An investigation of in-situ AFP process parameters using CF/LM-PAEK

Ines Schiel , Lukas Raps , Ashley R. Chadwick , Isabelle Schmidt , Manuel Simone & Sebastian Nowotny

Published online: 08 Oct 2020.

Submit your article to this journal ↗

View related articles ↗

View Crossmark data ↗

Taylor & Francis
Taylor & Francis Group

An investigation of in-situ AFP process parameters using CF/LM-PAEK

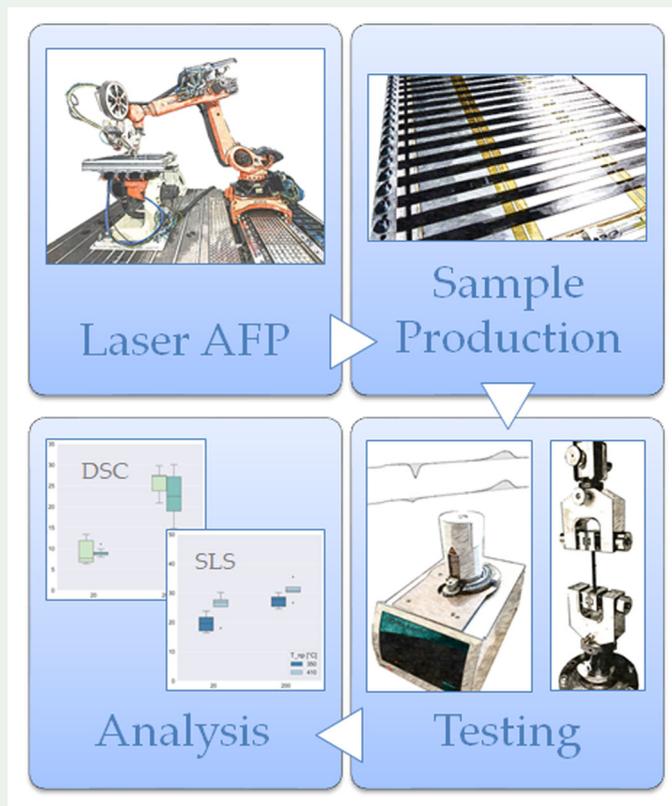Ines Schiel, Lukas Raps, Ashley R. Chadwick, Isabelle Schmidt, Manuel Simone and Sebastian Nowotny

Institute of Structures and Design, German Aerospace Center (DLR), Stuttgart, Germany

ABSTRACT

In recent years the use of thermoplastics has become popular in aerospace applications, with a primary focus on fiber-reinforced composites. Displaying greatly improved mechanical properties, new components using these materials still need to be characterized and their suitability for aviation applications demonstrated. A common restriction to the implementation of fiber-reinforced thermoplastic parts is the almost default autoclave manufacturing, which is both time consuming and expensive. Aiming for a more economical final product, this study utilizes a one-step in-situ Automated Fiber Placement (AFP) process to produce samples for mechanical and thermal characterization. The recently developed and highly popular material CF/LM-PAEK was used within this study, with the four major AFP processing parameters varied to assess material sensitivity. Test samples were manufactured using Design of Experiment (DoE). Subsequently, single lap shear (SLS) and differential scanning calorimetry (DSC) tests were performed to assess consolidation quality. With rising tooling temperature, both SLS strength and crystallinity increase up to 31 MPa and 25%, respectively. A post-manufacturing tempering process improved crystallinity of the tested CF/LM-PAEK specimens up to 29% and SLS strength up to 38 MPa. Within the tested parameter range, CF/LM-PAEK appeared to be unaffected by increasing layup speed, which is a promising aspect with regard to faster industrial production.

CONTACT Ines Schiel ines.schiel@dlr.de Institute of Structures and Design, German Aerospace Center (DLR), Pfaffenwaldring 38-40, Stuttgart 70569, Germany

1. Introduction

Aircraft structures have the requirements to be light, strong and reliable. To improve these structures, newly developed materials as well as fiber composites are under investigation. In addition, new and advanced manufacturing processes lead to a more economical production. Figure 1 depicts technologies which are studied at the German Aerospace Center's Institute for Structures and Design and can be used for manufacturing a thermoplastic fuselage. The fuselage skin is manufactured by means of the Automated Fiber Placement (AFP) Technology. The same process also enables the integration of local reinforcements. Stringers and frames are attached to the skin by ultrasonic and resistance welding, respectively.

Understanding of the tape laying process is the basic requirement for a future manufacturing of a fuselage skin. This paper deals with a new fiber-reinforced thermoplastic material with respect to the AFP process and the essential manufacturing parameters.

2. Materials and methods

2.1. AFP facility at DLR

The manufacturing of all specimens took place in the tape laying facility at the DLR in Stuttgart which is pictured in Figure 2. The used setup consists of a Multi Tape Laying Head (MTLH) developed by the company Automated Fiber Placement Technology (AFPT) mounted on a six degree-of-freedom robotic arm. The MTLH enables the placement of up to three 0.5 in unidirectional (UD) prepreg tapes or single tapes of varying width between 0.5 in and 1.5 in. The tapes are laid down on a two degree-of-freedom heatable planar metallic tooling surface. The layup speed can be increased to up to 15 m/min. The energy for heating up the tapes is provided by a 6 kW diode laser ($\lambda = 1000$ nm).

2.2. Fiber-reinforced thermoplastic material

The material examined in this study is a unidirectional thermoplastic prepreg tape consisting of standard modulus carbon fibers and a newly developed matrix material. This matrix material is a copolymer pertaining to the poly-aryl-ether-ketone (PAEK) family. It is called Low-Melt PAEK (LM-PAEK) and has been designed to exhibit a lower bulk melting temperature and melt viscosity than the standard PEEK material. The properties of the prepreg tape are listed in Table 1.

2.3. Critical process parameters

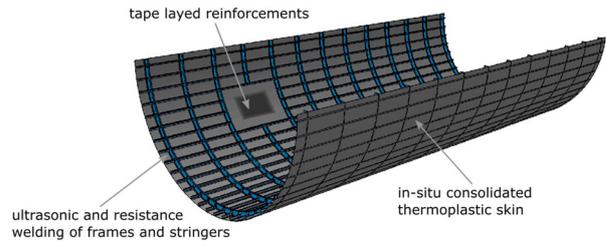The in-situ consolidation process as core aspect of AFP is illustrated in Figure 3. The incoming tape

Figure 1. Technologies from DLR's Institute of Structures and Design (BT) in a thermoplastic fuselage.

Figure 2. DLR's thermoplastic AFP facility in Stuttgart, Germany.

Table 1. Material properties of unidirectional CF/LM-PAEK tape [1].

Property	CF/LM-PAEK
Prepreg	Cetex TC 1225
Matrix	LM PAEK
Fiber	T700
Fiber mass fraction (w_f) (%)	66
Tape width (mm)	12.7 ($= 0.5$ in)
Manufacturer	Toray Advanced Composites
Melt temperature (T_m) (°C)	305
Glass transition temperature (T_g) (°C)	147
Crystallization enthalpy (H_{ref}) (J/g)	130

is directed toward the tooling surface. The laser beam heats up the tape and the tooling surface (or the substrate of already deposited tapes), respectively. The compaction roller presses the new tape onto the substrate. The temperature at the nip point, the point in the fusion zone under the compaction roller, is measured by an IR camera and is used as an input value for the control of the laser power.

The critical process parameters [2,3] pressure p, velocity v, tooling temperature T_{tool} and nip point temperature T_{np} are displayed in Figure 3. The investigated value ranges of these parameters are listed in Table 2. All specimens were produced with a pressure of 6 bar. Layup speeds of 7.5 and 15 m/min were investigated. The nip point temperature was set to 350 or 410 °C. The tooling was either kept at room temperature or heated to a tooling temperature of 200 °C. Furthermore the influence of a follow up tempering process was investigated. The specimens were kept in an oven at 290 °C for about 17 h.
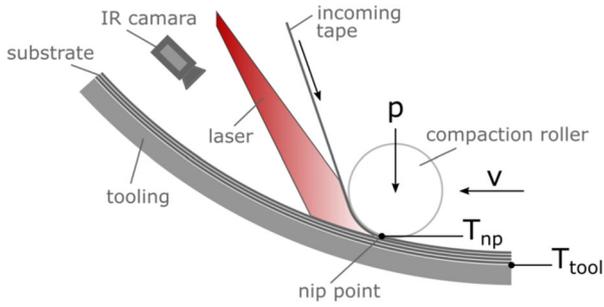
Figure 3. AFP process.

Table 2. Variation of the critical process parameter values in this study.

Parameter	Values	
p (bar)	6	
v (m/min)	7.5	15
T_{np} (°C)	350	410
T_{tool} (°C)	Unheated	200
T_{temper} (°C)	Untempered	290[a]

[a] Tempered for 17 h.

2.4. Characterization methods

Both thermal and mechanical properties of the material were investigated. Thermal properties were measured *via* DSC and used to determine the crystallinity. The interlaminar bonding strength was determined through a specifically developed single lap shear test which is described below.

2.4.1. Differential scanning calorimetry (DSC)

In order to determine the crystallinity, measurements were performed with a NETZSCH DSC 214 *Polyma* device. In the process aluminum crucibles were used. Nitrogen was chosen as purging and protective gas. Specimens were cut out with pliers and had on average weights of 6.7 mg. The respective sample and a reference were heated up to 350 °C with a heating rate of 10 °C/min, held isothermal for three minutes, and cooled down with a rate of 10 °C/min. Data evaluation was performed by using the NETZSCH Proteus® Software. With the determined heat flow differences between sample and reference (ΔH_m and ΔH_c), the degree of crystallinity X_c can be calculated with the following formula [4]:

$$X_c = \frac{|\Delta H_m| - |\Delta H_c|}{\Delta H_{ref}(1 - w_f)} \; [\%] \tag{1}$$

under consideration of the tape material properties given in Table 1.

2.4.2. Single lap shear (SLS) test

The method used in this paper to evaluate the interlaminar bonding strength was developed at the DLR Institute of Structures and Design in Stuttgart and is described in [2]. The procedure assesses the consolidation and bonding of the polymer matrix under shear conditions. For this purpose specimens with
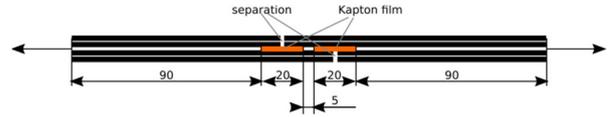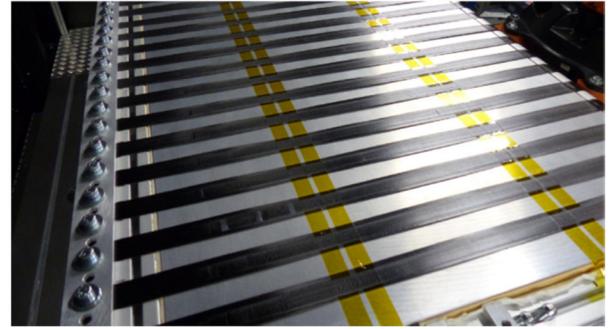

Figure 4. SLS sample geometry [2].

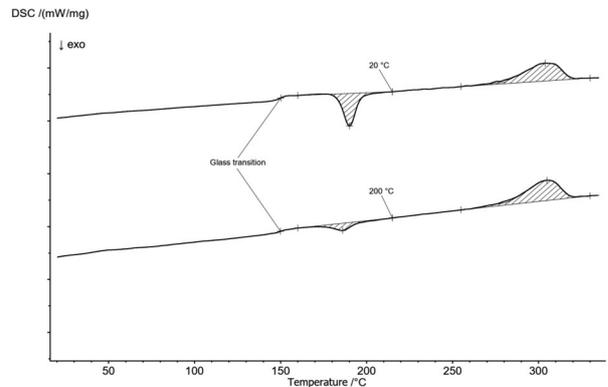Figure 5. Exemplary specimen production at DLR's AFP facility.

Figure 6. DSC measurements of AFP-produced samples. Top: unheated tool (20 °C), Bottom: 200 °C ($v = 7.5$ m/min, $T_{np} = 410$ °C, $p = 6$ bar, untempered).

the geometry displayed in Figure 4 were produced as shown in the image in Figure 5. Two layers of individual tracks of CF/LM-PAEK were placed on the tooling surface. Polyimide adhesive tape (Kapton film) with a thickness of 60 µm was subsequently placed in two 5-mm-apart double courses perpendicular over the stripes. After that, two more layers were placed on the first two tracks. To evaluate the bonding region the tapes were cut through on opposite lying sides. The shear force thus takes effect in the predefined region in between the Kapton films. To obtain tensile-shear values, the samples were clamped in a ZwickRoell 200 kN machine and were tensile tested until failure at a rate of 1 mm/min. The data was recorded with the testing software testXpert II.

3. Results

In the following sections the impact of the varying process parameters on the resulting crystallinity and single lap shear strength (SLS strength) values are
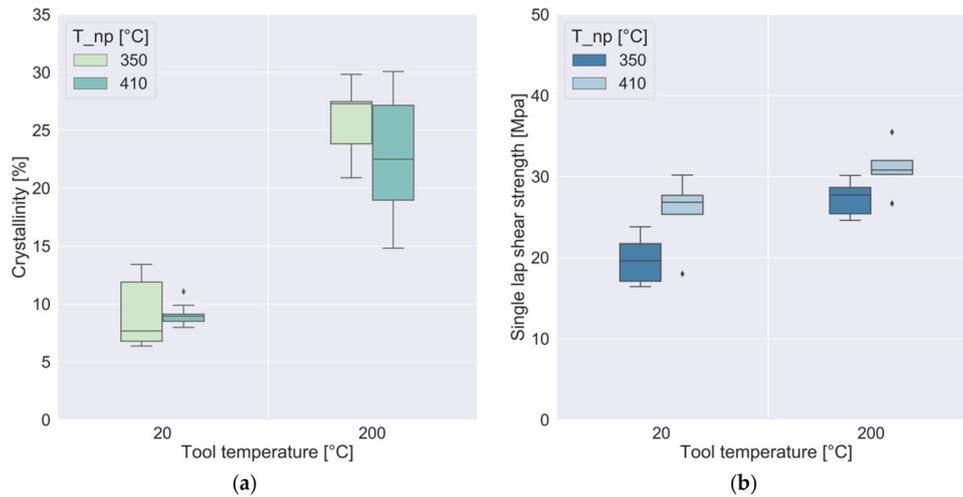
Figure 7. Crystallinity (a) and SLS strength (b) over tooling temperature ($v = 7.5$ m/min, $p = 6$ bar, untempered).

Table 3. Statistical values of crystallinity and SLS strength ($v = 7.5$ m/min, $p = 6$ bar, untempered).

T_{tool} (°C)	T_{np} (°C)	Median	Standard deviation
	Crystallinity (%)		
20	350	7.67	2.66
20	410	8.95	0.87
200	350	27.29	2.80
200	410	22.50	5.05
	Single lap shear strength (MPa)		
20	350	19.63	2.75
20	410	26.81	4.11
200	350	27.71	2.03
200	410	30.79	2.84

presented and compared. The median degree of crystallinity, deviation and outliers are displayed within box plots. Affiliated medians and standard deviation are reported in the related tables. SLS strength values are plotted and documented accordingly.

3.1. Effect of heated tooling

As a result of DSC measurements two entropy curves are displayed in Figure 6. The upper curve shows the test results of a specimen, produced with an unheated tooling. In comparison, the lower curve depicts the results of a specimen produced with a tooling temperature of 200 °C.

Both curves show a characteristic step at around 150 °C, marking the glass transition region. In the temperature range of 170 °C to 200 °C the cold crystallization takes place. The exothermic peak is more distinct for the unheated tooling configuration. The endothermic peaks above temperatures of 250 °C characterize the crystalline melting. Melting takes place at around 305 °C. Linear baselines were chosen for the area calculations. For comparability, the limits of 160–215 °C for the cold crystallization and 255–330 °C for the melting crystallization respectively were applied to all evaluations within this study.

Figure 7a points out an increase in crystallinity with rising tooling temperature. An increase of tooling temperature from 20 °C (unheated) to 200 °C yields proportional gains in crystallinity of 256% and 152% for 350 °C and 410 °C nip point temperature, respectively. A 41% ($T_{np} = 350$ °C) and a 15% ($T_{np} = 410$ °C) increase in single lap shear strength can be determined from Figure 7b.

The underlying statistical values of Figure 7 can be taken from Table 3. The statistic values are calculated based on nine DSC values and six SLS values per data set. The standard deviations vary. The maximum crystallinity standard deviation is 5.05%.

3.2. Effect of layup speed

The layup speed is a key parameter for consolidation, since it defines the time that the tape spends in the nip point area and under the compaction roller.

Figure 8 shows that both crystallinity (a) and single lap shear strength (b) remain at the same level with rising layup speed from 7.5 to 15 m/min.

Table 4 lists the statistical values of the parameter set pictured in Figure 8.

3.3. Effect of tempering

A set of specimens, produced with an unheated tooling, was subjected to a subsequent process step of tempering in an oven at a temperature of 290 °C for a duration of 17 h. Figure 9 shows a noticeable increase in both crystallinity (a) and single lap shear strength (b) after the tempering process. Proportional gains in crystallinity of 285% ($T_{np} = 350$ °C) and 219% ($T_{np} = 410$ °C) are achieved. The single lap shear strength values increase by 76% ($T_{np} = 350$ °C) and 45% ($T_{np} = 410$ °C), respectively.

The statistical values belonging to Figure 9 are listed in Table 5. The maximum single lap shear strength standard deviation is 6.19 MPa.

Table 4. Statistical values of crystallinity and SLS strength (unheated tooling, $p = 6$ bar, untempered).

Layup speed [m/min]	T_{np} (°C)	Median	Standard deviation
Crystallinity (%)			
7.5	350	7.67	2.66
7.5	410	8.95	0.87
15	350	8.89	0.75
15	410	9.40	0.96
Single lap shear strength (MPa)			
7.5	350	19.63	2.75
7.5	410	26.81	4.11
15	350	26.05	1.96
15	410	25.87	1.65

Table 5. Statistical values of crystallinity and SLS strength ($p = 6$ bar, $v = 7.5$ m/min, unheated tooling).

Tempering	T_{np} (°C)	Median	Standard deviation
Crystallinity (%)			
0	350	7.67	2.66
0	410	8.95	0.87
1	350	29.55	3.84
1	410	28.53	1.63
Single lap shear strength (MPa)			
0	350	19.63	2.75
0	410	26.81	4.11
1	350	34.49	6.19
1	410	38.84	6.11
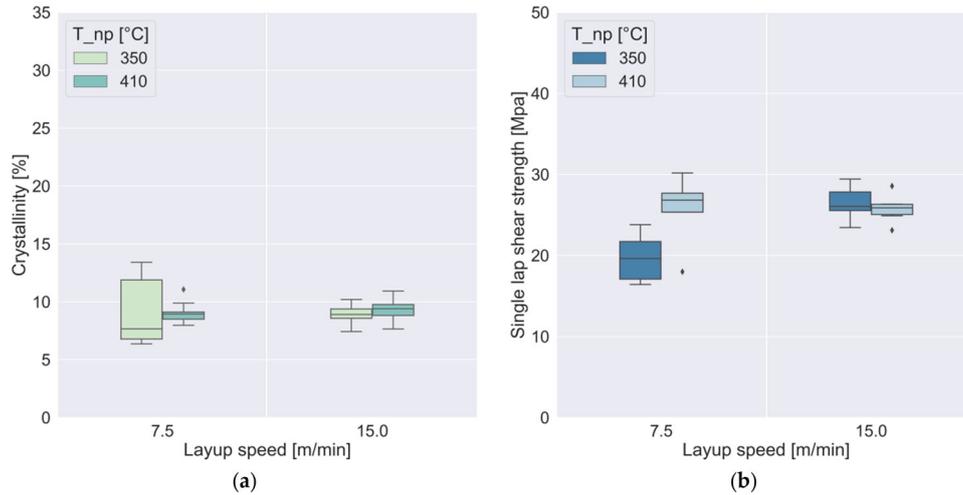
Figure 8. Crystallinity (a) and SLS strength (b) over layup speed (unheated tooling, $p = 6$ bar, untempered).

4. Discussion

The single lap shear strength and crystallinity values presented in the figures above show a somewhat synchronic behavior with respect to the varying parameters. As was to be expected, higher crystallinity values are accompanied by higher SLS strength values.

The tape laying process with unheated tooling results in a rapid cool down and because of the lack of time for crystallization the polymer stays partly amorphous [5]. This amorphous phase fraction crystallizes within the cold crystallization. Therefore the exothermic peak in Figure 6 is very distinct for the unheated tooling configuration. A higher tooling temperature promotes a lower cooling rate resulting in less amorphous areas and a less distinct exothermic peak.

The results from Figure 7 show that a heated tooling enhances both crystallinity and single lap shear strength. This makes sense considering that the polymer is less amorphous at a lower cooling rate. The comparatively low crystallinity values of the unheated tooling configuration around 8.5% contribute to small distortion and low internal stresses.

Looking at Figure 8, the bonding strength and crystallinity of CF/LM-PAEK appear to be unaffected by increased layup speed within the tested parameter range. In contrast, investigations of other standard thermoplastic materials e.g. CF/PPS and CF/PEEK showed sensitivity to the layup speed [2,3]. The strength values decreased with increasing layup speeds, thus low layup speeds are preferred. The insensitivity of CF/LM-PAEK to the layup speed is an advantage considering tape placement of big structures like aircraft fuselages since higher layup speeds can shorten production time and thus reduce manufacturing costs.

The additional process step of tempering increased both crystallinity and single lap shear strength of CF/LM-PAEK significantly (Figure 9). The polymer was fully crystallized after that additional process step. The tempering process raises the SLS strength of CF/LM-PAEK up to 38 MPa and the degree of crystallinity up to 29%.

First comparisons with other at hand standard materials as CF/PEEK and CF/PPS, which were investigated at the same facility in Stuttgart within the scope of prior research work [2], resulted in lower single lap shear strength values of the CF/LM-PAEK specimens. Due to differing process parameters and a lower fiber volume fraction of CF/PEEK and CF/PPS (55%) compared to 60% for CF/LM-PAEK the comparability is insufficient and needs to be investigated deeper in further studies.

5. Conclusions and outlook

The suitability of the new material CF/LM-PEAK for the tape laying process was investigated in this paper.
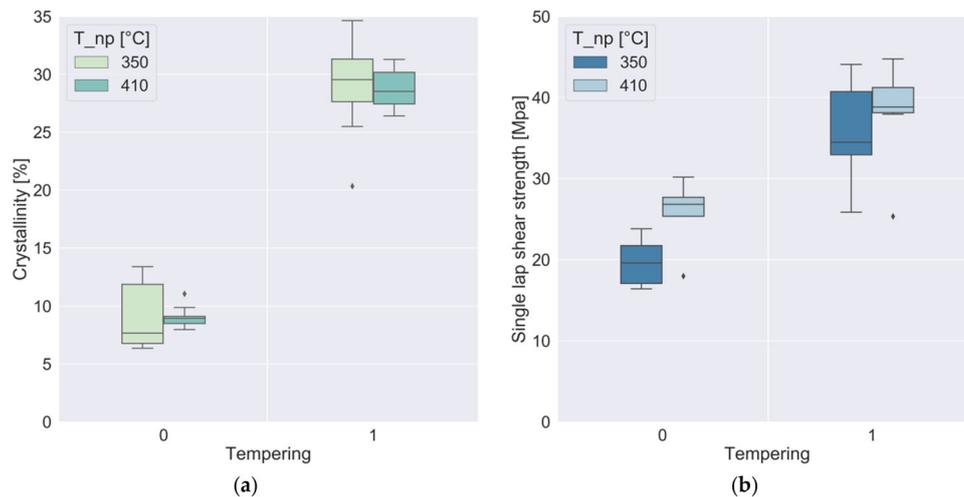
Figure 9. Crystallinity (a) and SLS strength (b) over layup speed ($p = 6$ bar, $v = 7.5$ m/min, unheated tooling).

For this purpose a range of specimens was produced using varying process parameters. It was demonstrated, that good consolidation can be achieved in the tape laying process with CF/LM-PAEK. Mechanical and thermal material properties were identified by means of single lap shear tests and differential scanning calorimetry. The correlation of crystallinity and single lap shear strength was pointed out. An increase in SLS strength and crystallinity was obtained with $200\,°C$ tooling temperature and also by a post manufacturing tempering process. Hence both these thermal treatments are conceivable to optimize the process. Its insensitivity to layup speed makes CF/LM-PAEK a promising material for AFP-production of large-scale components. For enhanced material properties, a tempering post process step can be added. Though the major advantage of in-situ consolidation, which is the one step AFP process, would be omitted, a tempering process in an oven with no need for a vacuum build-up is still favorable compared to an even more cost-intensive autoclave consolidation. The most cost-efficient process constellation might be fast AFP on a cold tooling with a free standing post cure process to increase crystallinity and SLS strength.

The impact of the fiber volume fraction of the tape material on interlaminar bonding of in-situ AFP-produced laminates needs to be investigated. The tradeoff between a higher fiber content and thus higher specific strength of the composite and a higher proportion of matrix, which might contribute to an increased interlaminar bonding, needs to be quantified. This will then also enable an adequate comparison with other prepreg materials.

The limit of the layup speed needs to be explored in subsequent studies. Investigations on the varying standard deviations are ongoing. Furthermore the behavior of CF/LM-PAEK throughout laminate production and the resulting laminate properties will be topics of interest in the upcoming studies.

Author contributions

Conceptualization, Ashley R. Chadwick; formal analysis, Isabelle Schmidt, Lukas Raps and Ines Schiel; investigation, Manuel Simone; data curation, Lukas Raps and Ines Schiel; writing – original draft preparation, Ines Schiel; writing – review and editing, Lukas Raps and Ashley R. Chadwick; supervision, Sebastian Nowotny; project administration, Sebastian Nowotny, Ashley R. Chadwick; funding acquisition, Sebastian Nowotny. All authors have read and agreed to the published version of the manuscript.

Disclaimer statement

The results, opinions, conclusions, etc. presented in this work are those of the author(s) only and do not necessarily represent the position of the JU; the JU is not responsible for any use made of the information contained herein.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Funding

References

[1] Toray Advanced Composites. Processing guidelines for TC1225 T700/PAEK UD tape. Data sheet. Internal document provided by Toray to DLR, 2019.

[2] Dreher PN, Chadwick AR, Nowotny S. Optimization of in-situ thermoplastic automated fiber placement process parameters through DoE. SAMPE Europe, Nantes, France, 2019.

[3] Khan MA, Mitschang P, Schledjewski R. Parametric study on processing parameters and resulting part quality through thermoplastic tape placement process. J Compos Mater. 2013; 47(4):485–499.

[4] Velisaris CN, Seferis JC. Crystallization kinetics of polyetheretherketone (peek) matrices. Polym Eng Sci. 1986;26(22):1574–1581.

[5] Frigione M, Naddeo C, Acierno D. Crystallization behavior and mechanical properties of poly(aryl ether ether ketone)/poly(ether imide) blends. Polym Eng Sci. 1996;36(16):2119–2128.