a much larger range of combinations virtually and to optimise the processing parameters in order to minimise defect sizes in a cured component [4]. Flow and compaction models have been proposed to predict the final thickness of composite laminates by accounting for the relative motion between resin and fibres when pressure is applied [5-7]. The model recently proposed by Belnoue et

al. [8] takes account of both bleeding and squeezing flow phenomenon that occur during consolidation and cure of composite materials. The model has been shown to capture the final fibre path geometry that arises due to parallel ply gaps and overlaps introduced during AFP deposition, and how the position of parallel ply gaps and overlaps can minimise waviness [9].

Process model validation is often hindered by the availability and fidelity of experimental data. Simulations have historically been compared to micrographs taken at discrete points in the manufacturing process, often the finished state. Microstructural information throughout manufacture would help validate existing process models and identify areas requiring improvement.

In-process visualisation techniques have been developed to observe material deformation during the manufacturing process. Transparent glass tooling can provide real time information of surfaces by video camera [10-12] to help understand how consolidation and evacuation process phenomena occur. However, to really see inside a material, X-ray computed tomography (CT) has become an increasingly attractive option to quantify 3D microstructural features of composite materials [13-15]. Ex-situ CT scanning has been demonstrated as a viable technique to evaluate composite materials at different stages of the manufacturing process by quenching semi-cured samples [16-17]. Variability between samples has been overcome with in-situ CT techniques that track a feature in a single sample through the entire manufacturing process, such as a bubble of water during the cure of neat resin [18] and swelling of unidirectional fabrics during infusion [19]. If higher magnification images and/or shorter time steps are required, synchrotron sources have been demonstrated to study resin infusion [20-21], prepreg debulking [22], and prepreg compression moulding [23] to provide a clearer picture of how materials flow and compact during the manufacturing cycle.

This paper builds on the existing literature investigating consolidation of uncured composite laminates. Gaps introduced during deposition of unidirectional preregs are investigated through the consolidation and cure stages of composite processing. The new contribution is an *in-situ* micro-CT testing capability to observe the 3D microstructure of prepreg laminate features using a lab-based CT scanner. The experimental observations are interpreted within the context of state-of-the-art numerical models through the consolidation and curing stages to address in-process data generation and underpinning process model assumptions in composites manufacture.

2 Experimental methods

2.1 Materials

The composite material used in this study is representative of the aerospace industry class of interleaf toughened carbon fibre-epoxy preregs that are used on the Boeing 787 and Airbus A350 XWB commercial airliners. The specific material was Hexcel IMA unidirectional carbon fibres with diameter of 5 microns, pre-impregnated with Hexcel M21 toughened resin. The prepreg had a nominal fibre areal mass of 194g/m^2 , resin content of 34% by weight, resulting in a nominal cured ply thickness of 0.184mm with a fibre volume fraction of 0.59 [24].

The recommended cure cycle for this material is a ramp of $1\text{-}2^\circ\text{C/min}$ to 180°C with a hold for 2h. To minimise through-thickness thermal gradients and resulting process-induced distortions, thick or variable thickness aerospace structures are often heated using slower ramp rates of $0.1\text{-}0.5^\circ\text{C/min}$ and include an intermediate dwell at lower temperatures. Previous studies investigating the consolidation behaviour of this material showed compaction to be independent of temperature above 70°C [25]. Therefore, a cure cycle was defined for the in-situ experiments with a target ramp rate of 0.5°C/min to a temperature of 150°C , where the sample was held at constant temperature for 2h before naturally cooling to

ambient temperature. This cure cycle advances the degree-of-cure to roughly 60%, estimated by the thermal model developed by Mesogitis et al [26], which used the same batch of prepreg as in this study.

The viscosity behaviour of neat resin was measured using a TA Instruments DHR-1 rheometer with an environmental thermal chamber. The sample was rotated between disposable 25mm aluminium parallel plates at a constant gap of 0.75mm, a frequency of 1Hz, and an oscillatory strain control of 10% strain – within the linear viscoelastic region of the material. A sample was ramped at 0.5°C/min to 150°C, and held at this temperature until a complex viscosity of 100,000 Pa·s was reached, in order to identify the timing of minimum viscosity and gelation of the cure cycle used in this study.

2.2 Sample configuration

Disk shaped samples were laid-up by hand using 22 plies of prepreg to form a 20mm diameter laminate; no intermediate vacuum consolidation (i.e. de-bulking) was applied. Two sample configurations were studied:

Parallel-ply gap: A gap approximately 2mm wide was introduced parallel to the fibre direction in the central 2 plies of a $[0/90]_{11S}$ laminate, as shown in Figure 1a.

Squeezing (fibres and resin moving together) is expected to be the dominating mechanism filling the gap in this configuration.

Ply-drop gap: A gap approximately 2mm wide was introduced perpendicular to the fibre direction in the central 2 plies of a $[0]_{22}$ laminate, as shown in Figure 1b.

Bleeding (resin movement relative to the fibre bed) is expected to be the dominating mechanism filling the gap in this configuration.

Bending of the 0° plies above and below each gap is expected to occur in both configurations.

Figure 1. Parallel-ply gap (a) and ply-drop gap (b) sample configurations. The 0° fibres are represented as horizontal lines, with 90° fibres as dots. The arrows in (a) identify the expected direction of fibre squeezing.

2.3 In-situ CT setup

A challenge encountered throughout this study was to include all relevant composite processing conditions (e.g. temperature and pressure) while maintaining a metal-free scan zone between the X-ray source and detector to avoid noise from X-ray scatter. A vacuum line, power extension lead, and USB cable were all passed into the scanner cabinet to enable the in-process CT experimental program and data acquisition. Photographs of the experiment are shown in Figure 2.

Pressure was applied to the sample by a flexible vacuum bag, as shown in in Figure 2a. A vacuum line was inserted into the CT cabinet and connected to a pressure pot, as shown in Figure 2b. A flexible glass fibre reinforced silicone hose was connected the vacuum bag and taped to the CT cabinet ceiling with enough excess length to allow the sample stage to rotate 360° during each scan. The stage counter-rotated to the start position before beginning the next scan to avoid entangling the vacuum line.

A 2mm acrylic disc was placed on top of the laminate and in contact with the breather fabric to replicate a caul sheet. An acrylic ring (approximately 2mm wall thickness) was placed around the laminate to laterally constrain the plies at the boundary. The onset glass transition temperature of the acrylic ring material was measured to be 115°C by dynamic mechanical analysis, but does not melt during these experiments, retaining an elastic modulus of 10MPa at 150°C. The measured elastic modulus of the acrylic was used as a boundary condition in the process simulation.

Figure 2. In-process CT experimental setup. The sample is placed on top of a ceramic rod in (a) with a vacuum line applying a vacuum bag pressure. A breather jacket covers the sample in (b) as X-rays pass through the sample to the detector (not shown). The temperature control and reader are located inside the CT cabinet with the vacuum pot.

The sample was cured under vacuum-bag-only pressure, with total applied consolidation pressure being roughly 100kPa, which is lower than the recommended 700kPa

autoclave pressure for this material. Nevertheless, the consolidation model [8] has been shown to capture the fibre and resin response in the full pressure range of 100–700kPa.

Heat was input to the prepreg laminate by a flexible silicone heater pad wrapped around a ceramic rod, as shown in Figure 2a. Heat was applied from one side only. Temperature at the boundary of the mould and prepreg was measured by a K-type thermocouple placed a few millimetres below the surface, in the centre of the ceramic rod. The temperature was recorded through a National Instruments thermocouple reader connected to LabView software.

The temperature profile in the centre of the laminate was measured in a preliminary heating trial with thermocouples placed at the bottom of the laminate where heat was applied, the laminate middle, and the top of the laminate under the acrylic disk. The measured temperatures from the heating trial are shown in Figure 3. Even though insulation was placed over the heating assembly, as shown in Figure 2b, a through-thickness thermal gradient began to arise around 75°C, reaching a maximum difference of 9°C between the middle of the laminate and the top/bottom plies at the 150°C hold temperature. As a result, the temperature in the middle of the laminate was used to control the in-situ CT experiments.

Figure 3. Temperature profile of laminate disk measured in a preliminary heating test.

2.4 CT scanning parameters and data reconstruction

The in-situ CT scanning was conducted using a Nikon XTH-320 CT scanner with a 225keV reflection head X-ray source and a flat panel detector. Based on the sample size, a resolution of 13.51 $\mu\text{m}/\text{voxel}$ was achieved for the parallel-ply gap sample, and 13.45 $\mu\text{m}/\text{voxel}$ for the ply-drop gap sample. The total power was 13W, resulting in a focal spot size of approximately 13 μm , just smaller than the resolution. Since the maximum magnification is limited by the geometry of the CT scanner, detector pixel density, X-ray focal spot size and sample size, there was no benefit in attempting to achieve better resolution by geometric magnification. Rueckel et al [27] have evaluated the dependence of spatial resolution on X-ray tube parameters for a CT scanner, demonstrating that at large magnifications the focal spot size becomes the limiting factor. At these scan setting, 14-16 pixels are available to reconstruct each ply, providing enough magnification to identify the gaps.

The CT scan captured 1600 projections with a single frame per projection, using an X-ray energy setting of 180KeV, and a 250ms exposure time per frame. Total scan time was 7 minutes. CT scans in less than 4 minutes were attempted using fewer projections and shorter exposure times, but the scans were very noisy. During a scan of 7 minutes, the temperature of the sample will increase by an average of 3.5°C. The slow temperature ramp allowed for more scans to be taken during the phases of viscosity decrease and sample consolidation, where a greater number of microstructural changes are likely to be seen. A CT scan was completed every 10 minutes for a total of 45 scans (7 hours and 20 minutes) as the cure proceeded.

Each scan was reconstructed using CT-Pro software. Regions of interest were processed and analysed by means of Fiji (ImageJ) [28] and a Python implementation of the 3D-Connected Component Labelling (3D-CCL) algorithm with a 26-connectivity [29]. The image processing methodology shown in Figure 4 was defined to improve the overall image quality, reduce noise and measure porosity. The final microstructure was rendered with Avizo[®] version 2019.1. Details of each step are described in the ensuing sub-sections.

Figure 4. Image processing flowchart used to segment porosity. Top-view and cross-section images of the gap regions are shown alongside the density histogram at each step of image processing.

2.4.1 Filtering and contrast improvement

The reconstructed scan images were noisy because of the high resolution and relatively fast scan time. For this reason, a $3 \times 3 \times 3$ Mean Filter, available in Fiji (ImageJ) [30], was applied to the original 3D images. This filter is especially well-suited to remove Gaussian noise, commonly found in CT images [31-33]. Subsequently, histogram normalisation, also called histogram stretching [34], was applied to improve the scan contrast, emphasising the

difference between the maximum and minimum grey value intensity, or brightness, of the displayed voxels. This transformation identifies the minimum (OL_{min}) and maximum (OL_{max}) displayed grey values in the original filtered images, and assigns new limits of NL_{min} and NL_{max} respectively. Each grey value present in the original image ($O_{x,y,z}$) is then linearly mapped to the new range of $[NL_{max} - NL_{min}]$ according to:

$$N_{x,y,z} = \frac{(NL_{max} - NL_{min})}{(OL_{max} - OL_{min})} \times (O_{x,y,z} - OL_{min}) + NL_{min} \quad \forall x, y, z \in 1, D \quad (1)$$

where $N_{x,y}$ is the new grey value of the voxel located at the coordinate (x, y, z) for an input image of size $D \times D \times D$.

Maximum contrast is desired to distinguish between phases. Therefore, new grey value limits have been assigned as follows: $NL_{min} = 0$ and $NL_{max} = 255$ for the case of an 8-bit image, as shown in Figure 5.

Figure 5. Histogram normalisation. Schematic of the original intensity range being stretched to the full 8-bit greyscale in (a), and application to the actual CT data in (b).

Histogram normalisation is not without drawbacks. This method is highly sensitive to X-Ray artefacts appearing in the form of abnormally high or low grey values that remain in the image even after filtering. The voxels displaying false grey values often represent an

extremely low portion of the total number of voxels, however, they can distort the mapping from the original image to the new image if they are chosen as the limits of the input image. This phenomenon was observed in the set of scans belonging to the parallel-ply gap configuration. Figure 6 shows bright spots that led to erroneous histogram normalisation, eventually creating false porosity segmentation.

Figure 6. Artefact effect on histogram normalisation observed in the parallel-ply gap sample. An artefact detected in the filtered scan (a). Effect of artefact on histogram normalisation (b). Method proposed to mitigate artefact influence on histogram normalisation (c).

To mitigate the effect of artefacts, pixels with abnormally high or low brightness are displayed as saturated voxels and excluded from the analysis. The number of voxels with abnormally high brightness accounted for less than 0.001% of the total number of voxels for any scan. Since the erroneous grey values are found in the higher end of the input histogram,

the corrected maximum original grey value limit corresponds to 99.999% of voxels, i.e., the 99.999th percentile ($OL_{\max_{99.999}}$). The minimum grey value limit was not changed since no artefacts related to low brightness voxels were found.

The final original limits have been established as follows:

- $OL_{\min_{corrected}} = OL_{\min}$
- $OL_{\max_{corrected}} = OL_{\max_{99.999}}$

These limits are then used for mapping the rest of grey values contained in the given scan as described by Eq. (1).

2.4.2 Segmentation

Segmentation is used to separate the air phase (gap + voids) from the composite phase (resin + fibres). This is a critical step since the segmented images were used to analyse the porosity level inside a given sub-volume. Otsu's threshold [35] is a common segmentation method consisting of minimizing the intensity variance inside two classes of pixels: background and foreground. This thresholding approach struggles to segment the air phase in low porosity samples due to the low number of pixels belonging to the air (foreground) phase. The methodology proposed by Tretiak and Smith [36] to set the value threshold in the middle of the greyscale range was applied. As can be seen in Figure 4, a threshold of 127 produces two equal segments containing 128 grey values, 0-127 for the air and 128-255 for the composite. This threshold gives satisfactory air phase segmentation after visual assessment between the input image and the resulting binary image.

2.4.3 Regions of Interest (ROI)

The composite microstructure was evaluated in-situ for five different sub-volumes within each laminate. The regions of interest (ROI) are shown in Figure 7, and selected as follows:

- i. ROI *Gap*: covers exclusively the 3D volume corresponding to the automated deposition feature in each scan. The ROI was defined in such a way that the

presence of micro voids surrounding the gap was minimised. The location and height of the ROI *Gap* were manually selected and adapted to the position of the gap in each scan. The width and length of the ROI *Gap* were kept constant throughout the set of scans for each configuration.

- ii.* ROI *Top* and ROI *Bottom*: cover the microstructure located above and below the gap, respectively.
- iii.* ROI *Right* and ROI *Left*: comprise the microstructure situated at the right and left side of the gap, respectively.

The position of the ROI Top, Bottom, Right, and Left were constant throughout the set of scans for each configuration. The dimensions of these ROIs were a length of 12.8mm, a width of 3.9mm, and a height of 0.95mm.

Figure 7. Reconstructed micro-CT images corresponding to the parallel-ply gap configuration (top) and ply-drop gap configuration (bottom) of the initial scan.

2.4.4 Porosity evaluation

The 3D binary images were analysed by a 3D-Connected Component Labelling algorithm (with 26-connectivity), developed in Python. This technique assigns a unique label at each set of connected air voxels (pixel value of 1) generating a “void object” with an exclusive label and the following properties: number of voxels that make up the void, void centroid, and void volume. The algorithm returns a list with all the voids contained in each ROI at each scan, as

well as their respective properties, enabling consistent computation of the porosity, the average void volume and the number of voids present in each ROI for a given scan. Performing such analysis over the whole set of scans provided information about the porosity, average void volume, and void count evolution throughout the manufacturing cycle.

2.5 Microscopy of cured samples

The final resin, fibre, and void distribution in the gap was analysed by confocal microscopy at 10x magnification to compare with the last in-situ CT scan. Cross-sections were cut with a diamond-coated water-cooled saw, mounted in room temperature cure casting epoxy, and polished for observation. The samples were cut off-axis to avoid fibre pull-out during polishing of the cross-section.

3 Theoretical background and numerical simulation

The experiments were modelled using the composite manufacturing simulation tools developed by Belnoue et al. [8,9,37-39]. The model follows a hyper-viscoelastic formulation for anisotropic materials to account for the very soft nature of prepreg in the transverse direction, and its much stiffer behaviour along the fibre direction [8]. One of the main advantages of this model is accounting for the effect of both bleeding and squeezing flow on the evolution of the thickness of prepreg stacks during manufacturing [40]. This means that the model can capture phenomena like fibre volume fraction evolution by reducing the size of the elemental volume, and the filling of pre-existing gaps or cavities by a mixture of fibre and resin [39].

Simulations were performed using the Finite Element package Abaqus/Standard and the model configuration is illustrated in Figure 8. A pressure of 100kPa was applied to the top of the disc and the ring was fixed at the bottom. A ply-by-ply treatment of the individual layers using linear brick elements with reduced integration (C3D8R) and enhanced hourglass control were adopted in all the components of the model. The initial internal ply geometry

was extracted from the CT-scan data and meshed. Frictionless interaction between the uncured composite plies and the acrylic ring and disc was assumed. A coulomb friction coefficient of 0.2 was applied to mimic the interaction between the composite plies [39]. This is an approximation as, strictly speaking, lubricated friction [41] should be considered. However, sensitivity analysis in previous work [42] showed that, in the case of consolidation-induced wrinkles, friction acts more like a switch i.e. it helps in triggering wrinkles, but their severity stays largely unaffected by different values of the friction coefficient. All the interactions in the model were set up so that contacting bodies can separate without any resistance (i.e. no tack).

Figure 8. Finite element representation of the consolidation experiments. The acrylic disc and ring that constrained the lateral motion of the sample plies at the perimeter was explicitly modelled (left). A ply-by-ply treatment of the individual layers extracted from the CT scan data (right).

A temperature history corresponding to the nominal temperature profile described in section 2.3 was applied. A uniform temperature was assumed by uncoupling the fibre volume fraction and the thermal conductivity or temperature gradient effects. The measured modulus of the acrylic as a function of temperature was used as a material input for the boundary condition with a constant Poisson ratio of 0.4. The uncured composite plies were modelled by a user material subroutine (UMAT) for Abaqus with the material parameters given in Table 1. The parameters are taken directly from references [8] and [39], where they were measured. The parameter k is a measure of the inter-fibres channel before compaction normalised by the

size of the unit cell, and parameters a and b control the resin viscosity. The bending rigidity in Table 1 was measured following the procedure described in [43]. Within the UMAT, the parameters at a given temperature are estimated through linear interpolation of the points provided in Table 1.

Table 1. Evolution with temperature of the model parameters for IMA-M21 prepreg.

IMA-M21							
T(°C)	30	40	50	60	70	80	90
k	0.96	0.8925	0.83	0.805	0.8	0.795	0.79
a	-0.934	-0.818	-0.752	-0.731	-0.752	-0.8	-0.864
b (squeezing)	-17.89	-15.78	-14.44	-13.9	-13.84	-13.98	-14.58
b (bleeding)	-34.62	-32.51	-31.17	-30.63	-30.56	-30.70	-31.31
Bending stiffness (N.m)	1.67E-02	1.48E-02	1.21E-02	8.31E-03	2.06E-03	5.05E-04	3.82E-04

4 Results

4.1 In-situ CT results

Reconstruction of key stages in the manufacturing process, including the initial, minimum, and final gap porosities are shown in Figure 9. The raw CT scan data is available from the University of Bristol research data repository (link follows Section 6). The gap porosity and height measured through the complete manufacturing process are shown in Figure 10.

As can be seen in Figure 9, a large initial pore was observed in both gap cases after the vacuum had been applied for approximately 10 minutes. As reported in Figure 10, the total porosity in the parallel-ply gap was approximately 24%, lower than the ply-drop gap at roughly 33% porosity. The difference was attributed to parallel fibres filling the gap once vacuum was applied. As the temperature increased in early stages of heating, resin and fibre squeezing were observed, reducing the gap widths, and total gap volume/porosity. The parallel-ply gap sample reached its minimum porosity of 0.24% after 130 minutes (at 73°C), whereas the ply-drop gap configuration registers a minimum porosity of 1.12% after 170 minutes (at 89°C).

Comparing the results of the parallel-ply and ply-drop gaps, the following observations are made:

- The parallel-ply gap porosity and height *reduce faster* than the ply-drop gap,
- The magnitude of the parallel-ply gap porosity and height *reach lower absolute values* than the ply-drop gap, and
- The porosity and height of the gap *increased after reaching a minimum* in both cases.

All dimensions are in mm.

Figure 9. Porosity evolution within ROI Gap for Parallel Ply Gap (left) and Ply-Drop Gap configuration (right) at their respective initial (top), minimum porosity (middle) and final scan (bottom). The microstructure reconstruction was made with Avizo® version 2019.1.

Figure 10. Porosity and height of ROI Gap evolution with temperature. The viscosity is also plotted to identify the timing of minimum viscosity and gelation.

4.1.1 Effect of gaps on surrounding microstructure

The influence of each gap type on the surrounding microstructure was analysed, and the results are shown in Figure 11. A statistical analysis was performed on the difference in the absolute value in porosity for the pairs of ROIs Top-Bottom and Right-Left, and the results

are shown in Figure 12. Comparing the results of the four ROIs in the parallel-ply and ply-drop gaps, the following observations are made:

- The porosity in the laminate containing a parallel-ply gap is *lower* than in a laminate with a ply-drop gap (Figure 12).
- A noticeable reduction in laminate porosity occurred in ROI Top and ROI Bottom in the parallel-ply gap during early stages of consolidation, but *increased near minimum viscosity*, similar to the gap itself (Figure 11).
- The microstructure beside the gap, ROI Right and ROI Left, have *lower porosity* than the microstructure above and below the gap, in ROI Top and Bottom (Figure 12), meaning the gap has a *more profound influence* on the laminate microstructure above and below the gap than on either side of the gap.

Figure 11. Porosity, average void and void count evolution within ROI Top, Bottom, Right and Left for parallel-ply gap (left) and ply-drop gap configuration (right).

Figure 12. Box and whisker plot of the difference in porosity (absolute value) between ROI Top-Bottom and Right-Left in each scan.

4.2 Microscopy of cured samples

Microscopy images characteristic of the final microstructure are shown in Figure 13. Figure 13a-b show two sections of parallel-ply gap samples, and Figure 13c-d show two sections of a ply-drop gap. Each image shows voids in the area of the original gap, with larger voids being visible in the resin-rich region of the ply-drop sample (Figure 13c-d). In both configurations, the original gap was filled by ply bending from above and below. However, fibre squeezing was observed in the parallel-ply gap sample with characteristic knife-edge plies moving into the original gap (Figure 13a-b). On the other hand, resin bleeding was observed in the ply-drop sample with rectangular ply edges (Figure 13c-d).

Figure 13: Microscopy images of parallel ply gap samples (a-b) and ply-drop gap samples (c-d) after the manufacturing cycle. Ply squeezing and bending is visible in (a-b) whereas resin bleeding and ply bending is the dominant mechanism to fill the gap in (c-d), where large voids remain.

Distinct resin-rich layers between plies, characteristic to the interleaf toughened class of material used are also visible by microscopy. The interleaf region is noticeably thicker above and below the gap because of the reduced consolidation pressure due to this feature. The influence of the gap on the interleaf regions is more predominant in the parallel-ply sample, increasing the interleaf thickness in four or five plies above and below the gap. In the ply-drop sample, the interleaf region is thicker in only three plies above and below the gap because of the lack of fibre squeezing.

4.3 Process simulation

The results of the simulation allows direct comparisons with the measured microstructures from the in-situ CT scans in Figure 14 and micrographs in Figure 15. The model and experimental data show a competing mechanism between the lateral filling of the gap through squeezing of the prepreg and the overlapping plies falling into the gaps through bending. In the case of the parallel-ply gap laminate, a significant portion of the original cavity was filled by fibre through squeezing and bending mechanisms. On the other hand, most of the fibres run transversely to the gap in the ply-drop gap sample, making the overlap region a lot stiffer than in the parallel-ply gap case. As a result, the original cavity only gets filled through transverse squeezing and consolidation of the central ply and most of the gap remains empty or gets filled by pure resin. In light of these mechanisms, the following observations were made from Figure 14 and Figure 15:

- The gap volume decreases *faster* in the parallel-ply gap than in the ply-drop gap (Figure 14).
- The ply-drop gap shows *very little* fibre squeezing (Figure 14 and Figure 15).
- The process simulation predicts a continuous reduction in gap volume until reaching steady-state, whereas the in-situ CT data shows an *increase* in gap volume after 130 minutes (Figure 14).

Figure 14. Parallel ply and ply-drop gap volumes fractions extracted from the FE models alongside the in-situ CT data.

Figure 15. FE simulation results for the fibre volume fraction alongside micrographs for the parallel-ply gap sample (a) and ply-drop gap sample (b) at 440 min.

5 Discussion

The in-situ CT experiments provided a powerful tool to analyse the evolution of the gaps and the surrounding laminate microstructures. The initially large parallel-ply and ply-drop gaps had a porosity between 20-30%, which reduced to nearly zero as the temperature was increased towards the cure temperature. As minimum viscosity was approached, the gap porosity slowly increased until constant temperature was achieved. The porosity level remained stable though gelation and until the end of the manufacturing process.

The characteristics of the final gap porosity were very different between the parallel-ply and ply-drop configurations. The parallel-ply gap had two needle-like pores running parallel, along the ply knife-edges due to squeezing and bleeding into the gap. On the other hand, the ply-drop gap had wider, non-continuous pores in the resin due to bleeding into the gap.

Adjacent regions of interest (ROI) in the laminate near the gap also showed a rise in the void count and porosity after minimum viscosity was reached. This behaviour suggests formation of new micro-voids within the gap and adjacent microstructure during the manufacturing process, or growth [44-46] of pre-existing micro-voids below the CT resolution of $13.5\mu\text{m}/\text{voxel}$.

Even though in-situ CT was successfully used to capture composite microstructure evolution using lab-based x-ray tomography, observation of flow and consolidation during early temperature ramp segment of the manufacturing process was challenging. The reconstructed images from scans performed at 10, 20, 30 and 40 minutes in the ply-drop gap sample were slightly blurred. The largest reduction in gap height was measured at this stage of the manufacturing cycle (see Figure 10), and this consolidation was faster than the CT was able to capture in a 7 minute rotation. As a result, values of porosity, average void volume and void counts for the ply-drop gap at the mentioned timesteps present some uncertainty. Future work is needed to reduce scan time in lab-based CT systems to better capture consolidation in-situ.

A potential drawback of the experimental set-up used in this study was a through-thickness temperature gradient above 75°C due to the single sided heating (see Figure 3). Given that the main feature of interest in this study was in the middle of the laminate, negligible consolidation effects due to temperature are considered. Consolidation would be expected to occur earlier in the hotter plies of the laminate as the resin is more mobile. However, the porosity analysis above and below the gap in Figure 11 show similar behaviour for both gap types – when porosity in ROI Bottom increases or decreases, porosity in ROI Top increases or decreases by the same extent. Therefore, the temperature gradient that arose in the laminate between 75°C and 150°C was deemed to have a negligible influence on the measured porosity and consolidation above and below the mid-plane. If the laminate in Figure 7 had multiple gaps positioned through the thickness, the temperature gradient would likely have a more significant influence and would need to be considered as part of the simulation. Alternatively, new experimental set-ups could be developed to improve how heat is input to the part.

Given the similarity in density of the materials used, current -lab-based CT capabilities struggle to distinguish resin from fibre. While synchrotron radiation sources can differentiate between two very much alike solid phases to provide a clearer picture of the flow and compaction mechanisms, the current models do not account for pure resin flow once it has left the fibre bed. As a result, some features of the gaps closure mechanism are not fully captured by the simulation capabilities. This mismatch was only highlighted by comparing the model predictions with the in-situ CT experiments to identify different flow mechanisms between the 2 different gaps configurations. The use of in-situ CT-scanning has allowed comparison of the process model throughout the process and not only based on the final state. Further model development to account for pure resin flow and void growth is an ongoing area of research.

6 Conclusions

In-situ micro-CT was developed using a lab-based CT scanner to analyse the evolution of parallel-ply gaps between courses and ply-drop gaps needed to change the thickness of prepreg laminates. The microstructure of composite laminates was observed while consolidation and cure were occurring, without interrupting the manufacturing process. A lab-based CT scanner has clear benefits in terms of cost and availability when compared to synchrotron x-ray sources but suffer from lower resolution and longer scan times.

The results showed that in-situ CT was able to distinguish between parallel-ply gap volume reduction as squeezing flow filled the gap, whereas the ply-drop gap volume filled more slowly due to resin bleeding. More importantly, the in-situ experiments revealed that initial gaps in the laminate that reduced during the early stages of processing reappeared at later stages of the manufacturing process. The return of previously consolidated voids in the gap would otherwise go unnoticed using ex-situ measurements of the manufacturing process.

The in-situ CT results were compared to state-of-the art process models simulating consolidation of the gaps, and microscopy of the final cured laminate. The process simulation predicts a continuous reduction in gap volume until reaching steady-state, whereas the in-situ CT data shows an increase in void volume after a porosity minimum was reached. The in-situ data emphasized the need for further compaction model developments to account for resin bleeding behaviour and also provides a means to obtain the validation data.

Overall, lab-based in-situ CT could be applied to many other features in composite manufacturing to better understand internal geometry development, validate process models, and support the move to full digital manufacturing of composite structures. Even though improvements are needed to reduce scan times and image contrast, the technique is sufficiently versatile to accommodate a wide range of set-up that will improve the understanding of complex material behaviour during the manufacturing process of advanced composites.

Data Access Statement

The raw CT data are available from the University of Bristol research data repository at: <https://doi.org/10.5523/bris.cpunp7ml1s5t2owhutk6hj7gd>. The consolidation code is available from the Bristol Composite Institute (ACCIS) Github page (<https://accis.github.io/DefGen/>) for the Finite Element Analysis software Abaqus.

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