EFFECT OF A NOVEL PROCESSING TECHNIQUE ON THE PHYSICAL AND MECHANICAL PROPERTIES OF 977-2A CARBON/EPOXY COMPOSITES

Laraib Alam Khan, Alan Nesbitt, Richard J. Day

Northwest Composites Centre, University of Manchester, Sackville St., Manchester M60 1QD, UK

ABSTRACT

A woven fabric carbon fibre reinforced epoxy matrix composite (Cycom® 977-2A/6KHTA) has been used to investigate the effect of different processing techniques on its physical and mechanical properties. Composites are manufactured by oven curing, hot press and the 'Quickstep' processes. Quickstep is a novel polymer composite processing technique designed for out-of-autoclave processing of high quality, low cost components in comparatively shorter cure cycle time. Mechanical (Flexural strength, Interlaminar shear strength (ILSS)) and physical properties (fibre volume fraction, void content, glass transition temperature) of composites manufactured using Quickstep were found to be comparable with those made using thermal oven and a hot press. Oven cured and hot press panels were manufactured by the recommended cure cycle from the material manufacturer, however, the cure cycle employed for Quickstep was different based on the higher ramp rate achievable through Quickstep. Manipulation of cure cycle for Quickstep was done for further optimization to get the best properties at the shortest possible time. The time at two dwell temperatures (130 ºC and 180 ºC) was manipulated and the effects on physical and mechanical properties were evaluated. It was observed that when the dwell time decreases, resin rich areas formed in the panels resulted in the reduction of physical and mechanical properties.

1. INTRODUCTION

Despite of the many applications of thermosets/thermoplastics composites, the disadvantages of these materials have been high processing and equipment costs, difficulty of manufacturing and lack of knowledge of composites long term properties [1-2]. Vacuum bagging and autoclave cure have been well established and standardized methods and as these methods get matured, the areas of need arise in terms of low production cost without sacrificing part quality [3]. Quickstep is a novel polymer composite processing technique designed and developed in Australia for out-of-autoclave processing of high quality, low cost components in comparatively shorter cure cycle time. The technique is based on a unique fluid filled, balanced pressure, floating mould technology for the curing, partial curing and joining of Prepreg and dry fibre/wet resin based composites [4-7].

The process functions by placing a rigid mould between upper and lower bladder which contains the heat transfer fluid (HTF) circulating in a low pressure (10 kPa) environment. This is schematically shown in figure 1. Due to convective heat transfer mode, quickstep provides much faster ramp rate than autoclave. Faster ramp rates have been avoided in the past due to evolution of dangerous exotherms; however, the HTF in Quickstep acts as a large thermal sink capable of removing any excess heat generated by resin volatiles.

Defects, such as voids and bubbles cause the detrimental effects on the mechanical properties of composites materials. It is reported in the literature that mechanical vibrations applied during curing of a laminated composites can reduce the number of voids [8-9]. Quickstep uses a vibrator on top of the pressure chamber which facilitates the dispersion of air bubbles and also reduces the residual stresses by vibrating the HTF inside the chamber.

977-2A is one of the most prominent aerospace resin systems and typically used in aircraft primary and secondary structure, space structure, ballistics, cryogenic tanks or many applications where impact resistance and light weight are required [10]. The objective of this work is to achieve optimal cure cycle parameters of this material for Quickstep, whilst attempting to maintain mechanical properties rivalling those obtained from other traditional processes.

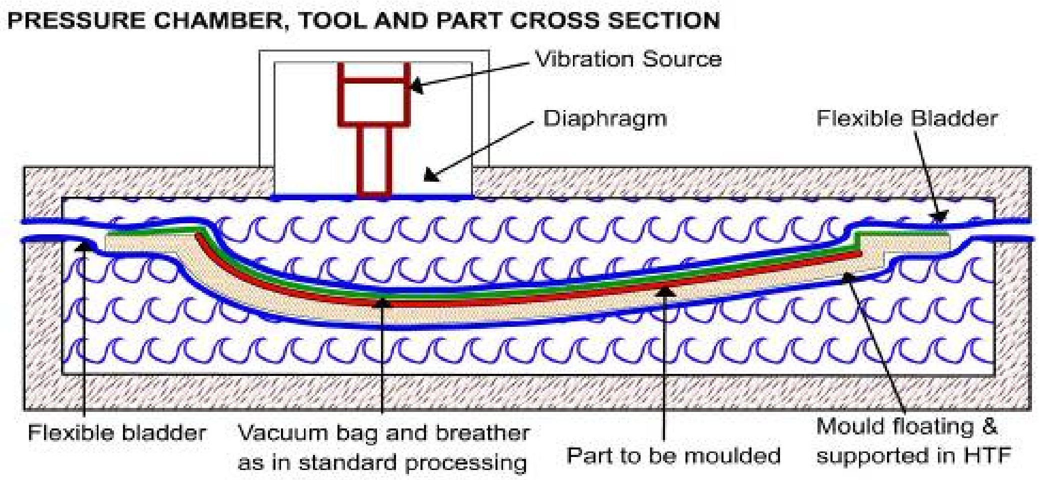

Figure 1: Schematic of Quickstep process

2. EXPERIMENTAL

2.1. Materials and lay-up procedure

As mentioned before, material being used in this study is Cytec Cycom®977-2A based prepreg, containing 177 ºC cure 977-2A epoxy resin (modified with thermoplastic) and five harness satin weave 6K-HTA carbon fibre reinforcement having volume fraction of 63%. The cured ply thickness is 0.362mm.

Since a five harness satin single ply is asymmetric about its mid plane and either the warp or weft tows run predominantly in one direction on either face, so plies are need to lay-up in such a manner to ensure that the laminate has balanced fibre direction through its thickness, to avoid any fibre distortion in the part after cure. For this purpose, it is assumed that side dominated by warp fibres is on the top while if it were on the bottom, it would be considered as flipped. In this study, the plies were stacked in the warp direction back to back like flipped pairs so that the whole stacking sequence was semi symmetric about the mid plane $[0/0_f]$ [11].

All prepared laminates consisted of six plies $[0/0_f]_3$ were 200mm in width and 160 mm in length with the fibres aligned in the warp directions.

2.2 Experimental Procedures

All the cured panels were first C-scanned using a Midas NDT System. A 5 MHz frequency probe with 720V excitation voltage was used to check and compare the panels qualitatively. The dynamic range and the scan speed were fixed at (0-74) db and 200 mm/sec respectively for all the scans.

The test coupons of different sizes were then cut from the panel for physical and mechanical characterization. Flexural and interlaminar shear strength (ILSS) testing were conducted on an Instron 4411 test apparatus using three point bend jig according to British Standard BS 2782 [12] and ASTM D2344 [13], respectively. Five specimens were tested from each panel for each test.

The fibre volume fraction and void content were determined by using hot acid digestion method according to BS 2564 [14]. Five specimens from different location of the panels were tested.

The glass transition temperature (T_g) was determined using a Perkin Elmer Pyris diamond dynamic mechanical analyser (DMA). The used experimental conditions were; heating rate (5 ºC/min), 1 Hz frequency, Double cantilever beam (DCB) based three point bending and temperature range of 30-300 ºC. The peak of tan δ curve was taken as T_g.

The viscosity of neat 977-2A resin was obtained using 25 mm diameter parallel plate in Rheometrics RMS 800 System. Two heating rates of 2 ºC/min and 10 ºC/min were employed with a constant frequency of 1 Hz. The resin was heated at prescribed heating rate to 175 ºC where the temperature was held until gelation of resin occurred.

SEM micrographs of fractured surface of flexural and ILSS specimens were taken from Hitachi S-3000N. The fractured specimens were carefully cut from the middle of the specimen and the surface was then examined in a SEM to analyse qualitatively the fibre/matrix adhesion.

2.3 Material Processing

2.3.1 Vacuum Bagging

Composites for oven and Quickstep curing were fabricated using a conventional vacuum bag lay-up as illustrated in figure 2. The laminates were de-bulked twice for 15 minutes after laying-up of two and six plies.

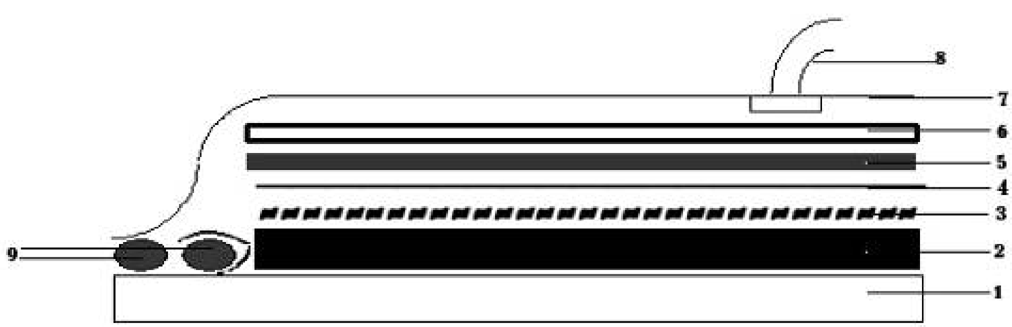

Figure 2: Vacuum bag assembly for Quickstep and oven curing (1) Tool plate (2) Laminate (3) Peel Ply (4) Solid release film (5) Caul Sheet (6) Breather (7)Bagging film (8) Vacuum Line (9) Sealant tape

2.3.2 Hot Press Curing

In hot press curing, prior to manufacturing, the mould was sprayed with Loctite 1711 Frekote (used as mould release agent). A steel window frame of size (160 X 210) mm was used to keep the laminate within the area under high pressure load. The laminate in the frame was then pressed between the two halves of a heated mould and the transformation into solid product was done under the effect of elevated temperature and pressure. The temperature was monitored and controlled through a PID controller during the entire cure cycle.

2.3.3 Cure Cycle

Since Quickstep uses higher process ramp rates which cause the reduction of resin viscosity, thus facilitating the proper wetting of fibres and application of high vacuum pressure remove the air bubbles produced during the process. The viscosity profile for neat 977-2A resin is shown in figure 3. Based on the viscosity profile, a spike cure is used for Quickstep processing. The cure schedule recommended by supplier for oven/press curing and those employed for Quickstep processing is given below.

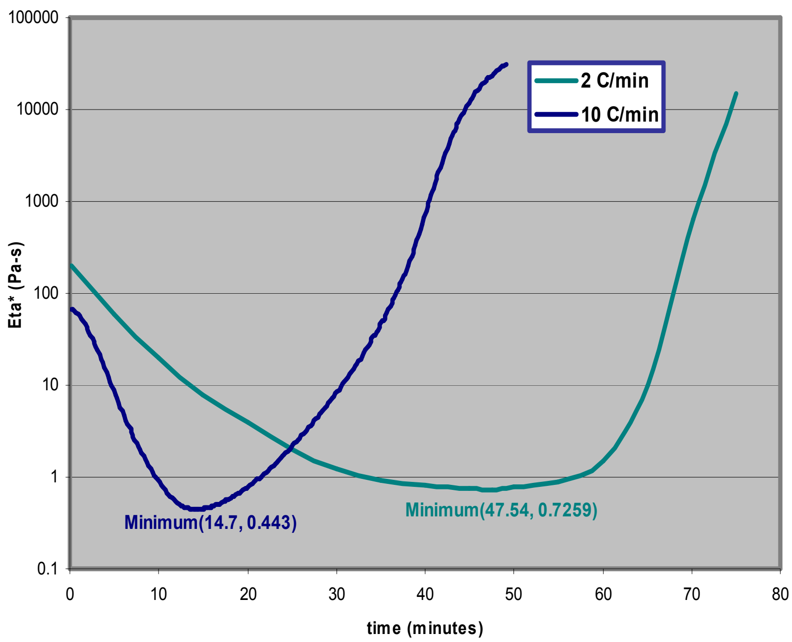

Figure 3: Viscosity Profile of 977-2A as a function of heating rate

2.3.3.1 Supplier Cure Cycle

A two-step cure cycle was recommended by the material manufacturer. The steps include;

1. Heat from room temperature to 130 °C at 2 °C/minute.
2. Isothermal Dwell for 60 minutes at 130 °C.
3. Heat from 130 °C to 180 °C at 2 °C/minute
4. Isothermal dwell for 120 minutes at 180 °C

2.3.3.2 QuickStep Cure Cycle:

The cure cycle used for Quickstep processing included the following steps.

1. Heat from room temperature to 175 °C at 10 °C/minute
2. Isothermal dwell for 10 minutes

3. Cool from 175 °C to 130 °C at 10 °C/minute
4. Isothermal dwell for X minutes
5. Heat from 130 °C to 180 °C at 10 °C/minutes
6. Isothermal dwell for Y minutes

Where X and Y were varying times and the effect of these times on the material properties was evaluated.

3. RESULTS AND DISCUSSION

Figure 4 shows the C- scanned images of hot press, Quickstep and oven cured panels. Uniformity of the fibre/resin distribution can be observed from hot press and Quickstep panels' images, however, the presence of resin rich areas (highlighted over the image) was observed in most of the parts of the oven cured panels.

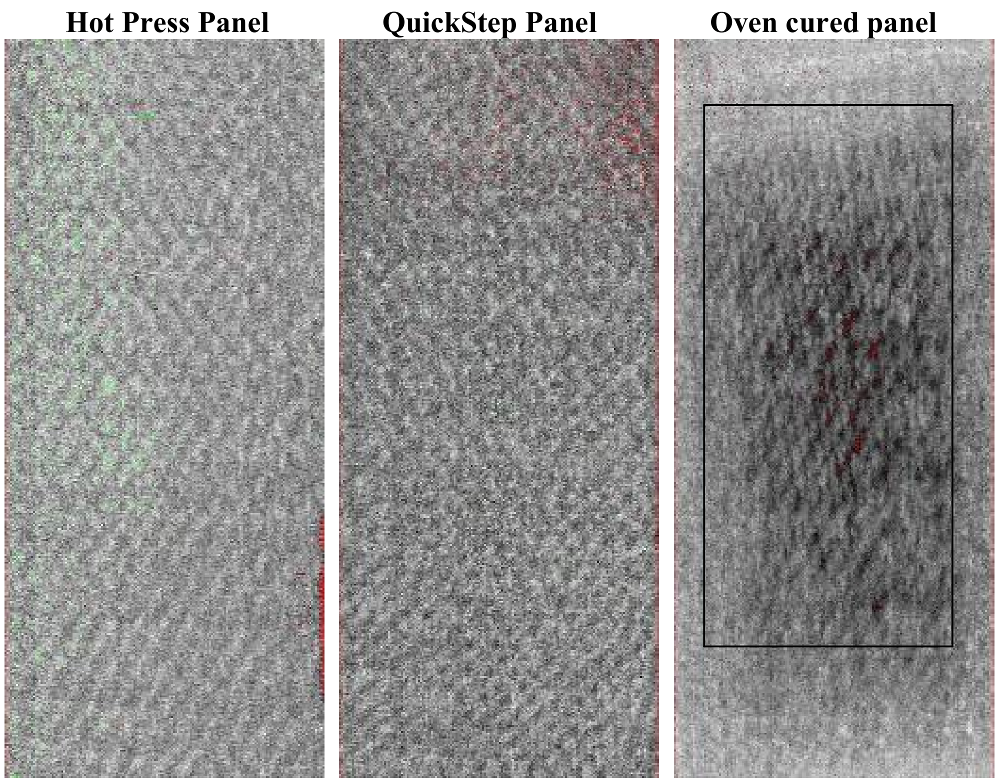

Figure 4: Ultrasonic Scanned images of hot press, Quickstep and oven cured panels.

Table 1 shows the average values of flexural strength, ILSS, fibre volume fraction, void content, glass transition temperature and processing time obtained for the panels manufactured from hot press, Quickstep and oven curing. The processing time excludes

-any set-up time

-cooling time from 180 ºC to room temperature after completion of cure cycle

Full curing schedule was applied to Quickstep Processing i.e. X and Y were fixed at 60 minutes and 120 minutes respectively.

Table 1: Physical and mechanical properties of oven cured, hot press and Quickstep specimens

Process	Flex. Strength (MPa)	ILSS (MPa)	Fibre Volume (%)	Void (%)	T_g (°C)	Avg. Panel thickness	Processing time (min)
Hot Press	1332 ± 23	82 ± 4	61.4	1.04	195	1.96	260
QS	1258 ± 66	84 ± 2	60	1.70	199	2.00	210
Oven	1093 ± 31	64 ± 3	55.9	3.56	195	2.06	260

As mechanical properties of composites are heavily dependent on the reduced void contents, it was observed that despite of slightly higher void content, the flexural strength and ILSS values for Quickstep panels were almost equal to hot press panels. The slightly superior flexural strength of hot press panel was however, primarily due to a higher fibre volume fraction as can be observed from C-scan images in figure 4.

The Quickstep cured specimens also exhibited a higher glass transition temperature. Also, on average, QuickStep specimens were 4mm thicker than press cured specimens. Since Quickstep works on low pressure (applied during cure), therefore thickness/volume fraction effects were attributed to reduced resin bleed out. Quickstep also showed a considerable reduction of 24% in processing time as compared to supplier's recommended cure schedule. This was achieved due to precise temperature control and high heating and cooling rates that are possible with Quickstep.

Since the hot press and oven curing used the same curing schedule as recommended by supplier, but the difference of pressure application highlighted the effect on physical and mechanical properties. The presence of resin rich areas in most part of the oven cured panel resulted in the decreased fibre volume fraction and thus can be correlated to an increased average thickness of the cured panels. Also, high void content and less fibre volume fraction produces poor fibre/matrix adhesion in oven cured panels, resulting in reduced flexural strength and ILSS values.

Figure 5 shows the SEM micrograph of cross sections of flexural fractured specimens from hot press, Quickstep and oven cured panels.

Hot Press Quickstep Oven

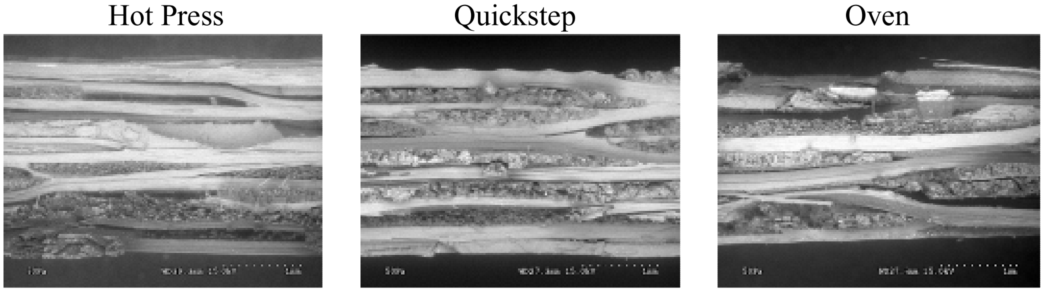

Figure 5: SEM images of bending fractured specimens

As resin viscosity plays an important role in the product quality during whole vacuum process, it was suggested that lower viscosity region over the duration of cure cycle can produce a part with low void content. Based on this suggestion, Quickstep cure cycle was manipulated with several dwell time and temperature combinations and was characterized. Presenting of all results in this paper was needless, however, some interesting results are provided here.

Table 2 presents the nomenclature and description of panels manufactured with different combinations of dwell times at 130 ºC (X) and 180 ºC(Y).

Table 2: Isothermal dwell times at 130 ºC (X) and 180 ºC (Y)

Nomenclature	X	Y
Q1	45	120
Q2	45	105
Q3	60	75

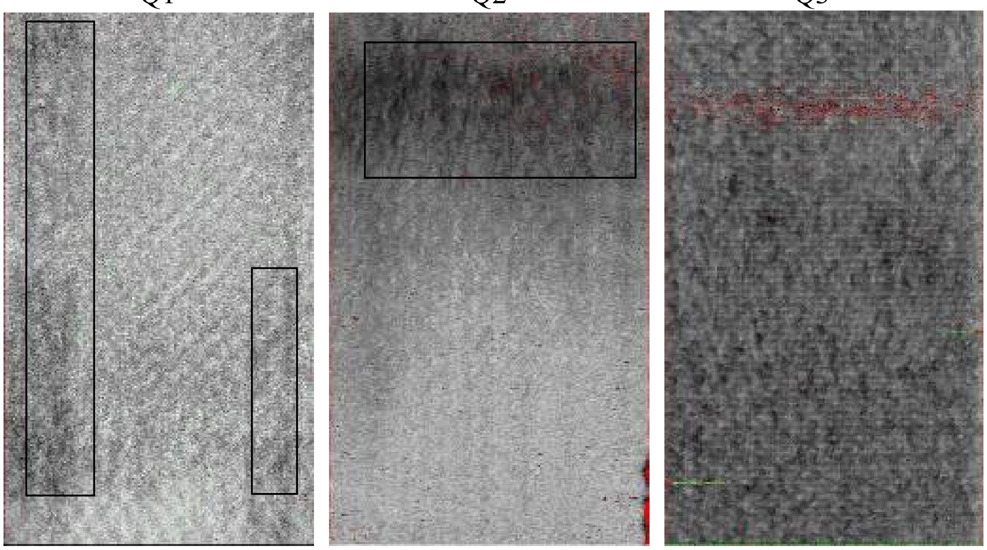

Figure 6: Ultrasonic Scanned images of Q1, Q2 and Q3

Table 3: Physical and mechanical properties of Q1, Q2 and Q3 Specimens

Panels	Flex. Strength (MPa)	ILSS (MPa)	Fibre Volume (%)	Void (%)	T_g (ºC)	Avg. Panel thickness	Processing time (min)
Q1	1301 ± 30	76 ± 7	60.4 ± 7	1.98	198	2.02	201
Q2	1200 ± 59	78 ± 7	59.8 ± 5	1.91	192	2.03	185
Q3	1259 ± 94	70 ± 1	60.2 ± 3	2.23	184	2.01	181

Reduction of dwell time was found to affect the quality of the panel, which are shown in C- scanned images in figure 6. However, the mechanical and physical characterization provided some interesting results.

Flexural strength, fibre volume fraction, void content and average panel thickness were found to be almost same irrespective of the cure cycle dwell time. However, the higher standard deviation values shows the non-uniformity of the fibre/matrix distribution, which is also depicted from C-scanned images. Two specimens were cut from resin rich areas and three from the uniform areas for the entire characterization. The average values with standard deviations are shown in table 3.

As expected, the ILSS values decrease with the void content of the laminates, however, the SEM analysis of ILSS fractured specimens shows that the tested specimens failed from the fibre/matrix interface rather than they emanated from voids [15] as shown in figure 7.

The glass transition temperature values also significantly effected and a reduction of 8% is found when the time is reduced to 75 minutes at 180 ºC (Q3). As the time increases, the T_g value increases as shown in table 3. This reduction was attributed to incomplete curing reaction.

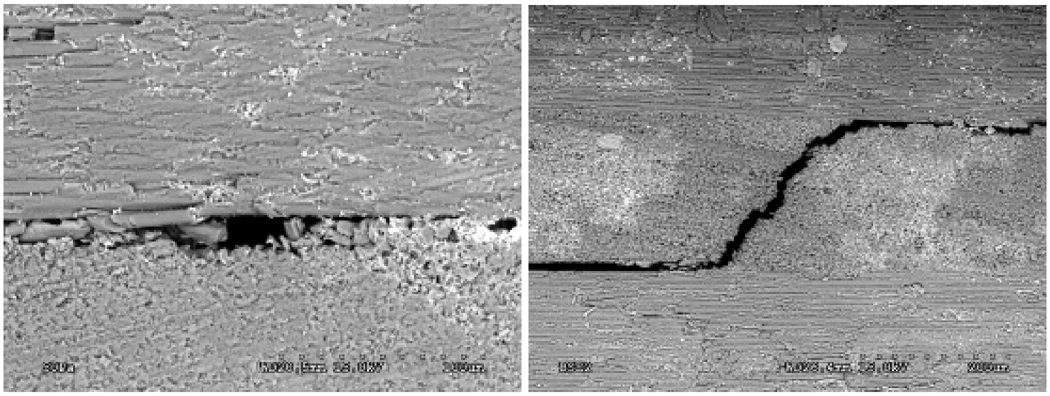

Figure 7: SEM images of ILSS fractured specimen

4. CONCLUSION:

977-2A is one of the prominent aerospace resin system and Composite laminated panels were manufactured and evaluated using Quickstep, hot press and oven curing from 977-2A carbon/epoxy prepreg. Quickstep panels were manufactured using a cure schedule which was proposed on the basis of minimum viscosity of resin, while hot press and oven cured panels were processed from material supplier's recommended cure cycle. The uncured laminates were stacked carefully due to asymmetric nature of five harness satin fabric.

Despite of slightly higher void content, the flexural strength and ILSS values for Quickstep panels were found to be almost equal to hot press panels. The Quickstep cured specimens also exhibited a higher glass transition temperature, 4mm thicker than press cured specimens with a considerable reduction of 24% in processing time as compared to supplier's recommended cure schedule.

A reduction of 13% and 24% in flexural strength and ILSS respectively were observed however, in the resin rich oven cured panels. These resin rich areas resulted in lower fibre volume fraction and higher void content thus depicted the poor fibre/matrix adhesion in the oven cured panels.

Quickstep cure cycle was then manipulated and reduction of dwell times at 130 ºC and 180 ºC were found to produce some resin rich areas on the corners of the panels. Despite of resin rich areas, the properties were found to be in respectable range, however, higher standard deviation values represented the non-uniform distribution of fibre/matrix in the cured laminate.

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