TECHNICAL PAPER

VICTREX AE™ 250 – A NOVEL POLYARYLETHERKETONE POLYMER SUITED TO AUTOMATED TAPE PLACEMENT AND OUT OF AUTOCLAVE PROCESSING

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victrex

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ABSTRACT

A Polyaryletherketone (PAEK) polymer from Victrex Ltd, Thornton-Cleveleys, UK has been created for automated lay-up and out of autoclave (OoA) consolidation. VICTREX AE™ 250 is a co-polymer based on polyetheretherketone (PEEK). It is a semi-crystalline polymer with mechanical properties and chemical resistance typical for this class of polymer. Combined with reinforcing carbon fibres it provides mechanical properties that are consistent with the requirements for structural aerospace applications.

Oven processed laminates (so called Out of Autoclave) made from stacked unidirectional tapes are shown to be well consolidated, substantially void free and with mechanical properties that are consistent with press consolidated product, opening a broader range of manufacturing options for aerospace parts including automated layup.

INTRODUCTION

The world of aerospace composite materials exists in two camps: those based on thermosets and those based on thermoplastics. Thermosets had a head start in the 1960's, but both polymer chemistries have been available commercially in the industry since the early 1980's with the introduction of PEEK by Imperial Chemical Industries (ICI). Both systems generally have comparable mechanical properties (thermoplastics being generally tougher) and both present manufacturing and processing advantages/disadvantages relative to each other. During the 1990's, thermosets won out during the development of significant aerospace civil aircraft development projects, largely based on cost and the ease of parts manufacture, as thermosets have tacky surfaces that enable them to be stacked without ply slippage, they are softer and more conformable to aid layup and their lower cost was attractive to aircraft build economics at that time.

Now, in 'the new era', things are changing and the emphasis is on build rate for mid-market single aisle aircraft, with manufacturers looking to significantly increase monthly output. Boeing estimate a build rate of fifty-seven aircraft per month in 2019 for their 737 aircraft [1] and Airbus are looking to produce sixty A320 aircraft per month in the same timeframe. [2] In part this is being facilitated with the development of automated tape laying (ATL) and automated fibre placement (AFP) suited to some thermoplastic prepregs. Interest in these materials is being boosted further with developments in out of autoclave processing, hot stamping and hybrid overmoulding; [3] processes that help manufacturers make parts more quickly and economically, avoiding the need for high numbers of expensive autoclaves and factories in which to house them. The prospects for thermoplastic composites in this sector are looking healthy. [4]

In this paper a composite prepreg manufactured using a relatively new high performance PAEK (polyaryletherketone) polymer from Victrex Plc (UK) will be described, which offers out of autoclave (OoA) processing at lower temperatures than Polyetheretherketone (PEEK) and polyetherketoneketone (PEKK) whilst displaying excellent consolidation with minimal voids and good mechanical properties.

It will also be demonstrated that AE 250/Carbon fibre prepreg laid by hand and by automated fibre placement (AFP) can be processed to form fully consolidated laminates that are essentially void free. Furthermore, it will be shown that kitted prepreg stacks can be compression moulded either in a low pressure press, or in an oven under vacuum to create compacted laminates that have substantially equivalent physical and mechanical properties.

EXPERIMENTATION

Polymer

The novel thermoplastic is a co-polymer based on polyketone chemistry which falls into the general class of PAEK polymers. In the world of Chemistry, this class covers a wide range of polymers comprising aromatic moieties connected by ketone and ether linkages at various ether to ketone ratios and with defined sequencing. VICTREX AE™ 250 retains the mechanical, physical and chemical resistance properties typical of PEEK with a lower crystalline melting temperature than PEEK, or PEKK. The mechanical properties of VICTREX AE™ 250 polymer are shown in Table 1.

The polymer is semi-crystalline (25-30 % typically) at cooling rates consistent with oven or press consolidation (5-10 °C/minute). The melting temperature is around 305 °C as measured using differential scanning calorimetry (DSC).

DSC thermographs for amorphous pressed films made from raw polymer (no carbon fibres) are shown in Figures 1 a-c after the first temperature cycle (a), the cooling phase (b) and the second cycle (c) respectively. These confirm key thermal transitions over the temperature range with a cold crystallisation peak at 189 °C, crystalline melting point in this example of 303 °C and recrystallization peak at 271 °C. The degree of crystallisation upon cooling reached 28 % and the glass transition temperature on the second cycle was 148 °C.

Mechanical Data				
Tensile Strength	Yield, 23°C	ISO 527	MPa	90
Tensile Elongation	Break, 23°C	ISO 527	%	15
Tensile Modulus	23°C	ISO 527	GPa	3.5
Flexural Strength	23°C	ISO 178	MPa	150
Flexural Modulus	23°C	ISO 178	GPa	3.3
Izod Impact Strength	Notched, 23°C	ISO 180/A	kJ m^2	5.0

Table 1. Typical mechanical properties of VICTREX AE™ 250 PAEK polymer

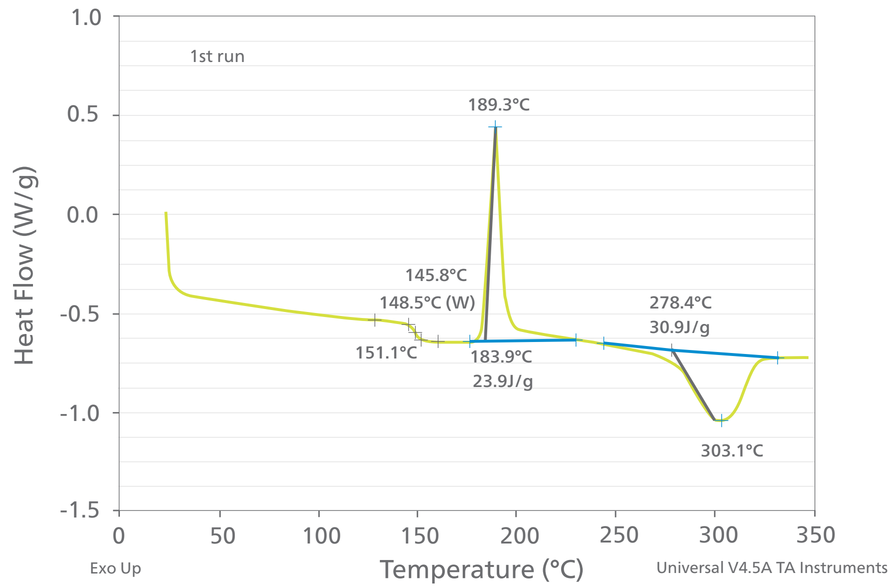

Figure 1 (a). First run DSC on raw polymer

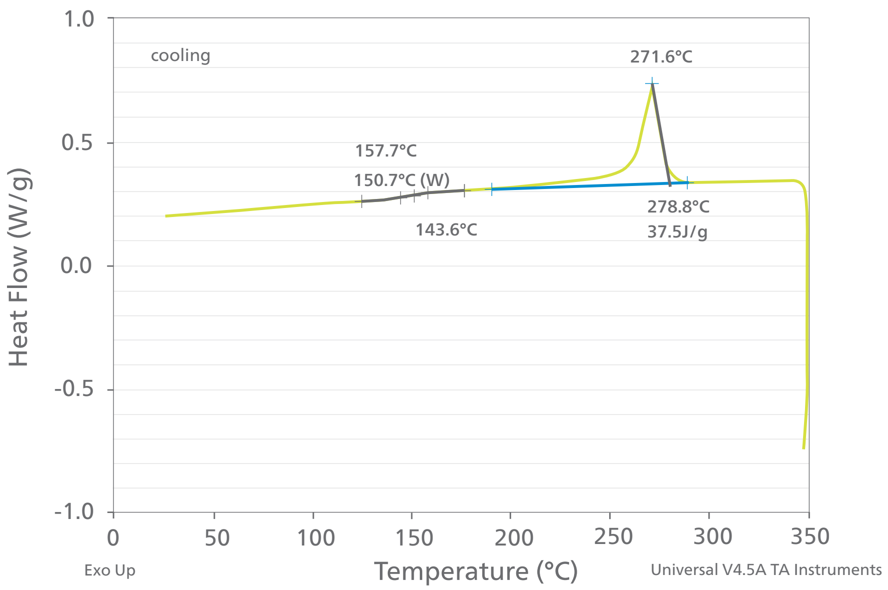

Figure 1 (b). Cooling curve DSC on raw polymer

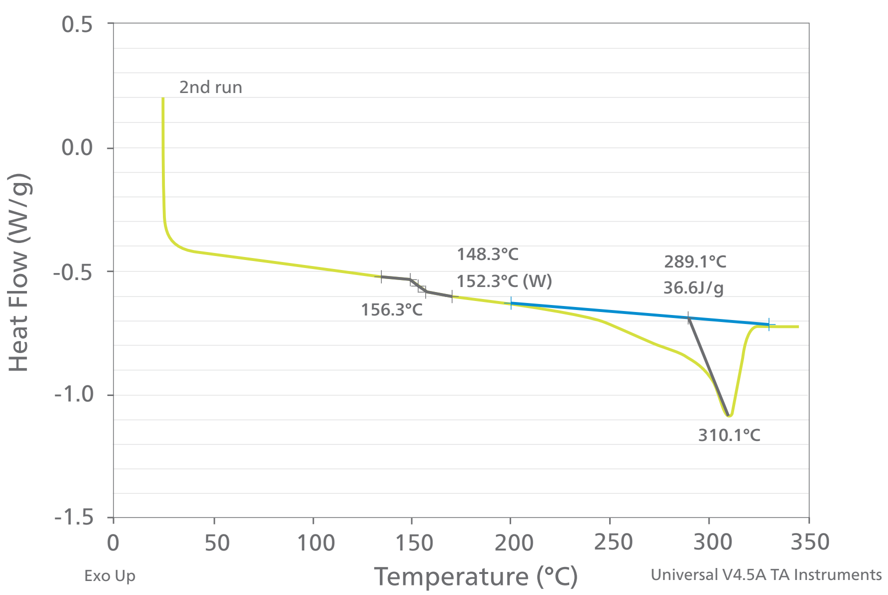

Figure 1 (c). Second run DSC on raw polymer

Composite Prepreg

Composite prepreg tapes were prepared by Victrex comprising unidirectional carbon fibres (Hexcel AS4 and AS7) impregnated with AE 250 polymer. Tapes for AFP were prepared by slitting to 6.35 mm ± 0.125 mm. Tapes for hand layup were prepared at a width of 50 mm ± 0.125 mm. The details are for these tapes are shown in Table 2.

Prepreg	Fibre	Fibre Area Weight (FAW g/m2)	Prepreg tape width (mm)	Process (layup and consolidation)
AE 250/CF	AS4	134	6.35 ± 0.125	AFP/Press
AE 250/CF	AS7	192	50.0 ± 0.125	Hand layup/Press
AE 250/CF	AS7	192	50.0 ± 0.125	Hand layup/Oven

Table 2. Prepreg tapes for AFP and hand layup

Processing

Automated Fibre Placement (AFP)

Tapes with a fibre area weight (FAW) of 134gsm (Hexcel AS4 carbon fibre) nominally 6.35 mm wide (within the width tolerance) were laid by a Coriolis AFP machine (CORIOLIS COMPOSITES TECHNOLOGIES S.A.S, France) to create partially consolidated laminates with fibre orientations and laminate thicknesses suitable for mechanical testing. These were fully consolidated at Victrex by pressing between steel caul plates and polyimide film treated with Frekote 55 mould release in a computer controlled Lauffer hydraulic press (Maschinenfabrik Lauffer GmbH & Co. KG, Germany) fitted with electrical platen heating and air/water cooling. The press was programmed with the requisite temperature and pressure sequence, as described below.

Hand Layup

Panels made from hand laid tapes were prepared as follows. Strips of prepreg tape (50mm wide with AS7 Hexcel carbon fibres at a FAW of 192 g/m2) were cut and stacked by hand using a hot iron operating above Tm, to tack weld and/or seam weld the tapes together. Welding was done at multiple points along and across the tape to form kitted ply lay-ups with ply stacking sequences and orientations that met the requirements for mechanical testing as detailed in Table 3.

Property	Lay-up	Number of Plies
In plane shear strength and modulus	[+45/-45]4S	16
Compression after impact (CAI)	[+45/0/-45/90]3S	24
Mode I (G1c) Fracture Toughness	[0]18	18
Plain tensile strength and modulus	[+45/0/-45/90]3S	24
Filled hole tensile strength (FHT)	[+45/0/-45/90]3S	24
Plain compression strength and modulus	[+45/0/-45/90]3S	24
Open Hole Compression (OHC)	[+45/0/-45/90]3S	24
Filled Hole Compression (FHC)	[+45/0/-45/90]3S	24
Bearing strength	[+45/0/-45/90]3S	24

Table 3. Layup sequence for hand-laid 50 mm wide prepreg tapes

These kitted stacks were then processed either by compression moulding in the Lauffer platen press operated by Victrex, or by oven processing using only vacuum and temperature to achieve consolidation. In practice an *autoclave* was used for this purpose (operated by the Thermoplastic Composite Research Centre (TPRC) in Enchede, Holland) although without any applied pressure the autoclave was used only as an oven to heat the prepreg. This process is referred to here as Out of Autoclave (OoA) processing, in common with industry standard nomenclature. Kitted prepreg stacks were positioned on a steel plate and covered with a breather layer (glass cloth) separated with Frekote 55 treated polyimide film. The whole stack and associated layers of breather and film layers were sealed within a covering layer of polyimide film, which was bonded to the base plate using a high temperature resistant sealing adhesive around the perimeter.

Process Thermal Cycles

The process cycles utilised in the preparation of test laminates are shown in Tables 4, 5 and 6. Table 1 illustrates the press cycle used to consolidate the prepreg material laid by AFP which is also illustrated for clarity in Figure 2.

Press	Hydraulic
Platen Heating	Electrical
Cooling	Water/Air mix
Heat up rate	7 °C/minute
Hold temperature	350 °C
Hold time at temp.	15 minutes
Hold pressure	Two step: ramp 2 Bar then 6 Bar at max. temp.
Cool down rate	-4.5 °C/minute

Table 4. Press consolidation cycle for AFP laid panels

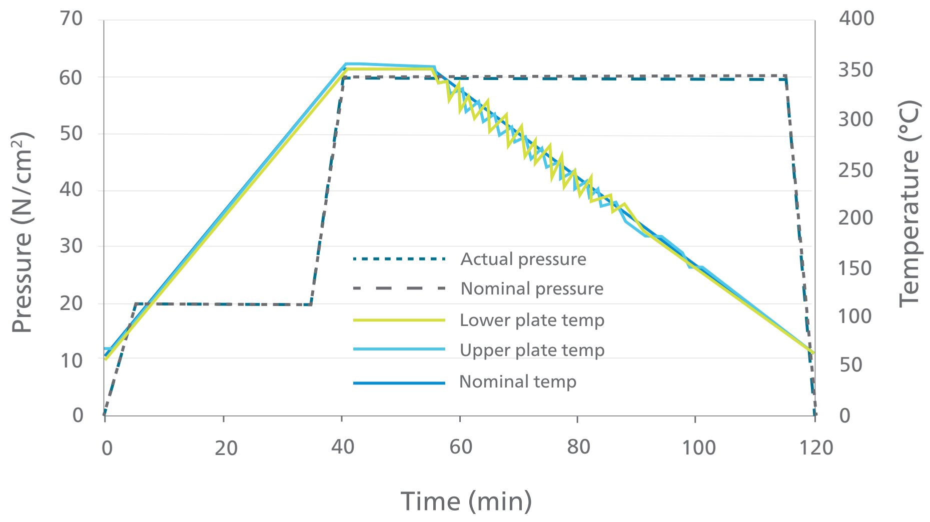

Figure 2. Press cycle for tapes laid by automated fibre placement (AFP)

Press Cycle for Hand Laid Panels

Press	Hydraulic
Platen Heating	Electrical
Cooling	Water/Air mix
Heat up rate	7°C/minute
Hold temperature	360 °C
Hold time at temp.	30 minutes
Hold pressure	1 bar
Cool down rate	-5 °C/minute

Table 5. Press consolidation cycle for hand laid panels

Oven Cycle for Hand Laid Panels

Platen Heating	Electrical
Cooling	Air
Heat up rate	7°C/minute
Hold temperature	360 °C
Hold time at temp.	30 minutes
Hold pressure	1 bar
Cool down rate	-6 °C/minute

Table 6. Oven consolidation cycle (using autoclave without added pressure)

Post Process Analyses

Physical and mechanical testing of all laminates was undertaken by GMA-WERKSTOFFPRÜFUNG GmbH (Stade, Germany). Laminates were ultrasonically C-scanned with an Omniscan MX unit using water coupling and then sectioned and imaged by optical microscopy to assess consolidation quality. Samples were analysed for fibre content, matrix content and porosity content by acid digestion. Density was measured and the thermal characteristics were recorded by DSC to ensure that the laminates met expectations.

Mechanical Testing- Hand Laid Laminates

Specimens were removed from both oven and press consolidated laminates using a diamond saw and in accordance with the relevant test methods. These were tested in triplicate under room temperature/ dry and in some cases elevated temperature wet conditions, as summarised in Table 7.

Properties	Specimen Geometry	Dry R.T.	Wet 70°C
In plane shear strength and modulus	230 mm x 25 mm	3	
Compression after impact (CAI) 35J	150 mm x 100 mm	3	
Mode I (G1c) Fracture Toughness	250 mm x 25 mm	3	
Plain tensile strength and modulus	340 mm x 32 mm	3	
Filled hole tensile strength (FHT)	280 mm x 32 mm	3	3
Plain compression strength and modulus	162 mm x 32 mm	3	3
Open Hole Compression (OHC)	162 mm x 32 mm	3	
Filled Hole Compression (FHC)	162 mm x 32 mm	3	3
Bearing strength	150 mm x 45 mm	3	

Table 7: Mechanical test matrix

RESULTS

Consolidation

AFP Panel

An example C-scan of an AFP laid/press consolidated laminate is illustrated in Figure 3. This shows that the laminate was fully consolidated without any areas of delamination and is typical of all laminates made by this process during this work.

Density measurements according to ASTM D 3171 method 1B-15 did not detect any porosity in the consolidated laminates.

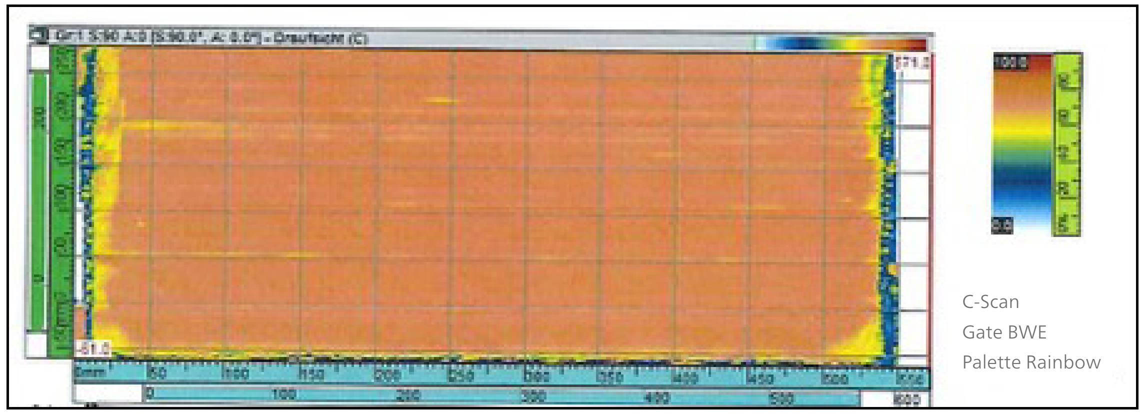

Figure 3. C-scan of AFP laid laminate consolidated in a press

Hand Laid Panels

Figures 4 a & b illustrate a typical C-scan result for laminates made by oven consolidation (a) and press consolidation (b). VICTREX AE™ 250 composite tape processed well and was fully consolidated in each case with no delamination evident.

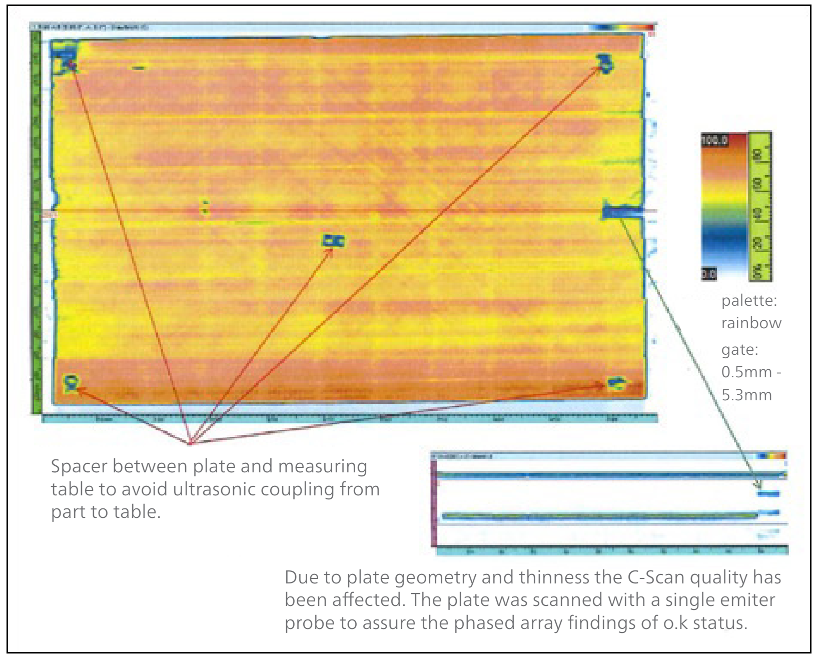

Figure 4 (a). C-scan chart for oven consolidated laminate

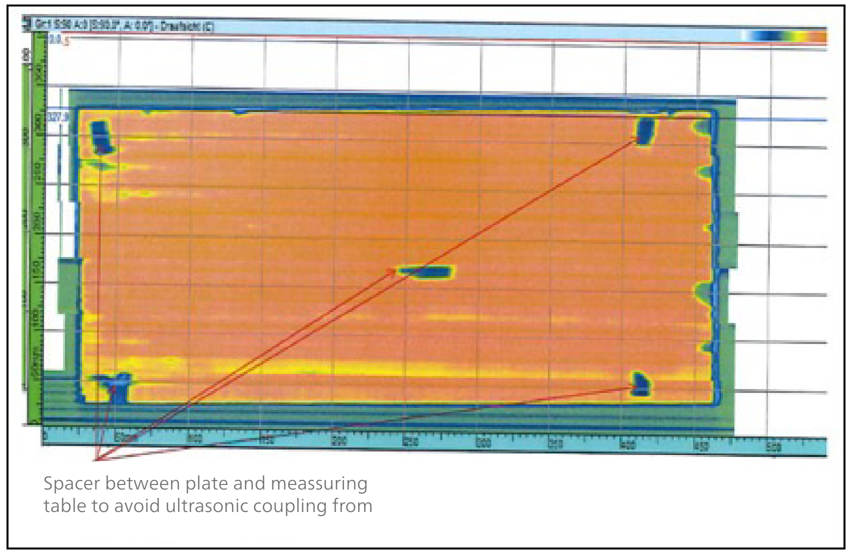

Figure 4 (b). C-scan chart for press consolidated laminate

For the oven and press consolidated laminates made using hand layup, sections of laminate removed, polished and imaged by optical microscopy confirmed that full consolidation had occurred in all cases. Example micro-cuts for the oven processed laminates are shown in Figure 5 a-c.

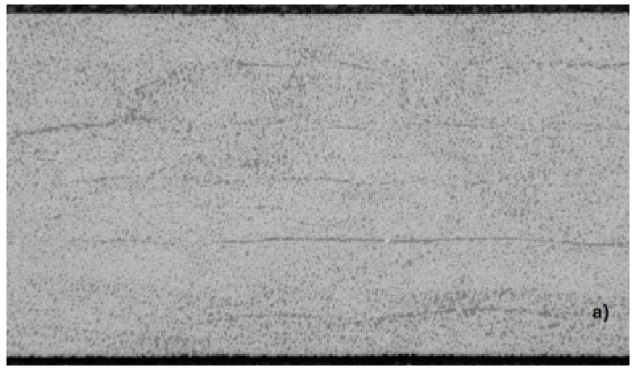

Figure 5a. Polished optical micro-sections of unidirectional laminate consolidated out of autoclave.

Figure 5b. Polished optical micro-sections of ± 45 laminate consolidated out of autoclave.

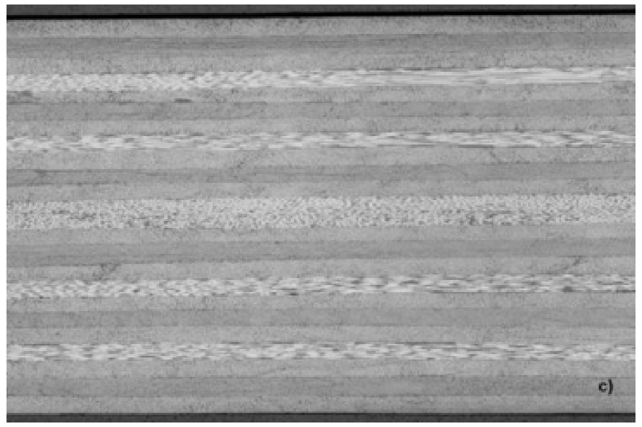

Figure 5c. Polished optical micro-sections of quasi-isotropic laminate consolidated out of autoclave.

Differential Scanning Calorimetry (DSC)

Thermographic analysis of the consolidated laminates by DSC (Universal V4.5A TA Instruments) confirmed that in *all cases* (AFP laid/pressed and hand laid OoA and pressed laminates) the material was fully crystallised after processing. This was evidenced by the absence of a 'cold' crystallisation peak (at 189 °C in amorphous polymer) for *any* of the laminates produced. Figures 6 and 7 are example DSC thermographs for hand laid oven processed and pressed laminates respectively. By calculation, using a heat of crystallisation of 130 J/g, the area under the melt peak in each case gave crystallinity levels of between 25-28 % for all laminates.

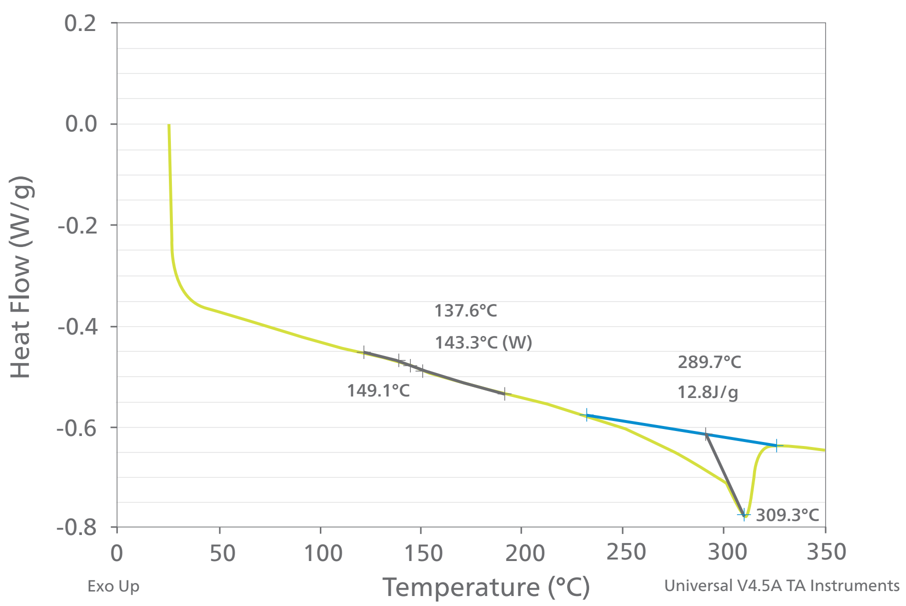

Figure 6. Example DSC thermograph for oven processed laminate

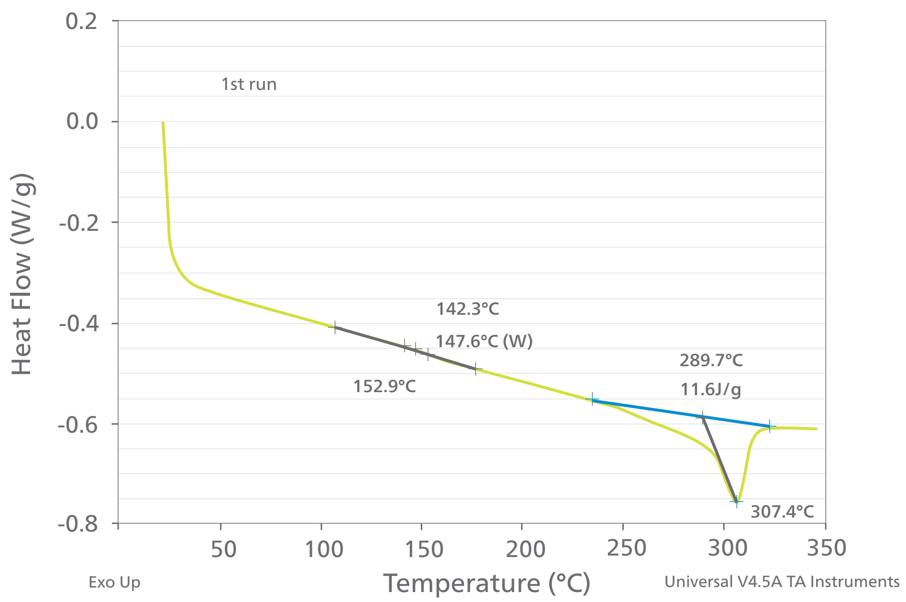

Figure 7. Example DSC thermograph for platen pressed laminate.

Mechanical Properties Test Results – Hand Laid Laminates Only

As this is a *comparison* between oven consolidated and press consolidated laminates only their *relative* performance will be presented, as shown in Table 8. Here the press consolidated laminates represent the baseline level of performance with oven consolidated laminates being compared with the baseline values on a percentage basis.

Properties		Oven Consolidated Compared with Press Consolidated (% Retained Properties)	
		R.T.	70 °C Wet
In plane shear strength and modulus	Strength	95%	-
	Modulus	104%	-
Compression after impact (CAI)		103%	
Mode I (G1c) Fracture Toughness		83%	
Plain tensile strength and modulus	Strength	100%	-
	Modulus	104%	-
Filled hole tensile strength (FHT)		103%	-
Plain compression strength and modulus	Strength	98%	101%
	Modulus	105%	103%
Open Hole Compression (OHC)		102%	-
Filled Hole Compression (FHC)		103%	103%
Bearing strength		102 %	-

Table 8. Comparative performance between oven consolidated and press consolidated (baseline) laminates.

DISCUSSION

The C-scan image presented in Figure 3 confirms that VICTREX AE™ 250 composite tapes can be laid by AFP and pressed to create fully consolidated laminates. Density measurements (ASTM D 3171 method 1B-15) confirm that laminates are fully dense, with no measureable void content.

Also this work has shown that VICTREX AE™ 250 composite tape can be fully consolidated by low pressure out of autoclave (OoA) processing, or with a low pressure platen press, using as little as 1 bar, as illustrated in sectional micrographs exemplified in Figures 5 a to c), creating fully dense composites with minimal voids (below detection). Laminate compaction quality has been confirmed by C-scanning, as shown in Figures 4a and b.

With its lower melting temperature compared with PEEK (305 °C vs 343 °C) VICTREX AE™ 250 unidirectional tape offers a wider processing window. DSC results (Figures 6 and 7) show that cooling at moderate rates, as with press and oven cycles (i.e. -5 °C/minute) allows full crystallinity to be achieved at levels of about 28 % which imparts maximum mechanical properties, heat and chemical resistance.

The mechanical properties of composite laminates made by either process are very comparable as shown in Table 8, illustrating that the material can be processed by either route. The low pressure processing ability of AE 250 composite prepreg tape is a major advantage for processors and opens the gates to the production of high quality parts utilising out of autoclave processing.

Preformed material can be pressed by hot stamping processes to produce parts rapidly, meeting the demands for higher throughput rates required to meet aircraft build targets discussed in the introduction.

CONCLUSION

The conclusion from this work is that VICTREX AE™ 250 polymer processes equally well under oven consolidation and press consolidation using hand layup or automated fibre placement to create highly consolidated laminates with substantially identical physical and mechanical properties, opening a broader range of manufacturing options for aerospace parts. The lower melting temperature of AE 250 polymer widens the processing window whilst still allowing fully crystalline morphology to develop through the cooling phase.

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