

Nonwoven fabrics with carbon nanotubes used as interleaves in CFRP

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Abstract. The goal of the present study was to implement thermoplastic nonwoven fabrics containing multi-walled carbon nanotubes as interlayers in Carbon Fiber Reinforced Polymers. These functional nonwovens were fabricated by a half-industrial scale melt-blown technique, starting with nanocomposite pellets of copolyamides doped with 3.5wt% of multi-walled carbon nanotubes. Three types of composite panels were fabricated using an out-of-autoclave technique (OoA): one without nonwovens and two with nonwovens. Incorporation of thermoplastic nonwovens doped with 3.5wt% of multi-walled carbon nanotubes increased the surface and volume electrical conductivity in direction K_z by about 2 and 3 orders of magnitude, respectively. Based on the images obtained from a Scanning Electron Microscope, it was found that melted nonwovens adhere well to the carbon fibers. It was also confirmed that carbon nanotubes are well dispersed in nonwovens, which results in an improvement of the overall electrical conductivity of the composite panels. The lack of homogenous layers of nonwovens between the carbon fiber layers decreased the interlaminar shear strength of the composite panels and affected the level of their electrical conductivity. Moreover, thermo-mechanical analysis showed an increase of the glass transition temperature of the resin in the presence of thermoplastic nonwovens and the appearance of an additional peak on the loss modulus curve caused by the polyamide 6 segments present in the copolyamides used.

1. Introduction

Lightweight non-metallic components made of Carbon Fiber Reinforced Polymer (CFRP) are commonly used in the automotive, aviation, defense and electronics industries, mainly due to their high strength and performance and low weight. On the one hand, CFRP offers increased manufacturing productivity, lower VOC emissions, better corrosion resistance, high durability and design flexibility. On the other hand, they suffer from insufficient electrical conductivity for lightning strike protection, static discharge, electrical bonding, grounding, interference shielding or current return through the structures required in many applications such as aircraft fuselages, wings, car bodies, satellites or antenna dishes [1][2].

Because of their low impedance and high reflectivity, carbonaceous structures like carbon fibers, graphite, graphene or carbon nanotubes (CNTs) are good candidates for increasing the electrical conductivity of CFRP. Moreover, in comparison to the copper meshes which are used nowadays in CFRP, they are much lighter but still show high mechanical strength [3][4]. Recently, much research has focused on incorporating single-walled (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) into CFRP. This can be achieved by various methods: mixing with the neat epoxy resin used in liquid technologies, e.g. infusion or RTM [5][6]; application of thin films of CNTs (buckypaper) [7][8];



deposits of nanofillers on commercial preregs [9][10]. Other approaches include modifying dry carbon fabrics by spraying epoxy and CNT on the carbon fabrics [11] or direct grafting of nanofillers onto carbon fabrics [12]. In each of the tested solutions, problems occur during fabrication and implementation into composites that in effect lead to insufficient enhancement of the electrical and mechanical properties of CFRP. For instance, excessively high viscosity of CNT-modified epoxy decreases its flowability and results in poor infiltration of CFRP layers [6].

In the composite industry thermoplastic nonwovens are commonly used as interlayers or surface finishing in CFRP. Applying a small amount of thermoplastic polymer makes the epoxy resin less brittle and improves the fracture toughness of the laminates [13][14]. Based on this, the new idea described in this paper involves fabrication of thermoplastic nonwoven fabrics containing CNTs which could be applied as interleaves in CFRP to improve not only the mechanical properties of composites, but also primarily their electrical conductivity. Such electrically conductive fabrics are of great interest nowadays due to their high potential for use not only in the composite industry, but also as electromagnetic absorbers [15], sensors [16], heating elements for aircraft de-icing [17] or in wearable electronics [18]. Recent studies have investigated various methods of producing nonwovens with conductive nanofillers, such as immersing the polymeric fabrics in a nanofiller solution [19][18], coating the fabrics with a thin layer of nanofiller [20], plasma treatment [21], ink printing [22], in an electrospinning process [23][24], by a melt-blown method [25] or by pressing thermoplastic fibers containing nanofillers [26].

Until now only a few papers have described the usage of thermoplastic nonwovens with CNTs as interleaves in CFRP manufactured by prepreg technology; however, they mainly describe the mechanical but not the electrical properties of CFRP. It was found that application of thin nonwovens containing CNTs produced by ultrasonication of polyethylene oxide and 15wt% MWCNTs resulted in an improvement of over 200% in Mode I and II interlaminar fracture toughness [27][28]. The authors of these papers suggested that this is due to increased adhesion between the epoxy and fibers of nonwoven fabrics. In other work, nanofibrous mats were produced from polysulfone by electrospinning and then kept in a carboxyl MWCNTs solution to cover the nanofibers [28]. Both Mode I and Mode II interlaminar fracture toughness were improved, but only by about 50%, and flexural strength was reduced due to the formation of microspheres of polysulfone and CNTs pulling out of the epoxy. The electrical conductivity of composite panels with inserted nonwovens containing CNTs has not been examined.

In the present study, we investigated the effect of thermoplastic nonwovens containing MWCNTs on the electrical, thermal and mechanical properties of CFRP. The nonwovens were produced by a half-industrial melt-blown pilot line by TMBK Partners company in Warsaw, Poland. In this technique, thermoplastic polymer is fed into the extruder, after which the melting material goes through the die orifices. Thin fibers are formed by application of high-speed hot air. These fibers are then deposited on a transport belt, thus creating the nonwoven web [29].

2. Materials and methods

Two types of thermoplastic copolyamides (coPA1 and coPA2) were selected as a polymer matrix; they have melting points of 108–118°C and 107°C, respectively. They were mixed with 3.5wt% of MWCNTs (NC7000) using a half-industrial twin screw extruder machine by Nanocyl, Belgium. Pellets containing 3.5wt% MWCNTs were processed directly using a melt-blown method. To decrease and homogenize the overall thickness of the nonwovens, they were thermally pressed below the melting point of copolyamides. The properties of the nonwovens are listed in Table 1, while macroscopic images are presented in Fig.1.

Table 1: Properties of nonwovens doped with MWCNTs.

Nonwoven designation	MWCNT content [wt%]	Areal weight [g/m ²]	Nonwoven thickness [mm]	Fiber diameter in nonwoven [μm]	Melting point measured by DSC [°C]
coPA1/3.5	3.5	30	0.12 ± 0.03	31.9 ± 10.7	113
coPA2/3.5	3.5	30	0.17 ± 0.05	40.9 ± 12.8	110

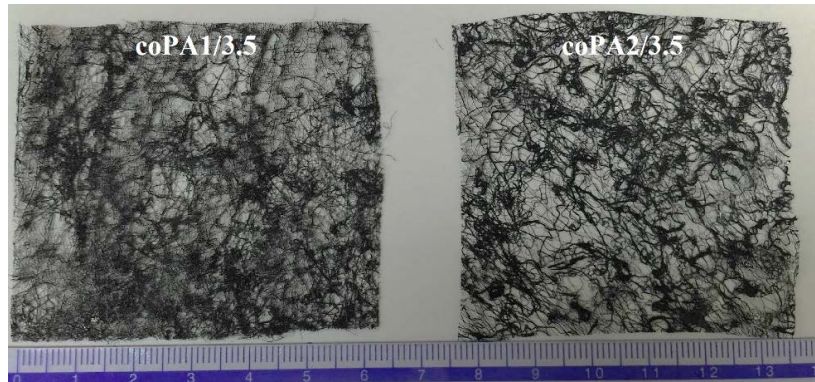


Figure 1: View of nonwoven coPA1/3.5 and nonwoven coPA2/3.5 produced by the melt-blown method.

The produced thermoplastic nonwovens with 3.5wt% MWCNTs were used as interlayers in prepreg technology. Three types of laminate were manufactured with the out-of-autoclave technique: a reference laminate without nonwovens (L1), a laminate with nonwovens made of coPA1+3.5wt% MWCNTs (L2) and a laminate with nonwovens made of coPA2+3.5wt% MWCNTs (L3). Each panel consisted of 13 layers of nonwovens placed between each layer of an unidirectional commercial prepreg with an areal weight 0.015g/cm². Laminates after lay-up were degassed, vacuum bagged on a glass tool plate and consolidated at 80°C for 60 minutes and then at 130°C for 120 minutes under a vacuum of 660–710 mmHg. After curing, composite plates were removed from the vacuum bag, trimmed, and cut into test specimen dimensions for electrical and mechanical measurements. The measured thicknesses of the laminates were as follows: 2.3mm (L1), 2.7mm (L2) and 2.7mm (L3). The calculated fiber weight fraction was 61% for L1, 55% for L2, and 54% 0.54% for L3.

Electrical conductivity of CFRP in directions K_z and K_x was measured. 5 samples with dimensions 85mm x 85mm were cut from different sections of the composite panels to measure the volume and surface electrical resistivity according to ASTM D-257 using a 6517B Electrometer/High Resistance Meter equipped with an 8009 Test Fixture. Before the test, each sample was degreased using acetone and the electrodes of the Test Fixture were cleaned with methanol. The sample was placed between stainless-steel electrodes, with a rubber pad which maintains perfect contact between the sample and electrodes. The voltage applied was 1V, time of the test was 15s, and the number of stored readings was adjusted to 5. Using this equipment, the electrical volume resistivity was measured throughout the laminate thickness. In order to measure the electrical volume conductivity along the carbon fibers (K_x), samples with 50mm length and 10mm width were cut from the composite panels. Before the test, each sample was degreased using acetone and covered with a silver paste tape to maintain good contact between electrodes and samples. The sample was placed between copper electrodes. Here, the variable current was set by a Keithley 6221 current source and the voltage change was read on a 2182A nanovoltmeter. In both tests, the final electrical conductivity was calculated, taking the dimensions and thickness of the samples into account.

Mechanical properties of the composite panels were analyzed in terms of the interlaminar shear strength (ILSS). A short beam shear test was conducted according to ASTM standard D2344 using an MTS Q/Test 10 universal testing machine. 8 specimens were cut from different places from each laminate in the direction of the carbon fibers, with dimensions of 25mm x 6.4mm. The test was performed at room temperature at a speed of 1 mm/min.

A Scanning Electron Microscope (SEM 3000, Hitachi, Japan) was used to analyze the microstructure and quality of the produced composite panels. For the observations, small pieces were cut from each laminate and finished using a polishing machine. Before the measurement, laminate cross sections were coated with a thin layer of gold. The applied voltage for SEM observations was 15kV. Using ImageJ software, the porosity in each panel was determined from a minimum 8 SEM images. The same microscope was used in the analysis of the composite panels after the ILSS test. In order to investigate

the dispersion and distribution of MWCNTs in the final composite panels, a high-resolution SEM SU70 (Hitachi, Japan) was used. For the observations, polished laminate samples were coated with an electrically conductive molybdenum layer of approximately 5 nm thickness. The acceleration voltage was 10kV.

Dynamic mechanical analysis (DMA) was performed using a Q800 DMA (TA Instruments, USA) in dual cantilever mode according to ASTM D4065-01. From each laminate, three rectangular specimens with 60mm length and 10mm width were cut and used in the test. The analysis was conducted from 0°C to 260°C with a heating rate of 2 °C/min at a frequency of 1Hz and amplitude of 30μm. The glass transition temperature (T_g) was determined from the peak value in the loss modulus curve.

3. Results

3.1. Electrical conductivity

Electrical properties of the CFRP panels were measured in terms of surface electrical conductivity and volume electrical conductivity along (K_x) and perpendicular (K_z) to the carbon fibers. As shown in Fig. 2a, it can be seen that implementation of nonwovens with 3.5wt% MWCNTs significantly improved the electrical conductivity throughout the laminate thickness. In comparison to the reference panel (L1), laminate L2 with nonwovens based on coPA1 has an electrical conductivity that is about 1882% higher. In the case of laminate L3, which has interlayers of nonwovens based on coPA2, this increase is much higher and was calculated as 8822%. The highest obtained electrical conductivity of CFRP with nonwovens containing 3.5wt% MWCNTs is 0.002 S/m through the laminate thickness. This was achieved for laminate L3, which incorporated nonwovens made of coPA2.

The in-plane electrical conductivity is higher than the out-of-plane electrical conductivity, which is obvious because of the parallelly laid carbon fibers. Incorporation of thermoplastic nonwovens with 3.5wt% MWCNTs leads to a tiny increase of about 47% and 63% for laminates L2 and L3, respectively, when compared to the reference panel (Fig. 2b). Such a small increase in electrical conductivity has already been reported in the literature: the increase of the in-plane conductivity by implementation of CNTs is only slight due to the presence of percolation pathways formed by carbon fibers [30]. Interestingly, the insertion of nonwovens with MWCNTs as interlayers also resulted in an increase of surface electrical conductivity, as shown in Fig. 2c. Electrical volume conductivity is dependent on the type of thermoplastic nonwovens used, but surface electrical conductivity is not. These differences will be explained in terms of the laminate microstructure in section 3.2.

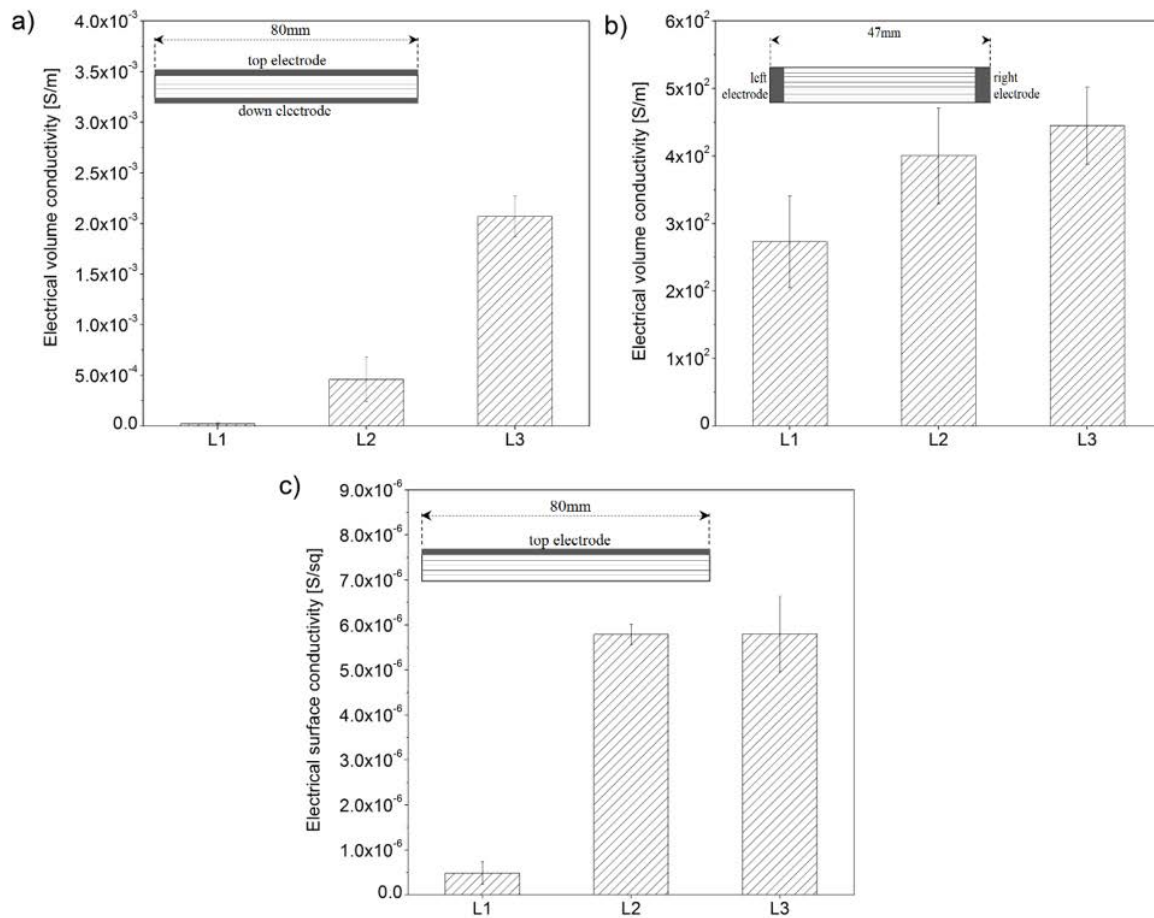


Figure 2: Volume electrical conductivity for CFRP panels in a) out-of-plane direction (throughout the thickness), b) in-plane direction (along the carbon fibers); c) surface electrical conductivity.

3.2. Microstructure

The typical microstructure of the CFRP reference panel cross-section is presented in Fig. 3a. There are visible layers of carbon fibers impregnated with the resin. The pore values determined by ImageJ, which are indicated as small black dots in the SEM images, equaled $0.53 \pm 0.25\%$, which proves the good quality of the panel. Microstructure images of the composite panel with inserted thermoplastic nonwovens are presented in Fig. 3b (L2) and Fig. 3c (L3). It can be observed that in both laminates the layers of melted nonwovens are not uniform in size through the whole panel cross-section. On one hand, this could be the result of insufficient compaction of the nonwoven layers. On the other hand, it could be due to the heterogeneous structure of the nonwovens. If the pores between the fibers are too small, the resin is not able to penetrate easily between the fibers, it is not mixed with the melted polymer, and therefore the formed layer is discontinuous. Hence, in some places the nonwovens remain as droplets. The other possible reason is that the nonwovens formed from the copolyamides have a trace amount of water which causes swelling of the polymer during CFRP manufacturing. Obviously, there are also layers which are homogenous and thin, thus confirming the influence of the nonwovens' structural parameters on the quality of the layers. Such variances in the thickness of the nonwoven layers affect the overall thickness of the CFRP panels after curing, which are higher for the CFRPs with nonwovens. Laminates L2 and L3 are about 0.5mm thicker than the reference panel. The creation of a non-uniform layer of melted nonwovens led to the formation in the epoxy of free spaces without thermoplastic polymer and therefore more voids. Hence, the determined porosity in the composite panel L2 is $0.58 \pm 0.40\%$, while in L3 it is only $0.31 \pm 0.11\%$, which is smaller than that calculated for the reference panel. Moreover, in the composite panels the pore shapes were not only circular but also elongated. Such

differences in laminate porosity and thickness can be related to the behavior of the nonwovens during CFRP manufacturing; this is related to the properties of the initial copolyamide + MWCNTs nanocomposites used to manufacture the nonwovens by the melt-blown technique. In particular, this could be an effect of the rheological properties and the varying dispersion of the nonwovens during melting. This means that the MWCNT cannot be uniformly dispersed in the CFRP, which affects the variances in the electrical conductivity improvement, as is presented in Fig. 2. Also, the non-uniform and local agglomeration of the thermoplastic copolyamides between the prepreg layers results in the varying electrical conductivity values of the laminates, which is difficult to explain in the context of the type of nonwovens used. Nevertheless, it indicates that the CFRP manufacturing process should be further improved to obtain laminates with the same thickness and porosity as well as uniform layers of melted nonwovens.

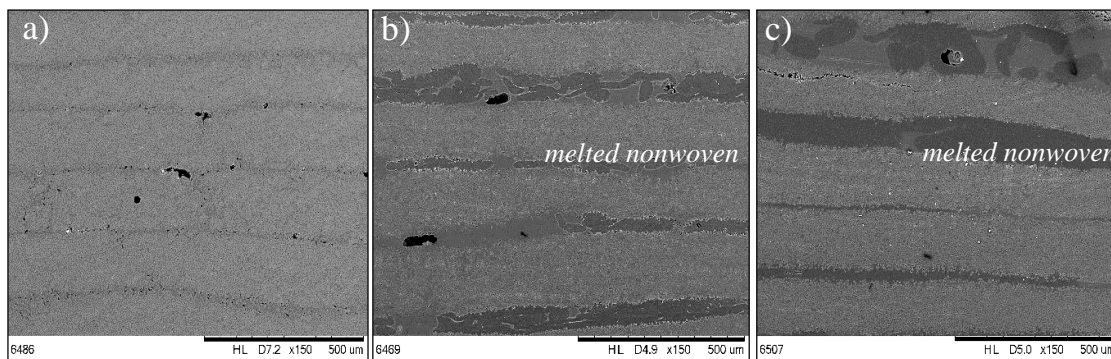


Figure 3: SEM images of cross sections: a) reference panel; b) panel with coPA1/3.5 nonwovens; c) panel with coPA2/3.5 nonwovens

In order to examine the dispersion and distribution of MWCNTs in CFRP, a high-resolution SEM was applied. The observations were performed for panel L2 and L3, but we did not find differences in the microstructure of these laminates. Therefore, the images presented in Fig. 4 show the microstructure of both L2 and L3 panels. Fig. 4a shows a melted layer of nonwoven that adheres perfectly to the carbon fibers. As has already been mentioned, it can be seen that the formed layers of nonwovens are not uniform in thickness. However, in some places the nonwovens occur as droplets after melting and the copolyamides used possess sufficiently high flow behavior to spread the polymer around the carbon fibers (Fig. 4b). The lack of voids in the boundary between the carbon fibers and the nonwovens confirms that the copolyamides used to produce the nonwovens have good adhesive properties and high compatibility with the carbon fibers. The dispersion of MWCNTs in the nonwovens after melting is shown in Fig. 4c. It can be seen that MWCNTs (white points) occur in a well-dispersed state and do not create agglomerates during the CFRP manufacturing process. Therefore, the electrical conductivity of these laminates can be improved because copolyamides doped with 3.5wt% MWCNTs are more conductive than the neat resin from the prepreg. MWCNTs create conductive pathways in the copolyamides and therefore the electric current which goes through the carbon fibers is easily conducted through the melted nonwovens. Obviously, to improve the overall electrical properties of the CFRP panel it is necessary to form continuous layers of the nonwoven throughout the whole laminate. In contrast, inhomogeneous layers of nonwovens introduce local agglomeration of the polymer without MWCNTs, which acts as an insulator. However, in such a situation single MWCNTs are pulled out of the thermoplastic polymer and have the tendency to create bridges, as presented in Fig. 4d. Thus, percolation pathways are created between neighboring nonwovens. Improving the electrical conductivity through the laminate thickness by formation of bridges between CNTs has been already suggested by Yamamoto et al. [31].

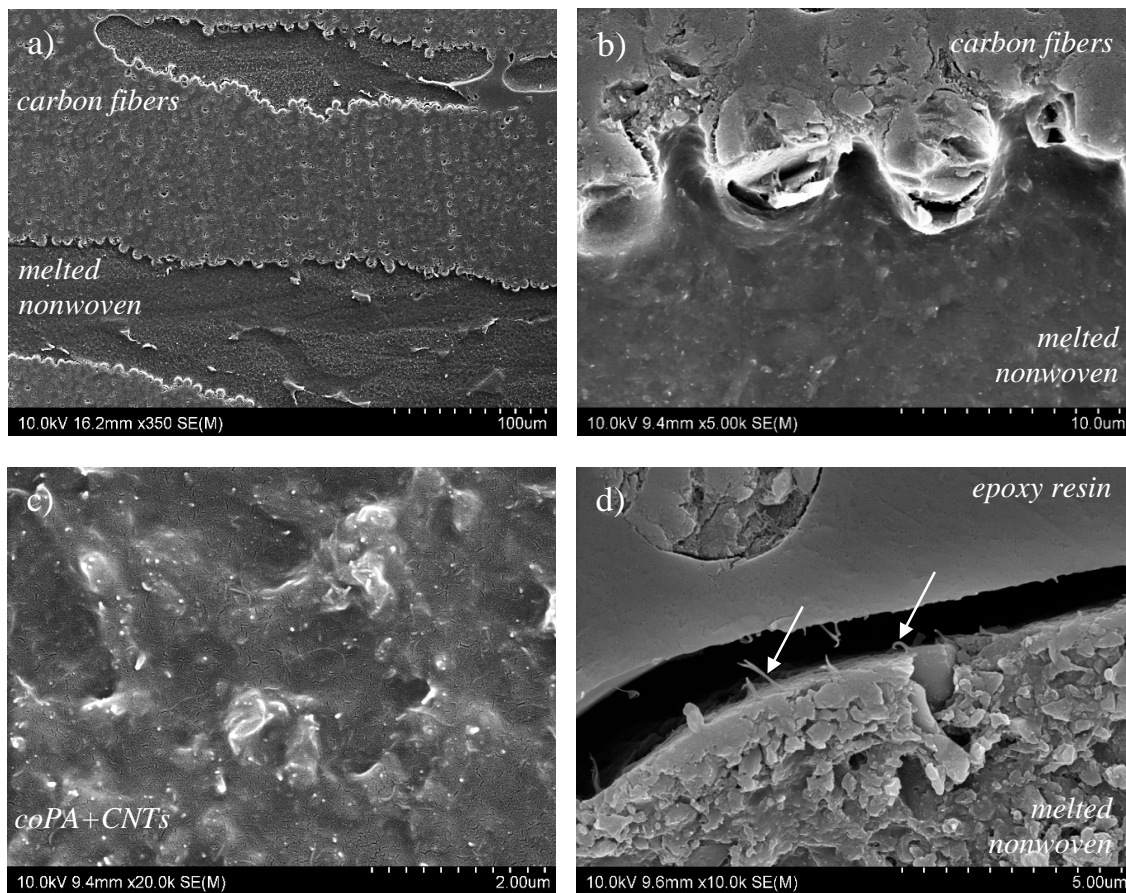


Figure 4: HRSEM images of CFRP panels containing nonwovens with CNTs: a) boundary nonwoven carbon fibers; b) melted nonwovens surrounded carbon fibers; c) well-dispersed CNTs in melted nonwoven; d) single CNTs (marked with arrows) between nonwoven layers.

3.3. Mechanical properties

In order to determine the resistance of the composite panels to delamination, an ILSS test was performed. It was expected that implementation of a small amount of thermoplastic polymer in the form of nonwovens between the carbon fabrics would prevent delamination of the CFRP. According to Fig. 5, the ILSS value determined for the reference panel L1 is 63.5 ± 5.4 MPa. In the presence of thermoplastic nonwovens containing 3.5wt% MWCNTs there is a slight decrease in the shear properties of CFRP for both types of nonwovens. For laminate L2 with nonwovens coPA1/3.5, the ILSS value is 53.7 ± 1.5 MPa, while for laminate L3 with nonwovens coPA2/3.5 it is 55.3 ± 3.01 MPa. The differences between these two values are negligible, which indicates no effect of the type of nonwovens used. Beylergil et al.[32] fabricated CFRP panels with interlayers of 17 and 50gsm thermoplastic PA 66 nonwovens. They observed only a slight increase in the ILSS of the reference panel that was independent of the areal weight of the nonwovens used. In the tested CFRP, the quality of the manufactured CFRP had a more significant impact on the interlaminar shear stress. Discontinuity of the nonwoven layers, local agglomeration of the polymer, and the presence of pores and voids affected the delamination process. As presented in the SEM images of composite panels after the ILSS test (Fig. 6), the reference panel cracked in one place along the whole length of the specimen (Fig. 6a). In the case of the laminates containing nonwovens and MWCNTs (Fig. 6b, c), the delamination occurred in a few places, but not in each layer between carbon fabrics. Presumably, the delamination is pronounced in the layers in which the nonwovens created droplets of melted polymer instead of a continuous layer. Moreover, in those layers MWCNTs were also not well-dispersed and therefore the interlaminar shear stress decreased.

According to the literature, the introduction of CNTs into CFRP prevents the delamination process, but this depends on the amount of nanofiller [33].

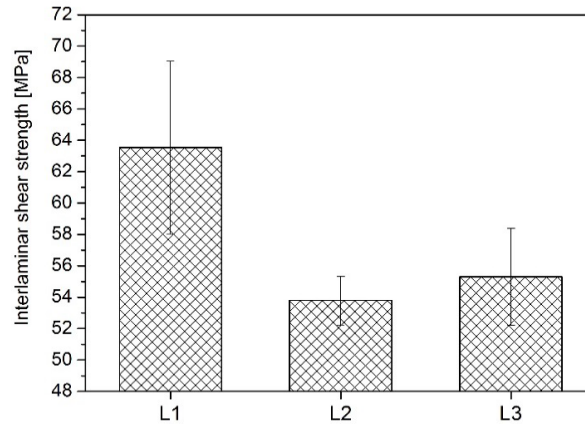


Figure 5: ILSS test results of fabricated CFRP panels without (L1) and with thermoplastic nonwovens containing 3.5 wt% MWCNTs (L2, L3).

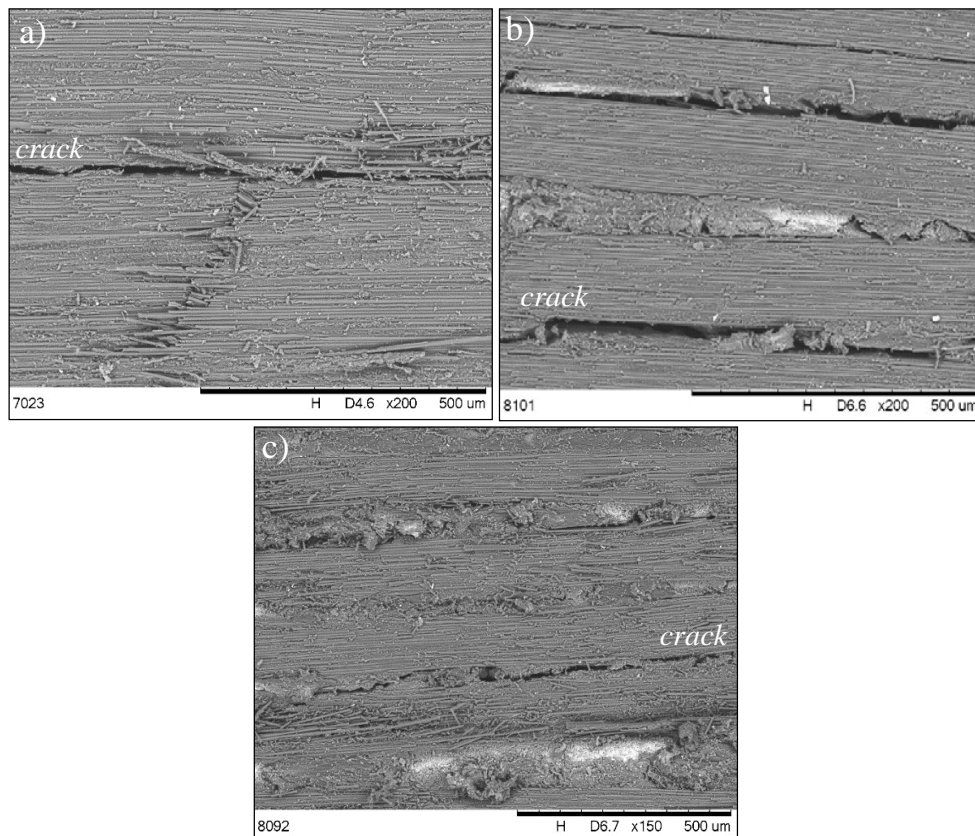


Figure 6: Microstructure of a) reference panel, b) panel L2 and c) panel L3 after ILSS test.

3.4. Dynamical Mechanical Analysis

The thermo-mechanical properties of the fabricated CFRP panels were measured using the DMA method as a function of temperature. From the loss modulus curves presented in Fig. 7, the changes in the glass transition temperatures as an effect of the addition of nonwovens was determined. The reference panel has T_g at 152.4°C. In the presence of thermoplastic nonwovens, the maximum of the T_g

peak increased. For laminate L2 containing nonwoven coPA1/3.5, it was around 161°C; for laminate L3 with nonwovens made of coPA2/3.5, it was around 170°C. The T_g values of the epoxy increased by a few degrees; this could be related to the presence of MWCNTs, as was also observed for CFRP fabricated with modified epoxy with CNTs [34]. The T_g temperature increase means that the composite panels containing copolyamide and MWCNTs should be stiffer than the reference panel. However, as presented in Fig. 7b, decreasing the storage modulus indicated that the introduction of thermoplastic polymer in the form of nonwovens increased the flexibility of the CFRP and therefore decreased their stiffness. This is also confirmed by ILSS results since laminates L2 and L3 have lower interlaminar strength (Fig. 5). An interesting phenomenon occurs in the loss modulus curves for the CFRP with implemented nonwovens. For both laminates the additional peak at around 41°C appeared, but this was not found in the CFRP panels reinforced by nonwovens made of PA66 [32]. This is due to the thermoplastic copolyamides used as a polymer matrix in nonwovens and indicates the presence of polyamide 6 segments in both copolyamides [35]. Presumably, the thermoplastic nonwovens used were too thick, which led to local agglomerations of the polymer. Hence, the overall mass of the introduced polymer was too high, as expressed by the additional peak in the loss modulus curve.

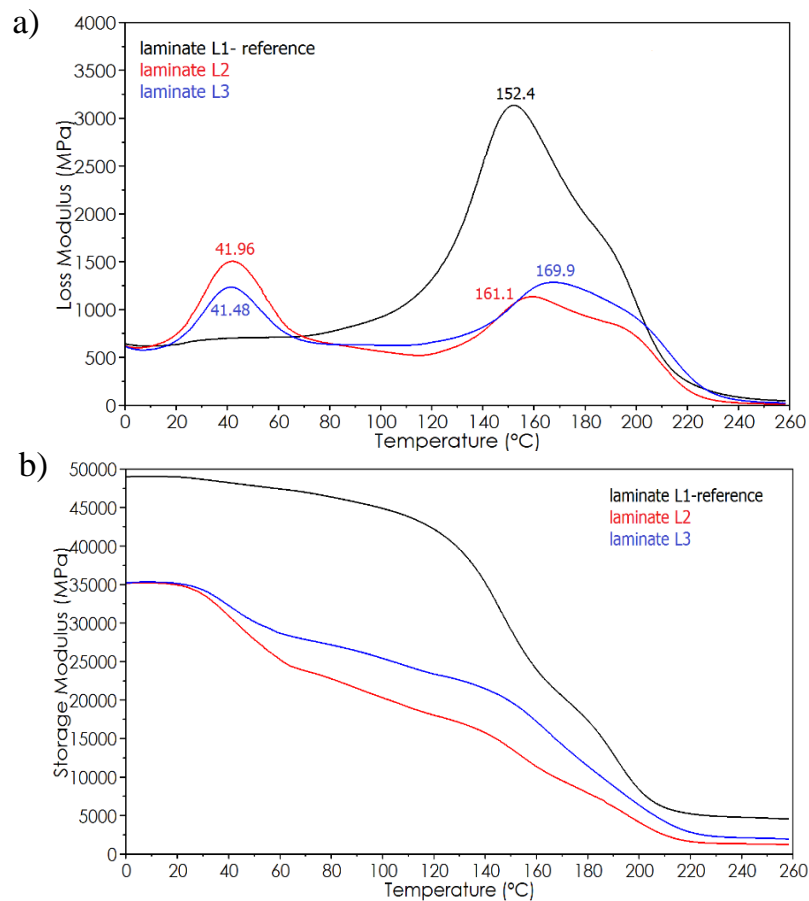


Figure 7: a) The loss modulus curves with marked T_g temperatures and b) the storage modulus curves obtained for CFRP composite panels by DMA test.

4. Conclusions

In this study, we demonstrated the effect of novel types of thermoplastic nonwovens containing 3.5wt% MWCNTs on the electrical and mechanical properties of CFRP. Two types of nonwovens based on different types of copolyamides (coPA1 and coPA2) and 3.5wt% of MWCNTs were fabricated directly from nanocomposite pellets by a half-industrial melt-blown method. Due to their high

flexibility, implementation of the nonwovens was easily achieved by a standard prepreg method; this resulted in good quality laminates with less than 1% pore content. The electrical properties measured along the carbon fibers increased slightly due to the existing percolation pathways. However, both laminates containing nonwovens made of coPA1 and coPA2 had electrical volume conductivity through the laminate thickness which was about 1882% and 8822% higher than the reference panel, respectively. When the electrical surface conductivity of the composite panels was not affected by the type of nonwoven used, the electrical properties through the laminate thickness were dependent on the structural parameters of nonwovens. The SEM images showed that the formed layers of melted nonwovens differed in thickness and were discontinuous in some places. It was suggested that the excessively small pores in the nonwovens prevented the resin from flowing freely between the nonwoven fibers, which led to insufficient impregnation. This caused the inhomogeneity in the layers and the variances in the electrical and mechanical properties of the laminates. Nevertheless, the high-resolution SEM images confirmed that copolyamides+3.5wt% MWCNTs have sufficiently high flow behavior and compatibility to surround and adhere to the carbon fibers. Moreover, it was confirmed that MWCNTs were well dispersed in the nonwoven fibers after the melt-blown process. The analysis of the mechanical properties in terms of the interlaminar shear strength showed that in the presence of thermoplastic nonwovens containing 3.5wt% MWCNTs, there was a slight decrease in the shear properties of CFRP from 63.5 MPa for the reference to 53.7MPa and to 55.3MPa for laminates interleaved with coPA1/3.5 and coPA2/3.5, respectively. Introduction of thermoplastic nonwovens into CFRP also increased the resin glass transition temperature by about 9°C for L2 and 17°C for L3, as examined by DMA analysis. What is more, the additional peak in the loss modulus curves for laminates with nonwovens at 41°C was caused by the thermoplastic copolyamide and appears to be caused by the excessively high overall mass of the polymer introduced into the CFRP.

Acknowledgments

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