AUTOMATED FIBER PLACEMENT OF THERMOPLASTIC MATERIALS: PURSUIT OF LOW POROSITY WITHOUT THE AUTOCLAVE

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1. Abstract

In-situ consolidation, where very low porosity content is sought, has been focusing attention of the aircraft industry for the past few years. This paper presents the latest advances made by Coriolis on such topic. A state of the art laser assisted automated fiber placement (AFP) head developed by Coriolis was used to produce in-situ and autoclave consolidated samples. Porosity content, degree of crystallinity and interlaminar shear strength were measured for each consolidation type. Manufacturing settings leading to very limited porosity content and acceptable degree of crystallinity are presented and improvement paths are proposed to highlight key features needed for the next generation of AFP machines.

2. Introduction

Through the past decade, thanks to its multiple advantages, composite materials have gained increasing attention to the aeronautical industry. Thermoset composite solutions are now widely selected by all the aircraft manufacturer. The proof can be found by looking at the material composition of the flag ships from both main aircraft manufacturers (Airbus A350 and Boeing B787) with more than 50% of composites materials (Figure 1).

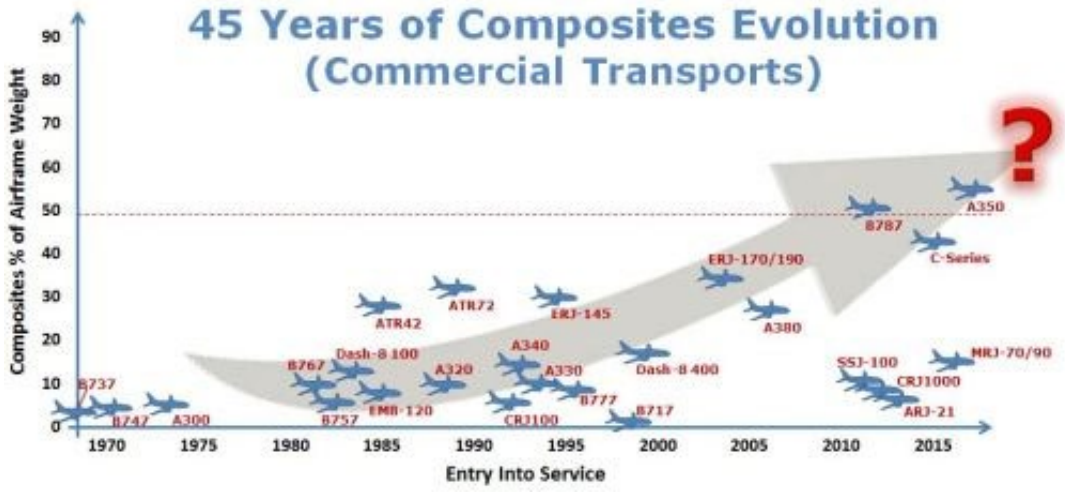


Figure 1 Evolution of the part for composite materials in aircrafts [1]

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In the meantime, automated manufacturing processes like automated fiber placement (AFP) have been developed to permit manufacturing of larger parts difficult to make by hand layup. Figure 2 shows a Coriolis AFP machine installed in the Airbus plant in Nantes manufacturing the keel beam of the A350-1000.

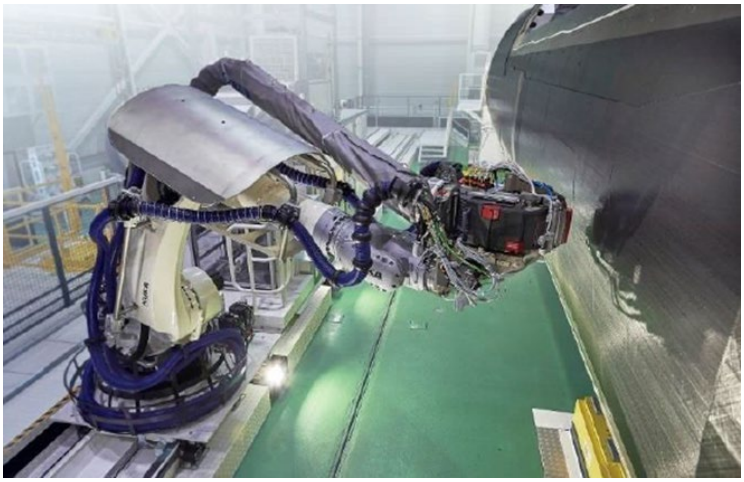


Figure 2 Coriolis AFP manufacturing a A350-1000 keel beam in the Airbus plant in Nantes, France

Thermoplastic materials can be remelted and reformed as they do not form three dimensional cross-linked structures. They also show higher performance with improved toughness, impact resistance, chemical resistance and low water absorption. Most of all, thermoplastic materials have unlimited shelf life and allow structures repairs and easy joining and welding operations.

The use of thermoplastic materials is yet limited in the aircraft industry. One can find flying parts such as wing ribs ([2]), small clips or the Airbus A380 wing leading edge [3]. Until now, most of TP parts are consolidated by autoclave oven or formed by stamping. This constraint associated with material price make TP material a suitable solution for only few applications.

For the past few years, alternative consolidation ways have appeared such as vacuum oven consolidation press stamping or in-situ consolidation. Oven consolidation allows to decrease the price of the consolidation device. However, this process requires a perfect control of the process to reach well consolidated parts. Press stamping allows to transform 2D preforms in 3D net shape parts with low cycle time. This process is limited to relatively small parts due to press capabilities and required compaction pressures. Finally, in-situ consolidation can be obtained directly from AFP layup, it allows large savings in part cost as the consolidation step can be removed. However, many challenges are associated with such consolidation type. The final part needs to be manufactured without any defect, which involves very low porosity content, perfect interlaminar bond, controlled degree of crystallinity and controlled dimensional geometry of the final part. Then, the impact of the layup parameters on laminate quality needs to be perfectly understood.

Thermoplastic AFP is a very complicated subject to study numerically as all the phenomenon are transitory and experimentally as it involves very high process temperatures. The holy grail of such process is to understand the impact of each parameters (layup, raw material) on the laminate microstructure and polymer chemistry. A better understanding of the physics involved in such process would permit to produce viable parts for the aircraft industry without a post consolidation process.

Pitchumani et al [4] set the scene in 1996 by highlighting the impact of process parameters on the quality of interfacial bonding and void content. They showed that, in the tow placement process, the time of residence under the compaction roller is key to ensure intimate contact development. The degree of bonding decreases dramatically with increasing layup speed. Increasing the compaction force increases the degree of bonding and reduces the void content. After this first work, many numerical models have been developed focusing on heat transfer through laminate thickness during fiber deposition. Chinesta et al. [5] proposed models helping to understand the different phenomenon happening during and after layup such as heat transfer resulting in residual thermal stresses buildup. The influence of material roughness on heat transfer and ply bonding has been investigated by Leon et al [6] and highlighted the need of a material with microroughness.

On the other hand, many experimental studies have been conducted to understand the most influencing parameters on the composite microstructure. Stokes-griffin and Compston [7] studied the relationship between layup speed and temperature. When, at 100mm/s, a process temperature of 400°C is sufficient, at temperature of 550°C is required to maintain composite performances at 400mm/s. with these parameters, they obtained in-situ composite that reached 95% of the short beam strength (SBS) of corresponding conventional composite. Tierney and Gillespie [8] showed that the bond strength, characterized by SBS, decreased with layup speed. They also demonstrated that an over exposition of the material to heat had the inverse effect by degrading the strength. Kok and al [9], showed the existence of deconsolidation with over exposure of the tape. This porosity increase was not recovered by the AFP process. Slange [10] explained that this deconsolidation can be due to the evaporation of moisture from the tape. Dolo et al. [11] proposed a model for thermal degradation of PEEK. They showed that the degradation temperature decreases with time, but the time/temperature couples found in AFP seem to remain acceptable. Hence, considering the short time of exposure in AFP manufacturing, it is possible to apply high processing temperatures. The heating device is also the source of many studies. Qureshi et al [12] demonstrated that the heat source used to melt the polymer matrix had an effect on the interlaminar shear stress (ILSS) properties of the laminate. 85% of the reference laminate ILSS was reach with laser heating whereas only 55% where obtained using hot Gas AFP systems. Di Francesco [13] demonstrated that a concentrated laser beam spot allowed to increase layup speed and developed a predictive model for power supply regarding head velocity.

Tierney [8] showed that at 400°C, a minimum contact time of 0.11s should be applied to allow the reptation phenomenon to occur. Compaction time, hence layup speed, is then critical to reach optimum consolidation with proper entanglement of polymeric chains. However, Stokes-Griffin and Compston [14] demonstrated that bonding can occur below melting temperature with the amorphous phase of the polymer. Whereas high degrees of crystallinity are sought in the industry, Sacchetti et al.[15] demonstrated on C/PPS that low degrees of crystallinity could lead to major improvement in interlaminar fracture toughness.

As a synthesis, Comer [16] compared the mechanical properties of two laminates laid up by AFP with "in-situ" parameters and consolidated by autoclave. The results showed lower properties of the in-situ laminate for every mechanical test (70% for ILSS and flexural strength and 90% for flexural stiffness). These results can be explained by the higher porosity content found in in-situ laminates (2.8%) compared to autoclave composites (0.08%).

There exist many types of thermoplastic composite automated deposition systems with different heating sources [7], [8], [13], [16]. Cartié et al. [17] conducted an experimental comparative study on the impact of the above parameters on the microstructure of the composite in situ consolidated. This study helped to evaluate the influence of each parameters on the composite laid up with the same AFP machine (Coriolis robot).

This study goes one step further into the understanding of the in-situ consolidation. When optimized layup parameters have been determined to obtain an optimal microstructure (low porosity content) [17], this paper evaluates the consequences of in-situ consolidation on the polymer crystallinity and resulting mechanical properties (flexural and interlaminar) of the composite. The results are compared with the same composite material consolidated with an autoclave. The objective of this study is to evaluate the suitability of in-situ AFP consolidation for aerospace structural parts.

3. Materials and method

3.1. Machine details

The machine used to manufacture the panels of this study is a state of the art CORIOLIS® C1, 8x1/4 Inch tows head mounted on an ABB IRB6650 robot, itself mounted on a GUDEL TM90 linear axis. This machine can layup simultaneously up to 8 continuous tows 6.35mm (1/4inch) wide at speeds up to 1000mm/s. This machine is the property of the ComposiTIC Research Centre based in Ploemeur, Brittany, France.

In order to heat the material to the required processing temperature, a laserline LDF6000-100 6kW Infrared laser source was used. A laser homogenizer optics (Figure 3.a) mounted on the fiber placement head is creating a rectangular laser beam of a cross section of 8mm height and 56mm wide. An optical fiber is guiding the laser energy from laser source to the fiber placement head.

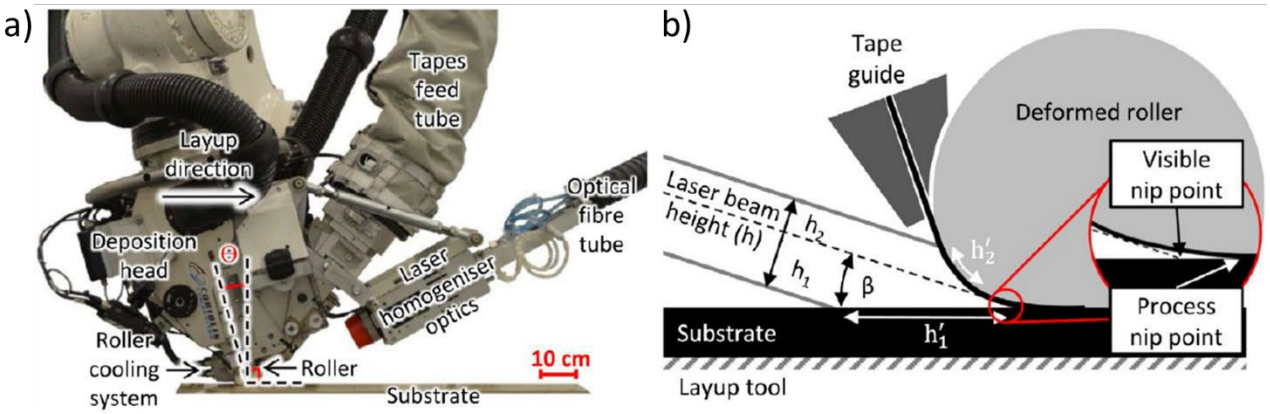


Figure 3 a) CORIOLIS Automated Fiber Placement (AFP) robotic machine in thermoplastic configuration b) Definition of layup parameters [13]

3.2. Test panels

Commercially available slit tapes (Tencate Cetex TC1200 PEEK AS4, 145 gsm, 34% RC, ¼ inch [18]) materials were used in this study. The physical properties are given in Table 1.

Material	Fiber	Fiber density (g/cm^3)	Matrix	Matrix density (g/cm^3)	Melting temp (°C)	Glass transition temp (°C)	Degradation temp (°C)	Areal fiber weight (g/m^2)	Resin content by weight (%)
Cetex TC1200	AS4	1.79	PEEK	To be determined	343	143	575	146	34

Table 1 Material properties

Test panels of 250x250mm² were laid up by AFP with a quasi-isotropic laminate sequence $[45/90/135/0]_{3S}$.

The settings applied to the manufacturing robot are described in Figure 3.b) and provided in Table 2. The compaction stress applied by the roller on the laminate is calculated as follows:

$$\sigma = \frac{F}{A} \tag{1}$$

where σ is the compaction stress, F is the compaction force exerted by the head and A the deformed roller area in contact with the substrate.

Panel	Process temp Tp (°C)	Tool temp Tt (°C)	Beam position h1/h2	Head tilt θ deg	Layup velocity V (m/s)	Compaction stress under roller C (MPa)	Laser power P (W)
Autoclave	450	RT	4	-7	0.05	0.73	1700
Insitu_Heated Tool		140					1725
Insitu_cold Tool		RT					1700

Table 2 Process parameters

One panel has been consolidated under nitrogen using an Olmar autoclave. The consolidation cycle is given in Figure 4.

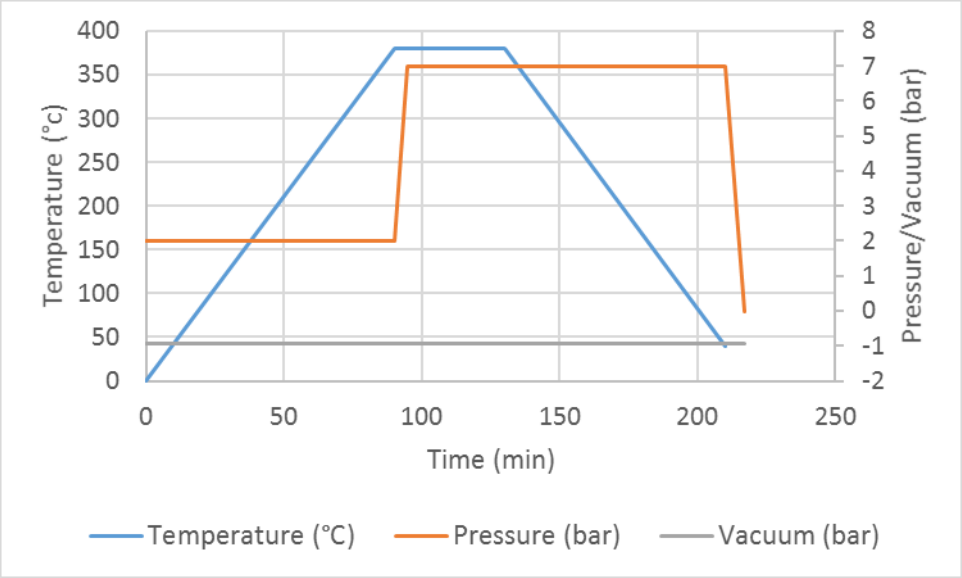


Figure 4 Autoclave cycle for C/PEEK consolidation

3.3. Characterization methods

3.3.1. Temperature measurement during layup

Process temperatures during layup are measured using an infra-red camera Optris PI4000 with an acquisition frequency of 120 Hz. The measured temperature is an average of the temperature values located at the nipoint (Figure 5). Material emissivity is supposed to be equal to 1 as it is supposed that all the wave reflections are absorbed by the material [7]. This measurement technique is very sensitive to surface defects and material quality (recorded temperature variations can reach up to 80°C at a constant laser power), hence the assumption of a material emissivity equal to 1 is reasonable.

The measured temperature is the average of the values measured on a length equal to around 6 tows and located at the nipoint. Considering the limitation given above, the layup temperature is given with an error equal to ±25°C.

Temperature is a function of laser power which is adjusted to reach the desired processing temperature (Table 2).

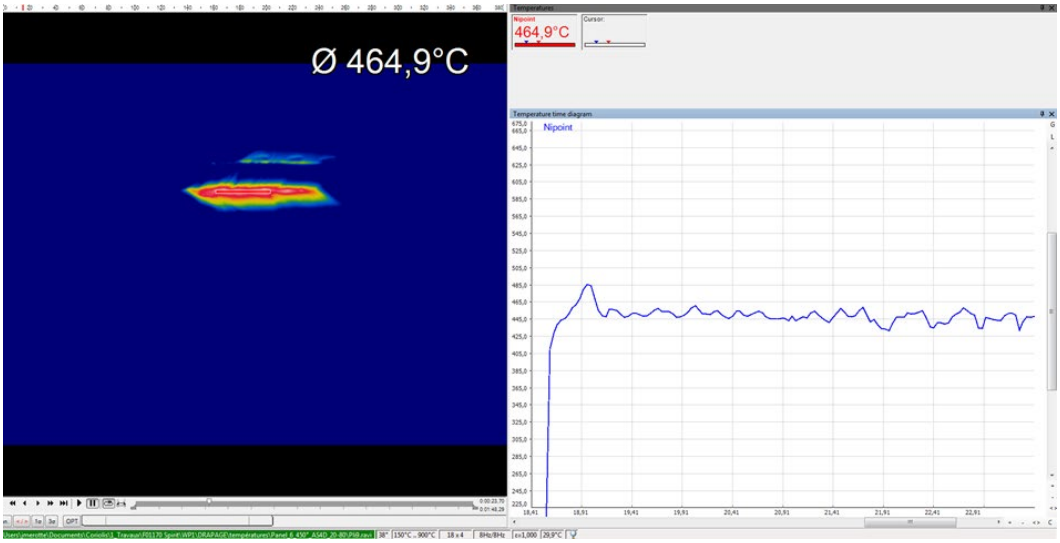


Figure 5 Temperature measurement using IR camera: recording during insitu layup

3.3.2.Matrix enthalpy measurements

The degree of crystallinity is evaluated for each material. First, melting enthalpy ΔH_f, cold crystallization enthalpy ΔH_{cc} have been measured using DSC Mettler Toledo 822^e. Samples of 7 to 20 mg are submitted to a heating cycle of 20°C/min from 20 to 400°C. From the first heating measurement curves (heat/cooling), glass transition temperature, melting temperature, cold crystallization enthalpy and melting enthalpy are determined (Figure 6). From the cooling measurement curve, crystallization temperature is determined.

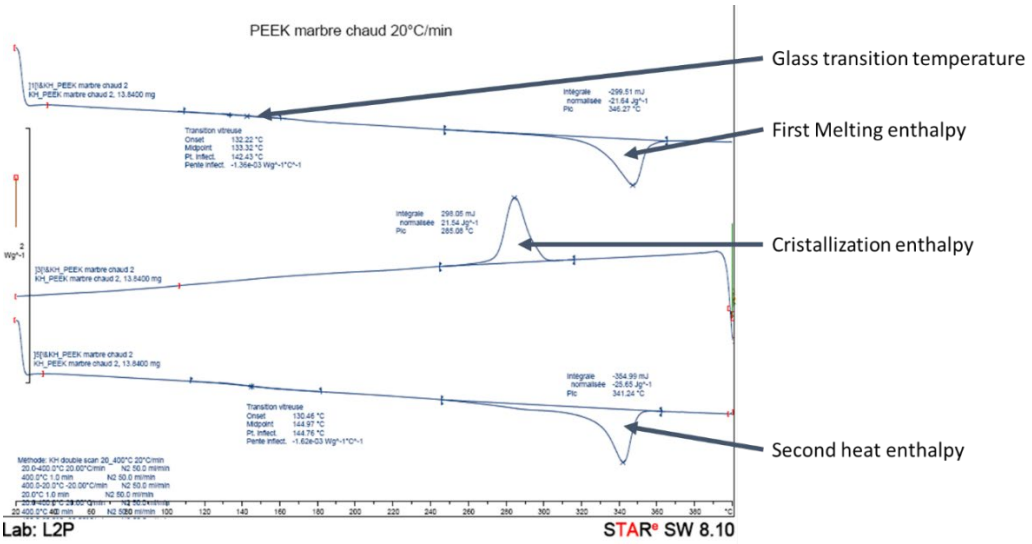


Figure 6 Typical DSC curve of PEEK material

The exact fiber mass fraction of the sample needs to be measured to calculate degree of crystallinity from DSC measurements.

3.3.3. Fiber volume fraction and matrix residual ashes

TGA Mettler Toledo ATG/DSC2 is used to calculate fiber volume fraction of the sample previously used in the DSC. Samples are submitted to a decomposition cycle of 10°C/min ranging from 20 to 900°C. The measurement is done under inert atmosphere with nitrogen. By this test, matrix is degraded, allowing to calculate residual weight represented by fiber and matrix residual ashes.

Residual ashes *ra* (constituted by carbon molecules) present in the PEEK matrix are evaluated by TGA by entirely degrading the raw matrix. The remaining weight represents the residual ashes and will be used in the calculation of matrix degree of crystallinity.

The residual ashes content is calculated as follows:

$$ra_{PEEK} = \left(\frac{m_{res}}{m_{ini}}\right) \tag{2}$$

Where m_{ini} and m_{res} are the initial and residual weight of the matrix sample submitted to TGA.

This leads to the following matrix mass fraction calculation:

$$w_{PEEK} = 1 - \left(\frac{\frac{m_{cini} - m_{cres}}{ra_{PEEK}}}{m_{cini}}\right) \tag{3}$$

Where m_{cini} and m_{cres} are the initial and residual weight of the composite sample.

3.3.4. Matrix crystallinity calculation

From enthalpy, fiber volume fraction and residual ashes measurements, the degree of matrix crystallinity of each laminate is calculated by the following equation:

$$x_{c\,red} = \frac{\frac{\Delta H_f - \Delta H_{cc}}{w_{PEEK}}}{\Delta H_f^*} \quad (4)$$

With x_{cred} the degree of crystallinity, r_{PEEK} the matrix weight ratio and ΔH_f^* the enthalpy of 100% crystalline PEEK (taken as 130J/g [19]).

3.3.5. Density and porosity measurements

Sample density measurements were performed using the immersion method according to the ISO1183 standard where the sample is weighted in the air and in an immersion liquid (ethanol). 10x10mm² samples are cut from the center of the panel in order to avoid manufacturing uncertainties due to fiber feeding and fiber cutting.

3.3.6. Cross section microscopy

As porosity content is not sufficient to fully describe the microstructure of the composite, cross section analysis of each sample is then performed. To do so, squared sample of 10x10mm² are cut in the middle of the panel, embedded into epoxy matrix and cured for 2h at 50°C. The sample is then polished using particle diameters down to 3µm.

Cross sections are then analyzed using an Olympus BS51 microscope coupled with an Olympus DP 26 camera and using the Olympus Stream Basic images processing software.

For plies where the cutting direction is similar to the fiber direction, fiber can be pulled out from the matrix during polishing. This results in apparent porosities that will not be considered as they were not present in the laminate at the beginning.

3.3.7. Interlaminar shrear strength characterization

Interlaminar shear strength were conducted on the autoclave consolidated sample and in-situ laid up on heated tool. The tests were performed according to the ISO14130 standard with an Instron E10000 mechanical testing machine equipped with a 10kN load cell. 5 samples of 30x15x3mm^3 were tested at 1mm/min speed. Testing span was 15mm.

4. Results

4.1. Fiber mass fraction and crystallinity

Autoclave consolidation allows to accurately control the cooling rate, thanks to multiple sensors in the oven and a very slow manufacturing process.

Inversely, even at slow layup speed, heating and cooling happen very quickly during AFP layup. It is also very difficult to experimentally assess the temperatures and cooling speed during the process as the localized applied energy is very high. Hence, in-situ consolidation makes it very difficult to control cooling rate, it is then mandatory to investigate the degree of crystallinity of the matrix of in-situ panels.

It is possible to modify the cooling rate by increasing the substrate temperature. Laying up on a heated tool set just below glass transition temperature (around 140°C), allows to decrease the cooling rate and to favorize crystallization.

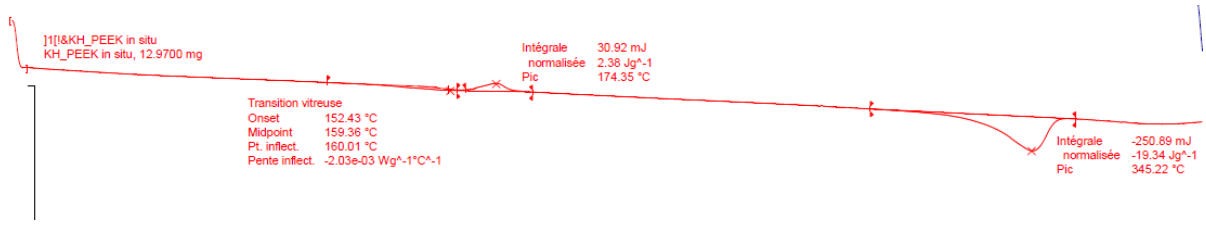


Figure 7DSC curve for the first heat phase of the in-situ cold tool composite

TGA and DSC measurements for plain matrix and the different composites are given in the table below:

	Initial weight	Final weight after TGA	Residual ashes	Fiber mass fraction	Cold crystallization enthalpy	Melting enthalpy	Crystallinity content
	mg	mg			(J/g)	(J/g)	
Raw PEEK	57.36	28.76	0.4986				
Raw Tape	9.64			0.66 (technical data sheet)	8.17	18.01	0.23
Autoclaved composite	98.87	82.64		0.6709	0	23.84	0.56
In-situ cold tool	89.84	72.25		0.6743	2.38	19.34	0.40
In-situ heated tool	93.54	78.57		0.6790		21.54	0.52

Table 3 TGA and DSC measurements for the different samples of the study

Table 3, presents the degrees of crystallinity derived from the melting enthalpy and reduced to the matrix content. The measured values are higher than those found in the literature [20], [21] which are usually located at around 40% on pure polymers. Fibers are known as initiators of transcrystallinity which modifies significantly the structure of the spherulites in the matrix. This could explain the high degrees of crystallinity found in the materials. Moreover, crystallinity over the thickness of the laminate is heterogeneous. Indeed, due to successive cold crystallizations happening during the deposition of plies above a given one, laminate plies keep crystallizing during the entire layup process. This makes very sensitive the sampling of the laminate for DSC/TGA measurements. For these reasons, further work needs to be performed to validate these measured values.

However, one can notice that AFP allows for an increase of crystallinity from the initial tape. Moreover, one can notice that laying up on a heated tool helps to improve the crystallinity of the sample. This is however, still not possible to reach crystallinity level found in autoclave consolidation.

Mehmet Alkan [20], [22] and Giants [21] showed that the degree of crystallinity of the matrix has a significant impact on the matrix density.

4.2. Composite Microstructure

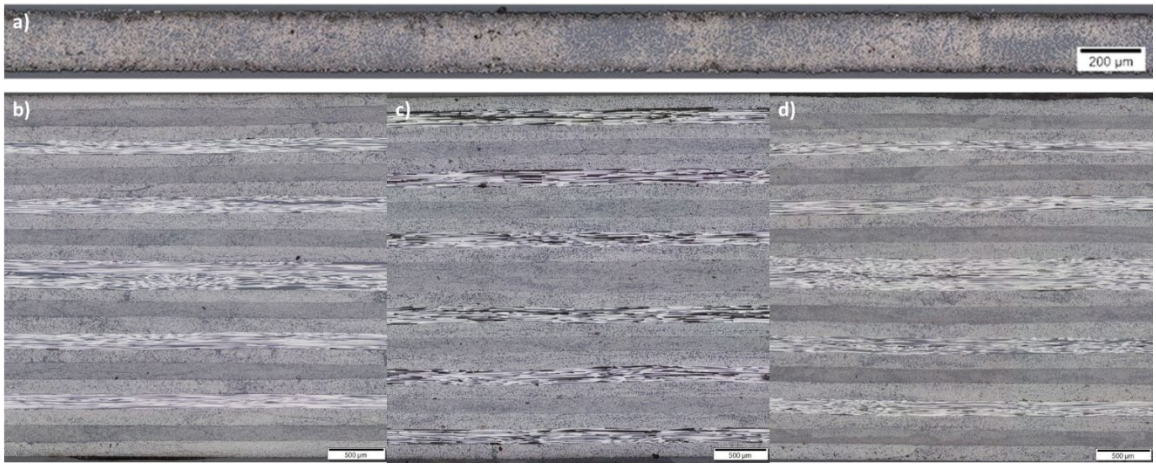


Figure 8 Micrographic cross sections of the laminates a) raw material b) laminates consolidated by autoclave c) laminate laidup on a cold mold d) laminate laidup on heated mold

Figure 8 shows composite cross sections of the raw tape (Figure 8.a), composite consolidated by autoclave (Figure 8.b), in-situ consolidated on a cold tool (Figure 8.c) and in-situ consolidated on a heated tool (Figure 8.d). From the observation of Figure 8.b), one can assume that the autoclave consolidated composite exhibits great consolidation with very little porosities. It is then fair to assume that the density of this sample can be considered as the reference density of the bulk composite.

Density measurements on raw tape, autoclaved composite and composite laid up on cold and heated tool, have been performed and are presented in Figure 9.a). In-situ consolidated panels exhibit higher density values than raw tapes. This means that the AFP process allows to improve the consolidation of

the initial tape. However, this improvement is still limited, and in-situ consolidation quality is very dependent on the quality of the initial tape.

From density measurement, porosity content can be derived using the following equation

$$\emptyset_c = 1 - \rho_c \left(\frac{1 - w_f}{\rho_m} + \frac{w_m}{\rho_f} \right) \tag{5}$$

Where $\emptyset_c$ is the porosity content, ρ_c, ρ_m, ρ_f the densities of the composite, the matrix and the fibers respectively and w_f the fiber weight ratio. From the literature, crystalline PEEK density is evaluated to be equal to 1.31 g/cm^3 and amorphous to be 1.27 g/cm^3. Considering the results presented in Table 3, porosity measurements are an evaluation as the polymeric state of the matrix is unknown (between amorphous and crystalline). Error bars correspond to the porosity values for these two extreme matrix states.

Taking into account the previous comment, porosity contents are calculated from density (Figure 9.a), measurements. From Figure 9.b), it is obvious that in-situ consolidation cannot provide a similar consolidation quality as autoclave. However, it is possible to reach porosity values lower than 2% with in-situ consolidated panels laid up on a heated tool.

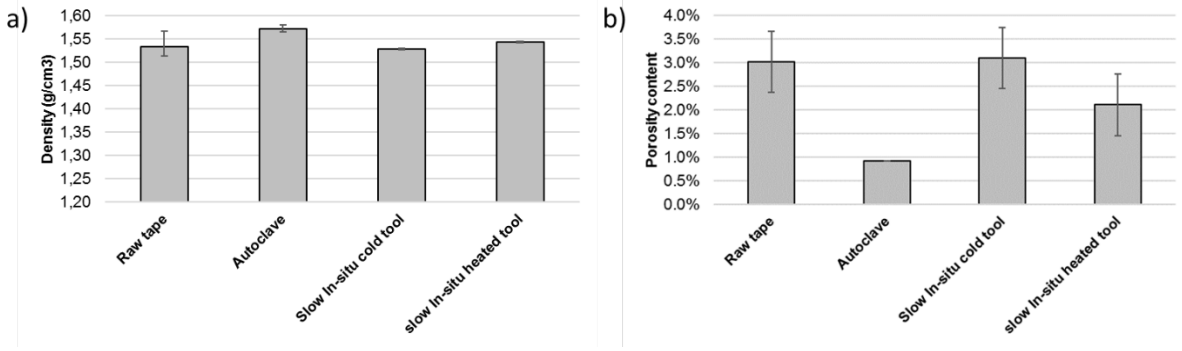


Figure 9 a) Density measurements and b) calculated porosity contents for the different samples

The micrographic observations confirm density measurements where only little porosity is found on in-situ consolidated panels. One can also notice a very good and homogeneous distribution of the fibers in the raw tape but also on the panels laid up by AFP.

4.3. Inter laminar shear strength

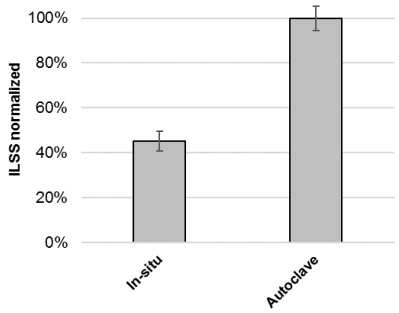


Figure 10 Inter-laminar shear strength (ILSS) of In-situ and autoclave consolidated laminates

Figure 10 presents inter laminar shear strength of laminates submitted to short beam bending tests. Despite a very good interphase between plies, in-situ laminates show much lower ILSS values than autoclave consolidated laminates. This low value can be explained by an incomplete bonding between the plies. Perfect plies contact was performed during layup but very limited reptation of the molecular chains took place during in-situ layup. Tierney and Gillespie [8] stated that the reptation time of PEEK under a temperature of 400°C is 0.11s. Interlaminar temperature decreases very quickly while the fed tows are compacted by the roller, temperature goes from 450°C to a temperature slightly above substrate temperature (Figure 11) in a couple of milliseconds [23]. Hence, the time above melting temperature, where reptation can occur [24], is not sufficient for it to happen (yellow color on Figure 11). While submitted to interlaminar shear stress, the laminate having low molecular chains entenglement exhibits early delamination behavior. To let the reptation phenomenon occurring, it is then necessary to layup at a slower velocity to increase the duration over melting temperature (green color on Figure 11). This would increase the time where the polymer is exposed above melting temperature.

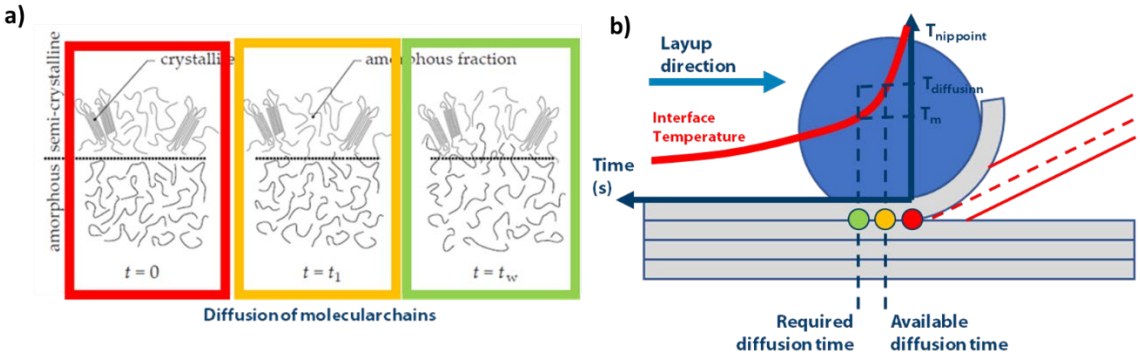


Figure 11 Phenomenon of temperature decrease during in-situ layup [24]

5. Conclusions:

For the past few years, machine and aircraft manufacturers as well as research centers have conducted tremendous work to demonstrate the great potential of thermoplastic composite materials for the aerospace industry. Moreover, AFP processes have demonstrated their ability to manufacture any kind

of parts, from large fuselage barrels to small complex parts. One of its great advantage is the very low buy to fly obtained thanks to the discretization of the surface into tow widths. This process also demonstrated its compatibility with historical consolidation process like autoclave but also emerging ones like vacuum oven consolidation or stamping. For the latest, it is even possible to predict the fiber motion during stamping and anticipate the location of fibers to avoid molding defects.

For AFP machine manufacturers, the holy grail consists of providing a solution permitting to reach in-situ consolidation. This would allow to save costs due to post consolidation processes. This necessitate an accurate control of the different manufacturing parameters. Layup temperature, compaction and speed allow to reach good adhesion between the plies, leading to very limited porosity contents. Layup temperature and speed and tool temperature allow to vary matrix crystallinity, leading to acceptable values (≈40%). However, interlaminar shear strength testing highlighted limited bonding of the different plies with an ILSS value two times lower than the corresponding autoclave composite. This low value can be explained by the very limited time of matrix exposure above melting temperature. Even at reduced speed, cooling rate is very large, leading to low molecular chains entanglement at the interphase. This issue could be solved by reducing the speed and increasing the temperature slightly below the degradation temperature. This involves an accurate control of the laser power to always preserve the material from degradation. Developments ongoing within Coriolis will lead in the near future to an innovative control approach for the heat source where not only the speed of the machine will be taken into account but also the orientation of the ply below, the stack already laid up and the geometry of part being manufactured.

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