STRUCTURE AND PROPERTIES OF LAMINATES MANUFACTURED USING THE QUICKSTEP TECHNIQUE

Leon. W. Davies, Alan Nesbitt and Richard Day

NORTHWEST COMPOSITES CENTRE
School of Materials, The University of Manchester, Manchester
Richard.Day@manchester.ac.uk

ABSTRACT

In the current work, Hexply 6376 has been used as a candidate aerospace grade prepreg material in an investigation aimed at optimising the cure cycle used in the Quickstep^{TM} process. The study attempts to match, or improve on, the physical and mechanical properties obtainable by the use of conventional autoclave processing.

Although the autoclave technique produces composite parts of high quality, the process is time consuming and has intrinsically high capital and operating costs; as a consequence, the application of such materials has been largely restricted to the aerospace and high performance automotive industries. Quickstep is a novel polymer composite manufacturing technique designed for out-of-autoclave, vacuum-bag-only processing of high quality, low-cost components with a reduction in cure cycle times.

Ramp rates of up to 15°C min^{-1} were achieved when composite parts were cured using the Quickstep process compared to 2°C min^{-1} for an equivalent autoclave process. The higher ramp rates resulted in a significant decrease in the minimum viscosity of the prepreg resin during processing. Starting from a basic cure schedule, the Quickstep cure cycle was manipulated in order to maintain resin viscosity at a low level for as long as possible in order to generate an extended processing window. The processing window was defined by a range of viscosity values during which fibre wet-out, and subsequent removal of voids from the system, was permitted before crosslinking caused the viscosity to rise rapidly. Through manipulation of the Quickstep cure cycle while the resin is at low viscosity, significant changes in the physical and mechanical properties of the product are demonstrated.

With the incorporation of a novel temperature spike into the Quickstep cure cycle, a panel void content comparable to that of autoclave produced panels was achieved (< 1% by volume). Based on this level of void content, a temperature spike, followed by a dwell period of 35 minutes, QSspike(35min), was found to be the optimum cure cycle. The flexural strength of QSspsike(35min) specimens was 6 % lower than those produced using an autoclave whereas the ILSS was 8 % higher. The lower flexural strength was postulated to be due to the presence of resin rich regions in the Quickstep panels, formed as a result of vacuum-only consolidation. The higher ILSS for the QSspike(35min) panels compared to the autoclave cured panels suggested an improvement in interfacial adhesion between fibres and resin. A Mode I fracture study also supported the idea of increased fibre/resin adhesion.

1. INTRODUCTION

Composite parts for the aircraft industry are often made using autoclaves. This is because good dimensional tolerances and low void contents, typically less than 1%[1], are required. Voids in the laminate can affect many properties including interlaminar shear strength [2-4], flexural strength [5-6] as well as the tensile and compressive properties [7-8]. Autoclaves are, however, expensive to purchase and operate due to the high pressures involved. Heat transfer between the nitrogen gas in the autoclave and the part to be cured is slow resulting in low heating rates being used. Hence the cure cycles can be quite long. Demand for volume production of aerospace components is increasing significantly due to the ACARE2020 vision and the economics of airlines both of which point to increased use of carbon fibre composites in aerostructures. It is widely believed that the volume of components would require a large investment in autoclaves, or else another out of autoclave route must be found in order to cope with this increase in demand. One technique which has been proposed is the Quickstep approach in which a heat transfer fluid is used, rather than nitrogen gas. The advantage of this change is that heat transfer from a liquid to a solid is typically twenty times better than heat transfer from a gas to a solid. Hence higher heating rates can be used without fear of uncontrolled reaction in the laminate. In order to prevent the heat transfer fluid from coming in contact with the laminate thin silicone rubber membranes are used. In this work processing conditions for a commercial prepreg, Hexply 6376, were established and the laminate properties were compared to those of laminates manufactured by other techniques.

2. EXPERIMENTAL

2.1 Materials

The prepreg used in this work was Hexply6376 and was supplied by Hexcel Composites plc. The resin is a toughened system primarily used for aerospace applications and consists of a mixture of epoxy resins with an amine hardener. The fibres used were unidirectional T800 carbon fibres.

2.2 Layup procedure

Laminates were laid up on a flat aluminium tool with dimensions 400 x 400 x 5 mm. All the composites prepared in this work were unidirectional and were constructed from 8 plies of prepreg. The dimensions of the composites were 130 mm wide by 160 mm long with the fibres aligned along the 160 mm length. As each layer was laid up a roller was used to exclude as much air as possible. A glass fibre reinforced PTFE was used as the release film and Elastomax 224 nylon was used as the bagging film. Both of these materials were supplied by Aerovac Systems Ltd. A nylon breather, Ultraweave 1032 was used and this was supplied by Tygavac Ltd. Debulking of the prepregs was performed after laying up the first 4 plies and then after all 8 plies had been laid up. In order to assist passage of trapped volatiles and air and also to reduce resin bleed a glass fibre membrane was positioned around the perimeter of the laminate.

2.3 Quickstep processing

The vacuum bag assembly was placed in the Quickstep chamber. A long tail with a vacuum connection was attached, thus allowing the vacuum connection to be made outside of the Quickstep chamber. This is illustrated in figure 1.

Figure 1: Quickstep chamber with vacuum bag

Consolidation was achieved using the vacuum and a pressure of 95 kPa or greater due to the vacuum was used in all cases. A further pressure of between 20 and 30 kPa was applied due to the head of fluid in the chamber above the laminate. The heat transfer fluid in the Quickstep equipment was preheated to the dwell temperatures required for processing. The fluid was then pumped into the chambers above and below the composite when required and heating rates of between 10 and 8 ^{o}C min^{-1}
Several curing cycles were investigated to observe their effect upon the mechanical properties and voids contents in the laminates. These were as follows:-

QSstraight

1. Room temperature to 175°C at an average of 10°C min^{-1}
2. Dwell at 175°C for 2 hours.
3. Cool from 175°C to room temperature at an average of 8°C min^{-1}

QSdwell

1. Room temperature to 130°C at an average of 10°C min^{-1}
2. Dwell at 130°C for 20, 30 or 40 minutes (QSdwell20, QSdwell30, QSdwell40, respectively).
3. Heat from 130°C to 175°C at an average of 10°C min^{-1}
4. Dwell at 175°C for 2 hours.
5. Cool from 175°C to room temperature at an average of 8°C min^{-1}

QSspike

1. Room temperature to 175°C at an average of 10°C min^{-1}
2. Immediately cool from 130°C to room temperature at an average of 8°C min^{-1}
3. Dwell at 130°C for 20, 35 or 45 minutes (QSspike20, QSspike35, QSdwell40, respectively).
4. Heat from 130°C to 175°C at an average of 10°C min^{-1}
5. Dwell at 175°C for 2 hours.
6. Cool from 175°C to room temperature at an average of 8°C min^{-1}

In each case three laminates were made under these conditions

2.4 Autoclave processing

The following cure schedule, as recommended by the manufacturer [9], was used to manufacture the composites.

1. Room temperature to 175°C at 2°C min^{-1}
2. Dwell at 175°C for 2 hours.
3. Cool from 175°C to room temperature at 2°C min^{-1}

The pressure was increased at the start of the cure at a rate of 0.5 bar min^{-1} until 8 bar was reached. At the end of the final dwell the pressure was reduced at the same rate. A vacuum of at least 95 kPa was maintained during processing. Three panels were prepared.

2.5 Oven processing

Laminates were manufactured in a Townson & Mercer recirculating air oven. The manufacturers recommended cure cycle was used. This is as follows;-

1. Room temperature to 175°C at 2°C min^{-1}

2. Dwell at 175°C for 2 hours.
3. Cool from 175°C to room temperature at 2°C min^{-1}

A vacuum of at least 95 kPa was maintained at all times. Again three laminates were made under these conditions.

2.6 Rheology

The viscosity of the resin used in the prepreg was measured at heating rates of 2, 5, 10 and 15 °C min^{-1} between 30 and 175 °C. A Bohlin Instruments rheometric scientific analyzer was used for all measurements.

2.7 Mechanical properties

Flexural testing was carried out using a Instron 4505 using three point bending according to the relevant British standard[10]. Interlaminar shear measurements were made using the same testing machine according the relevant ASTM standard [11]. Interlaminar fracture toughness was measured using a mode I double cantilever beam arrangement according to the ESIS standard [12]. Five measurements were made

2.8 Void content and fibre volume fraction

These were determined by hot acid digestion according to the ASTM standard[13]. The density of the composite samples was determined by pycnometry with nine samples being used. A density of 1.31 and 1.78 g cm^{-3} were used for the resin and fibre, respectively. These values were supplied by the manufacturers.

4. EXPERIMENTAL RESULTS

4.1 Viscosity

Figure 2 shows the viscosity of the resin against time at a number of heating rates. It can be seen that as the heating rate increased the minimum resin viscosity reduced by a factor of 3, thus making infiltration of resin and removal of voids easier, but the curing occurred much more quickly. The viscosity profiles for the Autoclave and QSstraight profiles are shown in figure 3

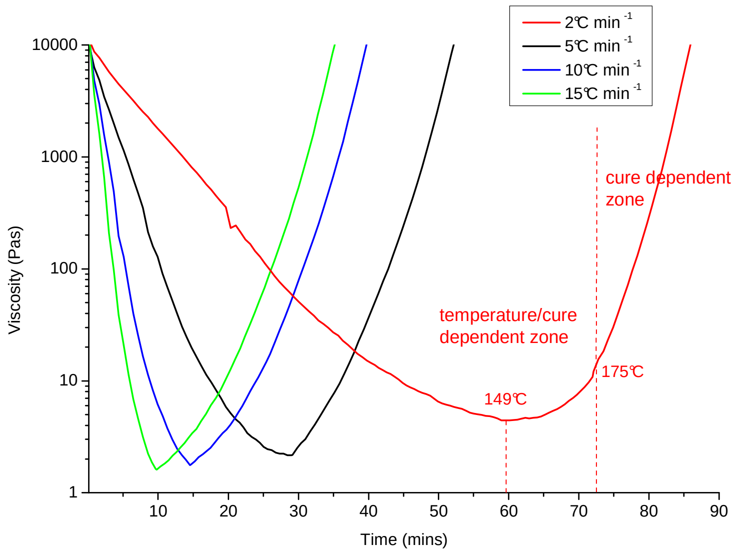

Figure 2: Resin viscosity as a function of temperature for a range of heating rates

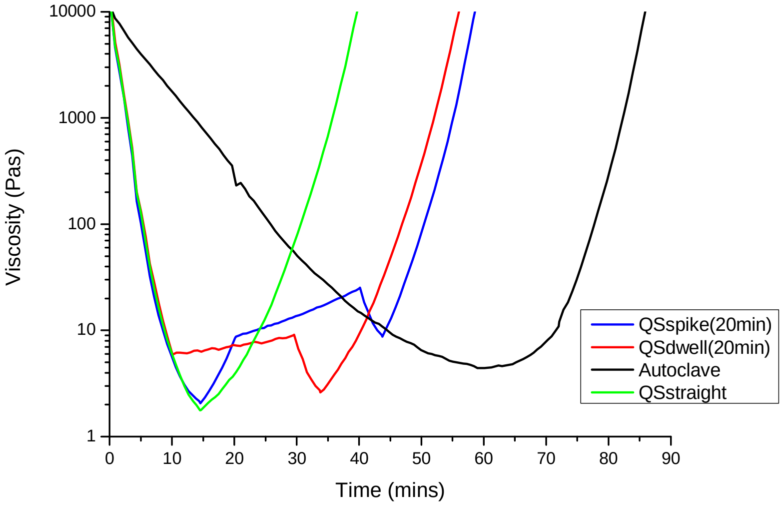

Figure 3: Resin viscosity for the autoclave and Quickstep cure cycles.

It is clear that using the QS spike cycle gives a lower viscosity for a longer period of time than the other cycles. Thus the removal of voids would be expected to be more complete for laminates cured using this cycle.

3.0 Mechanical properties

Table 1 below shows a summary of the mechanical properties of some the laminates manufactured under the different cycles described earlier. It can be seen that the includsion of a dwell in the Quickstep cycle improves the properties and that spiking the samples to 175 ^{o}C at the beginning of the cycle creates a further improvement in properties. Increasing the dwell in the spike cure cycle leads to properties that are similar to those obtained in autoclave cure. Figure 4 shows a polished section of a QSspike (35min) laminate. This contains few voids, but is slightly resin rich compared to the autoclave cured samples.

Table 1: Mechanical properties of the Hexply 6376 laminates

	QSstraight	QSdwell(20min)	QSspike(20min)	QSspike(35min)	Autoclave
Flexural Strength σ_{fs} (MPa)	1322 ± 65	1477 ± 99	1755 ± 47	1809 ± 53	1923 ± 39
Flexural Modulus E_f (GPa)	88.1 ± 3.5	93 ± 1.3	118 ± 3.9	119.0 ± 4	117 ± 1.1
ILSS (MPa)	71 ± 5	84 ± 5	115 ± 7	121 ± 3	111 ± 2
Void Content φ_v (%)	12.3 ± 1.7	8.1 ± 1.4	1.8 ± 0.8	<1	<1
Fibre Fraction φ_f (%)	49 ± 2.9	55 ± 2.0	60.2 ± 2.1	62.3 ± 2.4	64.1 ± 0.3

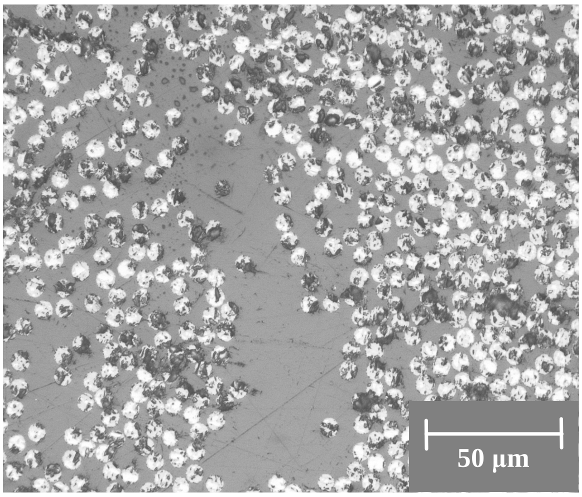

Figure 4: Optical micrograph of a polished section taken from a QSSpike (35mins) laminate

Table 2: Average panel thickness as a function of manufacturing route.

Panel	Average panel thickness (mm)
Autoclave	1.97 ± 0.02
QSstraight	2.25 ± 0.09
QSdwell(20min)	2.12 ± 0.10
QSspike(35min)	2.01 ± 0.08

It can be seen from figure 5 that the propagation values for the Quickstep and autoclave samples were similar, but the initiation value for the Quickstep samples was lower than that for the autoclave samples. This can be attributed to the presence of resin rich pockets close to the starter crack due to the lower resin viscosity during Quickstep processing and is thus probably an artefact.

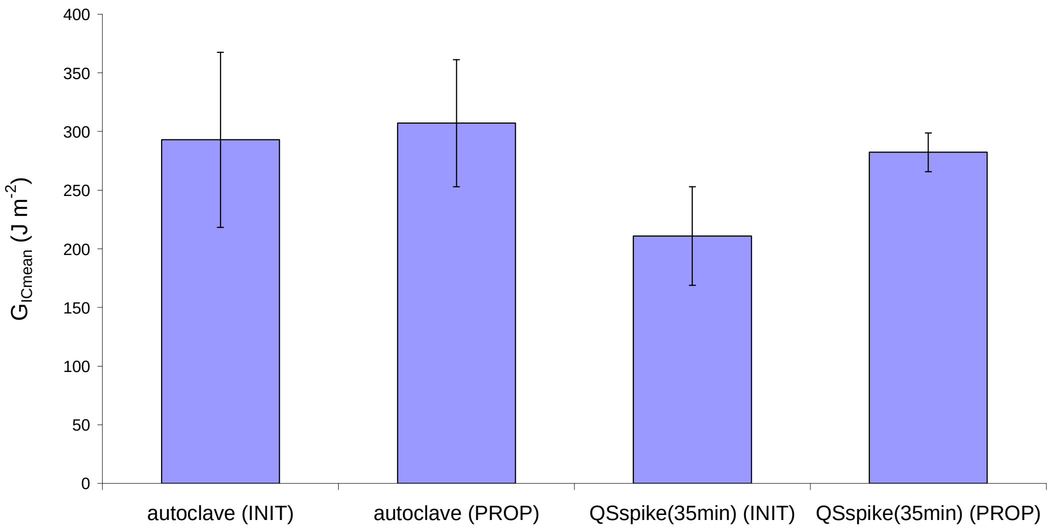

Figure 5: Comparison of the mean values of G_{IC}*(INIT)* and G_{IC}*(PROP)* for 6376 specimens fabricated using the autoclave and Quickstep processes.

5. CONCLUSIONS

It has been possible to use the Quickstep process to manufacture laminates of similar quality to those made in an autoclave. The prepreg used for this research was developed for autoclave curing. Further work will involve developing prepregs that are engineered for the quickstep liquid heat transfer process to optimize the mechanical properties and further reduce the cycle time.

ACKNOWLEDGEMENTS
LWD would like to that EPSRC for a scholarship to support this work. The authors are grateful for the support of Hexcel and Quickstep.

REFERENCES

1- Mouritz, A.P., “Ultrasonic & Interlaminar Properties of Highly Porous Composites”. *Journal of Composite Materials*, 2000. **34**(3): p. 219-239
2- Costa, M.L., Rezende, C.R., *Critical Void Content for Polymer Composite Laminates.* AIAA Journal, 2005. **43**(6): p. 1336-1341.
3- Hancox, N.L., *The Compression Strength of Unidirectional Carbon Fibre Reinforced Plastics.* Journal of Materials Science, 1975. **10**: p. 231-242.
4- Wisnom, M.R., Reynolds, T., Gwilliam, N., *Reduction in interlaminar shear strength by dicrete and distributed voids.* Composites Science & Technology, 1996. **56**: p. 93-101.
5- Almeida, S.F.M., *Effects of Void Content on the Strength of Composite Laminates.* Composite Structures, 1994. **28**(2): p. 139-148.
6- Hagstrand, P.O., Bonjour, F., Manson, A.E., *The influence of void content on the structural flexural performance of unidirectional glass fibre reinforced polypropylene composites.* Composites, 2005. **36**: p. 705-714.
7- Stone, D.E.W., Clarke, B., *Ultrasonic attenuation as a measure of void content in carbon fibre reinforced plastics.* NDT International, 1975. **8**(3): p. 137-145
8- Judd, N.C.W., Wright, W.W., Voids and their effects on the mechanical properties of composites. SAMPE Journal, 1978. **14**(1): p. 10-14.
9- *Hexcel Composites Plc: Hexply 6376 Product Data Sheet*, in *Duxford, Cambridge UK*.
10- *BS EN ISO 178 British Standard: Determination of Flexural Properties*. 1997.
11- *ASTM 2344/D: Standard test method for short-beam strength of polymer matrix composite materials and their laminates*. 2000
12- (ESIS), E.S.I.S., *Protocols for the Interlaminar Fracture Testing of Composites*. 1993: Delft, The Netherlands
13- *ASTM Standard D3171: Standard test method for constituent content of composite materials*. 2004