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EXPERIMENTAL ANALYSIS OF THE INFLUENCE OF THERMOPLASTIC VEILS DOPED WITH NANOFILLERS ON THE THERMAL PROPERTIES OF FIBRE-REINFORCED THERMOPLASTIC COMPOSITES

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Abstract. Fibre-reinforced polymers (FRP) have significant advantages over metals due to their excellent specific mechanical properties. However, their range of application is often limited by insufficient thermal properties. In order to expand the range of applications of thermoplastic composites in particular, it is necessary to improve their thermal properties, especially thermal conductivity. The use of novel veils doped with nanofillers offers a high potential for tailor-made modifications of composite properties depending on the filler used and the composite design. However, the integration of thermoplastic veils with nanofillers into composite structures is associated with some fundamental challenges: modification of the composite design and thus the change of material properties as well as change of the manufacturing process and the process parameters. To investigate these phenomena, polyphenylene sulphide (PPS) veils doped with multi-walled carbon nanotubes (MWCNTs) were integrated into carbon fibre-reinforced polymers (CFRP) with acrylic resin system. For this purpose, various lay-up setups of the fibre reinforcement and the modified veils were defined and the composite structures were fabricated using the wet compression moulding (WCM) process. The influence of the process parameters on the infiltration and consolidation of the composite structure with acrylic resin was investigated. The composite structures were evaluated using non-destructive testing methods such as ultrasonic as well as microscopic observations. In addition, extensive thermal and mechanical tests were carried out to determine the influence of the integrated veils on the composite properties and compared with reference structures. As a result, a basis for a model-based and integrated material development process was created.

Key words: nanomaterials; thermoplastic fibre composites; thermal conductivity; heat transfer;

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modified thermoplastic veils

1 INTRODUCTION

Fibre-reinforced polymers (FRP) are used in various industries such as aviation and aerospace due to their specific properties such as high strength and stiffness [1, 2]. In addition, FRP with thermoplastic matrix are also used in large-scale production like automotive due to their good processing properties. Matrix materials such as polybutylene terephthalate (PBT) or polyphenylene sulphide (PPS) in combination with glass fibres are also commonly used in the electrical and electronics sectors as insulation materials [3]. However, the processing of thermoplastic FRP is often associated with high investment costs, complex manufacturing processes and expensive mould systems [4].

An alternative is offered by novel liquid acrylic resins that combine the advantages of thermoplastic materials with the possibility of processing in classical manufacturing processes of thermoset materials such as injection, infusion or compression moulding [5]. Several studies have investigated the use of acrylic resins in fibre-reinforced composites. For example, FRP with acrylic resin matrix has been manufactured and the resulting composites shown to have excellent mechanical properties, including high stiffness, strength and fatigue resistance [6]. Similarly, the modification of an acrylic resin matrix with graphene oxide resulted in improvements in the mechanical properties of the resulting composites, including increased stiffness, strength and fracture toughness [7]. In addition, acrylic resins have been used as matrix materials in composite materials for wind turbines. For example, composite structures using glass fibres and an acrylic resin matrix were investigated here. The composites obtained exhibit good mechanical properties and showed potential for use in wind turbine blades [8]. Overall, the literature suggests that acrylic resins have great potential as a matrix material for fibre-reinforced composites, with excellent mechanical properties and good resistance to environmental influences.

However, the use of these materials in many applications is limited by their comparatively poor thermal and electrical properties especially in thickness direction, for example in electromobility, where targeted heat dissipation is required [9]. Several studies have investigated the improvement of the thermal and electrical properties of thermoplastic and thermoset FRPs. The addition of nanofillers to polymer matrices has been found to improve thermal and electrical properties and even lead to higher mechanical strength and stiffness. For example, the electrical conductivity of carbon fibre-reinforced thermoplastic composites was investigated and it was shown that the addition of single-walled carbon nanotubes to the matrix led to an improvement in electrical conductivity [10]. Similarly, studies on the thermal properties of graphene oxide modified epoxy composites were conducted and they showed that the resulting composites exhibited improved thermal stability and improved interlaminar shear strength ILSS values [11]. However, the use of nanofillers is associated with fundamental process-related problems such as the formation of agglomerates or filtration effects [12].

An alternative that enables the introduction of highly conductive particles and overcomes the typical processing problems is the use of modified thermoplastic veils. By introducing nanofillers into the polymer melt in processes such as the melt-blown process, nonwovens made of modified polymer fibres can be produced. These can be used as a layer between the reinforcing fibres and in particular influence the properties in the thickness direction of the FRP [13]. Several studies have investigated the effects of incorporating nanofillers into thermoplastic veils. A study investigated the effect of introducing carbon nanotubes (CNT) doped copolyimide nonwovens for fibre-reinforced composites [14]. They found that the addition of carbon nanotubes significantly improved the interlaminar shear strength and toughness of the composite.

In addition, the effects of processing conditions on the thermal properties of thermoplastic composites have also been studied. For example, effect of processing temperature on the thermal conductivity and as well as the use of PBT nano-composites were analysed and it was found that an increase in processing temperature led to an increase in thermal conductivity and a decrease in the coefficient of thermal expansion [15].

Overall, the literature suggests that the thermal properties of thermoplastic fibre-reinforced composites can be improved by various methods, such as the addition of nanofillers, the use of thermoplastic veils and the optimisation of processing conditions. Further research is needed to fully understand the mechanisms underlying these improvements and to optimise the properties of these composites for specific end-use applications.

2 MATERIAL AND METHODS

2.1 Materials

In order to achieve thermal properties modification in thickness direction of the FRP, PPS veil doped with 1 wt.% of multi-walled carbon nanotubes (MWCNTs) and an areal weight of 25 g/m² (TMBK Partners Sp. z o. o., Poland) was utilised. For reference, neat PPS veil with an areal weight of 25 g/m² was used. carbon fibre-reinforced polymers (CFRP) were manufactured using 0º stitched unidirectional carbon fibre fabric (UD-CF) with universal sizing and an areal weight of 314 g/m^2 (Saertex, Germany). Elium® 190 (Arkema, France), a thermoplastic acrylic resin, was used as a polymer matrix. The resin has a viscosity of 100 mPa·s at 20 ºC with a density of 1.01 g/cm^3. The resin can be processed by the commonly used methods for epoxy resins such as wet compression moulding (WCM). Dibenzoyl peroxide PEROXAN BP-25 WD (Pergan, Germany) was used to initiate the polymerisation process of the resin.

2.2 Fabrication method of modified CFRP

The analysis of the influence of the modified nonwovens requires the macroscale analysis of the individual layers on the one hand and the subsequent global analysis of the entire composite structure on the other hand. For this purpose, single-layer structures were prepared in the first step and the composite structures were then manufactured. The specific lay-up configurations are listed in Table 1.

Table 1. Lay-up configurations of analysed specimens.

Sample	Lay-up configuration
Single-layer structures	
SL_CF	1x UD CF 0º
SL_NPPS_CF	1x neat PPS veil; 1x UD CF 0º
SL_PPS_CF	1x PPS veil with 1 % MWCNT; 1x UD CF 0º
CFRP structures	
ML_CF	6x UD CF 0º
ML_CF_NPPS_V	6x UD CF 0º; 5x neat PPS veil; (PPS veil layers between the CF layers)
ML_CF_PPS_V	6x UD CF 0º; 5x PPS veil with 1 % MWCNT (PPS veil layers between the CF layers)
ML_CF_PPS_S	6x UD CF 0º; 4x PPS veil with 1 % MWCNT (PPS veil layers on both sides on the CFRP surfaces and between the first and second CF layer and the fifth and sixth CF layer.)

The specimen plates were manufactured using a WCM process. For this purpose, fibre and veil layers were prepared measuring 280 mm x 280 mm and the respective lay-ups were prepared. These were positioned in a dipping tool heated to 100 ºC. The resin system was mixed with 2 % by weight of peroxide at room temperature. The resin mixture was then distributed equally across the prepared layer structures. The test specimen plates were consolidated at a pressure of 8 bar for 10 min. The amount of resin was calculated and selected to achieve a fibre volume fraction of about 55 % in all specimens. In the case of the single-layer sheets, a fine peel ply was also used to facilitate demoulding.

2.3 Measurement methods

Non-destructive testing

The quality of the CFRP produced was investigated by ultrasonic analysis. A generator with a frequency of 250 kHz and a receiver located at a distance of 100 mm from the generator were used for this purpose. The signal gain used was 70 dB, while the attenuation level measured was -30 to 0 dB. The analysed structures were tested in air environment. The area investigated was 280 mm x 280 mm and the measurement had a resolution of 500 µm.

Non-destructive X-ray inspection methods such as computed tomography (CT) were used to investigate the veil position and fibre orientation in the CFRP. The high-resolution CT system V|tomex|-L 450 (GEPhoenix X-ray) with a 300 kV microfocus and a 450 kV macrofocus X-ray tube was used for this purpose. The CT consists of a flat panel detector with 2048 × 2048 14 bit pixels, with one pixel covering an area of 200 µm × 200 µm. Samples of 50 mm x 50 mm were prepared for the studies and 3D scans were obtained and analysed.

Optical microstructure observation

In order to determine the influence of the modified veils on the mechanical and thermal properties, an analysis of the positioning, structure and layer thickness is necessary. For this purpose, microscopic examinations of the manufactured CFRP structures as well as the single layers structures were carried out by a light microscope AxioTech 100HD. Samples were cut out from the fabricated plates and grinded with papers of 6 grit types: P180, P320, P500, P800, P1200 and polished using diamond slurries with 1 µm grit size.

Measurement of thermal properties

Mainly, the influence of the veils on the properties of CFRP in thickness direction was analysed. Therefore, thermal properties were measured using two methods: transient plane source method (HotDisk) [16, 17] and flash technique (LFA).

Single layer lay-ups were analysed using the flash technique [18] (LFA 447 Nanoflash, Netzsch), which requires thin square samples of 1mm to 2 mm thickness and about 10 mm side length. The applicability of this technique in heterogeneous materials was discussed by multiple studies [19, 20]. To reduce the negative effects of inhomogeneity, several three samples were taken and conclusions were drawn from the averaged results. Samples were cut from different areas of the plate. The specimens were further processed with sandpaper to obtain their final shape with smooth and parallel surfaces and then dried before the measurements. Thermal diffusivity was measured at 20 ºC.

The modified CFRP structures were analysed by the HotDisk method. For this purpose, sample geometries characterised by a thickness of 4 mm and a length and width of 100 mm were used. The thermal conductivity was determined at 20 ˚C. For this purpose, the setup of two sample plates and a HotDisk sensor with a diameter of 2 mm placed in between was chosen. The measurement parameters were set to a heating time of 10 s and adjusted heating powers of 25 to 30 mW. For this purpose, the samples were also prepared to obtain a smooth and parallel surface and six measurements were performed and the average value was taken for evaluation.

Mechanical testing

The influence of the embedded veils on the tensile and flexural properties of the CFRP structures was investigated. For this purpose, static tensile tests were carried out according to DIN EN ISO 527 standard. The specimen geometry was prepared in the dimension of 250 mm x 15 mm x 2 mm and longitudinal fibre direction. Additional load introduction tabs were used. The measurement was performed on a Zwick/Roell Z100 universal testing machine with a pre-load of 20 N and a traverse speed of 2 mm/min. The tensile tests were carried out for six specimens at room temperature (20 ˚C).

In addition, static 3-point bending tests were carried out according to DIN EN ISO 14125 standard. For this purpose, six specimens were prepared measuring 100 mm x 15 mm x 2 mm in the longitudinal direction of the fibres. The support distance was 80 mm, the diameter of the compression fin was 5 mm and the supports 2 mm. The tests were carried out by a Zwick/Roell Z2.5 testing machine at a testing speed of 2 mm/min at room temperature (20 ˚C).

3 RESULTS AND DISCUSSION

3.1 Ultrasonic test and computed tomography

In order to assess the quality of the analysed samples, ultrasound examinations of the manufactured plates were performed. This investigation allows the identification of manufacturing defects such as layer displacement, delamination or pores. Figure 1 a) shows an example ultrasonogram of the CFRP structure ML_CF_PPS_V. The sonogram confirms the good quality of the composite structures produced by wet compression moulding. No pores, delamination or dry areas were observed. The other composite lay-ups were of equally good quality. This confirms that the manufacturing process was carried out correctly and that the process parameters were chosen properly.

In addition, the manufactured samples were evaluated with the help of CT examinations. Here it was possible to analyse the distribution of the veils in the composite material and to detect any displacements, defects in the veil or agglomerations that could occur due to the manufacturing process. An example CT scan of CFRP structure ML_CF_PPS_V is shown in Figure 1 b). The individual fibres are clearly visible. No displacement or damage of the embedded veil fibres can be seen. This indicates the good quality of the manufactured structures and homogeneous material properties.

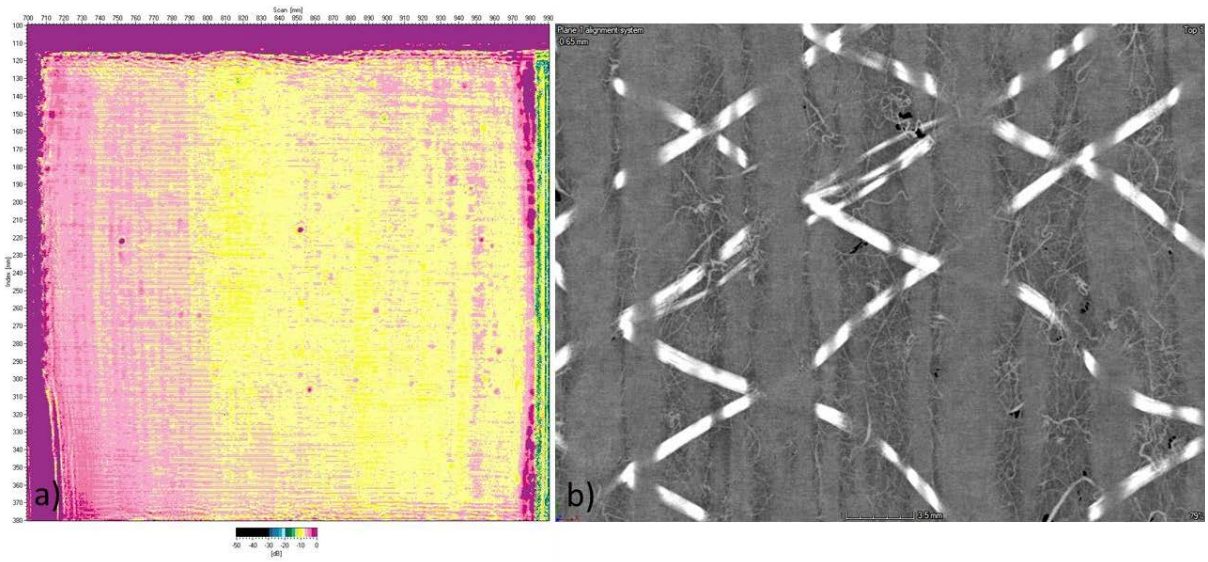

Figure 1. a) Ultrasonogram and b) CT-scan of the investigated CFRP structure with modified veils ML_CF_PPS_V

3.3 Analysis of single-layer structures

Thermal conductivity measurements

In order to design CFRP structures accurately and reliably, it is necessary to analyse the thermal properties of the individual layers. For this purpose, the manufactured single-layer structures were analysed with the help of LFA procedures. For this purpose, six measurements from different areas of each sample were performed and an average value calculated. The necessary density and specific heat capacity were determined using gravimetric buoyancy technique method and differential scanning calorimetry (DSC). The results of the measurements are shown in Table 2.

Table 2. Results of thermal measurements for single-layer structures

Sample	Density [g/cm^3]	Specific heat [J/(kg·K)]	Thermal diffusivity [m^2/s]	Thermal conductivity [W/(m·K)]
SL_CF	1.506	0.994	0.465	0.696
SL_NPPS_CF	1.469	0.996	0.464	0.679
SL_PPS_CF	1.475	0.967	0.547	0.780

The measurements have shown that the use of PPS veils has an influence on the thermal conductivity of the composite structure. Compared to the reference structure, the use of unmodified nonwovens leads to a minimal reduction in thermal conductivity. In contrast, the use of veils with CNTs improves the thermal conductivity in thickness direction. However, in order to be able to provide a precise conclusion, an analysis of the layer thickness is necessary and has to be considered in the assessment.

Microstructure observations

The samples with the single layers were additionally evaluated with regard to microstructure, in particular the layer thickness of the fibre layer, the layer thickness of the veils and the interlayer. For this purpose, microstructure observations were conducted and the layer thicknesses were measured in ten positions and the average values calculated. The respective microstructures are shown in Figure 2.

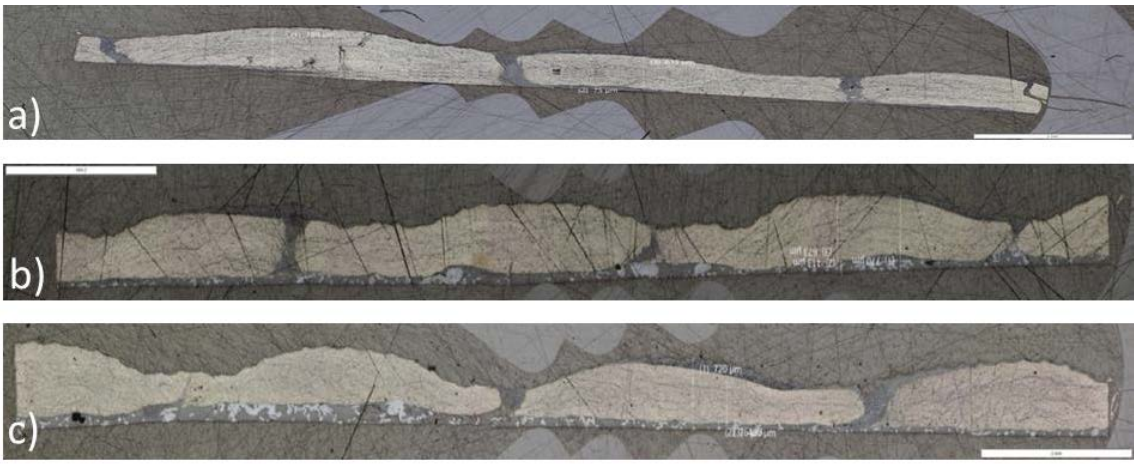

Figure 2. Microstructures of the samples a) SL_CF b) SL_NPPS_CF c) SL_PPS_CF

It can be observed that the average thickness of the carbon layer is 553 µm, 580 µm and 560 µm for the SL_CF, SL_NPPS_CF and SL_PPS_CF samples, respectively. The layer thickness of the nonwovens differs between the unmodified and modified PPS veils samples, averaging 55 µm and 120 µm, respectively. The use of CNT-modified PPS veils can lead to a significant increase in the thickness of the layer between fibre reinforcement layers.

3.2 Analysis of modified CFRP structures

Thermal conductivity determination of modified CFRP structures

To evaluate the influence of the veils used and the lay-up of the CFRP structure on the thermal conductivity in the thickness direction, measurements were carried out using the HotDisk method. For this purpose, six measurements were carried out on each of the CFRP structures at different positions and an average value was determined. The results of the measurements are shown in Table 3.

Table 3. Results of thermal measurements for CFRP structures

Sample	Thermal diffusivity [m^2/s]	Thermal conductivity [W/(m·K)]
ML_CF	0.3199	0.6398
ML_CF_NPPS_V	0.3034	0.6068
ML_CF_PPS_V	0.1626	0.3252
ML_CF_PPS_S	0.1328	0.2655

It is visible that the veils used have a negative influence on the thermal properties of the CFRP structure. The use of veils modified with CNT deteriorates the thermal diffusivity by almost 50 % compared to the reference structure. The use of unmodified PPS nonwovens also reduces the properties but this effect is minimal compared to the modified nonwovens.

This can be explained by the structure of the modified CFRP and the introduction of an additional resin layer between the highly conductive carbon fibres. This increases the contact resistance and leads to a reduction of the thermal properties in the thickness direction. The minimal influence of the unmodified PPS nonwovens can be explained by the significantly more homogeneous structure of the veils themselves. Due to the lower viscosity of the unmodified PPS during the production of the veils, smaller fibre diameters can be achieved, which ultimately reduces the layer thickness.

3.4 Static tensile and 3-point bending test

In order to analyse the influence of the embedded modified nonwovens on the mechanical properties of the CFRP structures, quasi-static tensile and 3-point bending tests were carried out. The tensile specimens were all fractured in the middle, indicating correct preparation of the specimens and performance of the tests. Table 4 shows the results of the tests on the tested specimens: tensile strength (σ), strain (ε), and Young's modulus (E).

Table 4. Results of static tensile tests.

Sample	σ [MPa]	ε [%]	E [MPa]
ML_CF	1670 ± 229	1.5 ± 0.45	132000 ± 7840
ML_CF_NPPS_V	980 ± 30.5	1.1 ± 0.19	109000 ± 9750
ML_CF_PPS_V	1240 ± 97.5	1.3 ± 0.23	115000 ± 12000
ML_CF_PPS_S	1340 ± 229	1.18 ± 0.12	121000 ± 8090

The flexural properties were determined using 3-point bending test. Table 5 shows the average result of the considered specimens: flexural strength (σ), strain (ε), and flexural modulus (E_f).

Table 5. Results of static 3-point bending tests.

Sample	σ [MPa]	ε [%]	E_f [MPa]
ML_CF	1030 ± 64.9	0.89 ± 0.064	122000 ± 3660
ML_CF_NPPS_V	733 ± 262	0.76 ± 0.081	111000 ± 14700
ML_CF_PPS_V	1010 ± 166	1.1 ± 0.20	109000 ± 10900
ML_CF_PPS_S	843 ± 80.3	1.1 ± 0.25	96400 ± 5160

It can be seen that the embedded veils in all variants reduce the mechanical properties, both tensile and flexural. However, the incorporation of veils with 1 % MWCNT improves both the strength and modulus compared to neat PPS nonwoven. The lay-up and positioning of the fleece also has a significant influence on the properties. The tensile strength and Young's modulus of the ML_CF_NPPS_S structure are higher than those of the structure where veils are placed between each layer of carbon fibre reinforcement. On the other hand, the flexural properties of the ML_CF_NPPS_S structure are lower than those of the ML_CF_PPS_V structure. However, the results obtained are encouraging and provide a basis for research into further material combinations.

4 CONCLUSIONS

The research work presented focused on investigating the influence of PPS veils modified with CNTs on the thermal and mechanical properties of CFRP structures with an acrylic resin matrix.

The veils were introduced into the structure using a stacking principle and the composite structures were fabricated using wet compression moulding processes. It was shown that this process is well suited to integrate such veils as interlayers in CFRP structures, also in combination with liquid acrylic resins. The NDT tests carried out confirmed the good quality of the structures manufactured and showed a homogeneous distribution of the veils.

The analysis of the thermal properties, in particular the thermal conductivity of the single layers, showed an improvement of the thermal conductivity from 0.696 W/(m·K) to 0.780 W/(m·K) when using CNT modified PPS veils compared to the reference structure. However, the microscopic investigation showed that the single layer thickness of the veil layer increases between non-modified and modified veils, which can have a significant influence on complex CFRP structures.

The investigations on the fabricated CFRP structures have shown a significant deterioration of the properties. Here, the thermal conductivity was decreased from 0.6398 W/(m·K) to 0.3252 W/(m·K) when using modified PPS veils between each layer of reinforcing fibres compared to the reference structure. This is due to the increase in the thickness of the resin-veil layer between the reinforcement fibre layers and the resulting increase in thermal contact resistance.

The mechanical tests have shown that the incorporation of veils in composite structures reduces the tensile and flexural properties. However, the use of CNTs in the nonwoven increases the mechanical properties compared to unmodified PPS nonwovens.

In summary, the modification of veils with CNTs and the incorporation of those as interlayers in composite structures is a complex problem. On the layer level, an improvement in thermal properties has been demonstrated but the problem of contact resistance and increased thickness of the resin-veil layers increases with more complex structures and can reduce the properties. This requires further investigation, especially to minimise the interlayer effects.

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