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Influence of the cooling behaviour on mechanical properties of carbon fibre-reinforced thermoplastic/metal laminates

Camilo Zopp[1)], Daisy Nestler[1)], Nadine Buschner[1)], Carola Mende[1)], Sven Mauersberger[3)], Jürgen Tröltzsch[1)], Sebastian Nendel[2)], Wolfgang Nendel[1)], Lothar Kroll[1)], Michael Gehde[3)]

1) Department of Lightweight Structures and Polymer Technology, camilo.zopp@mb.tu-chemnitz.de, daisy.nestler@mb.tu-chemnitz.de, nadine.buschner@mb.tu-chemnitz.de, carola.mende@mb.tu-chemnitz.de, juergen.troeltzsch@mb.tu-chemnitz.de, wolfgang.nendel@hrz.tu-chemnitz.de, lothar.kroll@mb.tu-chemnitz.de, Technische Universität Chemnitz, Reichenhainer Straße 31/33, 09126 Chemnitz, Germany

2) Cetex Institute of Textile- and Processing Machines non-profit GmbH, nendel@cetex.de, Altchemnitzer Straße 11, 09120 Chemnitz, Germany

3) Department of Conveyer Technology and Polymers, sven.mauersberger@mb.tu-chemnitz.de, michael.gehde@mb.tu-chemnitz.de, Technische Universität Chemnitz, Reichenhainer Straße 70, 09126 Chemnitz, Germany

Keywords

Thermoplastic Hybrid Laminates, Thermoplastic CFRP, Mechanical Properties, Cooling Rate

Abstract

For several years, thermoplastic hybrid laminates form a new class in the field of material compounds. These laminates consist of fibre-reinforced plastic prepregs and metal layers in alternating order. Compared to conventional thermosetting multilayer composites, these laminates are suitable for large-scale production and can be manufactured with significantly reduced cycle times in the thermoforming process.

In the framework of this contribution, the influence of the cooling rate of carbon fibre-reinforced thermoplastic composites and hybrid laminates was investigated with regard to crystallinity and the resulting mechanical properties. Polyamide 6 and thermoplastic polyurethane as matrix systems were examined, in particular.

Additionally, the differential scanning calorimetry was used in order to investigate the influence of the cooling rate on the crystallisation behaviour. It could be determined that the cooling rate has a limited influence on the crystallisation of polyamide 6 and this influences the mechanical properties. Furthermore, a reliance of process parameters on the characteristics profile of composite materials and material compounds with thermoplastic polyurethane could be identified. Depending on process conditions, tensile, bending, and interlaminar shear properties fluctuate up to 20 % in fibre-reinforced laminates and up to 32 % in hybrid laminates. Moderate to fast cooling rates result in optimum mechanical characteristics of tensile properties in fibre-plastic-compounds. Fast to very fast cooling rates are advisable for bending and interlaminar shear properties. Highest tensile and bending characteristics are achieved in hybrid laminates by using fast to very fast cooling rates, while interlaminar shear properties tend to be highest in slow to moderate cooling rates.

1 Introduction

Hybrid laminates consist of alternately layered fibre-reinforced plastic layers and thin metal sheets. These “Advanced Engineering Materials” provide a high degree of lightweight potential, especially where large volumes have to be moved.

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Thermosetting hybrid laminates or so-called fibre-metal-laminates are known from the state-of-the-art and are used primarily in aerospace industry, for example in fuselage and wing areas [1, 2]. Best-known representatives are GLARE (GLAss fibre-REinforced epoxy/aluminium laminate), ARALL (ARamid fibre-reinforced epoxy/ALuminium Laminate), and CARALL (CARbon fibre-reinforced epoxy/ALuminium Laminate). These laminates were investigated extensively with regard to their process conditions [3–5]. These material compounds are not suitable for large-scale production due to their thermoset resin system and the curing time. Furthermore, subsequent forming after the production process is not possible. For these reasons, thermoplastic-based hybrid laminates are in the focus of research and development.

Hybrid laminates with fibre-reinforced thermoplastic layers offer significant advantages compared to the conventional material connections. The production takes place without a time consuming curing process and comparatively short cycle times in the thermoforming process. As a result, the economic efficiency is considerably increased. Additionally, thermoplastic material compounds have an excellent damage tolerance and simple repair options similar to monolithic metal alloys [6, 7]. Thermoplastic-based hybrid laminates with glass fibre-reinforced and self-fibre-reinforced polypropylene (PP) are widely used [8, 9]. Glass fibre-reinforced polyamide (PA) multilayer composites are also established [6, 10, 11]. Publications on carbon fibre-reinforced PA prepregs and aluminium alloy sheets are issued under CAPAAL (CArbon fibre-reinforced PolyAmide/ALuminium laminate) [12–16]. New developments show investigations on the material compound CATPUAL (CArbon fibre-reinforced Thermoplastic PolyUrethane/ALuminium laminate) [17].

It is known that mechanical characteristics of fibre-reinforced plastics (FRP) can be influenced by processing conditions when semi-crystalline plastics are used [18, 19]. The processing speed plays an important role as it is related to the prevalent plant technology and the heating and cooling rates involved. For example, the interface shearing strength in glass fibre-reinforced PP and carbon fibre-reinforced polycarbonate (PC) could be increased with increased cooling speed [18, 20]. On the other hand, [18, 21, 22, 23] show a contradictory behaviour or no significant influence of the cooling rate in carbon fibre-reinforced PP, polyether sulfone (PES), or polyethylene terephthalate (PET). Further investigations, aside from PP and PA, were conducted on semi-crystalline polyetheretherketone (PEEK), thermoplastic vulcanizates (TPV) such as EPDM/PP, and amorphous polyetherimide (PEI) [24–26]. A fast cooling rate in bidirectional fibre reinforcement increased residual stresses resulting from the higher degree of crystallisation, as tests on PEEK showed [6]. Furthermore, strength and stiffness properties, impact behaviour, and fatigue characteristics can be significantly increased with a rising degree of crystallisation [7]. The interlaminar shear strength shows an analogous behaviour [18]. Additionally, a higher cooling speed is supposed to result in an improvement of impregnation behaviour, higher ductility, higher fracture energy and lower residual stresses in the matrix as well as a separation of molecular weights [18].

The mechanical characteristics profile of thermoplastic-based multilayer compounds can be influenced by variation of process parameters similar to fibre-reinforced plastics. In the framework of this contribution, the focus is laid on the cooling phase as it influences the crystallisation of plastics and thus affects the resulting mechanical properties [8, 18, 27, 28]. Further changes occur due to process temperature and pressure, matrix morphology, surface structure of the fibre, residual stresses, elasticity modulus of fibre and matrix as well as reactive functionalities [18].

The objective of the paper is to investigate the mechanical properties of carbon fibre-reinforced composite materials based of PA 6 and thermoplastic polyurethane (TPU) and the corresponding material compounds CAPAAL and CATPUAL subject to cooling rates. The matrix systems used are characterised by their semi-crystalline (PA 6) and non-crystalline (TPU) appearance and have excellent mechanical properties. In this context, the matrices are predestined for FRP [29, 30].

2 Experimental procedure

2.1 Materials and manufacturing process

The production of the multi-functional thermoplastic semi-finished products (Ce-Preg®) takes place on the Fibre-Foil-Tape Unit (FFTU). Its principle is based on the film stacking process. The plant technology is described in detail in [17, 29]. Polymer films (PF) with PA 6 (Cast-PA 6, Co. mf-Folien) or TPU (Dureflex X2311, Co. Covestro AG) with a film strength of 50 µm were used. The fibre component used was a 50 K HT- carbon fibre (Panex 35, Co. Zoltek) with a thermoplastic-based sizing system. The glass fibre-reinforced plastic interlayers (GFRP) were made from the E-glass fibre (TufRov 4588, Co. PPG). The Ce-Preg® semi-finished products have a thickness of 0.25 mm. Fibre volume content (FVC) is 53 % in the CF-PA 6 tapes and 58 % in the CF-TPU tapes. The glass fibre-reinforcement interlayers have a FVC of 49 %. The determination of the fibre volume content was performed by thermogravimetric analysis, according to DIN EN ISO 11358.

The carbon fibre-reinforced laminates consist of 5 and 11 layers of Ce-Preg® tapes. Thus, unidirectional fibre-reinforced laminates have a thickness of approx. 1 and 2 mm, respectively. Tensile, bending, and shear samples were taken from these laminates. In the multilayer compounds, the aluminium alloy (Al) EN AW 6082-T4 with a thickness of 0.5 mm was used as a metal layer. Two interlayers of polymer film as well as one layer of glass fibre-reinforced tapes were added in order to prevent contact corrosion of carbon fibre and metal alloy. The core layer of the hybrid laminate is comprised of 4 layers of carbon fibre-reinforced plastic (CFRP). In the framework of the study, a 2/1 lay-up structure [Al/2xPF/GFRP/4xCFRP/GFRP/2xPF/Al] was examined. Fibre reinforcement occurred unidirectionally. The measured thickness of the hybrid laminates was approx. 2 mm after the pressing process.

Fibre-reinforced laminates as well as hybrid multilayer compounds were manufactured in a Collin Press Type P 300 P. These were subjected to a surface modification before consolidation. The mechanical surface treatment proved appropriate due to its positive effects on the surface roughness, surface structure and the additional removal of surface contamination [13, 17]. The tool with an effective press area of 260 x 260 mm² was mutually heated by heating cartridges. Heat transmission took place through heat conduction to the core of the laminate. A temperature of 280 °C (PA 6) and 250 °C (TPU) was attained in the core of the laminate. Temperatures were measured with the temperature-measuring instrument Testo 735-2 (Co. Testo). Pressing pressure was 1.2 MPa in FRP and 1.5 MPa in hybrid laminates. Heating and cooling took place in the Collin Press or rather in the same tool. The plates were removed from the tool at a temperature of 80 °C and further cooled down at room temperature. The complete process time was between 23 and 36 min, subject to the cooling rate. Fig. 1 shows the qualitative process procedure of variants a), b), c) and d).

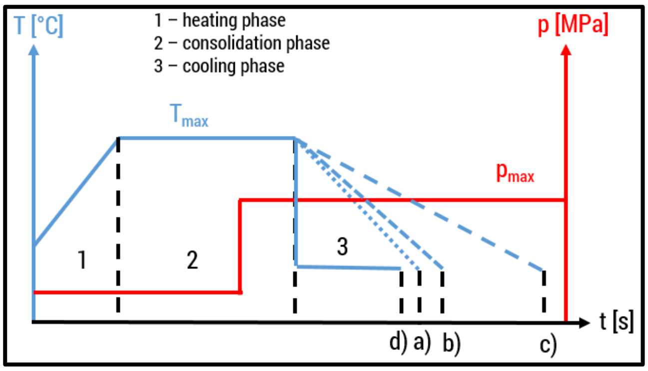

Figure 1: Process cycle of variant a) fast, b) moderate, c) slow cooling and d) very fast

Typical heating and cooling rates for example of glass fibre-reinforced PA 6 at approx. 3–6 K/min (in autoclave) [31]. In this study, a cooling rate between 0 and 30 K/min was chosen to increase the economy. Pressure and temperature profiles were kept constant. It is noted that variant d) equals a maximum cooling rate. Heating and cooling rates were equally used for the fibre-reinforced and correlating hybrid laminates. Tab. 1 summarises the heating and cooling rates of the manufactured systems.

Table 1: Heating and cooling rates of the manufactured FRP laminates and hybrid laminates

Variant	Heating rate [K/min]	Cooling rate [K/min]
a)	20	30
b)	20	20
c)	20	10
d)	20	0

2.2 Determination of crystallinity and investigation of microstructures

The degree of crystallinity describes the percentage of crystalline areas in semi-crystalline thermoplastics. Typical values for crystalline areas in PA 6 are 25–40 %, thus it is considered a kind of low-crystalline thermoplastics [28].

The influence of varying cooling rates on the crystallisation behaviour of the pure thermoplastic films were analysed using differential scanning calorimetry (DSC 204 F1 Phoenix, Co. Netzsch) and the degree of crystallinity was determined. For this purpose, a specimen with 6.5 mg was weighed in a 40 µl aluminium crucible and grouted with a perforated aluminium lid. Each specimen was heated in a nitrogen atmosphere with a volume flow of 20 mL/min for 20 K/min to its melting point, subsequently cooled down to 25 °C with a cooling rate of 2, 5, 10, 20, and 30 K/min. The heating rate was 20 K/min a second time. A double determination was carried out for each investigated cooling rate. In order to assess the influence of the respective cooling rate, the second heating was used for the evaluation and the degree of crystallinity (H_{mc}) determined using the following equation [6, 18]:

$$H_{mc} = \frac{H_m - H_c}{H_f(1-\alpha)} \tag{1}$$

H_m = Enthalpy of melt heat (experimental)

H_c = Enthalpy of crystallisation (experimental)

H_f = Enthalpy at full crystallisation (theoretical)

α = Fibre volume content of the FRP

The morphology of the crystallisation was characterised using polarised light filters in the optical microscope BX 51 with the digital camera DP 71 from Co. Olympus. In this context, microscopic thin sections were produced.

2.3 Investigation of mechanical properties

Mechanical tests regarding heating and cooling rate were conducted following DIN EN ISO 527-5 for tensile specimen, DIN EN ISO 14125 for bending specimen and DIN EN ISO 14130 for interlaminar shear strength (ILSS) specimen on FRP and respective hybrid laminates. For the statistical tests, 8 specimen were cut out of one sheet and tested in direction of the fibres. The characteristic values were determined on a Zwick/Roell Z100 (tension) and Zwick/Roell 5.0 (bending and ILSS). Test speed was set to 2 mm/min (tension), 5 mm/min (bending), and 1 mm/min (ILSS).

3 Results

3.1 Crystallinity and morphology

Fig. 2 shows the thermograms from the DSC measurements for each example, respectively. The first heating (20 K/min), cooling (10 K/min) and the second heating (20 K/min) of the appropriate unreinforced PA 6 and TPU films are shown.

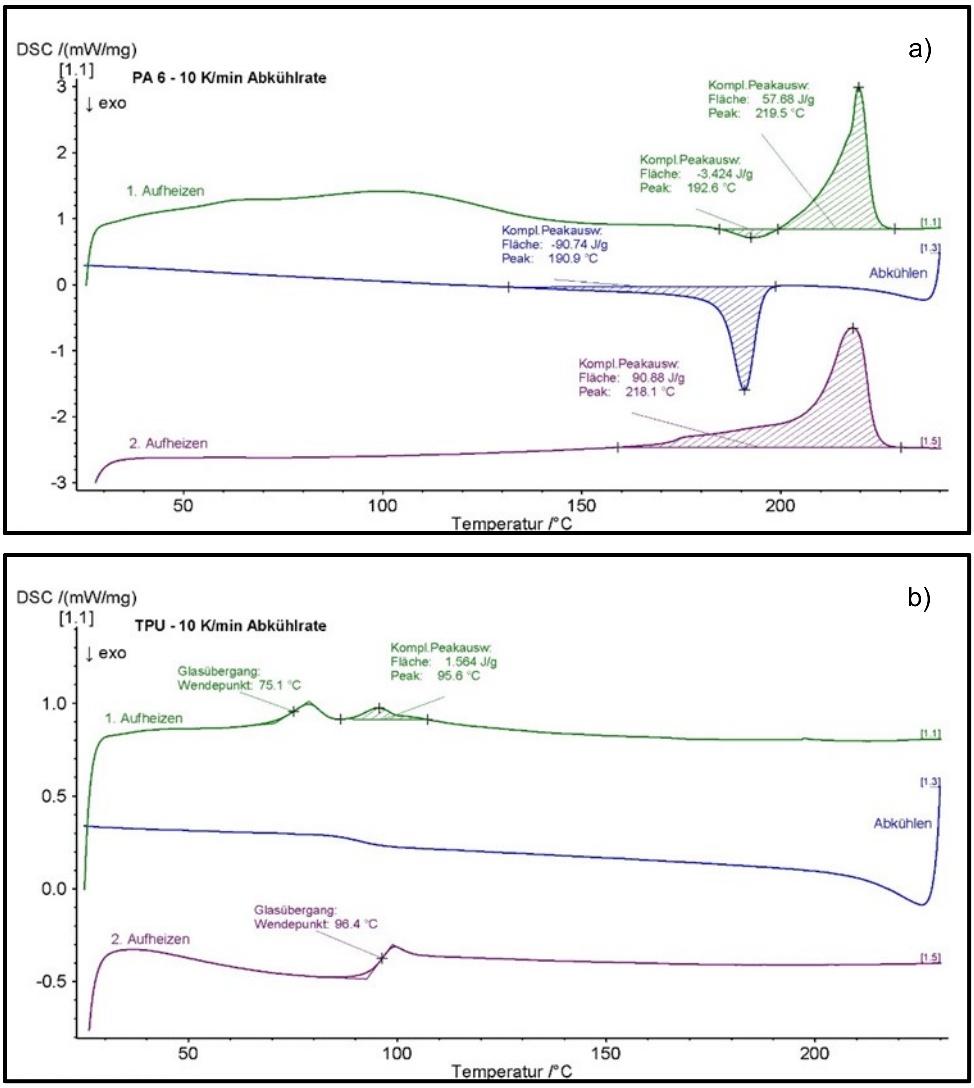

Figure 2: DSC thermogram for a) unreinforced PA 6 and b) unreinforced TPU with a cooling rate of 10 K/min

The thermogram of PA 6 (Fig. 2a) shows the escape of moisture at a wide endothermic peak of around 100 °C in the first heating. At 193 °C, the specimen shows an exothermic post-crystallisation that is subsequently followed by an endothermic melting of the crystallites at over 200 °C. During cooling the sample from the melt an exothermic solidification of the plastic occurs below 200 °C. The degree of crystallinity was determined from the peak area of the endothermic crystalline melting range in the second heating curve of the specimen. In PA 6, the enthalpy in full crystallisation was assumed at 230 J/g [32].

In the case of the TPU film (Fig. 2b), a glass transition temperature was detected in the first heating at 76 °C. The endothermic peaks in the 100 °C range can be explained with the typical TPU characteristic additivation. This TPU specimen is an amorphous thermoplastic plastic and no melting of crystalline areas or crystallisation during cooling can be observed. The degree of crystallisation cannot be determined. Thus, no dependencies can be determined for the investigated TPU film. The following results refer to the investigation of PA 6. Fig. 3 shows a summarised overview of the influence of cooling rates on the degree of crystallisation in the examined PA 6. A dependency between the degree of crystallisation and the cooling rate can be determined by means of DSC investigations on the PA 6 film in the second heating. A 45 % degree of crystallisation was determined at a cooling rate of 2 K/min and at a cooling rate of 30 K/min the lowest degree of crystallisation of 36 %, was established.

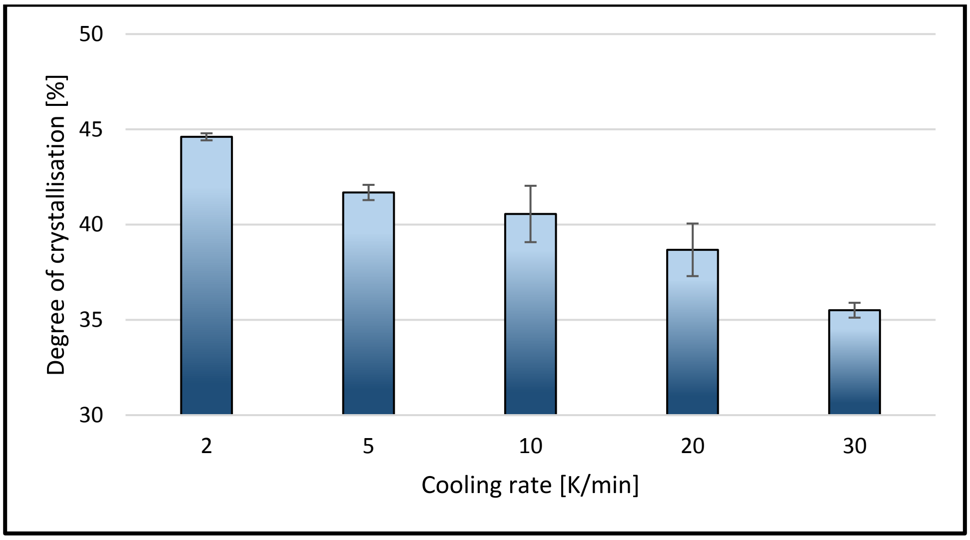

Figure 3: Influence of the cooling rates on the degree of crystallisation of unreinforced PA 6

The microstructural analysis for the determination of crystallinity is summarised in Fig. 4. As an instance carbon fibre-reinforced PA 6 and carbon fibre-reinforced TPU laminates, respectively, with the heating and cooling variant a) are shown. Thin section preparations with a thickness of 25 µm were examined in the polarised transmitted light. It is visible, that Fig. 4 developed a fine spherulitic structure which has optically typical material characteristics of PA 6. Fig. 4b) shows the matrix with no visible superstructures. Bright areas can be ascribed to process-related impacts (i.e. scratches).

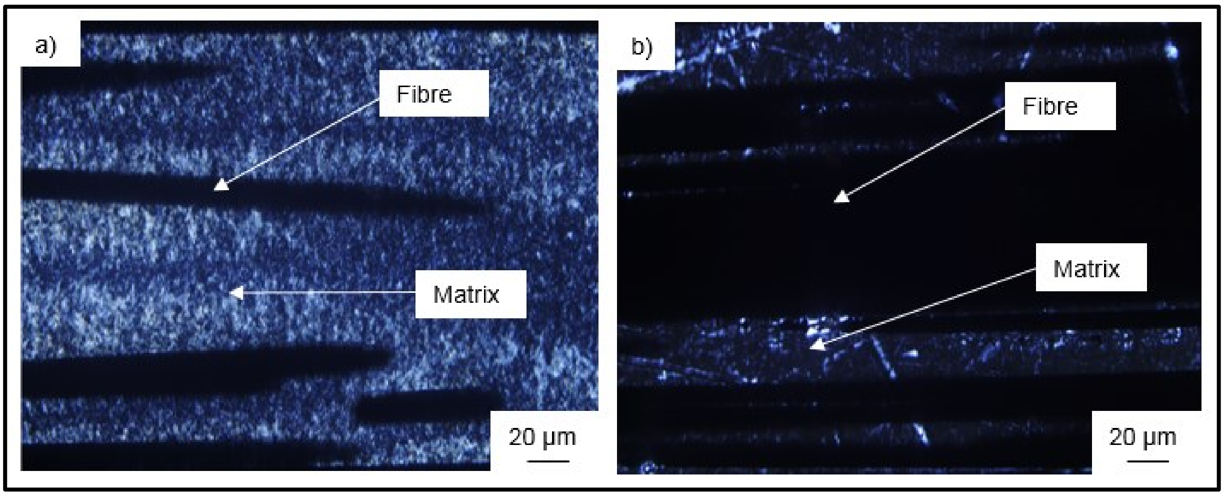

Figure 4: Thin section preparations at 20 K/min heating and cooling rate a) FRP with PA 6 b) FRP with TPU

3.2 Impregnation

The results of the impregnation are displayed as examples of selected FRP (1 mm) and hybrid laminates (2 mm) with a very fast cooling rate (0 K/min) in Fig. 5. A complete impregnation without pores of the carbon fibre-reinforced PA 6 as well as of the carbon fibre-reinforced TPU takes place. The multilayer composites CAPAAL and CATPUAL show an equivalent behaviour.

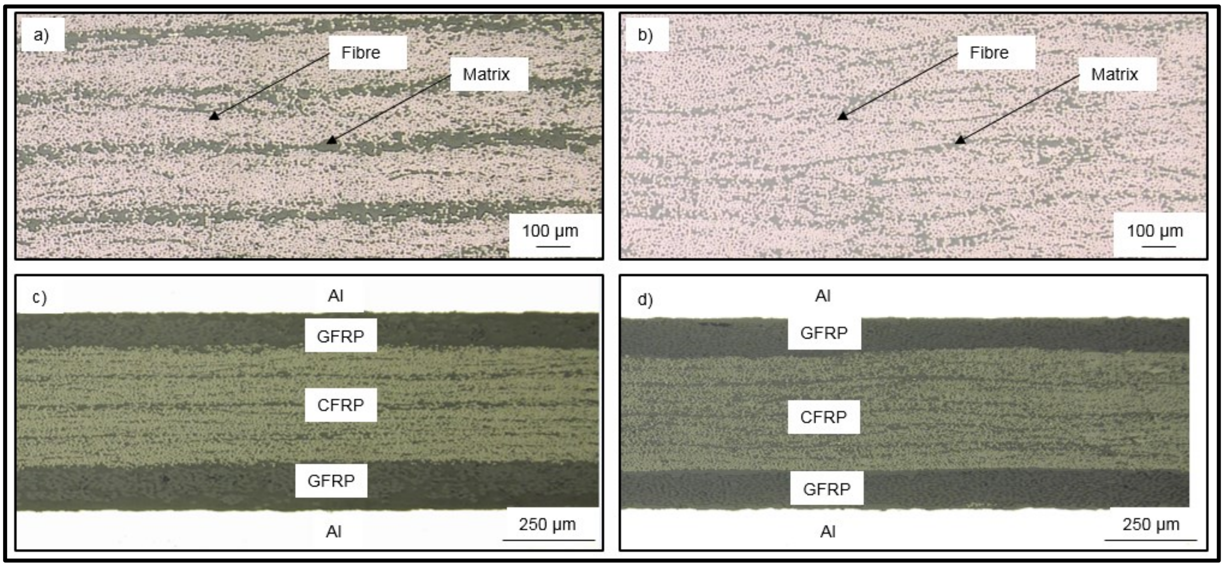

Figure 5: Microscopic investigations of the impregnation behaviour of a) CF-PA 6, b) CF-TPU, c) CAPAAL and d) CATPUAL

3.3 Mechanical properties

The mechanical characteristics of tensile modulus of elasticity, tensile strength, bending modulus of elasticity, bending strength, and interlaminar shear strength are summarised in Fig. 6, 7, and 8. The FRP specimen were conditioned according to DIN EN ISO 291 before testing. No conditioning was performed in the hybrid laminates as the aluminium alloy functions as a moisture barrier [3]. All characteristics were determined in longitudinal direction of fibre.

Fig. 6 shows, that the interlaminar shear strength in CF-PA 6 composites varies by up to 11 %. The highest characteristic of approx. 68 MPa could be achieved in a slow cooling rate. The characteristics between variant a) and c) are almost on the same level in the fibre-reinforced TPU laminates. A significant increase is visible under the influence of very fast cooling rate (71 MPa). Compared to the FRP laminates, the interlaminar shear strengths in hybrid laminates vary widely. The highest interlaminar shear strength can be achieved in the process cycle c) in the material compound CAPAAL equivalent to the FRP. A faster cooling rate results in a loss of strength. The interlaminar shear strengths in CATPUAL are all approximately on the same level except for variant c).

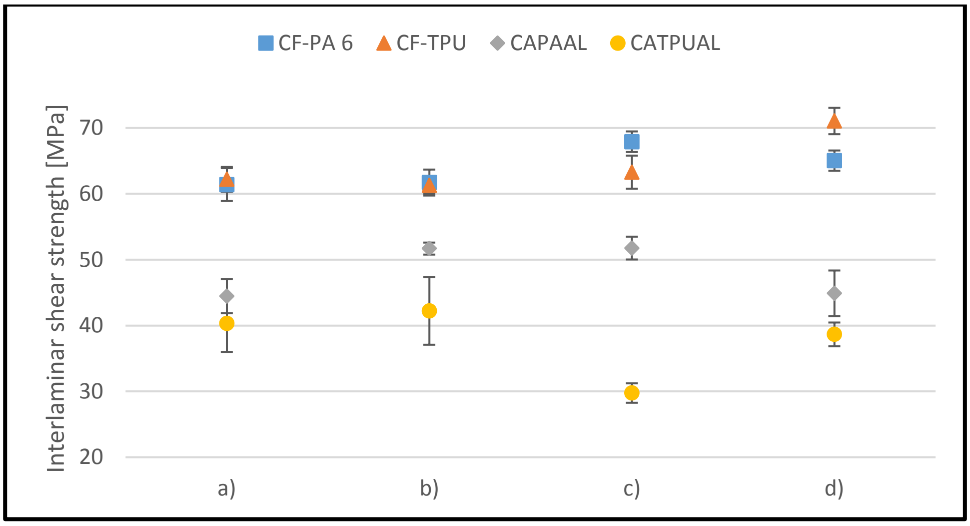

Figure 6: Interlaminar shear strengths of CF-PA 6 and CF-TPU as well as the hybrid laminates CAPAAL and CATPUAL

Fig. 7 shows that the bending modulus of elasticity only varies of around 3 % in the FRP with PA 6. Equivalent behaviour is visible in the carbon fibre-reinforced TPU composites. The hybrid laminate CAPAAL shows a variation of 15 % in dependency on the cooling behaviour. A significant increase with regard to the bending modulus of elasticity is observed in a very high cooling rate (variant c)). However, stiffness values in the material compound CATPUAL are approximately on the same level. The highest bending stiffness is achieved with about 60 GPa. The highest characteristics with regard to bending strength in FRP with PA 6 and TPU can be set at a very fast cooling range. The mechanical properties vary compared to reference variant a) between 8 % (CF-PA 6) and 10 % (CF-TPU). This trend can be transferred to the bending strengths of the material compounds.

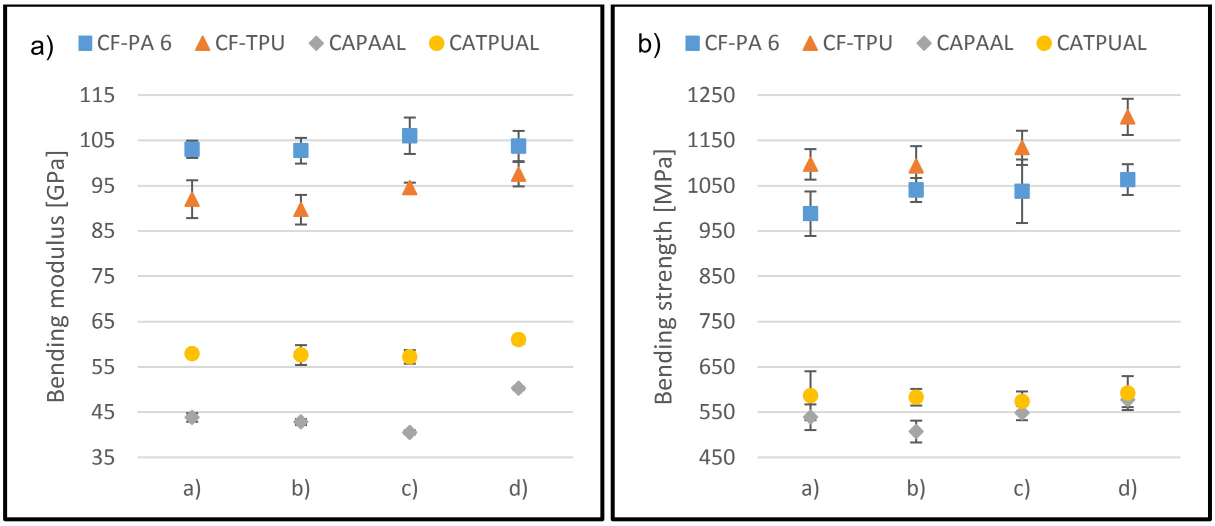

Figure 7: a) Bending modulus of elasticity and b) bending strength of the CF-PA6 and CF-TPU as well as the hybrid laminates CAPAAL and CATPUAL

The characteristics of the tensile modulus of elasticity and the tensile strength are summarised in Fig. 8. The highest characteristics in regard to tensile modulus of elasticity in CF-PA 6 and CF-TPU are noted in cooling variant b). Analogous to the bending properties CATPUAL exceeds the properties of CAPAAL. The highest characteristics are at approximately 70 GPa. The bending strength of the CF-PA 6 composites varies between 1370 MPa and 1665 MPa. In the case of the carbon fibre-reinforced TPU composites, variant c) shows a difference. This can be ascribed to measurement inaccuracies. CATPUAL shows an equivalent behaviour.

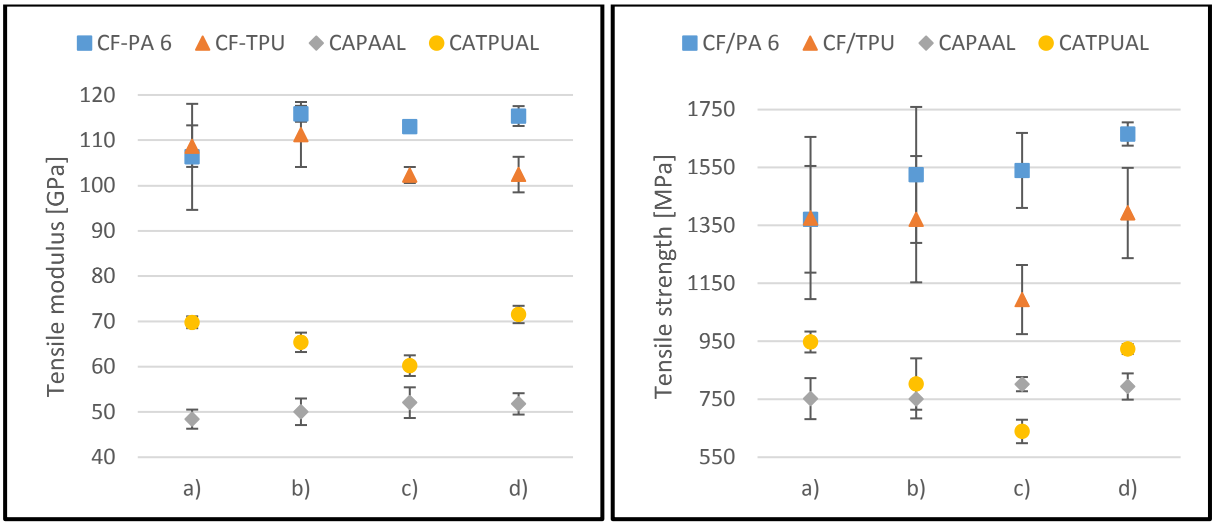

Figure 8: a) Tensile modulus of elasticity and b) tensile strength of CF-PA 6 and CF-TPU as well as hybrid laminates CAPAAL and CATPUAL

4 Conclusion

The investigations showed that the cooling rate has only a limited influence on the mechanical characteristics. Nevertheless, a DSC analysis of the unreinforced PA 6 samples showed that a decrease in the cooling rate increases the crystallinity. This has no significant influence on the tensile and bending properties of the fibre-reinforced plastics. This can be ascribed to the comparatively low degree of crystallisation, which only varies around 9 % in the individual cooling rates from 2 to 30 K/min. A significant increase of the mechanical characteristics with an increased degree of crystallinity could not be verified, especially not in the stiffness properties. However, the cooling rate has a significant influence on the strength. Furthermore, a significant difference could also be determined in the interlaminar shear strength. Concerning the hybrid laminates, the mechanical characteristics are more strongly affected by the varying cooling rates. Especially in the interlaminar shear strength, the differences become significant.

Due to the relatively high moisture absorption of polyamide, the mechanical properties are influenced up to saturation. This influence can be reduced by fibre reinforcement. The absorption of moisture can also be reduced with an increased degree of crystallinity [33]. The applied process cycles show a complete impregnation of the fibre-plastic composites as well as of the hybrid laminates. Thus, no difference in the dependency of cooling speeds was observed.

The used TPU primarily consists of amorphous areas so that no crystalline areas could be identified. But it was observed in the investigations that the process conditions influence the mechanical characteristics. This influence is most notable in the tensile strengths and interlaminar shear strengths. It is noteworthy, that the stiffness values are higher in the CF-PA 6 composites than in the carbon fibre-reinforced thermoplastic polyurethane. However, CATPUAL in combination with the aluminium alloy has a higher bending modulus of elasticity of up to 10 GPa in comparison to CAPAAL. This can be ascribed to the higher pressing temperature and the corresponding over-ageing of the aluminium alloy which influences the mechanical characteristics significantly [6].

5 Summary and Outlook

In the study, the influence of the cooling rate on mechanical characteristics and interlaminar shear strengths in carbon fibre-reinforced polyamide 6 and carbon fibre-reinforced thermoplastic polyurethane and their corresponding hybrid laminates CAPAAL and CATPUAL was analysed. It was shown that a cooling rate between 10 and 30 K/min only cause a change of 5 % in the crystallinity when using polyamide. In this context, no significant influence on the characteristics of the fibre-plastic composites could be detected. The greatest difference was observed only in the tensile strengths. On the other hand, the hybrid laminates showed a significant difference especially with regard to the interlaminar shear strength.

Thermoplastic-based hybrid laminates can be manufactured in a one-shot procedure and are predestined for large-scale production. The usage of a thermoplastic matrix can significantly shorten the manufacturing process in comparison to conventional hybrid laminates. The optimisation of the process cycle in the heating and cooling phase further increases economic efficiency and mechanical characteristics. Their high degree of forming and excellent mechanical characteristics make hybrid laminates such as CAPAAL and CATPUAL not only suitable for the aerospace industry but also for the automotive industry.

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