

TRACKING THE EVOLUTION OF A DEFECT, CHARACTERISTIC OF AFP LAYUP, DURING CURE WITH IN-PROCESS MICRO-CT SCANNING.

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ABSTRACT

An increased understanding of how features such as voids change during the cure process can inform design and manufacturing process choices. This paper presents the detailed evolution of a prepreg defect throughout the cure process, using a novel method of In-Process Micro-CT.

A Nikon XTH-320 industrial CT scanner was equipped to allow curing of an uncured prepreg sample under vacuum. Short scans of approximately 7 minutes were performed repeatedly during cure.

Here we discuss the evolution of a cylindrical sample of a carbon fibre prepreg containing a 2 ply thick 2mm gap along the fibre direction across the centre, typical of defects found in AFP processes. The evolution of the gap is compared to the theoretical resin behaviour.

In-process micro-CT has the potential to provide far more detailed information on the behaviour of composites during cure than has been available prior to today. This work provides both proof of the principle, as an experimental method, and shows detailed results measuring the evolution of a gap during the cure of carbon fibre prepreg materials.

1 INTRODUCTION

Composite parts may be designed to exacting standards, with layup guides requiring accuracy of a fraction of a millimetre. Achieving this in practise, without any form of defect, is very difficult. Both manual layup and automated processes can and do produce parts with a variety of defects [1], which can significantly affect the structural properties of the final product. Understanding how a defect introduced in the initial stages of manufacture evolves and changes as the composite is cured should inform our design and manufacturing choices, particularly with respect to defining tolerances for defects.

Tracking the evolution of a defect during cure is one way to do this. While it has previously been possible to take CT scans at different stages of the cure by quenching several partially cured samples at different stages of the cure cycle [2], there is inevitably variation between samples and the quenching process may affect the result. An in-process method gives us the ability to track a defect, in a single sample, through the entire manufacturing process, providing a clearer picture of how the material micro-structure evolves during cure.

In an industrial CT scanner, the sample is rotated through 360 degrees while exposed to X-rays. The number of projections, or images, per scan and the exposure time of each image determine how long the scan will take. As longer scans average the image detected over a larger period of time- with

multiple hour scans being the norm for a static composite sample- the signal to noise ratio is better; however in order to capture the changes in a curing sample, we need a very short scan time.

Industrial CT scanners have been used to track the changes in objects over time before, with the most relevant being Plank et al.'s 2016 work [3] following a bubble of water during the cure of neat resin. Our experimental method, developed independently of their work, bears some similarities to theirs- in that a sample is heated while under vacuum- but the requirements for a composite prepreg sample and evaluation of the voids therein are very different. In-process CT scanning of composites has been carried out using synchrotrons, including for resin infusion [4] and compression moulding [5]. Synchrotrons can produce a high quality and high magnification image, but are expensive to use and not widely available in industry, therefore an industrial CT scanner is more widely accessible.

Results presented in this paper show the evolution of an approximately 2mm wide, 2 ply deep gap along the fibre direction at the centre of a (0,90) layup, of the type which might reasonably occur during manufacture with either manual layup or Automated Fibre Placement. This can be compared to the tow gaps studied by Rhead et al. 2012 [6], which had a significant effect on sample compression after impact.

2 MATERIALS

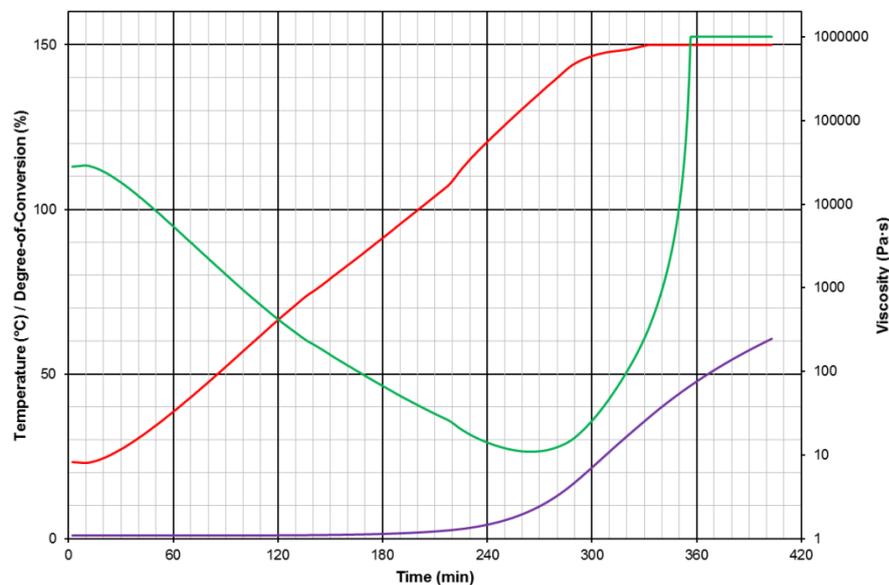


Figure 1: M21 viscosity and degree of cure over selected cure cycle

The test samples used in this study were made from Hexply M21 prepreg, as this is a widely used material, representative of aerospace grade carbon fibre-epoxy based prepreps. The prepreg was reinforced with IMA fibre with an areal density of 194 g/m², a nominal resin content of 34% by weight, leading to a nominal fibre volume fraction of 59%.

The cure cycle was as shown in Figure 1- a slow ramp of approximately 0.45 degrees Celsius per minute to a temperature of 150 degrees Celsius, hold for 2 hours then cool naturally by turning off the heat source. The prepreg material passes through gelation and reaches a 60% degree of conversion during this experiment [7]. The major processing effects, including resin flow and fibre bed consolidation are captured in this cure cycle. The hold temperature was chosen to ensure the heat was sufficient for gelation and some curing to occur, but not likely to risk a fire in the highly expensive XTH-320 CT scanner.

A typical autoclave has a temperature accuracy of plus or minus 2 to 3 degrees Celsius, meaning variation of 4 to 6 degrees Celsius can be considered 'constant temperature' by industrial standards. During a short scan of 7 minutes, the temperature of the sample will increase by an average of 3.15 degrees Celsius, well within this variation. Furthermore, the slow ramp allows numerous scans to be taken during the interesting phases of viscosity decrease and sample consolidation where the most movement is likely to be seen, without compromising too much on scan quality.

3 EXPERIMENTAL SETUP

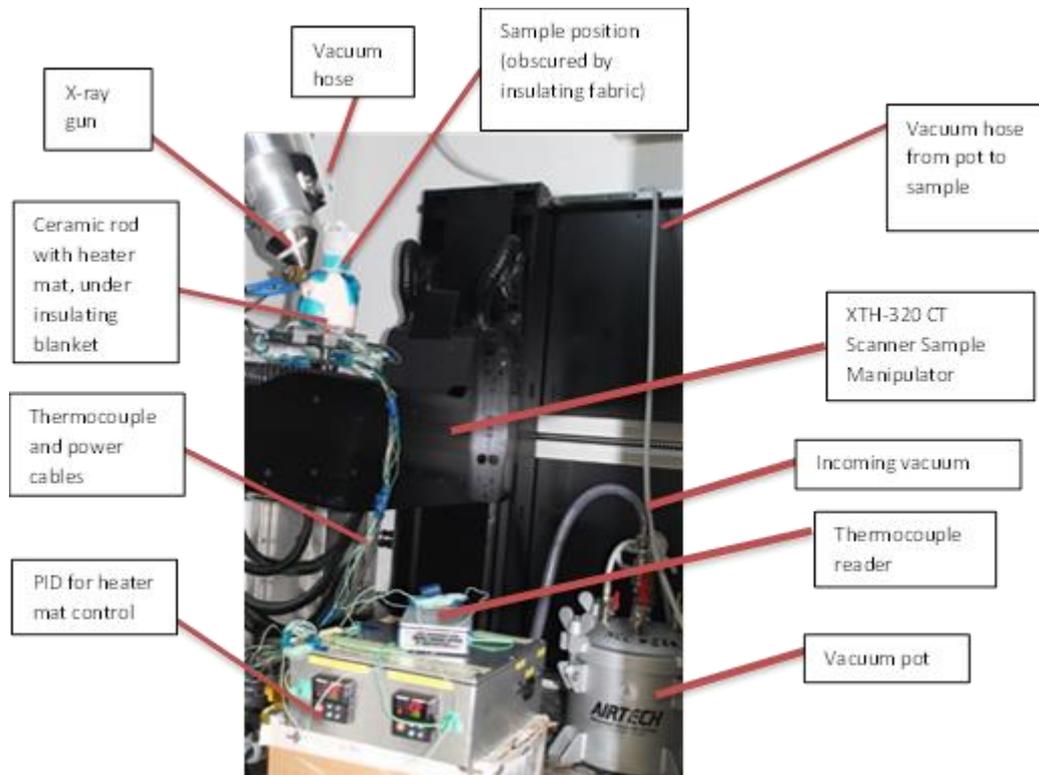


Figure 2: Experimental equipment in XTH-320 CT scanner cabinet.

Figure 2 and Figure 3 show the experimental setup with the sample ready for curing. To keep the heat only where we want it, the heater mat and tool assembly is covered by a 'blanket' of breather contained within a sealed piece of vacuum bag- to prevent breather fabric becoming caught in the clamp screws. Another piece of breather is used to loosely insulate the sample. Breather is not very dense, hence is nearly transparent to X-rays and so has a minimal effect on the resulting images.

A vacuum line passes into the cabinet from outside and is connected to a pressure pot. A hose from the pressure pot then runs up to the ceiling and is affixed by tape, hanging loosely down to meet the sample. This keeps the sample under an even vacuum without requiring a metal fixing (which would scatter the x-rays and cause noise) in the x-ray zone.

Both the vacuum hose and the cables connecting the heater mat to the PID and thermocouple reader must be arranged in a manner which allows the sample to rotate 360 degrees. The software is set to then counter-rotate it back to the start position before the next scan, so that the hoses and cables do not become overly tangled. They must be affixed to the cabinet and manipulator in a manner that keeps them out of the line between the X-ray gun and detector.

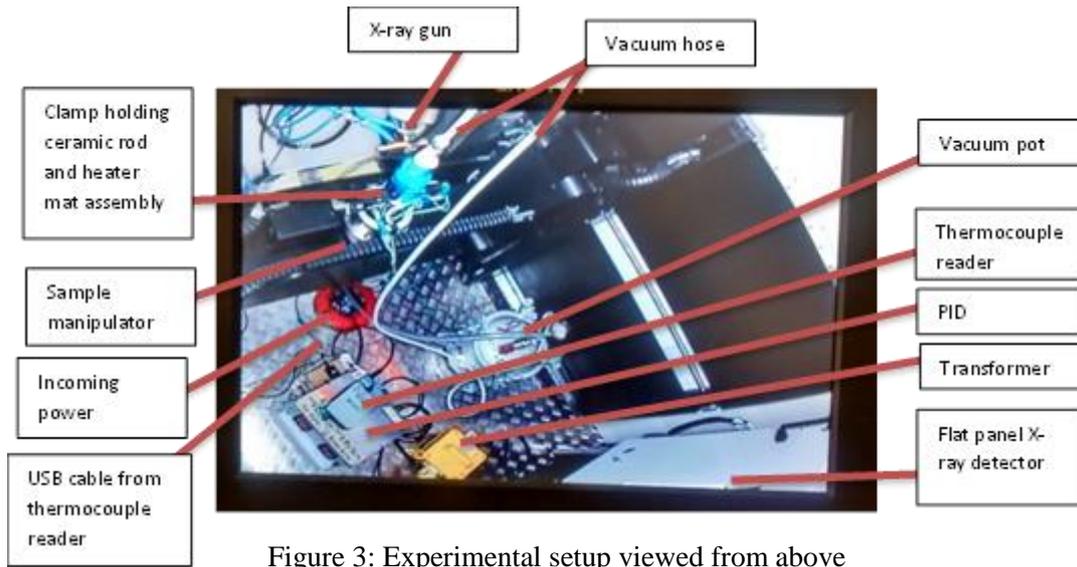


Figure 3: Experimental setup viewed from above

The 225KeV reflection X-ray head was used. While it may be possible to achieve better results using a rotating target head- allowing higher power to be used- the time required to swap the heads for each day on a shared use machine made this impractical. The results achieved with the 225KeV head were more than sufficient for our purposes. The flat panel detector was used, again for speed.

The heater mat is controlled based on temperature readings from a thermocouple in a slot in the base of the ceramic. Thermocouples in a second slot, taped to the middle of the base, taped to the mat itself and in the air (to provide a warning if the cabinet heats up) pass to the thermocouple reader, which is connected via USB extension cord to a PC outside the cabinet.

Figure 4 shows a sample before cure. The heater mat is wrapped around the ceramic rod, between two tooling blocks (to protect the rubber on the clamp). The sample is placed on the top of the ceramic, which protrudes just enough above the heater mat to allow the vacuum bag to be sealed to the tool with half width tacky tape. The sample is lined up with the X-ray gun behind it.

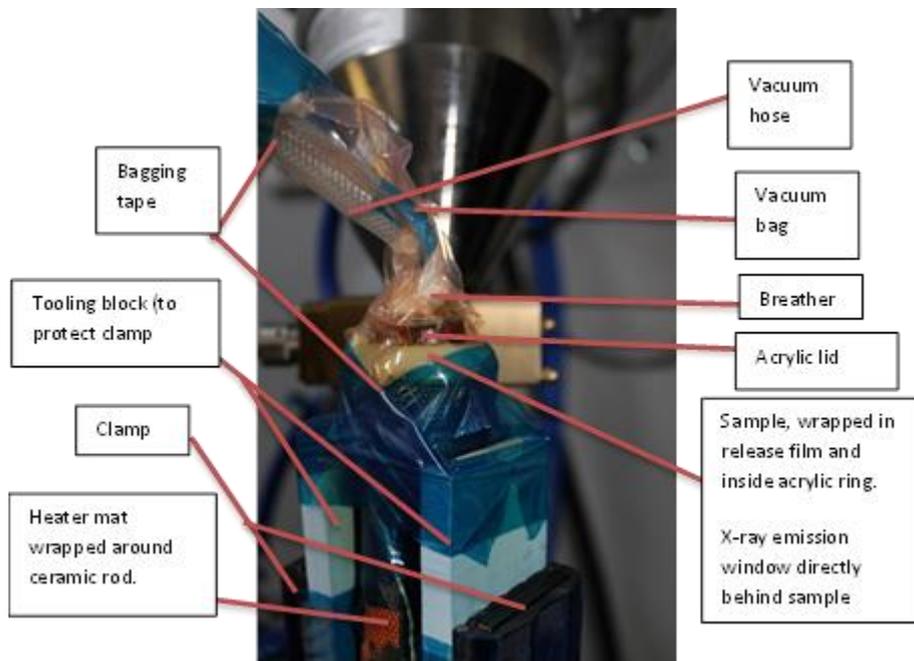


Figure 4: Close-up view of bagged sample, in position for curing

The sample is contained within an acrylic ring, to provide structure, so that it might be considered representative of a defect in the centre of a larger part. Atop the sample is an acrylic disk which ensures the consolidation pressure from the vacuum is even across the centre of top of the sample. A piece of breather connects this to the vacuum hose, around which the other end of the envelope bag is secured. The small envelope bags were made from standard vacuum bag plastic formed into cylinders using a hot seal press. The sample is bagged on top of a ceramic rod, which is heated with a silicone heater mat.

4 SAMPLE GEOMETRY

Each sample consisted of 22 plies of M21 carbon fibre prepreg, laid up in a balanced manner [0,90...0,0...90,0]. The sample was cylindrical, for maximum scanning efficiency, and approximately the size of a pound coin. Each of the two central, 0 degree plies was two truncated semicircles, laid up with a gap of approximately 2mm between them. Figure 5 shows an example.

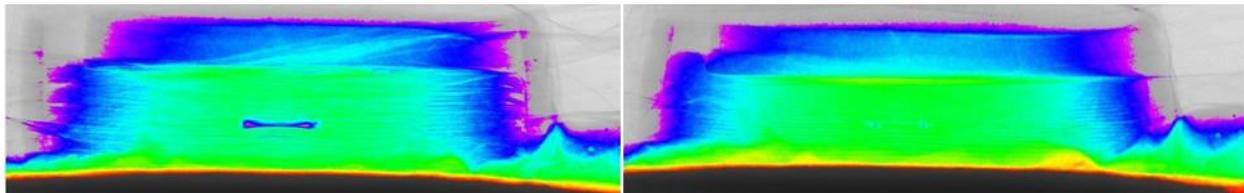


Figure 5: Scatter free radiographs of sample before (left) and after (right) cure in false colour, with shading correction. Looking end-on at the gap.

The black is the ceramic and the material to the side and in front of the sample is bagging tape. The acrylic lid is in a bluer colour above. The sides of the acrylic ring and the bag can be seen in grey.

5 METHOD

5.1 Temperature Measurement Baseline

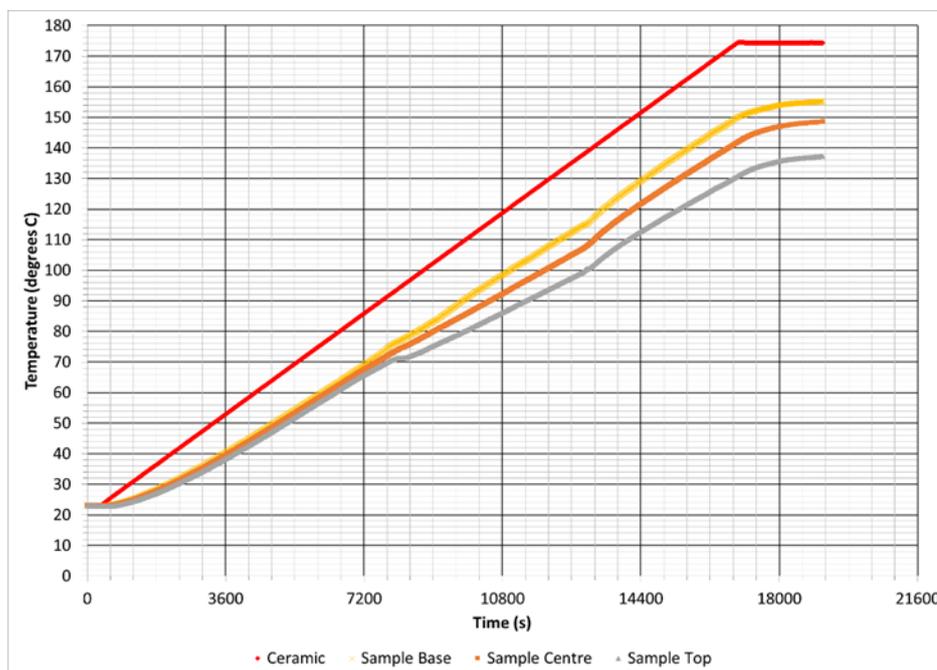


Figure 6: Temperature measurement baseline

During a scan, it is not advisable to place thermocouples in or around the sample, as the metal scatters x-rays at these energies, making the resulting scan too noisy to be of much use.

For this reason, the temperature of the setup is controlled and measured by thermocouples embedded in the bottom of the ceramic cylinder, well away from the x-ray zone. In order to estimate the temperature of the sample from this, a temperature calibration experiment was carried out, with the result shown in Figure 6. Thermocouples were placed in the base of the ceramic, between ceramic and sample, embedded in the middle of the sample, and on top of the sample. No scans were taken, but otherwise the same setup was used as during an In-Process CT batch scan. The results of this were used to estimate the sample temperature based on the measured ceramic temperature during the actual experiments.

5.2 X-Ray CT Settings

The fastest scan attempted with the XTH-320, using the 225KeV reflection head, took just under 4 minutes. Achieving such a fast scan meant using a low number of projections and very short exposure. It is possible to identify the deliberate, large gap using this setting, but the scans were-unsurprisingly- very noisy. A scan of just under 7 minutes showed significant improvement in the quality of the results without any blurring from any changes during that time. This scan was 1600 projections with a single frame per projection and an exposure of 250ms per frame. A resolution of 13.45 microns or less was found to be suitable, being enough magnification to clearly identify the voids while allowing a power of 13W to be used (which results in a focal spot size of approximately 13 microns, just smaller than the required resolution). A higher power means a greater x-ray flux, making the most of a short exposure time. The X-ray energy was set to 180KeV.

For each sample, the position and settings were checked and a shading correction taken. A batch program started a scan every 10 minutes for a total of 45 scans (7 hours and 20 minutes) as the cure proceeded. This timing was sufficient to see each successful run to gelation and allowed two runs to be done per day, with the second left on overnight.

Each scan was automatically reconstructed using CT pro software. The best results were achieved using a noise reduction filter on level 2. The voids were then analysed using VG Studio Max.

5.3 Void Analysis

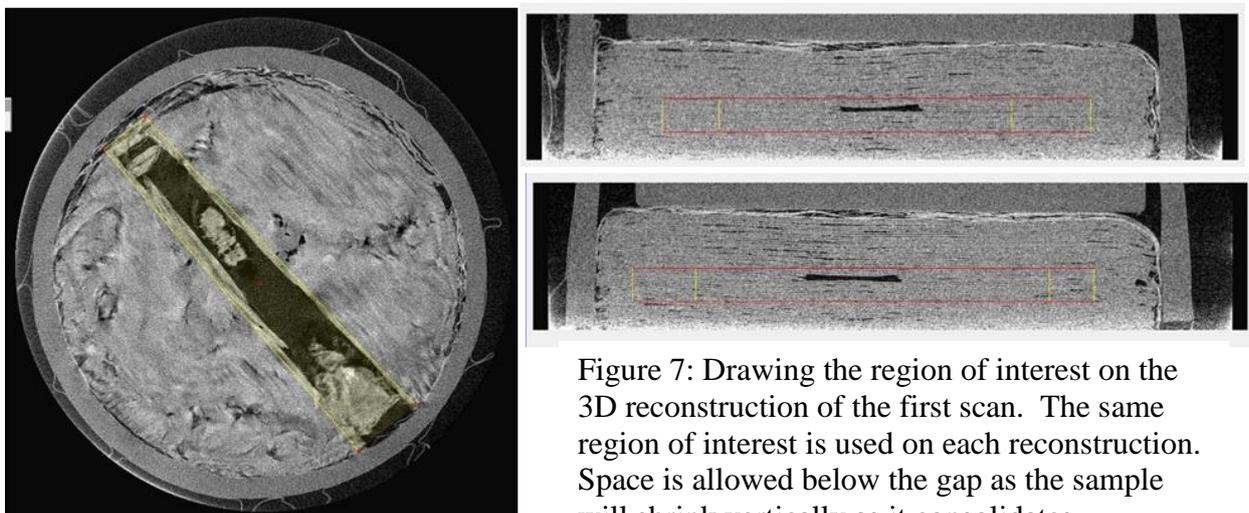


Figure 7: Drawing the region of interest on the 3D reconstruction of the first scan. The same region of interest is used on each reconstruction. Space is allowed below the gap as the sample will shrink vertically as it consolidates.

A region of interest is defined for the void analysis, containing the main gap, as shown in Figure 7 and Figure 8. As the sample consolidates and changes the gap position relative to the scan baseline moves, so the region must be large enough to allow for this. Hence, while any smaller voids and gaps in the region of interest will also be detected, the volume and geometry over which the voids are measured is constant throughout and dominated by the deliberately introduced gap. All voids in this region are measured as, at later stages, it is difficult to tell which of the smaller voids were initially part of the large one.

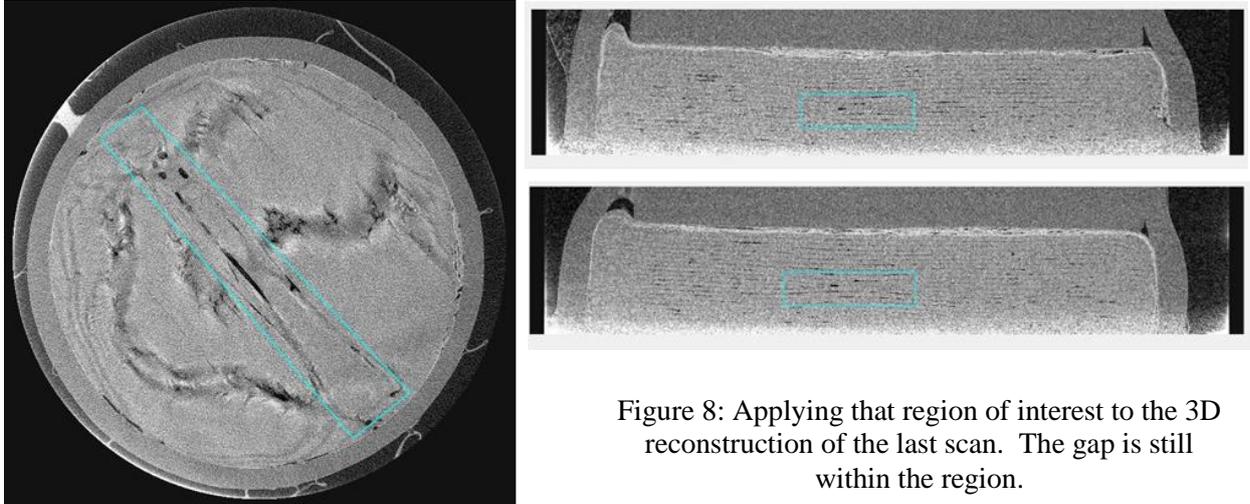


Figure 8: Applying that region of interest to the 3D reconstruction of the last scan. The gap is still within the region.

After defining the region of interest and saving it as a template, a macro was created to automate application of the void analysis process to all 45 reconstructions. The process is as follows:

- Open .vgi reconstruction file
- Apply a median filter (level 5) to the reconstruction. This helps to remove noise, and does not change voids and gaps of the size we are looking for.
- Use VG Studio's surface determination tool to automatically locate surfaces- this uses changes in density to find the surfaces of the object being scanned, including inner surfaces around voids and gaps.
- Define a region of interest from this surface.
- Invert the region of interest, so that the gaps and voids are 'inside'.
- Extract this region of interest, containing the gaps.
- Apply the previously defined region of interest template to this extracted region.
- Extract the resulting region. We now have a volume consisting only of the gaps and voids in the defined zone.
- Use VG studio's void analysis tool (set to "only threshold" to detect voids as we now have a simple volume of voids only) to measure all these gaps and voids. Voids of volume 0.01 to 100 mm³, with a probability of 0.1 or more, are measured. This means anything too small to be reliably identified as a void is ignored, and any spuriously large measurements- indicating errors- would also be ignored.
- Extract these results as a .csv file, showing measurements of all the voids in the target zone.

Figures 11-13 are taken from the early part of the run shown in the darkest colour in Figure 10. These are slices through a 3D volume reconstructed from the CT scan. The images on the left are slices partway through the vertical height of the sample, always at the same position (slice 143). As the sample consolidates and shrinks vertically, different layers move through this slice. The images on the right are slices halfway through the x axis of the sample. Note that the gap does not run directly along an axis. The consolidation of the sample as a whole can clearly be seen, along with the filling of the gap.

The images on the right are slices halfway through the x axis of the sample. Note that the gap does not run directly along an axis. The consolidation of the sample as a whole can clearly be seen, along with the filling of the gap.

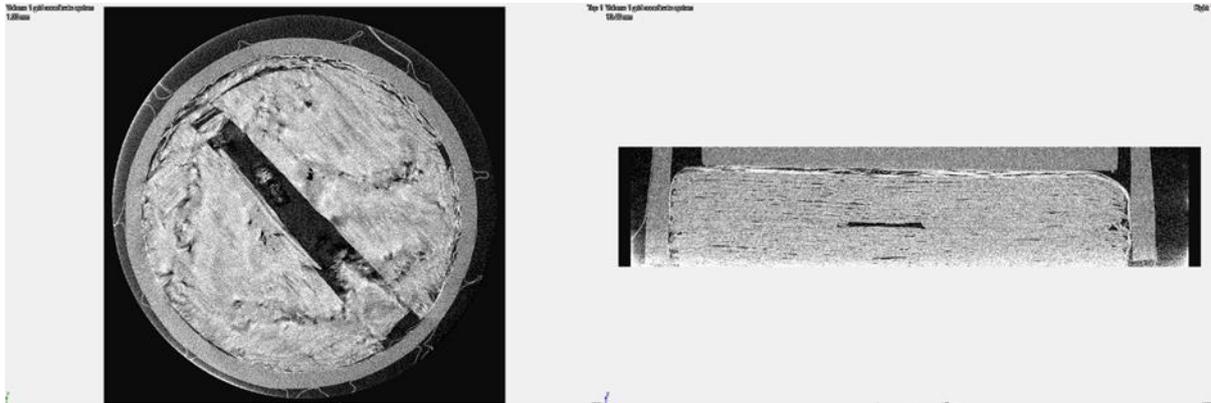


Figure 11: Slices through the z and x axis of the sample at time 00:00, temperature ~ 23 degrees C

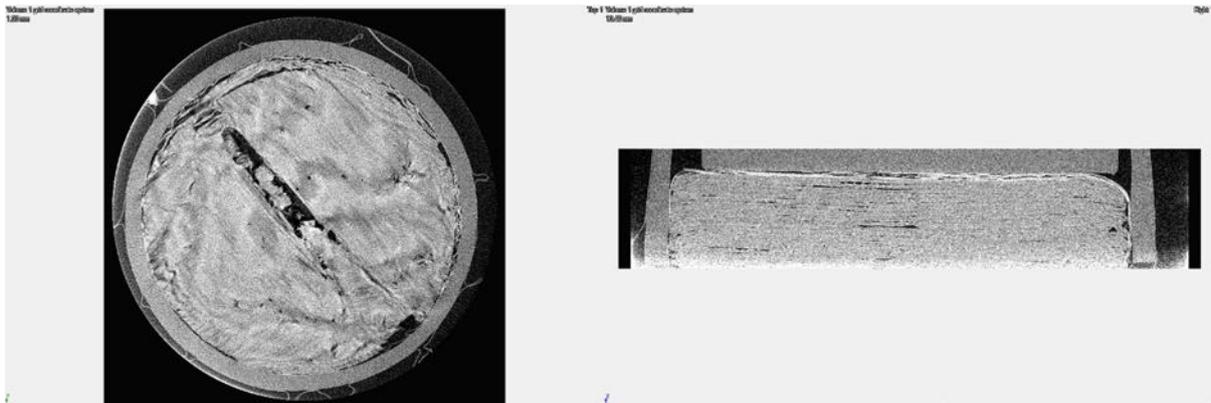


Figure 12: Slices through the z and x axis of the sample at time 01:00, temperature ~ 39 degrees C

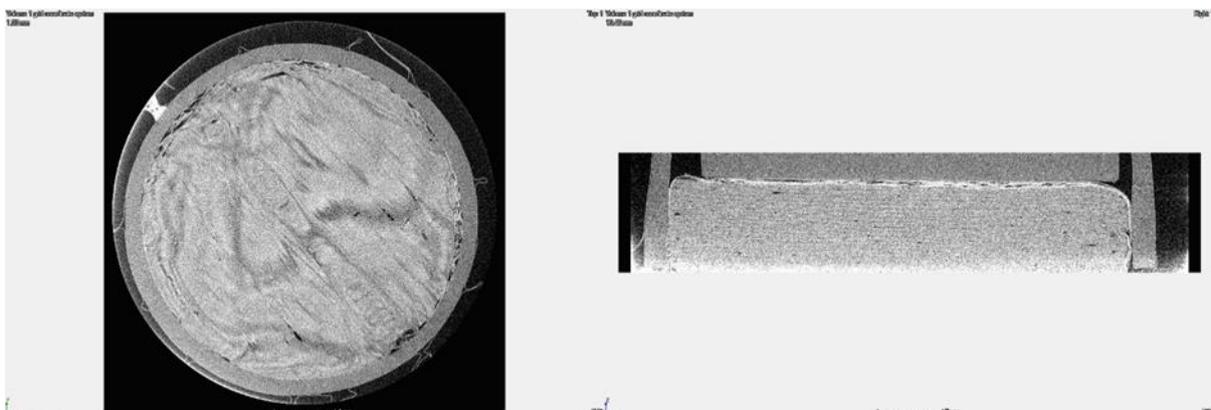


Figure 13: Slices through the z and x axis of the sample at time 02:00, temperature ~ 66 degrees C

After this point, the sample becomes fully consolidated and the voids become small or, in this case, below the detection limit of 0.01mm^3 .

Later in the scan, smaller voids appear along the line where the original gap edges were. Figure 14 shows the final scan of the same run.

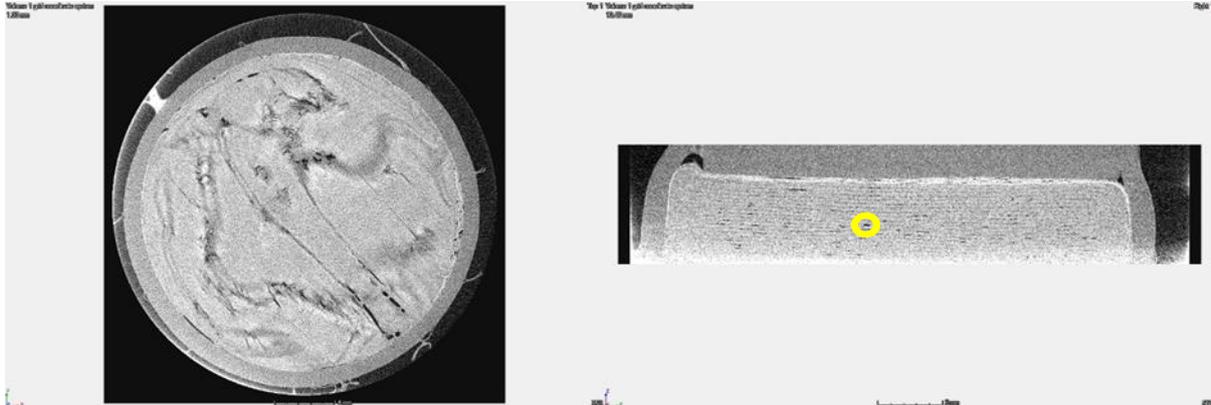


Figure 14: Final scan of the batch. Small voids have re-appeared. The void at the corner of the original gap is highlighted in the x axis view. The sample is now noticeably shorter and the acrylic ring has softened and joined onto the lid.

Figure 15 shows the evolution of the voids in the region of interest for the same run shown above. The voids are binned by volume on a logarithmic scale. The single large, deliberate void is seen at the start, along with a number of smaller voids. As the run progresses, the voids shrink and the large void splits into smaller voids, before all vanish below the threshold (0.01mm^3) in the middle of the run. The small voids at the edges of the original gap then appear and stabilise.

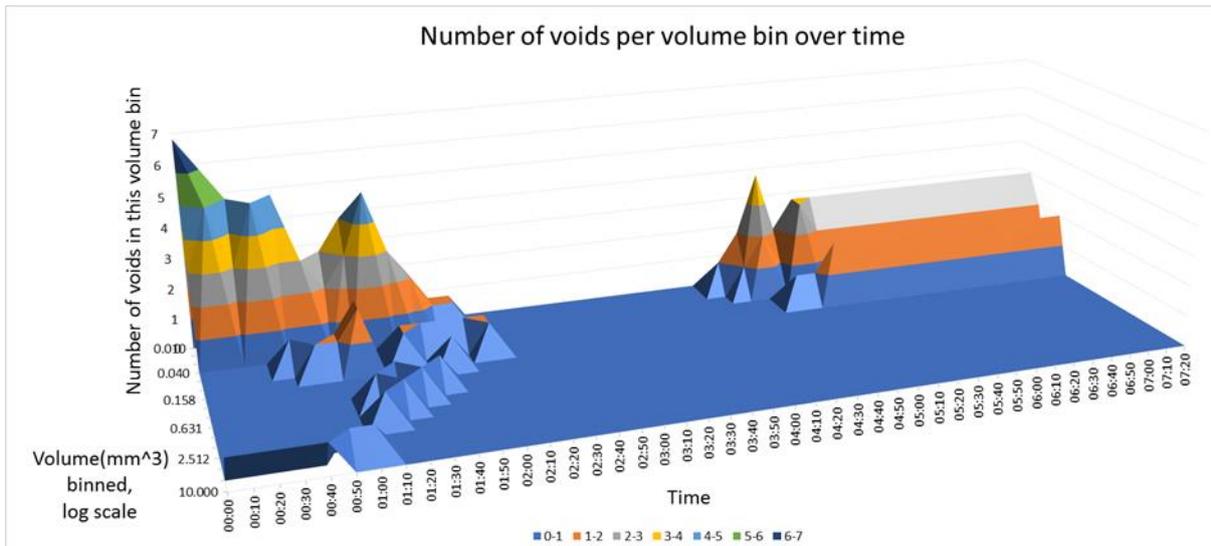


Figure 15: Distribution of void volumes

7 COMPARISON TO RESIN BEHAVIOUR

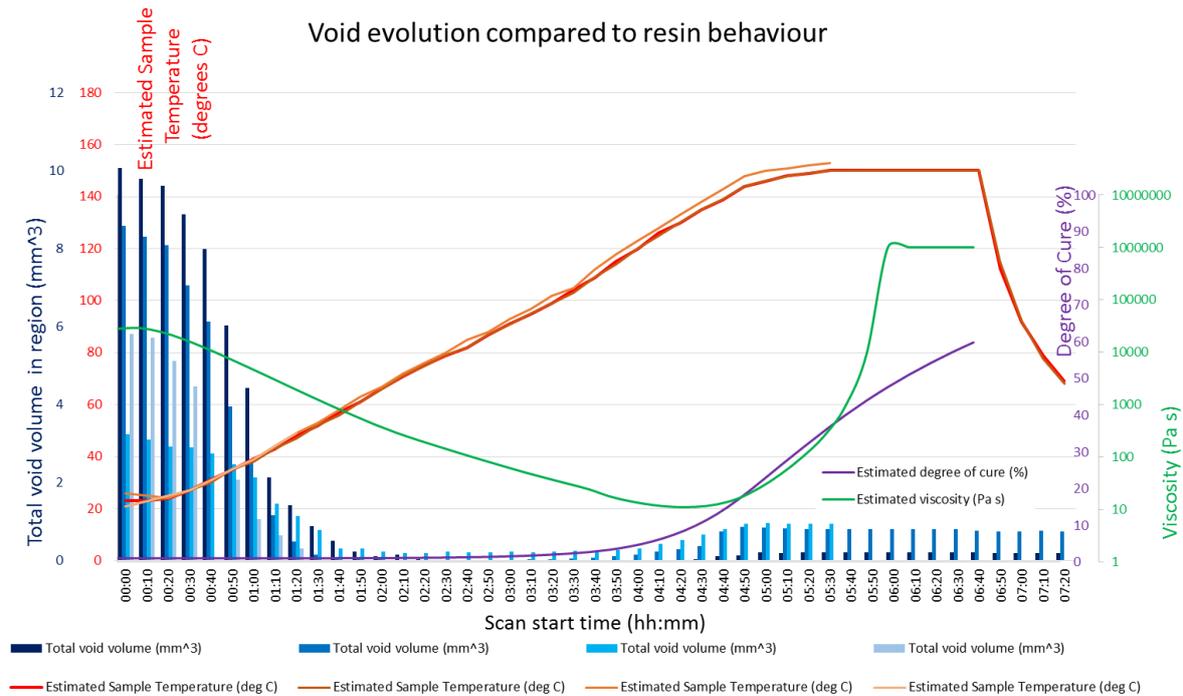


Figure 16: Comparison of void evolution with M21 behaviour

Figure 16 shows that the small voids which appear after the sample has consolidated do so around the region of minimum resin viscosity. We therefore theorise that these voids may be caused by release of moisture from within the resin as the resin pressure lowers. These voids appear to stabilise upon gelation, but may shrink very slightly as the sample cools- and hence shrinks as a whole- the change is too small to be considered definitive.

8 CONCLUSIONS AND FUTURE WORK

In-process micro-CT has the potential to provide far more detailed information on the behaviour of composites during cure than has been available prior to today. This work provides both proof of the principle, as an experimental method, and shows detailed results measuring the evolution of features during the cure of M21 prepreg.

From these results we can see that a 2mm wide AFP-style prepreg gap will essentially disappear as the sample heats up and consolidates, but the edges of the cut plies provide points where a void is likely to appear further on in the cure, and these small voids will remain in place, showing that despite being mostly filled, the original gap location remains a point of weakness.

The secondary voids appear at a time consistent with the lowest viscosity of the resin. We theorise that these voids are created by release of moisture from within the resin as it reaches its lowest pressure when the viscosity is low. It would be interesting to develop an X-ray transparent pressure chamber so that samples could be cured under pressure rather than relying solely on vacuum- this may affect the formation of the secondary gaps seen here.

Only a single defect type has been presented here. Study of different gap geometries and other defect types will give us greater insight into the evolution of defects during cure. Some such defect types will be presented in a future paper.

The CT scanning results could potentially be improved by use of a rotating target head. A different experiment may allow longer scans- or require shorter ones- and the general tradeoff of noise levels versus scan time must be considered when using this method. Using a standard industrial CT scanner has clear benefits in terms of cost and availability when compared to a synchrotron and the resulting scans were of more than sufficient quality for our purposes.

The method of In-Process CT scanning could also be applied to many other problems in composite manufacture, such as consolidation in a corner or tracking the effects of foreign bodies laid up into the laminate. As a proof of concept we consider this a success, and only the start of a potentially very interesting area of research.

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