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SYNERGISTIC ENHANCEMENT OF CFRPs: COMBINING CNT-DOPED THERMOPLASTIC VEILS AND SWCNT-MODIFIED THERMOPLASTIC MATRIX

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The multifunctional enhancement of carbon fibre-reinforced polymers (CFRPs) is critical for their expanding applications in aerospace, automotive, and electronics industries. This study examines the combined effect of thermoplastic veils doped with multi-walled carbon nanotubes (MWCNTs) and a polymer matrix modified with single-walled carbon nanotubes (SWCNTs) on the mechanical, thermal, and electrical properties of CFRPs. Liquid thermoplastic acrylic resin Elium®, modified with 0.02 wt.% SWCNTs served as the matrix, while thermoplastic veils based on polyphenylene sulphide (PPS) and polybutylene terephthalate (PBT) doped with 1.0 wt.% MWCNTs were interleaved into the composite structure. Characterisation revealed that the SWCNTs formed conductive networks in the polymer matrix, enhancing electrical conductivity in-plane (X and Y directions) but not improving it through the thickness (Z direction) due to resin-rich regions introduced by the veils. The impact resistance improved across all the composites, particularly for the PPS-based veils, attributed to effective fibre bridging mechanisms. The glass transition temperature (T_g) also increased due to strong adhesion at the veil-matrix interface and molecular interactions between the nanofillers and the polymer matrix. The results highlight the potential of combining nanofiller-modified matrices with thermoplastic veils to achieve tailored multifunctional CFRPs. However, optimising the interlayer resin content remains crucial for further enhancing through-thickness conductivity. These findings contribute to advancing CFRPs for high-performance, multifunctional applications in diverse industries.

Keywords: **CFRP, Elium® resin, thermoplastic veils, CNT**

INTRODUCTION

The demand for carbon fibre-reinforced polymers (CFRPs) in industries such as aerospace, automotive, construction, electronics, and renewable energy continues to grow, with projections estimating the total CFRP market to be worth $41.4 billion US in 2025 [1]. In addition, literature reports indicate a drive to develop CFRPs with enhanced applicability by designing materials that can perform additional functions beyond their role as a structural component [2-5]. Extension of the applicability of CFRPs can be achieved, for example, by increasing the electrical or thermal conductivity of the composites, which will allow them to be utilised as electromagnetic shielding elements, provide increased protection against lightning strikes or enable damage detection of the composite [4, 6]. In order to improve the functionality of CFRPs, modifications are used, among which are modifications of the polymer matrix and the introduction of conductive material into the composite structure [7].

Modification of the composite matrix involves the introduction of a filler into the polymer to increase electrical conductivity. Various types of fillers are employed to improve conductivity: carbon-based (graphene, graphite, carbon nanotubes), metallic (Ag, Cu), and ceramics (BN, Si) [8]. Carbon nanotubes (CNTs) have high electrical conductivity (10^2 S/cm - 10^6 S/cm), high thermal conductivity (2000 W/mK - 6000 W/mK), a high Young's modulus (1.7 TPa - 3.6 TPa), high tensile strength (50 GPa - 200 GPa), and a high aspect ratio [9-11]. Due to these properties, CNTs are often used as CFRP modification fillers. The most common problems encountered when using polymer matrix modification are low filler dispersion, a lack of adhesion between the filler and the matrix, and a significant increase in viscosity with an increasing filler content. A second way to modify the properties of CFRPs is to introduce additional conductive material between the carbon fabric layers (interleaving). The introduced material is intended to improve the interlaminar fracture strength and conductivity of the composite [7]. The materials introduced into the composite can be in various forms: paper sheets with a layer of silver nanofibres [12], a layer of polyurethane modified with multi-walled carbon nanotubes (MWCNTs) [13], buckypaper (a carbon nanotube sheet) [14] or thermoplastic veils modified with MWCNTs [15]. Regardless of the form of material introduced into the composite structure, it is vital to ensure adequate adhesion between the matrix and the introduced material as it can alter the mechanical properties of the composite [16]. In addition, the introduced layers of conductive material can also perform additional functions, e.g. sensors to monitor damage to the composite [17].

Studies on modifying epoxy resin are widespread in the literature due to its frequent use in industry [7, 18]. The literature also describes the use of thermoplastics as a matrix for CFRPs, which have the advantage of increased recyclability or the possibility to reshape components into different parts under temperature. Compared to thermoset matrix composites, they offer lower mechanical properties and lower thermal resistance but improve fracture toughness and impact resistance. Because of the need for high temperatures during manufacturing and the high viscosity of melted thermoplastic polymers, manufacturing methods typical for thermoset polymers cannot be used. Additionally, modification with fillers further increases the viscosity of the thermoplastics, which makes the manufacturing of CFRPs much more complex [19]. Owing to the problems associated with manufacturing CFRP with a thermoplastic matrix, new thermoplastic materials have been developed, including a liquid thermoplastic acrylic resin with the trade name Elium®. The liquid form of Elium® resin allows the use of CFRP manufacturing methods typical of thermoset resin-based composites, such as the infusion process, RTM, or pultrusion. Additionally, it has a low viscosity (100 mPa·s) and thanks to that, it can be easily modified by the introduction of fillers. Previous publications show that researchers have mainly focused on the mechanical and thermomechanical properties of Elium® resin, conducting impact [20-22], tensile [23, 24], bending [25, 26] or dynamic mechanical analysis [27-29] tests. Based on these tests, it can be concluded that Elium® resin exhibits thermomechanical and mechanical properties comparable to commonly used epoxy resin systems [30]. Nevertheless, Elium®, like thermoset resins, has low electrical and thermal conductivity. Due to these properties, the applications of Elium® may be limited. Some studies on Elium® resin have addressed topics such as its use as an insulating material [31], the introduction of phase change materials into the resin [32] or increasing the electrical conductivity of CFRPs by introducing single-walled carbon nanotubes (SWCNT) into the polymer matrix [33] to develop new applications.

This work investigates the influence of two methods of CFRP modification – introducing thermoplastic MWCNT-doped veils based on polyphenylene sulphide (PPS) and polybutylene terephthalate (PBT) into the composite structure as well as modifying the polymer matrix with SWCNT – on selected properties of composites. The second aim is to examine whether achieving a synergetic effect of the performed modifications is possible.

MATERIALS AND METHODS

Materials

The acrylic liquid thermoplastic resin Elium® 188XO (Arkema, France) was used for nanocomposite preparation and as the CFRP matrix. A powder form of dibenzoyl peroxide (Acros Organics, Geel, Belgium) was employed as the polymerisation reaction initiator. To modify the polymer matrix, Tuball™ SWCNTs (OCSiAl, Leudelange, Luxembourg) were used as the filler.

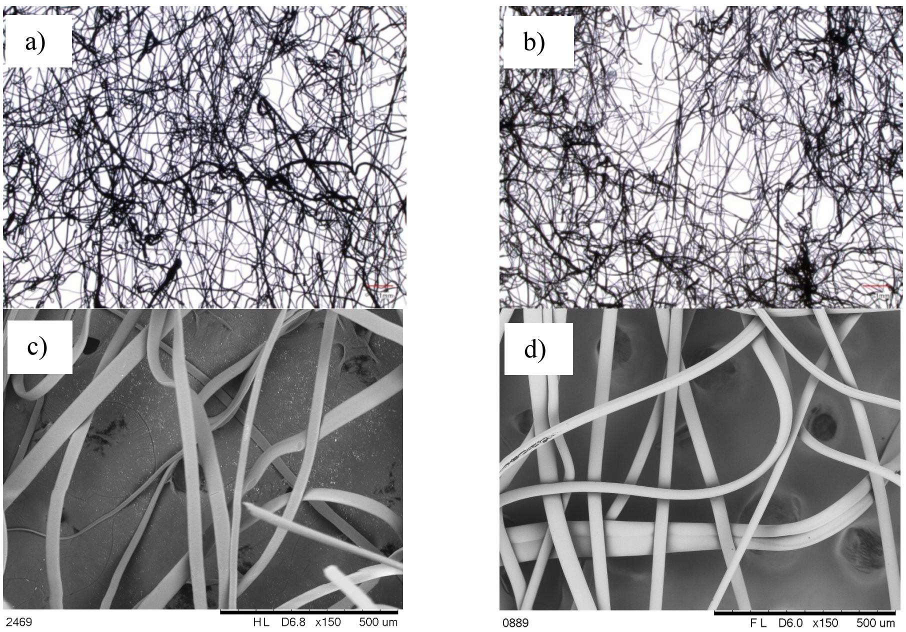

Fig. 1. Macrostrucutre of MWCNT-doped veils based on PBT (a), based on PPS (b) and microstructure of MWCNT-doped veils based on PBT (c), based on PPS (d)

The chosen SWCNTs have an average diameter of < 2 nm, length > 1 μm and purity of 75 %. Two thermoplastic materials were used for veil production – Fortron® PPS and Celanex® 2008 PBT, provided by Celanese (Irving, TX, USA). NC7000 MWCNTs (Nanocyl, Sambreville, Belgium) were utilised for thermoplastic veil modification. The used MWCNTs have an average diameter of 9.5 nm, an average length of 1.5 μm and a purity of 90 %. The microstructure of the veils is presented in Figure 1. A unidirectional (UD) carbon fabric (Saertex, Saerbeck, Germany) with an areal weight of 600 gsm was employed for CFRP manufacturing. Additionally, the fabric had polyester stitches.

Manufacturing

The nanocomposites were prepared by mixing Elium® resin with 0.02 wt.% SWCNT. After mechanical mixing, the SWCNTs were dispersed by ultrasonication. The process was performed utilising an ultrasonic processor VCX 1500 (Sonics & Materials, Newtown, CT, USA) with a maximum frequency of 20 kHz. The ultrasonication process was conducted with the following parameters: process time 1 h, amplitude 40 %, ultrasonic wave activity 10 s, and time between ultrasonic waves 14 s. Afterwards, the prepared mixtures were used for CFRP manufacturing.

The thermoplastic veils of the areal weight of 25 gsm, based on PPS and PBT, were manufactured by the melt-blown method. TMBK Partners Sp. z o.o (Warsaw, Poland) supplied the thermoplastic veils modified with 1.0 wt.% MWCNTs.

To manufacture the CFRPs, a stack of six carbon fabrics with dimensions of 300 mm × 300 mm was prepared. The CNT-doped veils were inserted between each layer of carbon fabric. The CFRPs were manufactured by the resin infusion process under the vacuum pressure of 0.9 bar. Before the process, unmodified and modified Elium® resin was mixed with 1.0 wt.% benzoyl peroxide. Five different CFRPs were prepared, and the composition of the laminates is presented in Table 1.

TABLE 1. **CFRP composition and symbols**

Symbol	Elium® SWCNT modification	Veils material	Veils MWCNT modification
C_REF	No modification	Without veils	No modification
C_vPBT	No modification	Four PBT veils	Modified with 1.0 wt.%
C_vPPS	No modification	Four PPS veils	Modified with 1.0 wt.%
C_ES_vPBT	Modified with 0.02 wt.%	Four PBT veils	Modified with 1.0 wt.%
C_ES_vPPS	Modified with 0.02 wt.%	Four PPS veils	Modified with 1.0 wt.%

Methods

Microstructure observations were performed by means of two scanning electron microscopes. The first microscope, TM 3000 Hitachi (Tokyo, Japan), was used to observe the manufactured CFRP microstructure. The specimens were sanded and polished with diamond slurry to prepare the surface. Afterwards, specimens were coated with a 3 nm Pd-Au electroconductive layer. Observations of the CFRP microstructure were conducted at 15 kV acceleration voltage. A Hitachi SU-70 (Tokyo, Japan) was employed to observe the SWCNT distribution in the Elium® matrix. The observations were made at the acceleration voltage of 5 kV.

The impact strength was determined by Charpy's test using a pendulum impact tester RKP 450 (Zwick-Roell, Ulm, Germany). Ten unnotched test specimens 15 mm × 75 mm (width × length) were tested for each manufactured CFRP. The impact strength was calculated from the absorbed energy obtained at break and according to the PN-EN ISO 179 standard.

The influence of the SWCNT modification and thermoplastic veil introduction on the glass transition temperature (T_g) and storage modulus at room temperature (E`@25°C) of CFRPs was analysed by dynamic mechanical analysis (DMA). The research was performed using a DMA Q800 (TA Instruments, New Castle, DE, USA) in dual cantilever mode from 0 °C to 180 °C, at the heating rate of 2 °C/min with the amplitude of 20 μm and frequency of 1 Hz. The studies were performed according to ASTM D7028.

The electrical conductivity of the fabricated CFRPs was measured in three directions: parallel to the carbon fibres (X direction), perpendicular to the carbon fibres (Y direction) and through the sample thickness (Z direction). Test specimens with the dimensions of 60 mm × 10 mm (X and Y direction) and 10 × 10 mm were cut from different sections of the CFRPs. Five test specimens for each direction represented each material.

a)

b)

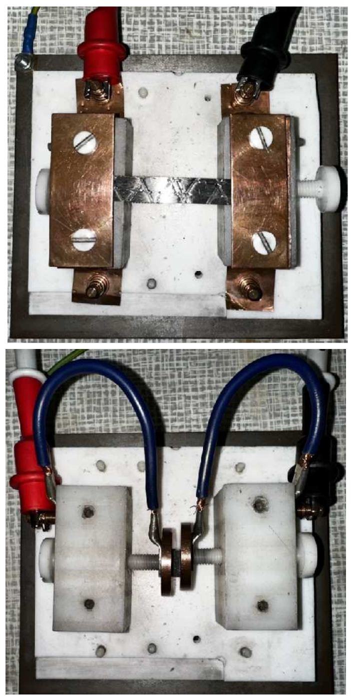

Fig. 2. Measuring stand for electrical conductivity test in configuration for test in X and Y direction (a) and configuration for test in Z direction (b)

The electrical conductivity was measured by means of a Keithley Model 2450 SourceMeter connected to a measuring stand with copper electrodes (Fig. 2). Silver paste (CW7100 from Chemtronics®, Kennesaw, GA, USA) was utilised to ensure better contact between the specimens and the electrodes.

RESULTS

Microstructure observations

The microstructure observations of the manufactured CFRPs, presented in Figure 3, were conducted to evaluate the influence of the introduced veils on their microstructure. Figure 3a shows the microstructure of the composite with SWCNT modified Elium® resin at higher magnification. It can be observed that the introduced SWCNTs are distributed in the polymer matrix and have contact with the carbon fibres. Such distribution of the introduced nanofiller leads to the formation of electroconductive paths, which can increase the electrical conductivity of the composites [34].

The microstructure observations showed that the manufactured CFRPs have few defects such as pores. The small number of defects indicates that the carbon fabric is well-infiltrated by the Elium® resin even after SWCNT modification. Moreover, delaminations at the fibre/matrix and veil/matrix interface were not observed. Introducing the thermoplastic veils into the composite structures increased the distance between the two carbon fabric layers (Fig. 3c and d), resulting in higher resin-rich regions in the modified composites than C_REF.

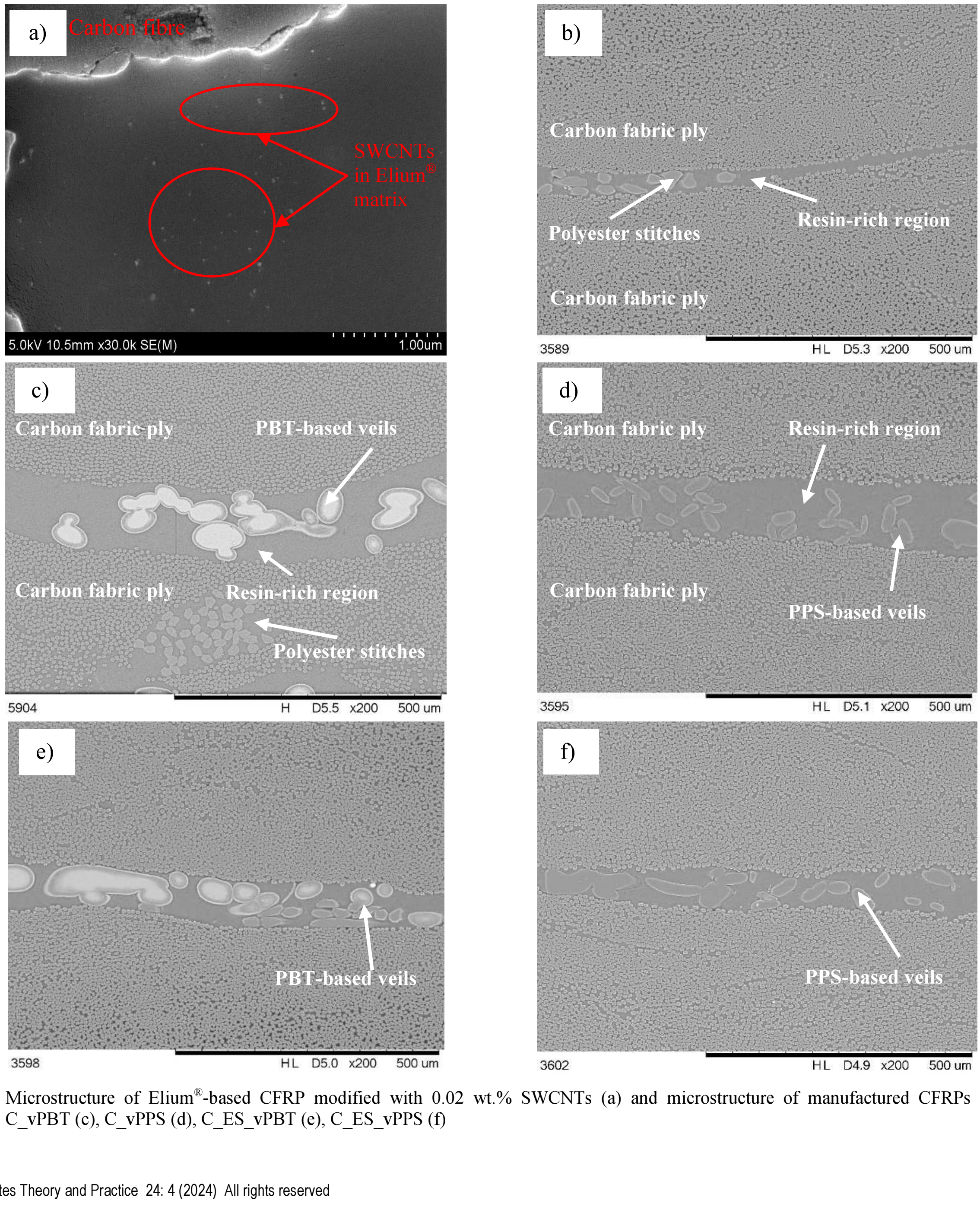

Fig. 3. Microstructure of Elium®-based CFRP modified with 0.02 wt.% SWCNTs (a) and microstructure of manufactured CFRPs C_REF (b), C_vPBT (c), C_vPPS (d), C_ES_vPBT (e), C_ES_vPPS (f)

Impact resistance

The results of Charpy's test are presented in Figure 4.

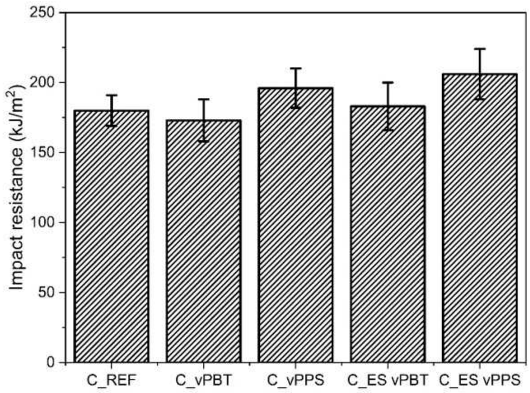

Fig. 4. Impact resistance of manufactured CFRPs

Introducing the thermoplastic veils generally improved the impact resistance of the composites compared to C_REF. Only for C_vPBT was a decrease of around 4 % observed. Quan et al. [35] proved that introducing thermoplastic veils could improve the mechanical properties of composites through mechanisms such as fibre debonding bridging. Such an effect was observed for the introduced PPS veils. Moreover, a synergetic effect of the two modifications was observed. The modification of Elium® resin by introducing SWCNTs further improved the impact resistance of the veil-modified composites. Good chemical interaction between the introduced nanofiller and matrix in addition to mechanisms such as CNT bridging and pull-out are the causes of the impact strength increase [36].

Thermomechanical properties

The results determined from the obtained DMA curves are presented in Table 2. The highest storage modulus was obtained for the composite without modifications (C_REF). The introduction of thermoplastic veils caused a decrease in the modulus as a consequence of the lower fibre volume in the composite resulting from the increased resin-rich regions. Resin modification raises the storage modulus of composites with veils introduced into the composite structure (C_ES_vPBT and C_ES_vPPS). The high stiffness of the SWCNT and molecular interactions between the nanofiller and the polymer matrix is responsible for the storage modulus growth [37, 38]. The glass transition temperature of modified composites rose when compared to C_REF. The thermoplastic materials used for the veil production possess a higher T_g than the Elium® resin. Good adhesion at the veil/matrix interface causes an increase in the T_g of the modified composites. The addition of the SWCNTs to the polymer matrix further raises the T_g due to the high thermal properties of the nanofiller and its molecular interaction with the matrix.

TABLE 2. **Storage modulus (E') at room temperature and glass transition temperature (T_g) of CFRP determined from tan delta**

Composite	E'@25°C [GPa]	Glass transition temperature [°C]
C_REF	39.6	98
C_vPBT	32.3	102
C_vPPS	30.8	102
C_ES_vPBT	34.5	106
C_ES_vPPS	37.8	105

Electrical conductivity

The results of the electrical conductivity test conducted in three directions for the composites are presented in Figure 5. Generally, introducing the MWCNT-modified veils into the composite structures reduced the electrical conductivity of the composites.

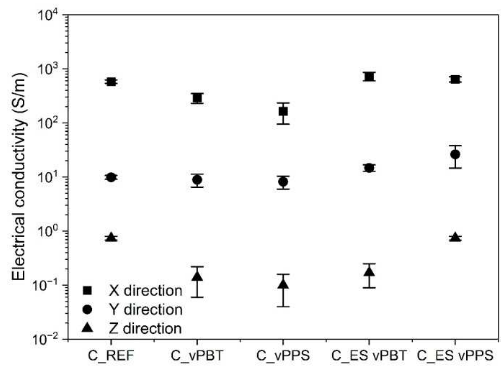

Fig. 5. Electrical conductivity in three directions (X, Y and Z) of manufactured CFRPs

In the composites with the unmodified resin (C_vPBT and C_vPPS), a decrease of about 60 % in the X direction, about 15 % in the Y direction and about 80 % in the X direction were observed. It was caused by the increased distance between the carbon fibre plies and the increased number of resin-rich regions, which is an insulator. Additionally, the electrical conductivity of the MWCNT-modified veils was not sufficient to raise the electrical conductivity of the CFRPs. The resin modification increased the electrical conductivity values as the introduced SWCNT created conductive paths in the polymer matrix [39]. 25 % and 10 % increments in the X direction and 50 % and 165 % in the Y direction were observed for the C_ES_vPBT and C_ES_vPPS composites, respectively. As for the Z direction, a decrease of 77 % was observed for C_ES_vPBT, and the electrical conductivity value for C_ES_vPPS stayed at the level of C_REF. The conductive paths of the SWCNTs could not raise the electrical conductivity of composites in the Z direction owing to the increased distance between the carbon fabric plies after the introduction of the thermoplastic veils. The authors' previous work [33] proved that the modification of Elium® resin with a small content of SWCNTs could improve electrical conductivity in the Z direction.

CONCLUSIONS

This work investigated the combined effect of SWCNT-modified Elium® resin and the incorporation of thermoplastic MWCNT-doped veils on the selected properties of CFRPs. The introduction of thermoplastic veils based on PPS and PBT did not cause an increased number of defects in the composite structure but simultaneously increased the number of resin-rich regions. The introduction of thermoplastic veils generally improved the impact resistance of the composites. The PPS veils, in particular, performed better than the PBT veils, likely because of their more effective fibre bridging mechanisms caused by better interaction at the veil/matrix interface. Adding SWCNTs further enhanced the impact resistance by means of chemical interactions and bridging effects. The glass transition temperatures of all the modified composites grew, indicating improved thermal properties due to strong veil-matrix adhesion and nanofiller-matrix interactions. Nonetheless, resin modification with SWCNTs enhanced conductivity in both the X and Y directions, attributed to the formation of conductive paths. The Z-direction conductivity remained unchanged or reduced as the resin-rich regions and the increased interlayer distance hindered the conductive network. The study demonstrates that while individual modifications enhance specific properties, achieving a synergistic effect depends on balancing the microstructure of the composite and modification with CNTs. The simultaneous use of SWCNTs and MWCNT-doped veils presents a promising approach for multifunctional CFRPs, but requires further optimisation to mitigate conductivity losses caused by resin-rich regions.

Future research could explore improving the nanofiller dispersion in the resin and thermoplastic veils, alternative veil materials, or using veils with a lower areal weight. Reducing the thickness of the resin-rich regions in the composite structure can be achieved by lowering the areal weight of the veils or thermopressing them before CFRP manufacturing. The findings contribute valuable insights into the design of multifunctional CFRPs for high-performance applications across industries.

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