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Article:

Qureshi, Z., Swait, T., Scaife, R. et al. (1 more author) (2014) In situ consolidation of thermoplastic prepreg tape using automated tape placement technology: Potential and possibilities. *Composites Part B: Engineering*, 66. pp. 255-267. ISSN 1359-8368

<https://doi.org/10.1016/j.compositesb.2014.05.025>

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Manuscript Number: JCOMB-D-14-00158R1

Title: In-situ consolidation of thermoplastic prepreg tape using automated tape placement technology: potential and possibilities

Article Type: Full Length Article

Keywords: A. Carbon fibre; B. Mechanical properties; E. Automation; E. Autoclave

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Abstract: The key parameters of the in-situ consolidation of carbon fibre reinforced poly-ether-ether-ketone (AS4-CF/PEEK) by automated tape placement (ATP) process were investigated by manufacturing of continuous rings and by laying tape onto pre-consolidated laminates. In order to establish and understand correlations between the process parameters and mechanical properties, a number of parametric studies were performed by manufacturing and testing the interlaminar shear strength, single lap shear strength and fracture toughness samples. The main process parameters investigated were the compaction force, tape laying speed and tool temperature. To achieve a uniform heat distribution across the thermoplastic tape, a new nozzle was designed. Baseline samples were also manufactured using the autoclave process to provide a comparison for the ATP composites produced. Optical microscopy was used for investigating the microstructure of composites compared. It was found that increasing the tool temperature reduced the temperature gradient between the incoming tape and substrate, resulting in better lap-shear strength and fracture toughness properties.

To: Editor of Composites: Part B

Dear Prof. Feo

Hope you enjoyed the Easter break.

I would like to resubmit the revised version of manuscript ref. JCOMB-D-14-00158 and entitled "In-situ Consolidation of Thermoplastic Prepreg Tape Using Automated Tape Placement Technology: Potential and Possibilities" for possible publication in Composites Part B: Engineering.

We met all the reviewers comments and changes, a list of author response and answers to comments including the changes and additions to the manuscript, is made and enclosed with the revised manuscript.

Yours Sincerely



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Authors Response to Reviewers Comments for Manuscript JCOMB-D-14-00158

Dear Editor - Please find below the author response and answers to the reviewers' comments (given in [blue](#)) and the corresponding changes in the revised manuscript (highlighted in [blue](#) as well).

Reviewer #1

1. Page 2, para 2: do you have any references or data to support this claim?

[Author:](#) New reference [2] is added to support this statement.

2. Page 8: line 40: what is the justification for using two 8 micron films at the interface?

[Author:](#) Two layers of PEEK film were used in order to obtain noticeable differences in mechanical performance. This resin rich interface was used to tape place the first ply onto using the maximum tool temperature of 150°C.

In addition to justify why two layers of PEEK film (8um each) were used, because the following; the resin content (RC) in the prepreg is 33% by weight which was just enough for the interfacial bonding between the plies, but to increase the resin on the surface for the mentioned purpose (above and in page 8), a reasonable amount (approximately 10%) of the resin (2 layers of PEEK film) was added to enrich the laminate surface, where one layer of PEEK film (10.4gsm) was not noticeable w.r.t the prepreg areal weight (218gsm).

3. Page 11, line 13: It is showed... Language needs correction

[Author:](#) The sentence "It is showed that the gas flow rate has insignificant effect on temperature" has corrected to "It is found that the effect of gas flow rate on temperature was not significant"

4. Page 12, line 2: baseline ILSS values need to be presented in the tables (table 8?)

[Author:](#) done

5. Page17: excluding ASTM standards, there are limited references

[Author:](#) New references are added

Page 18: Figure 2 a,b: not easy to see the differences between conventional and new nozzle -may benefit form a simple sketch

[Author:](#) Figure 2 is modified and a new schematic sketch for the new nozzle and the conventional is added as; d) schematic diagram of new nozzle (left) compared to the conventional one (right).

Figure 2c: I cannot see temperature profile

Author: Figure legend 2(c) [2(b) in the revised version] has corrected to show where the temperature readings were taken of the roller not the temperature profile of the roller. i.e. Temperature profile → b) Zones at where the temperature readings were taken of the nip roller.

Page 19,21: there are several small tables: it may be advantageous to combine them

Author: Tables 1 and 2 are merged into one table (Table 1 in the revised version) and Tables 3 and 4 are also merged into one table (Table 3 in the revised version).

Page 20: table 3: incoming tape tension: should this be in N?

Author: The unit of tension is converted to Newton and its value changed to 75.84kN/m² instead of 10Psi (please see Table 2 in the revised version).

Page 22: figure 4 not clear: close-up views recommended

Author: done

ILSS and LSS values for in-situ consolidated samples are significantly lower than autoclave samples. Some discussion is required whether these values are acceptable for potential applications

Author: A new paragraph has been added in page 14 “It is obvious that the ATP processed ring samples exhibited approximately 55% of the ILSS and LSS values compared to the baseline autoclaved samples. In order to achieve the optimum of autoclave ILSS, the ATP samples have to be post-consolidated to fulfill for example the aerospace requirements, but without post-consolidation the ATP ILSS might be acceptable for commercial applications such as oil and gas pipes, which are normally made of low melting thermoplastic polymers”

In addition, to carry out like-to-like comparison, a second baseline sample (ring) was made from the same tape (12.7mm wide) using ATP and autoclave post-consolidated (Please see [page 12](#), Figure 5.b and Table 6 in the revised version).

Reviewer #2

It is not clear from the description that, apart from the difference in nozzle width, all other nozzle design parameters are the same. A diagram showing the essential dimensions of the nozzles would be more informative than the photographs presented. I think the dimensions of the roller should also be given, so that the reader can gain an understanding of the importance of the nozzle width in relation to the roller length.

Author: Figure 2 has been modified to include a schematic diagram of the two nozzles: [Conventional: 12mm wide (inner diameter = 10mm) and New: 20mm wide (inner width=18mm)] used and their dimensions, the roller dimensions (diameter = 12mm and surface width = 19mm) are also added into the figure.

The temperature readings were taken at the bottom and the middle of the roller, as indicated in Fig.2. Bearing in mind it is stated that (p5 paragraph 3) the heat source is 'directed at the nip-point of the incoming tape, substrate ply and pressure roll', then I think it is important to know the distances marked x in Fig.2, in particular of that referred to as the 'Bottom'. Some explanation should be given as to why the authors think measuring the pressure roller surface temperature is a good indication of what the material temperature would be.

Author: It was difficult to measure the material temperature at the nip point as this zone is always glowing and wasn't suitable to use the infrared thermometer. That is why we decided to measure the temperature across the roller surface which will be close the contact point (roller-material) considering the heat dissipation to the surrounding.

Fig 2.c is modified to show the distances between the marked points of measuring the temperatures on the roller surface.

The temperature measurements were made according to tables 1 and 2. I note that $T_{NG} = 975^{\circ}\text{C}$. Yet the results given in Figs 9, 10, 11 & 12 show different values of T_{NG} (Vertical axes). This may be a minor error of notation, but it is confusing for the reader.

Author: The T_{NG} of 975°C is the nitrogen gas temperature measured by a thermocouple fitted inside the nozzle (please see figure 2), but to avoid this confusion the "Temperature of nitrogen gas (T_{NG})" is changed to "Roller temperature (T_R)" on figures 9-12.

The notation of T_{HG} is corrected to T_{NG} and the same for the gas flow rate.

In the text of the results (p10, 1st paragraph, 1st sentence) the description of the distances at which the nozzles were placed do not concur with tables 1 and 2. The tables and text should be consistent. It would also be useful to know why a wider range of distances were chosen for the wider nozzle.

Author: A range of separations (12, 15, 20, 25mm) was studied but Tables 1 and 2 (Table 1 in the revised version) just give a selection of results at two distances 12mm and 20mm for like-to-like comparison between the two nozzles. Same distances were used for both nozzles and we didn't use wider range with the wider nozzle (please see Table 1 in the revised version).

Fig 2 shows (poorly) the location of X – the position of the nozzle from the roller. This distance is a key variable in these initial experiments. Looking at Fig.1, I think it would be more informative to show the X position here. This is important because it would appear that as X changes, the nozzle angle of inclination to the nip point changes, and therefore the impact area of the gas may also change. If this is so, the authors should indicate why the angle of inclination was not considered important in their work. It may be that the holder of the nozzle had to be adjusted to ensure the gas direction was still focused at the nip line. However, this is not clear from the text.

Author: The distance X was measured from the nozzle tip to the nip line.

Yes that is right the nozzle angle of inclination appears when the nozzle-roller distance changes, but to correct this, the nozzle holder was smoothly adjusted forward/backward and rotated up/down to ensure its tip (hot gas flow) is always directed toward the nip line between the incoming tape and the pressure roller (impact area of the gas) [such paragraph is added on [page 5](#) of the revised version].

The discussion of the results given in Figs 9 to 12 is very limited, and leaves the reader with a number of unanswered questions. In the text of their discussion, the authors suggest that the increased gas flow increases the roller temperature because the heat loss is reduced owing to the shorter “interaction time between the nitrogen gas and the surroundings” (p11, 1st paragraph, 1st sentence). However, no temperature measurements of the “surroundings” – immediate environment?? – were taken. It seems more likely that with increased gas flow the rate of heating becomes greater than the initial rate of heat loss.

Author: A new paragraph including some temperature measurements and infrared image is added to [page 11](#).

“On processing the prepreg tape, trials were also carried out to determine the material temperature. By considering the main source of heat (Nitrogen Gas) having temperature of 975°C and this heat has been transferred into surroundings: nip roller, tape and mandrel. It was difficult to measure the actual temperature of the tape and the nip region due to the poor infrared reflection from the glowing zone. But by measuring the temperatures of nip roller at the closest point near to the glowing zone, the mean value of temperature was found to be 514.33±12.5°C. This is confirmed by another infrared image analysis made by the ATP manufacturer (ADC) [2], which reported that the temperature of the nip region was estimated to be around 500°C (See Fig. 13 in the revised version).

I think it is important to indicate how many readings were taken per data point and, whether repeatability of results was considered for each experiment. The data points would tend to show that although there are differences in temperature between locations on the roller, the effect of gas flow rate is very small, and could indeed be negligible. Therefore statistical tests of significance should have been carried out. Nevertheless, the effect of gas flow seems too small to have any meaningful effect.

Author: The temperature was measured three times per data point and the mean value was taken. One of the parametric study aims was to figure out the effect of nitrogen gas flow rate on the heat distribution and roller temperature profile. By studying different range of gas flow rate (50 - 100L/min) with different nozzles, it was concluded that the effect of gas flow was not significant and the consistent results obtained by the new (wider) nozzle at 100L/min was the preferable.

The distance X would seem to have had a positive effect only with the use of the narrow nozzle, and then the resulting data points show a confused picture, i.e. the front middle position increasing in temp with distance, but other locations decreasing. Fig.11 and 12 clearly shows that the wider nozzle gives a more uniform temp, but the values are much lower, particularly with increased distance. In general the bottom roller position has much lower temps, yet this is near the all important nip point. The inference is that the nip point temperature could be even lower. In view of this, I think the authors should give consideration to the resin rheology, in respect of the changes in its viscosity with temperature. This seems fundamental to their work and may account for the poor mechanical results associated with the ATP part of their subsequent experiments. In other words, unless the melt flow viscosity is suitably low for good impregnation, their whole experiment is of little value since a high void content would be inevitable in the resultant composite.

Author: It is good point and it is very useful to carry out the rheological studies for the resin used in the tape which is PEEK-150. In this work we were not able to do this study as we don't have the raw polymeric material of resin (PEEK-150 powder) that the prepreg manufacturer used, but the company has provided the rheological data of this resin, new paragraph and figure 14 out of this are added into [page 11](#) in the revised version.

“In addition and in order to consider the shear thinning of the PEEK (due to the dynamic and rapid application of pressure of ATP), polymer squeeze flow (in the nip zone), and other reasons which significantly enhance the resin inter-diffusion over the static (autoclave or press forming) case of in intimate contact, a rheological study was made by ADC [2]. Figure 14 [2] shows the shear viscosity versus the shear rate at three processing temperatures: 360, 380 and 400°C. August et al. [2] found that a dramatic shear thinning was occurred in PEEK resin even at low process temperatures.”

On page 11, 2nd paragraph, last sentence, the authors state that “Therefore, all the thermoplastic rings made from the 12.7mm wide tape were manufactured by using the new designed nozzle instead of the narrower one which was suitable for processing 6.3mm or less wide tapes.”, the question is if they knew the narrower width nozzle was only suitable for processing tape widths of 6.3mm or less, why use this nozzle for the experiment with the 12.7mm tape?

Author: This was the only available/provided nozzle (Ø12mm) to use and when we tried to use it for processing 12.7mm tape, we found (based on the parametric studies) that this nozzle was not appropriate to heat the full width of 12.7mm tape and therefore it was decided to design a new (wider) nozzle of rectangular tip (cross section) for processing ½” (12.7mm) and ¾” (19mm) tapes. Processing wider tapes offers higher material deposition rates (kg/hrs.). One of the key findings out of this; the stated sentence/conclusion “..... *the narrower one which was suitable for processing 6.3mm or less wide tapes*”

The experiment comparing the ATP process and autoclave baseline uses two differing tape widths. These I assume are slit tapes, i.e. tape widths cut from UD prepreg. If this is the case, then there is likely to be broken filaments at the edges of the tapes. Since the tape used for the hand layup autoclave process was 12.5 x wider than the tape used for the ATP, I think this would immediately place the ATP process at a disadvantage in terms of resultant mechanical properties; in which case this part of the work may be flawed.

Author: To avoid this problem and to carry out like-to-like comparison in addition to the flat panel, a new baseline ring is made using ATP at high speed and then post consolidated in autoclave. The baseline ring is made from the same tape (12.7mm wide); hence curved specimens are mechanically tested to give fair comparison with the in-situ consolidated samples. (Please see [page 12](#), Figure 5.b and Table 6 in the revised version).

Additions by Author:

A new [Figure 24](#) is added to show a comparison between the ILSS values obtained from three different processing technologies: ATP (Hot gas), ATP (Laser) and Autoclave. This is to figure out how far the current technology ATP (hot gas) from the state-of-the-art technologies (ATP-laser and Autoclave), the following paragraph is also added to [page 16](#)

“Generally and in order to compare the current ATP hot-gas system used in this study with the state-of-the-art laser ATP technology, an ATP laser processed sample (by ADC) was provided. Figure 24 shows a bar-chart comparison in terms of the ILSS values of three samples processed by three different technologies: ATP hot-gas, ATP laser and Autoclave. From Table 6 and figure 24, it found that the ATP hot-gas (Gas-ATP) only achieved 55% ILSS of the autoclave baseline ILSS but still 30% less than the laser-ATP one, which achieved 85% of the autoclave ILSS. According to the aerospace requirements, the laser-ATP still needs post consolidation (using autoclave) to gain the remaining 15% of the baseline ILSS, i.e. ATP’s (Gas and Laser) exhibit unacceptable ILSS compared to the autoclave one and both need post consolidation to achieve the optimum of ILSS. The current ATP (hot-gas and laser) system can be used as a rapid preforming process that might be cost effective technology compared to the hand layup.”

In-situ consolidation of thermoplastic prepreg tape using automated tape placement technology: potential and possibilities

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Abstract

The key parameters of the in-situ consolidation of carbon fibre reinforced poly-ether-ether-ketone (AS4-CF/PEEK) by automated tape placement (ATP) process were investigated by manufacturing of continuous rings and by laying tape onto pre-consolidated laminates. In order to establish and understand correlations between the process parameters and mechanical properties, a number of parametric studies were performed by manufacturing and testing the interlaminar shear strength, single lap shear strength and fracture toughness samples. The main process parameters investigated were the compaction force, tape laying speed and tool temperature. To achieve a uniform heat distribution across the thermoplastic tape, a new nozzle was designed. Baseline samples were also manufactured using the autoclave process to provide a comparison for the ATP composites produced. Optical microscopy was used for investigating the microstructure of composites compared. It was found that increasing the tool temperature reduced the temperature gradient between the incoming tape and substrate, resulting in better lap-shear strength and fracture toughness properties.

Keywords: A. Carbon fibre; B. Mechanical properties; E. Automation; E. Autoclave

Introduction

High performance thermoplastic composites are highly attractive to the aerospace industry due to their combination of high fracture toughness, high damage tolerance, long (indefinite) out life and high recyclability compared to thermoset systems [1]. However, they are difficult and expensive to process using the methods traditionally used for thermosets (vacuum bagging and autoclave cure) due to their lack of tack and high temperature requirements. This has led to increasing research in the area of in-situ consolidation by using automated fibre placement (AFP). Additionally, the ability to co-consolidate complex parts as one provides for a reduction in part count and assembly cost as mechanical fasteners are eliminated, particularly useful for skin-stiffener attachment [1, 2].

Rapid manufacturing of composites is an area of great interest to the industry. The automation of the manufacturing process for thermoplastics by automated tape placement (ATP) can achieve an increased production rate, reduction in labour cost and improved geometric repeatability when compared to conventional hand layup [3].

Previously, Sonmez and Akbulut [4] carried out a computational study to develop a process optimisation scheme for tape placement of APC-2 (PEEK) thermoplastic prepreg composites. Two optimisation studies were performed with the objectives of minimising peak residual stress and maximising tape laying speed. Results from the first optimisation study revealed a minimum peak residual stress of 70.4MPa without violating the two quality constraints (degradation weight loss % and degree of bonding, D_b) imposed. Results from the second study showed maximum speeds of above 80mm/s with D_b constraint at its lower limit of $0.8D_b$ (80% consolidation).

Pitchumani et al [5] studied a methodology for determining process design windows and optimum operating conditions for ATP. They concluded that the processing window itself was shown to get narrower with increasing main heater torch temperatures, while the preheater one was found to be insignificant. They also found that the time-optimal solutions do not lead to maximum strength and conversely, strength-optimal solutions require longer than optimum fabrication times.

Schledjewski and Latrille [6] presented a development study to automatically process the unidirectional (UD) fiber reinforced thermoplastic tapes, APC 2 (PEEK). They reported that the most important phenomena during the ATP and the filament winding processes are; heat

1 transfer (within the tape and substrate) and consolidation (bonding of incoming tape to
2 substrate. It is also stated that the consolidation is a key step during the processing of fibre
3 reinforced thermoplastic tapes. Pressure and temperature are applied to the incoming tape and
4 the substrate in order to eliminate spatial gaps, to eject out any entrapped air, and to place the
5 incoming tape next to previously laid ones without gaps or overlapping, leading to a low
6 porosity monolithic laminate.
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11 It has been known that more resin on the tape surface, or a resin rich surface, has a significant
12 effect upon the speed at which the tape can be processed and still achieve good to near
13 autoclave comparable properties. It was recommended that the surface resin content should
14 equal to one filament diameter. This free resin, unencumbered by fibers, enables resin flow
15 and chain entanglement during the ATP consolidation process, promotes intimate contact
16 between the tape and substrate in the consolidation process, and seems to reduce the
17 entrapment of inter-ply voids relative to a resin poor surface [7].
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25 Lang et al [8] reported a comparative study on the mechanical behavior of AS4/PEEK
26 (APC2) manufactured by a reference process including an autoclave consolidation compared
27 to ATP consolidation. They found that the autoclave consolidation results in the best known
28 quality level of the material considering void content and compaction. They concluded that
29 the filled-hole compression test results were on the same level for both consolidation
30 processes. However, filled-hole tension tests showed a loss of characteristics and higher
31 scattering of the results for ATP, though the residual strength remained at an acceptable level
32 for design. The third test comparison was on stringer pull out where the ATP showed a lower
33 discrepancy of the results with a lower average value, however the pull out stress was much
34 higher than for classical epoxy bonding.
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44 Recently, Kim et al [9] developed an alternative tow-placement head for ATP called
45 continuous tow shearing (CTS) using the in-plane shear deformation of the tow material.
46 Because the CTS head is equipped with an in-situ impregnation device for producing hybrid
47 tow materials, which were semi-impregnated tows with the combined flexibility of the dry
48 tow, they reported that a good impregnation quality of the prepreg was developed and tested.
49 Through the prototype tests, they concluded that CTS could significantly reduce process-
50 induced defects such as fibre buckling, fibre wrinkling, resin rich areas and fibre
51 discontinuity.
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In this work, in-situ consolidations of AS4-CF/PEEK rings and skin panels onto pre-consolidated laminates are carried out. For correlating the process parameters and mechanical properties, parametric experiments were performed by manufacturing and testing the interlaminar shear strength, single lap shear strength and fracture toughness samples. The compaction force, tape laying speed and tool temperature were studied as the process parameters. A new heating nozzle was designed to investigate heat distribution along the roller. An autoclave was used to manufacture baseline samples to compare with the ATP in-situ consolidated composites. ATP Laser processed sample by ADC was provided for comparison with the ATP hot-gas manufactured samples.

Material

The thermoplastic prepreg (sourced by TenCate) used in this study was carbon fibre-reinforced poly(ether-ether-ketone) (AS4-CF/PEEK). The commercial name is CETEX[®] TC1200 PEEK AS4. The AS-4 fibre areal weight, FAW = 146g/m². The PEEK resin content, RC = 33% by weight or 41% by volume. The prepreg areal weight = 218g/m². Two different widths of the thermoplastic prepreg UD tape were used; 12.7mm (i.e. narrow tape) and 158.75mm (i.e. wide tape), for ATP and hand lay-up of flat panel samples, respectively. Both tapes have the same as-received thicknesses of 0.15mm.

Experimental: Techniques and Test Methods

Automated Tape Placement (ATP) System

One of the methods for processing thermoplastic composites is to use the automated tape/fibre placement (ATP/AFP) as shown in Figure 1. ATP is currently an area of interest for the aerospace industry due to its ability to improve part quality, repeatability and production rate. In addition, automating the thermoplastic layup process reduces labour cost and can eliminate the need for autoclave processing and the associated bagging challenges.

ATP consists of a computer numerical control (CNC) deposition head which automatically heats and consolidates material in-situ. During the tape placement process, a single tape is passed through the feed rollers with a predefined tension and feed rate. Temperature of the incoming tape is increased to its T_M as it passes in front of the heating system before being

1 compacted by the consolidation roller. It is important to have adequate heat flux for both the
2 incoming tape and substrate ply in order to achieve a good level of resin diffusion. This is
3 achieved by directing the heat source at the nip point (tape-substrate contact area) to ensure
4 that the two interface plies reach their T_M . Typical heating systems for melting the resin
5 include laser, infra-red and hot gas torch (HGT) system. In the HGT system, nitrogen gas is
6 heated by passing through a series of heating elements (Fig. 1, [10]). An HGT system
7 developed by Automated Dynamics Corporation (ADC) was used in this work.
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13 **Hot Gas Torch (HGT) System**

14 During the initial processing of narrow tape, there was continuous accumulation of material
15 onto the nip roller. By measuring temperature at the roller (Fig. 2.b), it was found that the
16 conventional (narrow) nozzle (Fig. 2(a,d)) concentrated the heated at the middle of roller
17 rather than providing a uniform temperature across the roller width. Therefore, to overcome
18 this problem, a new wider nozzle (Fig. 2(c,d)) was suggested and then developed by ADC.
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26 In order to ensure a sufficient heat flux for processing 12.7mm wide thermoplastic tape,
27 preliminary studies were performed to study relationships between the temperature at the
28 roller and the nitrogen gas flow rate (V_{NG}), and nozzle-roller distance (\times) (see Fig. 2.a). **To**
29 **change the nozzle-roller distance, the nozzle holder was smoothly adjusted forward/backward**
30 **and rotated up/down to ensure its tip (hot gas flow) is always directed toward the nip line**
31 **between the incoming tape and the pressure roller (impact area of the gas)**
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38 Temperature readings were taken at six different points on the roller as shown in Figure 2.b
39 using a thermocouple probe with 5mm diameter. Preliminary Study 1 was carried out using
40 the conventional (narrower) nozzle and study 2 was executed using the new wider nozzle.
41 Widths of the conventional and new nozzle were 12mm and 20mm, respectively. **Table 1**
42 **gives the variables for the two studies.**
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51 **Mechanical Tests**

52 **1. Short-beam Strength of Composites**

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54 To carry out the interlaminar shear strength (ILSS) test, ASTM D2344 [11] standard test
55 method was used. Test software calculated the beam strength using Equation 1 [11];
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$$F^{sbs} = 0.75 \times \frac{P_m}{b \times h} \quad (1)$$

Where F^{sbs} is the short-beam strength in MPa; P_m is the maximum flexure load in Newton; b is the specimen width in mm; h is the specimen thickness in mm.

2. Lap Shear Adhesion Test

ASTM D5868 [12] standard test method was used for single lap shear strength test. No adhesive was used to produce the test sample as the aim was to assess the in-situ consolidation quality of thermoplastic composites using ATP. A loading rate of 13mm/min was used as per test standard and Equation 2 [12] was used to calculate the lap shear strength.

$$LSS = \frac{P_m}{A} \quad (2)$$

Where LSS is lap shear strength in kPa; P_m is the maximum load in Newton and A is the overlap/bond area in mm^2

3. Mode I Interlaminar Fracture Toughness Test

ASTM D5528-01 [13] was used to produce the double cantilever beam (DCB) test samples. Rectangular loading blocks were used to apply tensile load to the sample. In order to minimise errors as a result of the applied moment arm, the distance from the loading block pin to the center line of top specimen arm (t) was kept as small as possible [13]. This distance (t) was calculated via the following Equation 3 [13];

$$t \leq \frac{h}{4} + 0.01 \sqrt{\frac{0.0434h^3 E_{11}}{G_{Ic}} + a^2} \quad (3)$$

Where E_{11} is the tensile modulus in MPa, G_{Ic} is the expected fracture toughness in kJ/m^2 and a is the delamination length in mm.

As recommended by the test method, fracture toughness (G_{Ic}) was calculated using the modified beam theory (MBT) given by Equation 4 [13];

$$G_{Ic} = \frac{3P\delta}{2b(a+|\Delta|)} \quad (4)$$

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2 Where P is the load in Newton, δ is load point displacement in mm, and $|\Delta|$ is the correction
3 factor obtained from the cube root of compliance against delamination length graph.

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5 As recommended by the test method, three initiation G_{Ic} values were calculated using the
6 initial delamination length (a_0) and are explained as follows: i) Deviation from Linearity or
7 Non-Linearity (NL): G_{Ic} is calculated from the load-displacement curve at the point where the
8 graph deviates from linearity. This assumes that the delamination starts in the interior of the
9 specimen. ii) Visual Observation (VIS): this corresponds to the load and displacement values
10 recorded for the point at which delamination is first visually observed to grow. iii) 5% Offset:
11 G_{Ic} is calculated by determining the intersection point between the load-displacement curve
12 and a line drawn from the origin and offset by 5% increase in compliance (δ/P) from the
13 original linear region.
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22 **Manufacture of Thermoplastic Rings and ILSS Test Samples**

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24 ATP attached with the new designed nozzle (20mm wide) was used to manufacture AS4-
25 CF/PEEK rings for establishing the relationships between the mechanical properties and
26 process parameters of monolithic in-situ consolidated laminate. [Table 2](#) lists the consolidation
27 conditions used for manufacturing the rings. Different experiments were performed to study
28 the effect of process parameters on the ILSS (interlaminar shear strength) of rings produced.
29 Figure 3 shows one of the obtained AS4- CF/PEEK thermoplastic ring and three-point-bend
30 test sample for ILSS measurements (according to the ASTM D2344).
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38 To study the effect of the tape laying speed (V_R) on ILSS, five rings (1-5) were manufactured
39 at different speeds as given in [Table 3](#), and to study the effect of both speed (V_R) and roller
40 compaction force (F_R) on the ILSS, another six rings were also produced. [Table 3](#) also gives
41 the variables for this study. To carry out these studies, 6mm thick rings made of 40plies were
42 manufactured using the ATP's aluminum mandrel (outer diameter of 146mm, see Fig. 1.a). A
43 single ply along the length of mandrel was first placed to act as an adherent for the rings.
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50 All the rings were first hand-sanded using a 60-grit sand paper and cut into approximate test
51 sample chord lengths using a diamond coated band saw. To achieve finished tolerances, the
52 test samples were machined by 5-axis CNC machine to 24mm chord lengths to fulfill the
53 ASTM test method requirements.
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58 The test specimens were tested at the AMRC's Advanced Structural Testing Centre (ASTC)
59 using an Instron 50kN test machine in accordance to ASTM D2344. The specimen was
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1 loaded into a test fixture specifically designed to perform the ILSS test (see Fig.4).
2 Specimens were tested at a loading speed of 1mm/min in displacement control mode.
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7 **Manufacture of Single Lap Shear and Double Cantilever Beam Samples**

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10 The purpose of this study was to investigate the effects of tool temperature and resin rich
11 interface on lap shear strength (LSS) and fracture toughness of double cantilever beam
12 (DCB). For both, the base laminate was processed in the autoclave and the top half laminate
13 was tape placed in order to assess the in-situ bonding quality obtained using the ATP process.
14 In addition, this process represented the tape placement of a thermoplastic aerodynamic skin
15 onto a pre-consolidated stiffener. Again, baseline samples were manufactured using the
16 autoclave process to act as a comparison for the ATP produced samples. The list of fixed
17 ATP process parameters are given also in [Table 2](#).
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26 To study the effect of tool temperature on LSS and interlaminar fracture toughness or DCB, a
27 total of eight AS4-CF/PEEK panels were manufactured at different tooling temperatures. Due
28 to the upper temperature limit of the heater, the maximum tool temperature was limited to
29 150°C. [Table 4](#) gives the tool temperature range used in accordance with the panels produced.
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34 To study the effect of resin rich interface (enhanced resin mixing at the interfacing plies) on
35 single LSS and DCB, top surface of the base laminate was enriched with two layers of 8µm
36 thick APTIV PEEK 1000 series film obtained from Victrex (<http://www.victrex.com>). Two
37 layers of PEEK film ([approximately 10% extra RC](#)) were used in order to obtain noticeable
38 differences in mechanical performance. This resin rich interface was used to tape place the
39 first ply onto using the maximum tool temperature of 150°C.
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46 **Manufacture of Panels**

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48 The AMRC's high temperature autoclave was used to manufacture the base laminates of LSS
49 and DCB composite panels. 17 plies of the wider (158.75mm) AS4-CF/PEEK prepreg tape
50 were hand-laid and tacked together using a soldering iron to produce 635mm x 635mm x
51 2.5mm thick UD panel/laminate. The tapes were staggered to avoid overlapping joints and
52 potential weak spots. [Figure 5.a](#) shows the bagging arrangement used to autoclave process the
53 panels. Details of the autoclave process cycle are listed in [Table 5](#).
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1 A number of small plaques were machined from the autoclave processed panel using the
2 CNC machine to be used as the base laminates. On the top half of each, the narrower
3 (12.7mm) tape was placed to produce the final LSS and DCB panel configurations. The LSS
4 and DCB panels were made large enough to extract at least five coupons from each. To make
5 the resin rich interface panels, two layers of PEEK film were placed on top of the hand-laid
6 panel before bagging and processing in the autoclave.
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11 In order to carry out parametric studies, which required heated substrate laminates, an
12 aluminum tool was designed. This tool measured 500mm x 500mm x 12mm and contained a
13 central pocket measuring 250mm x 250mm with 2.5mm depth. The central pocket was
14 surrounded by a thermoplastic picture frame to act as an adherent when tape placing the first
15 ply onto the bottom laminate. To produce the LSS panel configuration, an aluminum plate
16 was inserted inside the pocket to create the step feature required for LSS samples. This tool
17 was heated using four silicone rubber heater mats that were attached to the bottom of the tool
18 and covered by a 2.5mm thick aluminum plate. Each heater measured 230mm x 230mm with
19 230V rating and watt density of 1.53W/cm².
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29 To produce the DCB samples, a thin steel shim was first placed to act as the crack initiator.
30 This was due to the kapton film being thermally damaged when exposed to the hot nitrogen
31 gas. After tape placing the first ply, steel shim was replaced with a 25µm thick kapton film.
32 Thin strips from DCB panels were machined to measure the panel distortion (radius of
33 curvature) caused by the residual stresses (Fig. 6.a). Figure 6.b shows the LSS panel
34 configuration with aluminum plate inside the tool pocket. Both figures show that poor tape
35 quality was obtained at the start of tape lay-up. This was due to an excessive tape pre-feed as
36 the roller started to move and was later minimised by decreasing the pre-feed value.
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47 **LSS and DCB Samples Preparation and Testing**

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50 Both LSS and DCB panels were first cut to approximate coupon sizes using a diamond
51 coated band-saw, followed by CNC machining to the final dimensions, using designed
52 fixtures that provided with movable clamps for obtaining the final sample dimensions.
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56 The DCB samples were prepared using a specially designed bonding fixture. The aluminum
57 tabs and corresponding bonding areas on the sample were first keyed with 60-grit sand paper
58 and then bonded using room temperature cure epoxy adhesive.
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1 The assembly was held securely using two clamps. After bonding the first tab, the sample
2 was flipped over and the bonding process was repeated to attach the tab on the other side.
3 ASTM D5528-01[10] was used to perform the Mode I fracture toughness of DCB test as
4 described above (Fig. 7). Samples were tested at a loading rate of 2.5mm/min. Three
5 initiation G_{Ic} values (NL, VIS and 5% Offset), were calculated.
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9 For the LSS samples, tabs were first attached to the samples and then machined to the final
10 dimensions. The tabs were needed to ensure that the samples are loaded through the
11 centerline (overlap area) during the testing process. The 50kN Instron test machine was also
12 used as in the case of ILSS testing. ASTM D5868-01[12] test method was used to perform
13 the LSS tests. A loading rate of 13mm/min was used to test the samples.
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17 Baseline samples were also manufactured using the autoclave to provide a comparison
18 against the ATP manufactured samples. Flat ILSS samples were manufactured instead of the
19 curved coupons (Fig. 4.b) as they were readily available from the base laminates processed in
20 the autoclave. A processed sample thickness of approximately 2.3mm was obtained and this
21 led to a loading span length of 9.2mm.
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32 **Optical Microscopy**

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35 Optical microscopic studies were conducted on polished sections of specimens cut from the
36 rings and laminates. Alicona-IFM microscope was used to examine the specimens and optical
37 images (micrographs) were captured. Using two-part epoxy compound the samples were
38 cured in 30mm diameter resin blocks. The polishing procedure is as follows: silicon carbide
39 SiC-220 for 20 seconds and SiC-1200 for 2 minutes until flat, 9-micron (MD-Plan) for 4
40 minutes, 3-micron (MD-Dac) for 4 minutes and OP-S for 2:40 minutes plus 1minute on water
41 to clean OP-S off. Rotation speed was 150rpm and applied force per sample was 30N.
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52 **Results and Discussion**

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54 The temperature (T_{NG}) at the nip roller was measured with varying the nitrogen gas flow rate
55 (V_{NG}) at different fixed distances ($x = 12, 15, 20, 25$ mm). Figures 9 and 10 show a selection
56 of these measurements; $x=12$ mm and $x=20$ mm, respectively. It is noticed that as the nitrogen
57 gas flow rate is increased, the temperature at the roller (T_R) is also increased. In other words,
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1 increasing the flow rate decreases the interaction time between the nitrogen gas and the
2 surrounding and hence a lower heat loss to the surroundings. Temperatures measured in the
3 middle of roller were significantly higher than the sides since the nozzle was relatively
4 narrow, concentrating the heat flow to the centre of roller. For distances of 15mm and
5 beyond, there is a significant temperature difference (approx. 200°C) between the front
6 middle and other locations of the roller.
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11 Using the new (wider) nozzle, the temperature (T_{NG}) was measured at different nitrogen gas
12 flow rate (V_{NG}) at roller-nozzle distances of $x = 12\text{mm}$ and $x = 20\text{mm}$ (Fig. 11 and 12). It is
13 showed that the gas flow rate has insignificant effect on temperature. Temperature at the front
14 was always higher than the bottom of roller. A much better heat distribution is obtained along
15 the nip roller owing to less temperature difference between the sides (edges) and middle of
16 roller compared to Figures 9 and 10. Therefore, all the thermoplastic rings made from the
17 12.7mm wide tape were manufactured by using the new designed nozzle instead of the
18 narrower one which suitable for processing 6.3mm or less wide tapes.
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27 On processing the prepreg tape, trials were also carried out to determine the material
28 temperature. By considering the main source of heat (Nitrogen Gas) having temperature of
29 975°C and this heat has been transferred into surroundings: nip roller, tape and mandrel. It
30 was difficult to measure the actual temperature of the tape and the nip region due to the poor
31 infrared reflection from the glowing zone. But by measuring the temperatures of nip roller at
32 the closest point near to the glowing zone, the mean value of temperature was found to be
33 514.33±12.5°C. This is confirmed by another infrared image analysis made by the ATP
34 manufacturer (ADC) [2], which reported that the temperature of the nip region was estimated
35 to be around 500°C (See Fig. 13).
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44 In addition and in order to consider the shear thinning of the PEEK (due to the dynamic and
45 rapid application of pressure of ATP), polymer squeeze flow (in the nip zone), and other
46 reasons which significantly enhance the resin inter-diffusion over the static (autoclave or
47 press forming) case of in intimate contact, a rheological study was made by ADC [2]. Figure
48 14 [2] shows the shear viscosity versus the shear rate at three processing temperatures: 360,
49 380 and 400°C. August et al. [2] found that a dramatic shear thinning was occurred in PEEK
50 resin even at low process temperatures.
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58 Figure 15 shows the ILSS specimens that were tested in accordance with the ASTM D2344
59 [8]. A mixture of interlaminar shear (ILS) and plastic deformation failure modes were
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obtained for the ATP and baseline samples (See Table 6). Table 6 gives the summary of ILSS test results from parametric study 1.

There are likely to be three competing factors relating the tape laying speed to the ILSS. Firstly, the lower the speed the greater the time the polymer has to flow and the greater the time for molecular diffusion across the interface, which should result in a higher ILSS for a lower speed. Secondly, the lower speed the greater the intensity of the heating so the higher the temperature reached, which will also increase the flow and diffusion of the polymer and so the ILSS. However, thirdly, the increased temperature will also lead to increased thermal degradation of the polymer (at least above a certain point) which will decrease the ILSS. Of the three competing factors, two will tend to increase the ILSS with a lower speed and one will tend to reduce it. However, all three factors are non-linear and so in combination will be very much so.

The maximum ILSS value of 50.58MPa \pm standard deviation (SD) was obtained for the lowest tape laying speed of 57mm/s, with the higher speeds being considerably lower. Even at lower speeds, similar results were reported by Shih and Loos [14]. The trend is clearly not a linear one, but this is not surprising given the non-linear nature of the physical processes described above, although the direction of the trend suggests that thermal degradation is not the dominant process [2], suggesting that an even higher heat flux may be beneficial, further supported by the baseline sample. The baseline ILSS value of 109.84MPa demonstrated a well-consolidated laminate that experienced a good level of resin diffusion during the autoclave process. These results indicate that with the current HGT system available on the ATP machine, insufficient heat energy is provided, which limits the maximum tape laying speed.

For a fair comparison, a baseline ring was processed using ATP at high speed, then post consolidated in autoclave. Same autoclave cycle as the flat panel was used (Table 5) and a similar bagging arrangement as shown in Figure 5.b was also used. By testing this ring as a curved specimen (like to like comparison) it was found that the ILSS value of 92.69MPa (Table 6) which is slightly lower than the flat panel but still significantly higher compared to the ATP in-situ consolidated ring.

To investigate the difference in the ILSS values obtained, the microstructure of ATP and baseline samples was studied. Figure 14 shows a selection of optical cross sections for the baseline (a) and ATP (b) specimens. The optical micrographs would tend to confirm the

1 measured difference in the ILSS of the ATP composites manufactured compared to the
2 autoclaved baseline sample. It is found that the baseline panel (Fig. 16a) is free of voids but
3 the ATP sample (Fig. 16b) is not. The voids appear to be located between the laid tapes
4 owing to lack of debulking/consolidation, affecting the inter-ply intimate contact, hence
5 reducing the shear strength. Material development, e.g. thermoplastic tape of resin rich
6 surface is recommended for future work of composites processing by ATP. Such
7 development could improve the in situ consolidation, reducing porosity and enhancing the
8 ILSS.
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15 Table 8 presents the test results for parametric study 2. A maximum ILSS of 49.23MPa was
16 obtained for roller compaction force (F_R) of 125lbs (556N) and speed of 65mm/s. For
17 $F_R=75$ lbs, there is a decrease followed by an increase in ILSS as the speed increases. But for
18 $F_R=125$ lbs, there is an increase followed by a decrease in ILSS as the speed increases. The
19 results (Table 7) did not show a specific correlation between the compaction force and ILSS.
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21 Theoretically, an increase in compaction force should increase the ILSS of the part as
22 increased pressure enhances resin diffusion. The compaction forces used may perhaps already
23 be higher than required, limiting any further improvements in ILSS. There was some
24 inconsistency between the tape laying speeds for given compaction force which may have
25 affected the results.
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34 The tested ATP and baseline LSS samples are shown in Figures 17. Light fibre tear (LFT)
35 failure mode was mostly noted in the ATP manufactured samples. Failures occurred at the
36 first ATP ply that was placed onto the base laminate. Much neater and mid-plane failures
37 were obtained for baseline LSS specimens. The LSS test results are listed in Table 8. A
38 maximum LSS of 13.62MPa was obtained for ATP which was approximately half of the
39 baseline LSS value (25.70MPa). It is found that there is an overall increase in lap shear
40 strength as the tool temperature was increased. Increasing the substrate laminate's
41 temperature reduces the temperature gradient between the incoming tape and substrate.
42 Increasing the tool temperature allows the substrate to reach its melting temperature in a
43 shorter period of time upon heating by the HGT system. This enhances the resin diffusion
44 between the interface plies and hence improves the level of consolidation. The LSS values for
45 tool temperatures of 25°C and 65°C did not follow the overall trend (see Table 8). However,
46 tool temperatures ranging from 65°C to 150°C showed a linear increase in strength values.
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48 The baseline LSS value of 25MPa was 50% higher than the maximum ATP LSS value,
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2 indicating that the autoclave processing provides a better consolidation when it is compared
3 to the ATP process.

4 The resin rich interface sample showed a slight decrease in LSS value compared to its
5 corresponding sample without the PEEK film. Enriching the bonding area with resin
6 increases the bond-line thickness which would enable larger strain deformations to occur in
7 the overlap/bonding area. This may have a net effect of lowering the joint strength even
8 though the material toughness is increased. Due to the large standard deviation obtained, the
9 effect of resin rich interface may be negligible.

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16 It is obvious that the ATP processed ring samples exhibited approximately 55% of the ILSS
17 and LSS values compared to the baseline autoclaved samples. In order to achieve the
18 optimum of autoclave ILSS, the ATP samples have to be post-consolidated to fulfill for
19 example the aerospace requirements, but without post-consolidation the ATP ILSS might be
20 acceptable for commercial applications such as oil and gas pipes, which are normally made of
21 low melting thermoplastic polymers.

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28 Figure 18 shows the tested ATP DCB samples. The samples were delaminated to a total
29 delamination length of 100mm. Most of the samples, except for the baseline ones, had some
30 level of curvature along their length.

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Figure 19 shows a typical load against load-point displacement graph for testing of DCB
samples. On the graph, visual onset of delamination (VIS), first deviation from non-linear
(NL) and 5% offset values for initial delamination (fracture) are plotted.

Figure 20 shows a typical cube root of compliance against delamination length graph. From
the graph, a correction factor ($|\Delta| = x$ -axis intercept) is used to calculate the G_{Ic} . A resistance
(R) curve for a typical DCB test is shown in Figure 21. The initiation NL value is
recommended to be the most reliable value for reporting fracture toughness (G_{Ic}) [15].
Propagation fracture toughness values are artificially raised due to fibre bridging in the crack
opening area (Fig. 22). Therefore, only VIS, NL and 5% initiation G_{Ic} values were analysed
in details, with special attention paid to the NL values.

Tables 9, 10 and 11 list the initiation G_{Ic} values for VIS, NL and 5% offset methods
respectively for DCB samples. For all methods used, relatively large coefficients of variation
were obtained. There is an overall increase in initiation G_{Ic} values as the tool temperature is
increased from room temperature to 150°C. There is an approximately 50% decrease in G_{Ic} as

1 the tool temperature increases from room temperature to 65°C, but as the tool temperature is
 2 increased from 65°C to 150°C, toughness increases linearly for all initiation toughness
 3 methods used. The resin rich G_{Ic} values, based on NL and 5% offset methods, were 30%
 4 higher than the values obtained from coupons without the enriched resin surface. VIS
 5 initiation G_{Ic} values were given minimum significance as visual observation is highly
 6 dependent on the test operator. Maximum G_{Ic} values for VIS, NL and 5% offset were 0.271,
 7 0.485 and 0.570kJ/m² respectively. Maximum values for NL and 5% offset were both
 8 obtained from the resin rich interface samples which were manufactured using T_T of 150°C.
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Toughness is mainly a resin dominated property and a high level of resin diffusion is necessary to achieve acceptable impact resistance characteristics. NL toughness values are the most reliable means of determining the fracture toughness as VIS values are highly dependent on the visual observations of test operators. Use of PEEK films to enrich the surface with resin and high tool temperature both improve the resin diffusion process that led to the maximum value for toughness.

Figure 23 shows the curved strips machined from the DCB panels manufactured using different tool temperatures, 15°C (RT), 65°C, and 150°C. Radii of 700, 750 and 1800mm were obtained for tool temperatures of room temperature; 65°C and 150°C, respectively. This curvature is caused by a residual strain between the primary layers of the laminate (the pre-consolidated plies versus the ATP consolidated plies. This is analogous to the curvature of an asymmetric 0/90 laminate. An expression to estimate the residual strain in such a laminate was developed by Bailey et al [16] and is shown in Equation 5.

$$\varepsilon_r = \frac{E_l b p}{12r(E_l b + E_t d)} \left(\frac{E_l}{E_t} + 14 + \frac{E_t}{E_l} \right) \quad (5)$$

Where ε_r is the residual strain, E_l and E_t are the moduli in longitudinal and transverse directions and b , d and p are all ply thicknesses. However, in this example, we do not have a 0/90 laminate, so we do not have a longitudinal and a transverse ply, we have two transverse plies. The thickness of the pre-consolidated layer is equal to the thickness of the ATP layer, so b , d and p are also all equal. Equation 5 can therefore be greatly simplified to Equation 6.

$$\varepsilon_r = \frac{2b}{3r} \quad (6)$$

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Substituting in the layer thickness of 2.5mm and the radii of curvature recorded above this then gives the residual strain in flat laminate as 0.24%, 0.22% and 0.09% for the pieces produced with the tool at room temperature, 65°C and 150°C respectively.

It can therefore be seen that raising the temperature of the tool reduces the level of residual stress in the laminate. This can be explained as the temperature gradient between the incoming tape and substrate laminate was lowered as a result of the increase in tool/substrate temperature.

Generally and in order to compare the current ATP hot-gas system used in this study with the state-of-the-art laser ATP technology, an ATP laser processed sample (by ADC) was provided. Figure 24 shows a bar-chart comparison in terms of the ILSS values of three samples processed by three different technologies: ATP hot-gas, ATP laser and Autoclave. From Table 6 and figure 24, it found that the ATP hot-gas (Gas-ATP) only achieved 55% ILSS of the autoclave baseline ILSS but still 30% less than the laser-ATP one, which achieved 85% of the autoclave ILSS. According to the aerospace requirements, the laser-ATP still needs post consolidation (using autoclave) to gain the remaining 15% of the baseline ILSS, i.e. ATP's (Gas and Laser) exhibit unacceptable ILSS compared to the autoclave one and both need post consolidation to achieve the optimum of ILSS. The current ATP (hot-gas and laser) system can be used as a rapid preforming process that might be cost effective technology compared to the hand layup.

Conclusion

Based on the results and discussion, the following remarks were concluded;

- The new wider nozzle provides a much better heat distribution along the roller and it is recommended to use for processing the tape (12.7mm wide).
- An overall decrease in ILSS as the tape laying speed was increased.
- An overall increase in both LSS and DCB test performance as the tool temperature was increased. Although, there is believed to be some anomalies at lower tool temperatures.
- An increase in interlaminar toughness property by using resin rich interface. However, LSS value was slightly reduced by using resin rich surface.

- 1 • Consistent heat flux is an issue with the current HGT system and this also limits the
2 maximum tape laying speed that can be achieved to produce an acceptable quality part.
3 The inconsistency in heat flux was caused by the nozzle deviating from the desired
4 position as the head touched the part before placing the tapes. ATP produced samples
5 exhibited mechanical properties of approximately 45% less than the autoclaved samples.
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- 8 • With post consolidation the ATP processed composites will be acceptable for aerospace
9 applications, but without post consolidation (in-situ consolidation only), they may be
10 suitable for commercial applications and ATP could be used over the hand layup to offer:
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 - 12 - Rapid manufacturing
 - 13 - Less time consuming
 - 14 - Cost effectiveness
 - 15 - Complex preform
- 16 • To improve the properties of ATP processed composite parts, the following points could
17 be studied in the future work;
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 - 19 - Study of alternatives to the HGT for the provision of greater heat flux with
20 improved control and consistency, potentially followed by a secondary
21 consolidation stage.
 - 22 - Determination of the degree and extent of any thermal degradation of the
23 thermoplastic material.
 - 24 - It is thought that the first few ATP plies may be poorly consolidated compared to
25 the bulk. This could be investigated by machining away the inner plies of the
26 curved ILSS coupons and re-testing to determine any changes in performance.
 - 27 - Thermoplastic tape of resin rich surface could be considered

43 **Acknowledgment**

44 The authors would like to thank the AMRC's partners for their continuous support and
45 feedback during the course of this project. Special thanks go to ADC
46 (<http://www.automateddynamics.com>) and the AMRC Composite Centre research staff (A.
47 Smith, K. Mee, R. Smith & T. Kilham) for their help.
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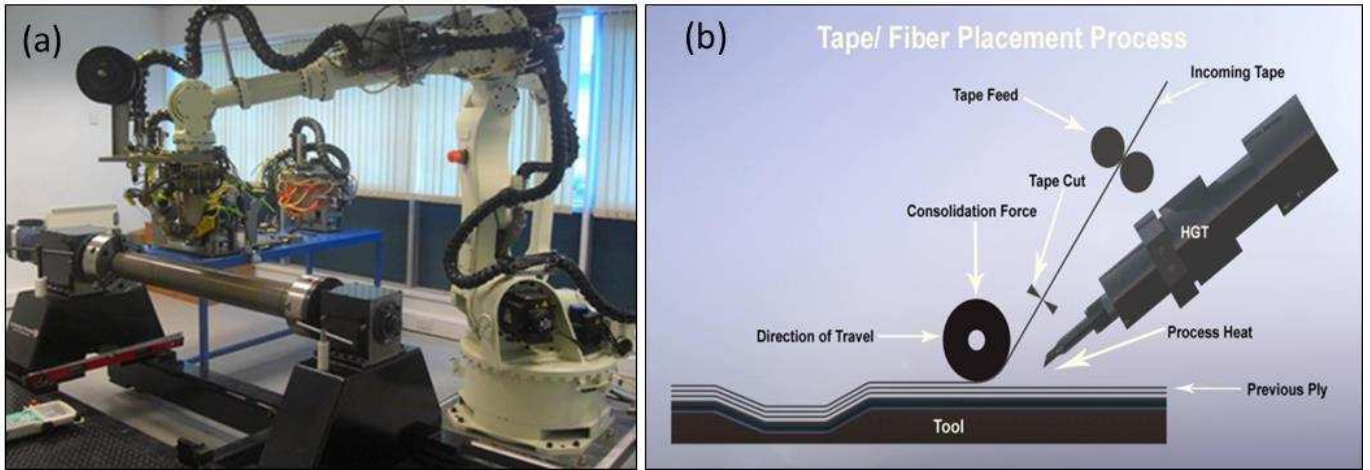


Figure 1: a) Automated Tape Placement (ATP), and b) Hot Gas Torch (HGT) heating system of ATP (adapted from Ref. [5])

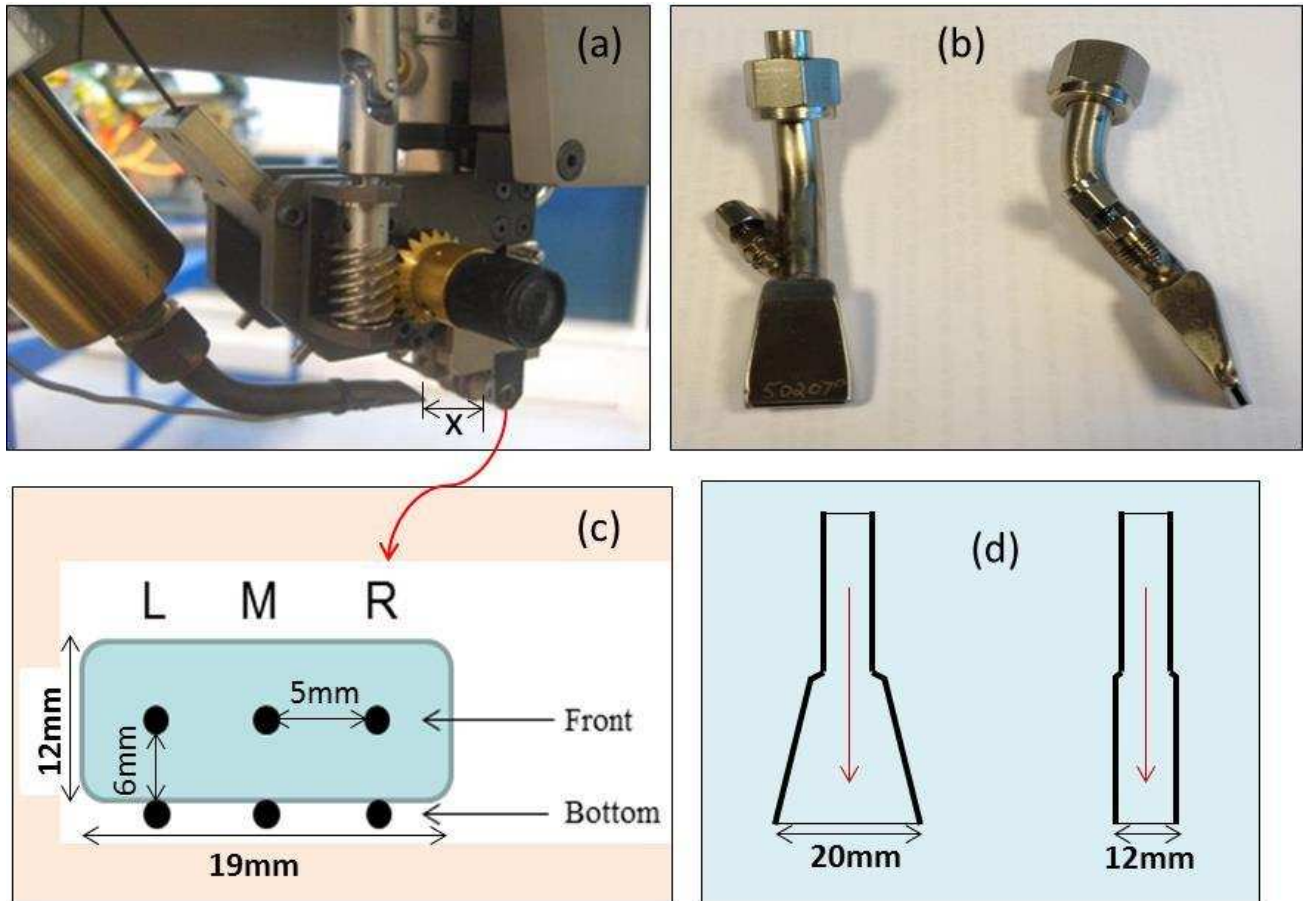


Figure 2: a) HGT heating system of ATP including the conventional nozzle, b) Zones at where the temperature readings were taken of the nip roller, L (left), M (middle), R (right), c) photograph of new nozzle, d) schematic diagram of new nozzle (left) compared to the conventional one (right).

Table 1: The distances used to measure the temperatures at the nip roller at $T_{NG} = 975^{\circ}\text{C}$ and different V_{NG} (Preliminary study 1: conventional nozzle and Preliminary study 2: new nozzle)

Distance X (Nozzle – Nip roller)	Nitrogen Gas Flow rate, V_{NG} (L/min)	
	Conventional nozzle	New nozzle
12	50, 60, 70	50, 75, 100
20	50, 60, 70	50, 75, 100

Table 2: Consolidation conditions (fixed parameters) of AS-4 CF/PEEK rings, LSS and DCB panels using ATP

Nozzle type	The new wider (20mm wide)
Incoming tape tension per unit area	75.84 kN/m ²
T_{NG}	975°C
V_{NG}	100 L/min
Roller – Nozzle distance (×)	7mm



Figure 3: Thermoplastic ring with machined test sample.

Table 3: Tape laying speed and roller compaction force settings for determining ILSS

Parametric study	Ring	Tape laying speed V_R (mm/s)	Roller compaction force F_R (lbs)
1	1	57	100 or (444.82N)
	2	68	
	3	73	
	4	82	
	5	95	
2	6	60	75 or (333.62N)
	7		125 or (556.03N)
	8	65	75 or (333.62N)
	9		125 or (556.03N)
	10	75	75 or (333.62N)
	11		125 or (556.03N)

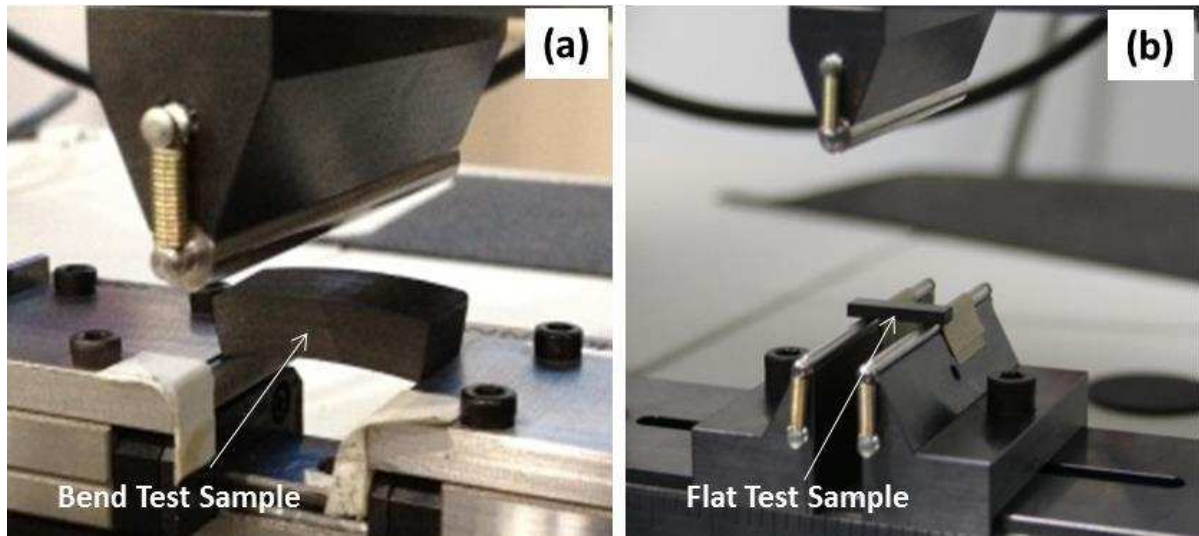


Figure 4: Test fixture for ILSS test; a) Curved/bend sample machined from ring and b) Flat sample machined from panel

Table 4: Tool temperatures used for LSS and DCB measurements

LSS Panel	DCB Panel	Tool temperature, T_T ($^{\circ}\text{C}$)
1	1	Room Temperature
2	2	65
3	3	105
4	4	150

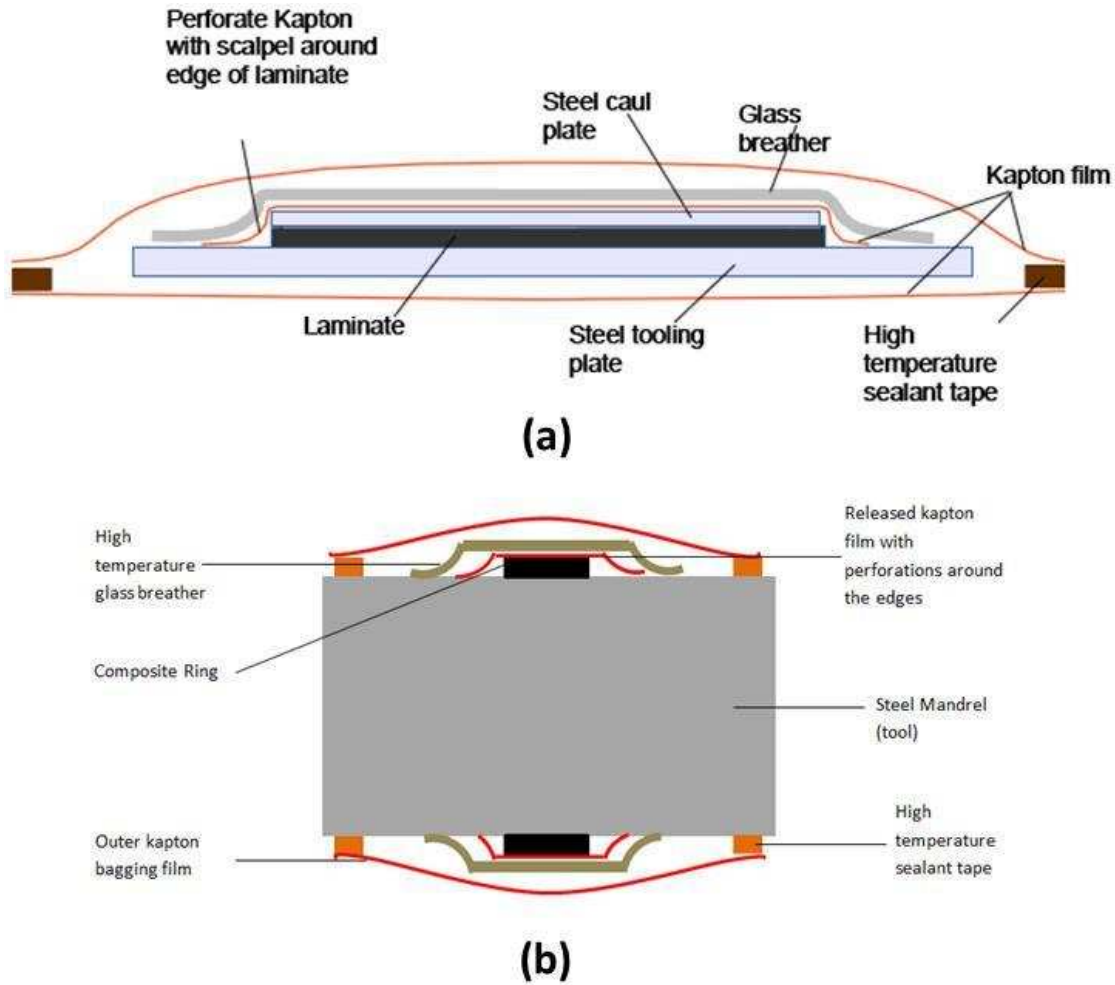


Figure 5: Bagging arrangement for autoclave processing, a) Flat panel and b) Baseline ring

Table 5: Autoclave cycle details

Stage	Details
1	Ramp air temperature to 200°C at 10°C /min
2	Ramp pressure to 6.5 bar at 0.5 bar/min
3	Ramp air temperature to 385°C at 10°C /min
4	Dwell air temperature at 385°C for 3hours
5	Cool air to 60°C at 15°C/min or less
6	Release pressure

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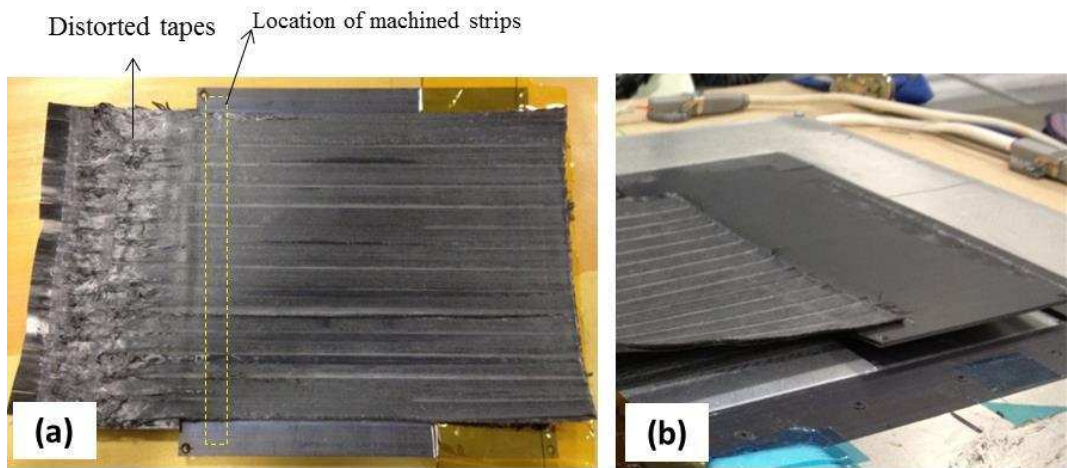


Figure 6: Manufacture of a) DCB and b) LSS panels

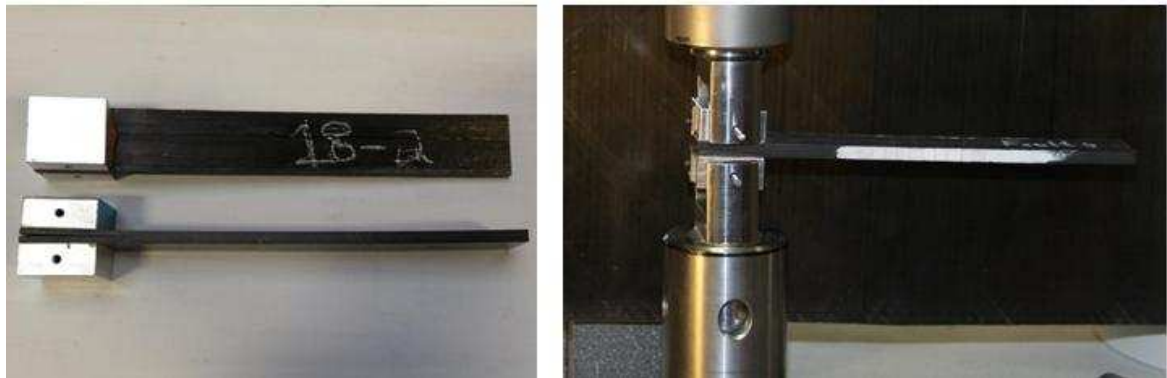


Figure 7: DCB test samples



Figure 8: LSS test samples

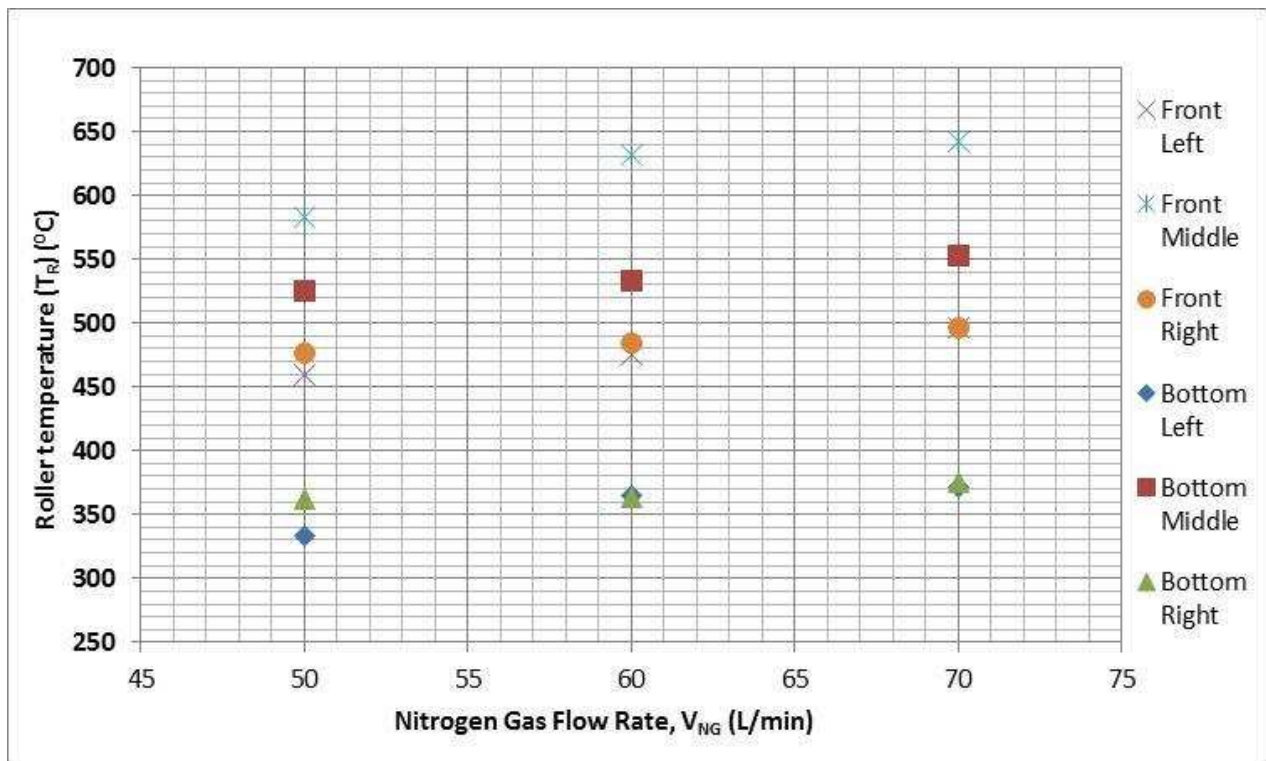


Figure 9: Roller temperature (T_R) versus nitrogen gas flow rate (V_{NG}) at roller-probe distance $x = 12\text{mm}$ using the conventional nozzle.

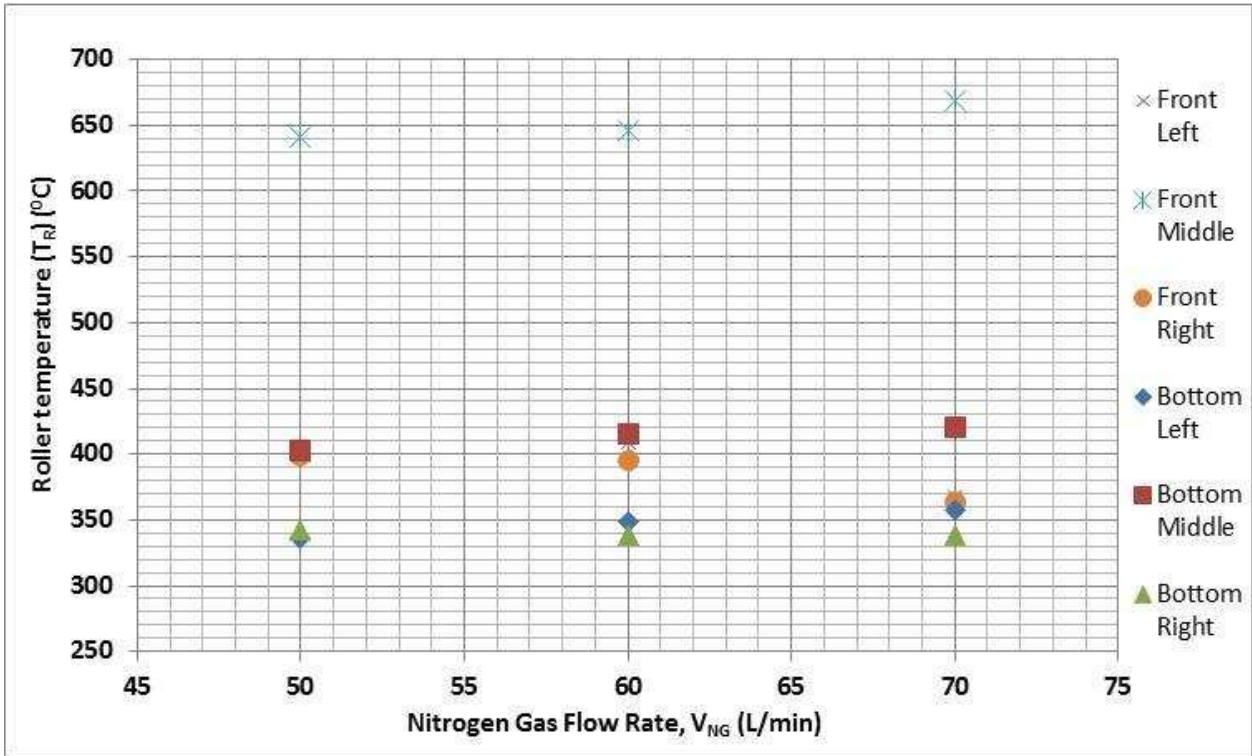


Figure 10: Roller temperature (T_R) versus nitrogen gas flow rate (V_{NG}) at roller-probe distance $x = 20\text{mm}$ using the conventional nozzle.

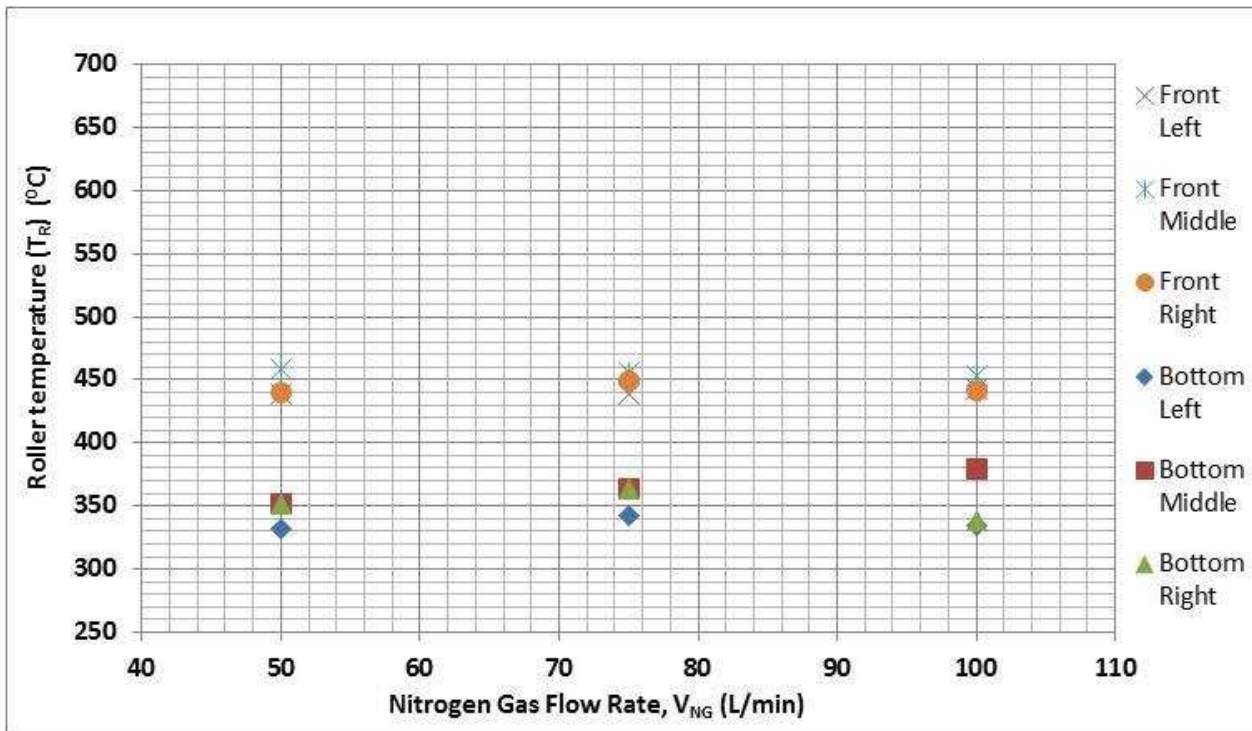


Figure 11: Roller temperature (T_R) versus nitrogen gas flow rate (V_{NG}) at roller-probe distance $x = 12\text{mm}$ using the new nozzle.

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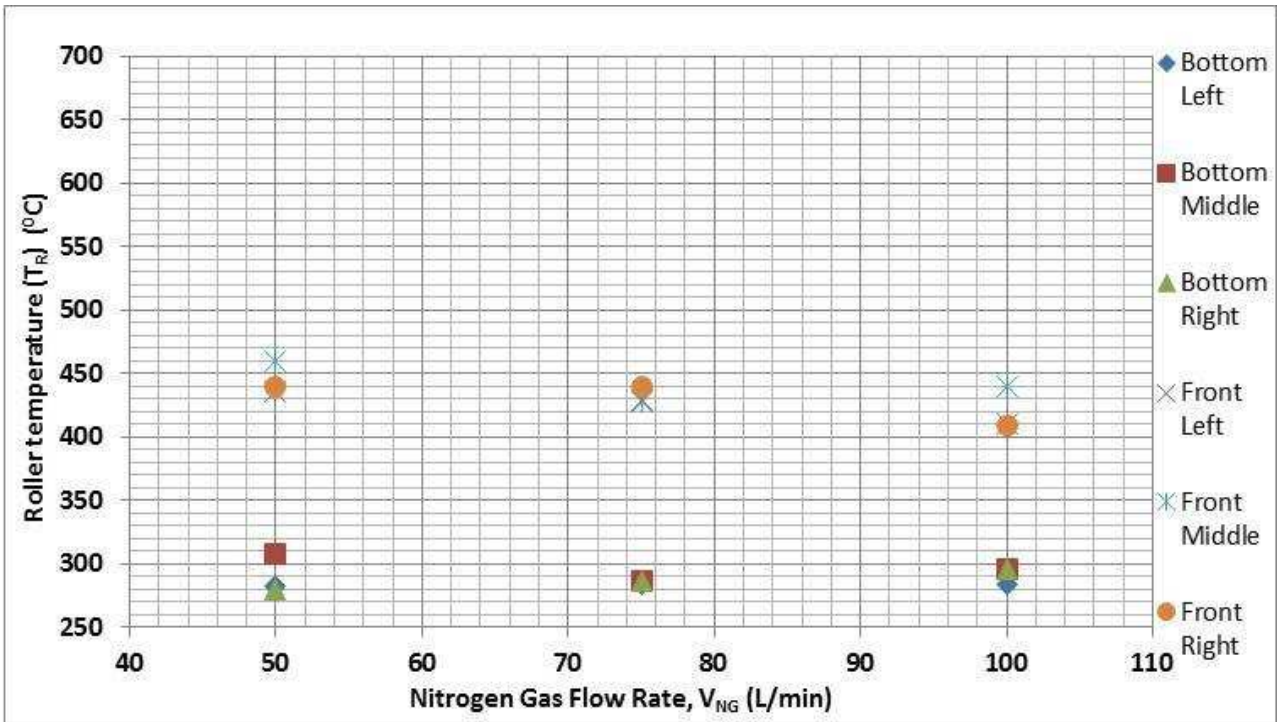


Figure 12: Roller temperature (T_R) versus nitrogen gas flow rate (V_{NG}) at roller-probe distance $x = 20\text{mm}$ using the new nozzle.

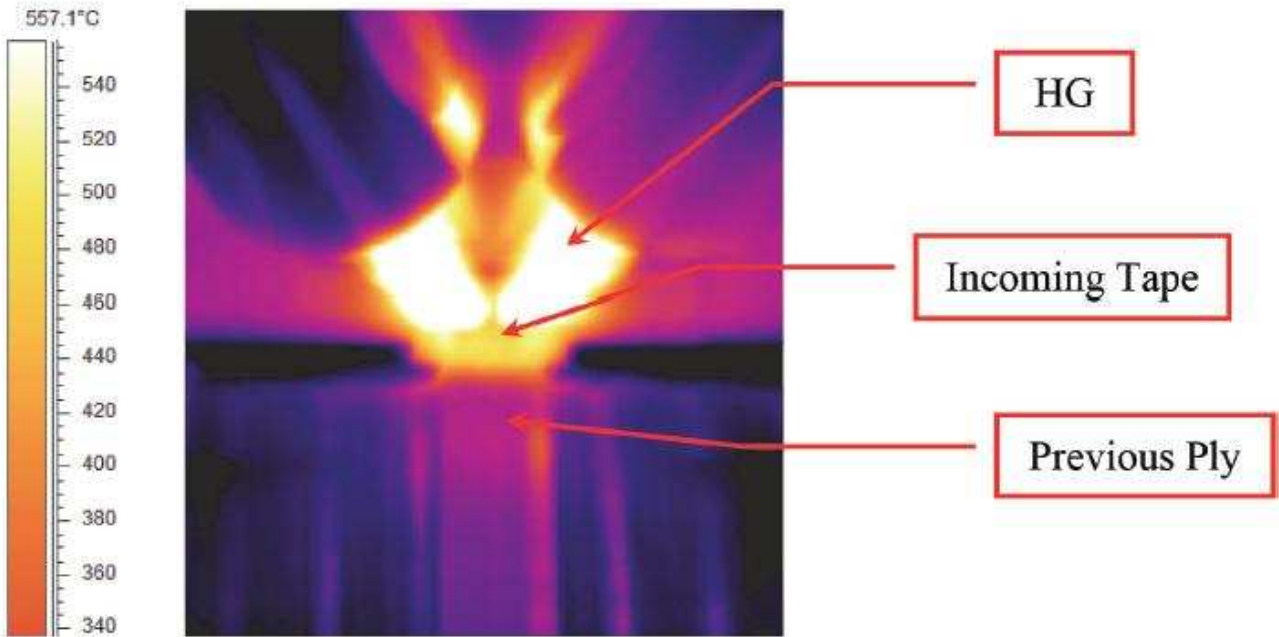


Figure 13: Thermal image of the nip region including the incoming tape [2].

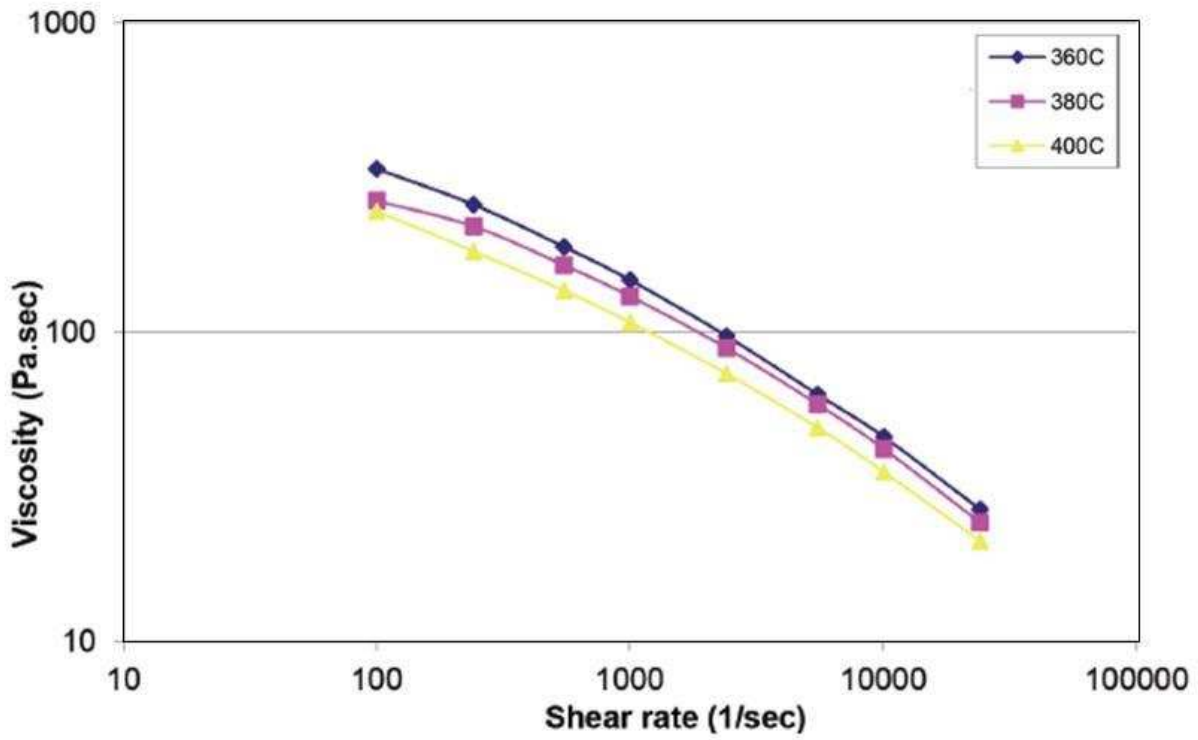


Figure 14: Viscosity versus shear rate for PEEK-150 [2].



Figure 15: Tested ILSS samples; a) Curved specimens and b) Baseline flat samples

Table 6: Summary of the test results of parametric study 1

Ring	Tape laying speed, V_R (mm/s)	ILSS (MPa)	Failure mode
Baseline panel	NA	109.84 ± 2.80	ILS
Baseline ring	100	92.69 ± 3.17	ILS
Laser ring	127	78.93 ± 2.38	ILS
1	57	50.58 ± 1.37	ILS
2	68	38.22 ± 1.92	ILS
3	73	44.73 ± 1.07	ILS
4	82	45.65 ± 1.78	ILS
5	95	46.55 ± 1.89	ILS

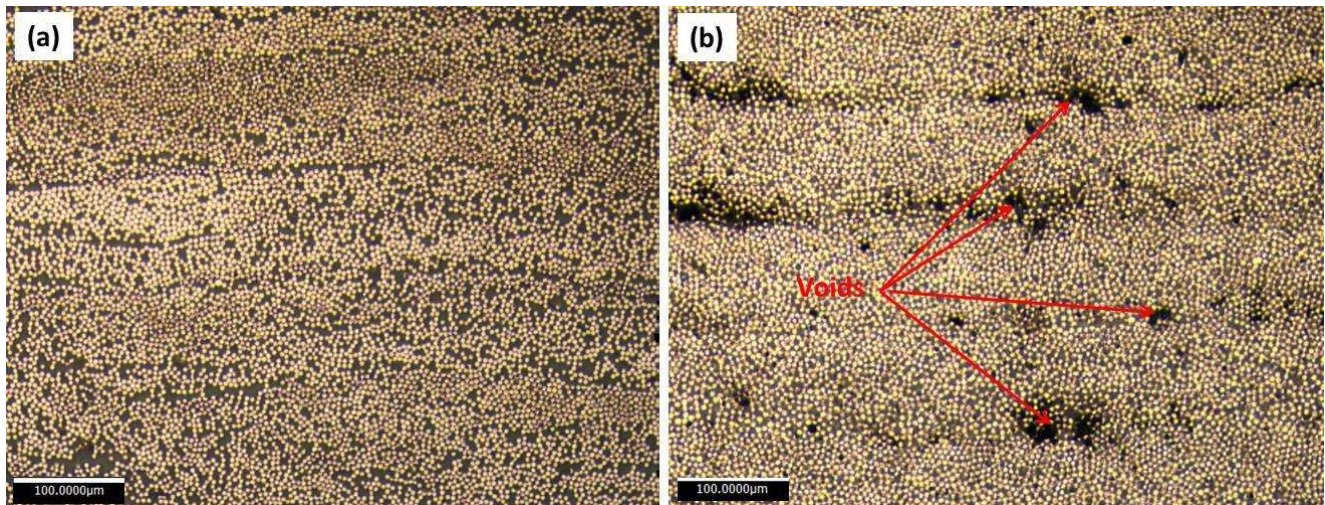


Figure 16: Optical cross sections of a) Autoclaved baseline and a) ATP in-situ consolidated composite samples

Table 7: Summary of the test results of parametric study 2

Ring	Tape Laying Speed, V_R (mm/s)	Roller compaction force (F_R)	ILSS (MPa)	Failure mode
6	60	75	45.18 \pm 0.60	ILS
7		125	44.26 \pm 1.82	ILS
8	65	75	44.32 \pm 2.02	ILS
9		125	49.23 \pm 0.88	ILS
10	75	75	47.39 \pm 1.20	ILS
11		125	47.59 \pm 1.59	ILS

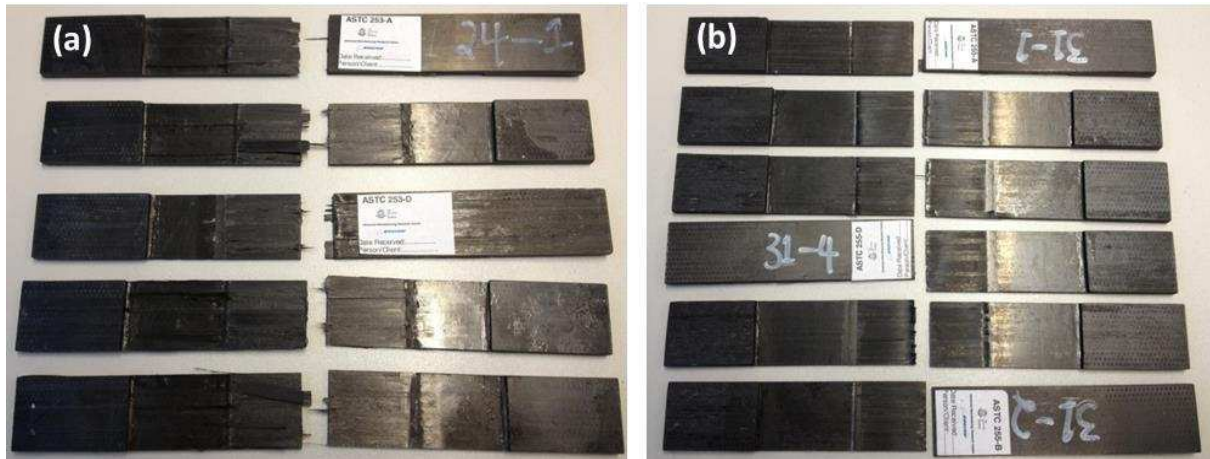


Figure 17: Tested LSS samples; a) ATP samples and b) Baseline autoclave samples

Table 8: Summary of Lap shear strength (LSS) test results

Tool Temperature, T_T ($^{\circ}\text{C}$) & Baseline + Resin Rich	Lap Shear Strength (LSS) MPa	Failure mode
Room temperature	9.24 ± 1.11	LFT
65	8.15 ± 0.66	LFT
105	11.47 ± 1.00	LFT
150	13.62 ± 2.05	LFT
Resin rich interface with $T_T=150^{\circ}\text{C}$	12.29 ± 2.76	LFT
Baseline (Autoclave processed)	25.70 ± 3.76	Midplane/cohesive failure



Figure 18: Tested ATP manufactured DCB samples

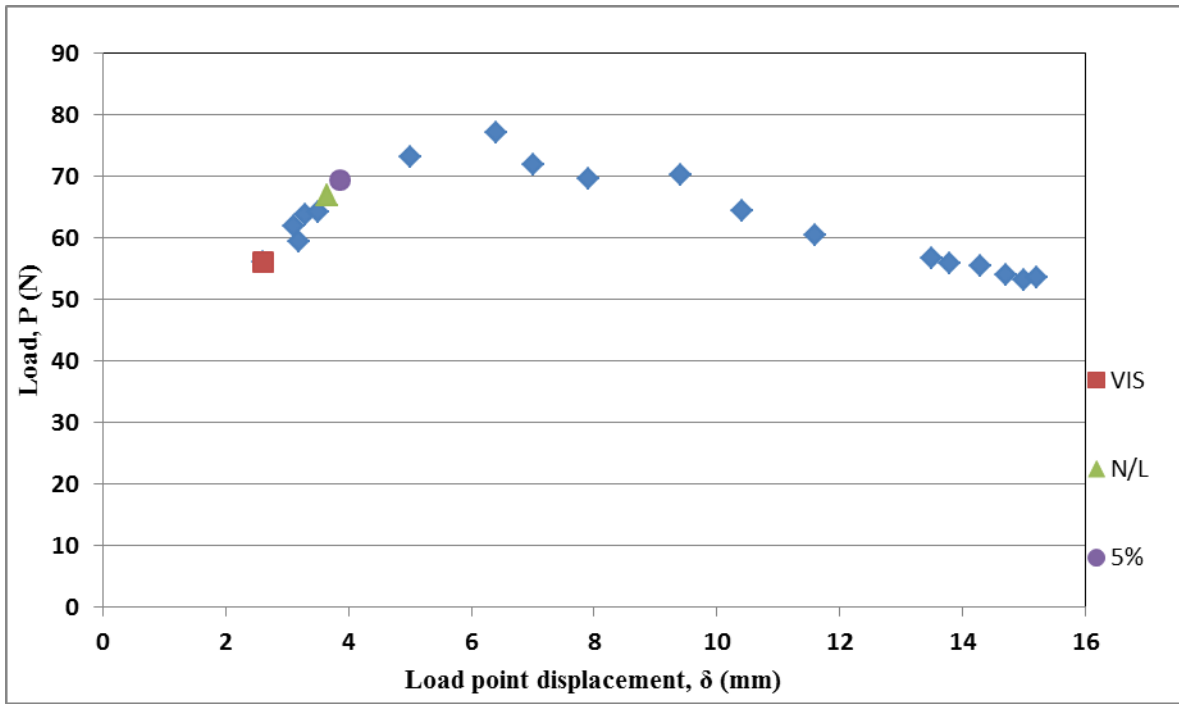


Figure 19: Typical load against load-point displacement

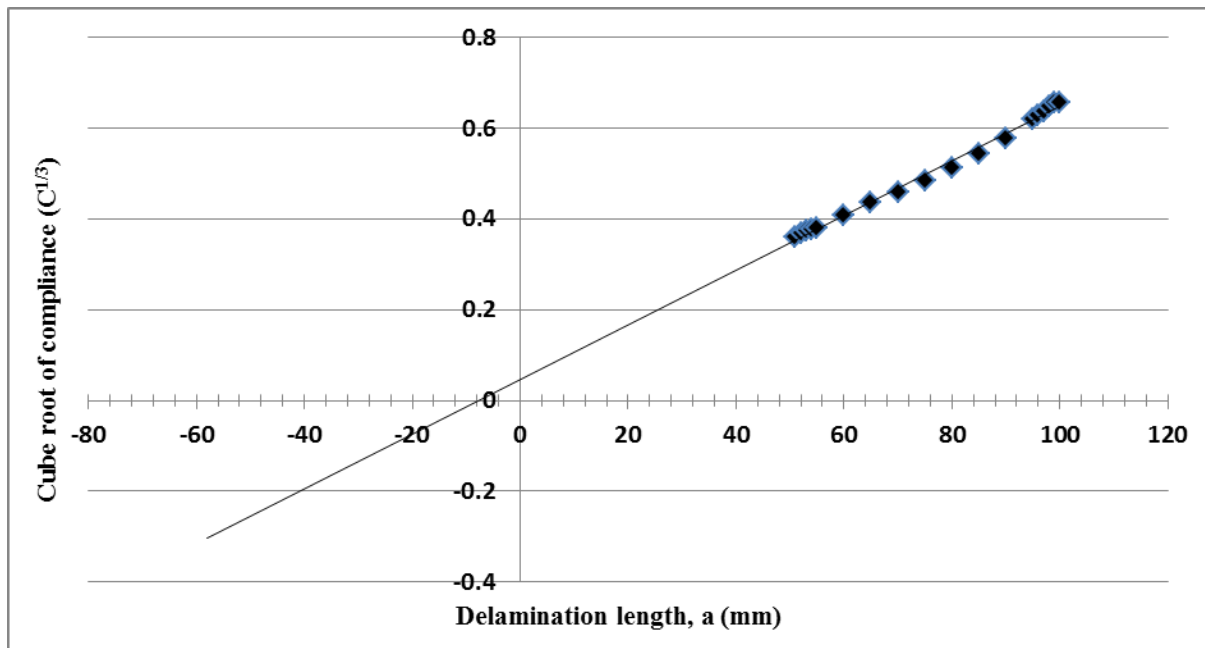


Figure 20: Typical cube root of compliance against delamination length

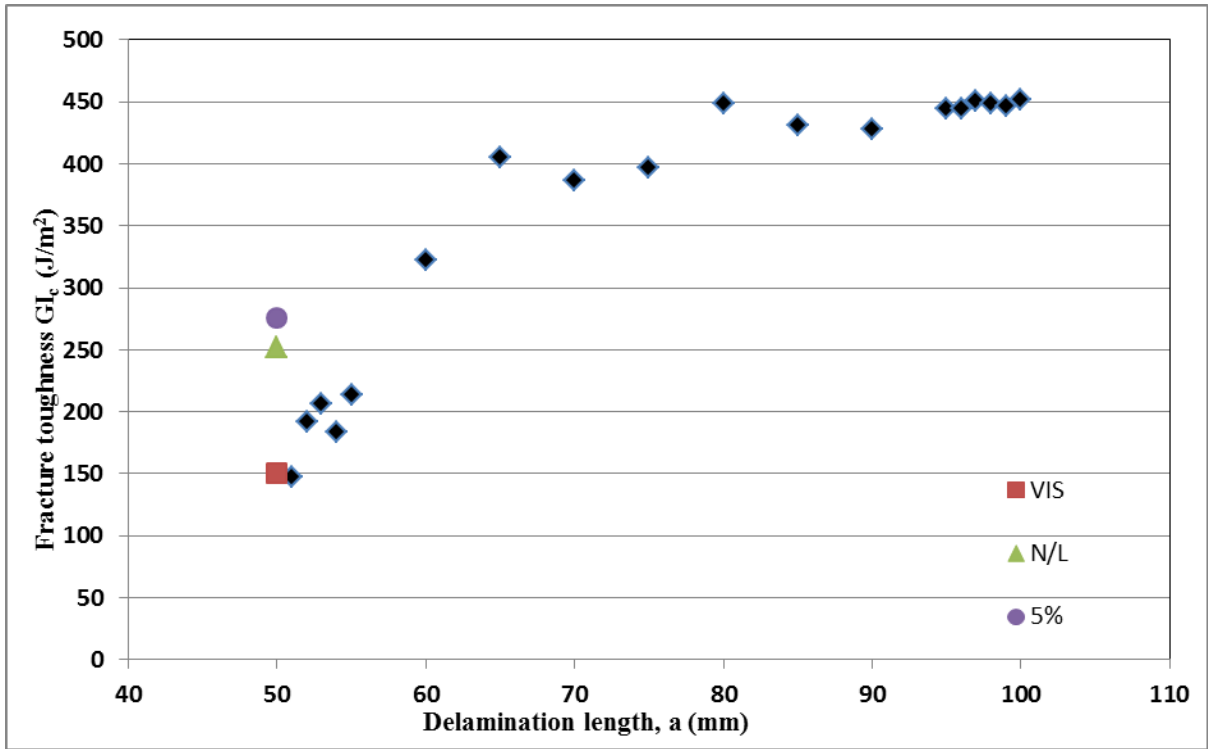


Figure 21: Typical resistance curve

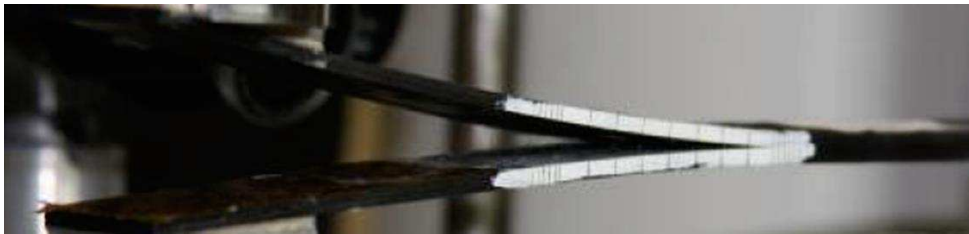


Figure 22: DCB sample during testing

Table 9: Summary of the results for visual fracture toughness (VIS) test

Tool Temperature, T_T ($^{\circ}\text{C}$) & Resin Rich	Visual Fracture Toughness, VIS (kJ/m^2)	Coefficient of Variation (%)
Room Temperature	0.199 ± 0.087	44
65	0.084 ± 0.052	63
105	0.165 ± 0.096	58
150	0.271 ± 0.071	26
Resin Rich with $T_T=150^{\circ}\text{C}$	0.255 ± 0.154	60

Table 10: Summary of the results for Non-Linear fracture toughness (NL) test

Tool Temperature, T_T ($^{\circ}\text{C}$) & Resin Rich	Non-Linear Fracture Toughness, NL (kJ/m^2)	Coefficient of Variation (%)
15 (Room Temperature)	0.331 ± 0.108	33
65	0.174 ± 0.103	59
105	0.312 ± 0.121	39
150	0.376 ± 0.090	24
Resin Rich with $T_T=150^{\circ}\text{C}$	0.485 ± 0.098	20

Table 11: Summary of the results for 5% offset fracture toughness test

Tool Temperature, T_T ($^{\circ}\text{C}$) & Resin Rich	Mean 5% Offset Fracture Toughness, (kJ/m^2)	Coefficient of Variation (%)
15 (Room Temperature)	0.381 ± 0.106	28
65	0.204 ± 0.113	55
105	0.372 ± 0.135	36
150	0.435 ± 0.102	24
Resin Rich with $T_T=150^{\circ}\text{C}$	0.570 ± 0.104	18

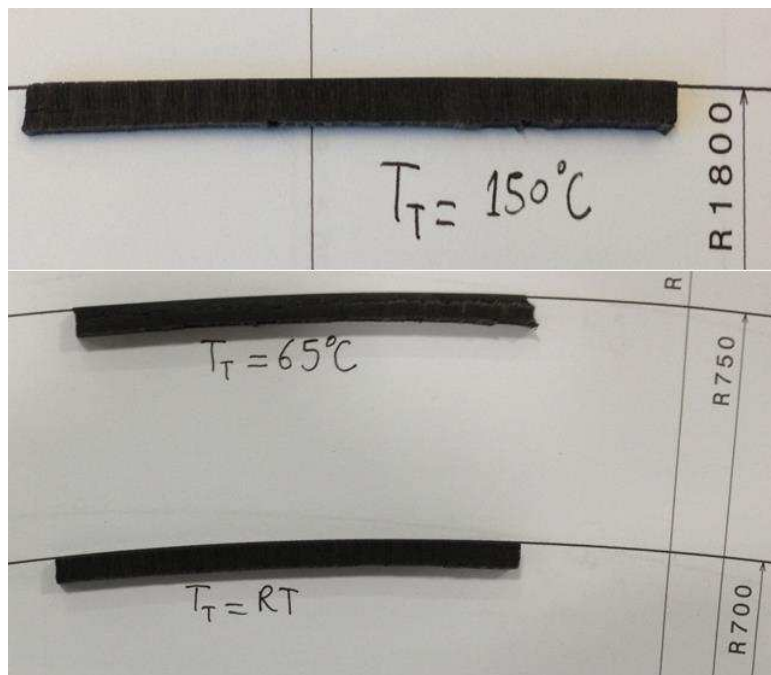


Figure 23: Curved strips from 150°C , 65°C and 15°C (RT) DCB panels

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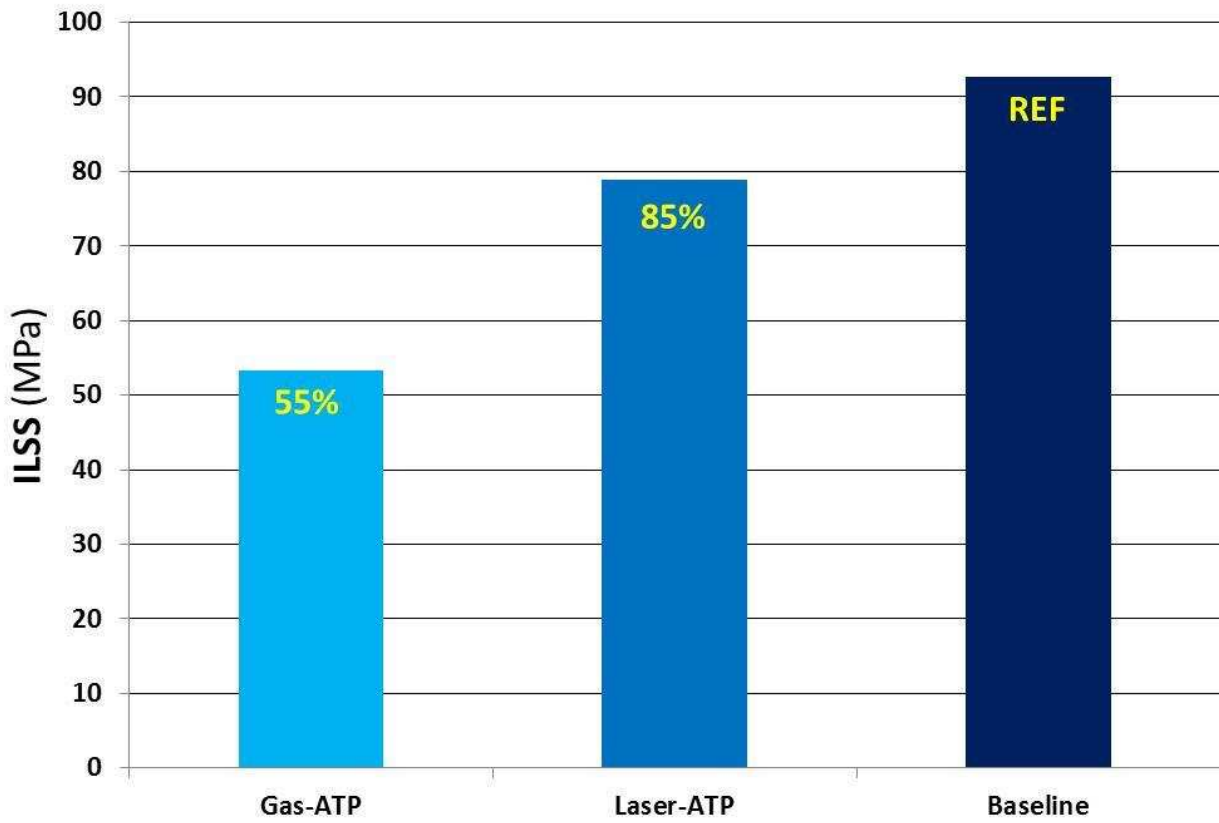


Figure 24: Comparison between three processing technologies in terms of ILSS: ATP-Hot Gas, ATP-Laser and Autoclave (baseline).