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RELATIONSHIP BETWEEN ELECTRICAL, MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF CFRPS BASED ON THERMOPLASTIC ACRYLIC RESIN

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This study examines the effect of incorporating single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) into carbon fiber reinforced polymers (CFRPs) based on Elium® thermoplastic acrylic resin and investigates the relationship between the studied properties. SWCNTs exhibited better dispersion in the matrix, which leads to better electrical conductivity (2.72 ± 0.34 S/m) and impact resistance (154 ± 14.6 kJ/m²) compared to MWCNTs. Microstructural analysis revealed a defect-free architecture of the SWCNT-modified laminates, while the MWCNT laminates showed small voids and agglomerates. The increased dispersion and interconnectivity of the SWCNTs contribute to an EMI shielding efficiency of 24.6 dB, a 30% improvement over the unmodified samples. These findings highlight the potential of SWCNTs to improve the multifunctional properties of thermoplastic CFRPs, including mechanical strength, electrical performance and EMI shielding capability, making them highly suitable for advanced aerospace, electronics and power applications. Moreover, the recyclability and lightweight nature of the Elium® resin matrix make these composites environmentally friendly and an alternative to traditional materials in a variety of industrial contexts.

Keywords: **CFRP, Elium® resin, carbon nanotubes**

INTRODUCTION

In today's rapidly advancing technological era, the prevalence of electromagnetic fields (EMs) is becoming increasingly common due to the widespread use of electronic devices. EMs are generated by various electrical and electronic devices such as televisions, mobile phones, computers, and wireless networks [1, 2]. While these technologies bring numerous benefits, there is growing concern about the potential risks associated with exposure to electromagnetic fields. The adverse effects of electromagnetic fields may compromise people's health and safety, including interference with medical devices, alterations to the nervous system, and even potential risks of developing cancer [3,4]. As a result, researchers, engineers, and scientists are striving to develop methods and materials that can protect against these electromagnetic fields. One such material with great potential is carbon fiber reinforced polymers (CFRPs) [5-7], which are widely used in the aerospace, automotive, construction, sports and energy industries [8-12].

CFRPs may be effective electromagnetic shields for several reasons. Firstly, carbon fibers in the composite possess high electrical conductivity, allowing efficient absorption and dissipation of electromagnetic energy [13]. Secondly, the composite structure is well-organized, facilitating the conduction of electrical current and preventing the penetration of electromagnetic waves [14]. Moreover, CFRPs are corrosion-resistant, providing durable protection against environmental factors, making them more versatile than metallic materials [15]. Lastly, owing to their light weight and strength, CFRPs can be used as thin and efficient electromagnetic shields that can be easily tailored to different applications [16]. Modifying the structure of CFRPs by incorporating conductive nanoparticles into the polymer matrix is one way to improve the effectiveness of electromagnetic interference (EMI) shielding. Examples of such nanoparticles include graphene, carbon nanotubes and metallic powders. The addition of these conductive materials creates a conductive path inside the CFRP, resulting in effective EMI shielding [17,18]. Furthermore, the addition of carbon nanotubes to the polymer matrix improves the mechanical properties of CFRPs, including impact resistance [19, 20]. Accordingly, carbon nanotubes improve the interfacial bonding between the carbon fiber and the matrix, and consequently, improve the mechanical properties of CFRPs.

Conventional CFRPs are manufactured based on thermosets; however, thermoplastics are increasingly being used in production. CFRPs based on thermoplastic resins offer unique properties and numerous advantages. These composites combine the strength and stiffness of carbon fibers with the versatility and processability of thermoplastics [21, 22]. They exhibit excellent mechanical properties such as high tensile strength, stiffness and impact resistance, making them ideal for structural applications [23]. They can be repeatedly processed without significant loss of mechanical properties, allowing easy repair, recycling and reshaping of composite parts, leading to cost savings and sustainability benefits [24-26]. One approach under development is the use of Elium® thermoplastic acrylic resin, which combines the characteristics of both thermoplastics and thermosets. It offers advantages such as low viscosity, high strength, light weight, adjustable reactivity, excellent UV resistance, and the ability to cure at room temperature. In addition, Elium® resin is environmentally friendly because it can be recycled by means of chemical and mechanical processes, and is free of toxic components such as styrene and bisphenol A (BPA). These properties make Elium® resin suitable for a variety of industries, including wind power, marine, construction and transportation.

This study investigates the effect of adding single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) as a nanofiller in CFRP laminates based on Elium® thermoplastic acrylic resin. The system proposed in this study may solve the difficulties of weakening EMI shielding using a conductive resin. The main aim of this study is to compare the properties of composites with MWCNTs and SWCNTs as nanofillers as well as evaluate the EMI shielding ability of such materials.

MATERIALS AND METHODS

Materials

Elium® 188XO acrylic liquid thermoplastic resin (Arkema, France) was used as the CFRP matrix, where dibenzoyl peroxide powder (Acros Organics, Geel, Belgium) was employed as the initiator of the polymerization reaction. Tuball™ SWCNTs (OCSiAl, Leudelange, Luxembourg) and MWCNTs (Nanocyl, Belgium) were selected as the nanofillers to modify the polymer matrix. Unidirectional (UD) carbon fabric with a 600 gsm aerial weight (Saertex, Saerbeck, Germany) was utilized to manufacture the CFRPs.

Manufacturing

The addition of SWCNTs or MWCNTs in a proportion of 0.02 wt% to the Elium® acrylic resin was dispersed by an ultrasonic process, which was conducted by means of a VCX 1500 ultrasonic processor (Sonics & Materials, Newtown, CT, USA) with a maximum frequency of 20 kHz. The ultrasonic process was carried out with the following parameters: process time 1 h, amplitude 40%, ultrasonic wave activity 10 s and time between ultrasonic waves 14 s. The mixture prepared in this way was used to produce CFRPs by the infusion technique under a vacuum pressure of 0.9 bar. Unmodified and modified Elium® resin was mixed with 2.0 wt% benzoyl peroxide before the process. Three different laminates were prepared: a reference sample (L_REF), with the addition of SWCNTs (L_SWCNT), and MWCNTs (L_MWCNT).

Methods

The macrodispersion state of the SWCNT or MWCNT and Elium® acrylic resin mixtures was evaluated by transmission light microscopy. The prepared mixtures were placed on glass slides and observed under a PZO Biolar transmission microscope (Biolar, Warsaw, Poland). Microstructure observations of the fabricated laminates were performed utilizing a scanning electron microscope (SEM, SU-70 Hitachi, Tokyo, Japan), where the accelerating voltage was 5 kV. To prepare the samples (10 × 10 mm), they were ground with sandpaper of five different grain sizes (P240, P600, P1200, P2500, P4000) and then polished with diamond suspension containing 1 μm particles. A 3 nm thick Au-Pd layer was then deposited using a voltage of 2 kV, a current of 15 mA and a deposition time of 90 s to ensure electrical conductivity.

The influence of introducing SWCNTs and MWCNTs into the structure of the laminates on their impact resistance was evaluated using a Charpy test performed on a Zwick Roell RKP450 (with a maximum impact force of 300 J). All the tests followed the EN ISO 179 standard. Ten unnotched specimens were tested for each material, and the impact resistance was calculated based on the energy absorbed during fracture under EN ISO 179.

The electrical conductivity of the fabricated CFRPs was measured by means of a Keithley 6221/2182A device connected to a measuring stand with copper electrodes. The measurement was performed using the four-point method and in delta mode to ensure a reduction of noise and thermoelectric effects. The electrical conductivity test was performed through the laminate thickness, where the test specimens (5 pieces for each laminate) were 10 x 10 mm in size and were cut from different locations on the panel.

Shielding effectiveness (SE) was measured in GTEM cell (gigahertz transverse electromagnetic cell). A signal with an amplitude of 40 dBm from 300 MHz to 1 GHz generated with the NSG4070 was fed into the cell. The measurement consisted of two steps: 1) The measurement of magnitude of reference electric field E_{ref}: in this step measured by the probe (Loomi loop LS PROBE 1.2), which was placed in the cell at half of the distance between the floor and the septum to obtain the maximal field strength. 2) The measurement of electric field magnitude transmitted through the shielding: in this step, the measuring probe was placed behind the composite sample to be measured. The measuring probe was connected to the transducer with an optical fiber cable. Measurement control (setting of the measurement parameters, calibration, the triggering of individual measurements and recording of results) was performed employing WIN 6000 software from TESEQ. SE was defined as the logarithm of the ratio of the magnitude of the electric field E_{ref} incident on the shielding material to the magnitude of the electric field transmitted through it. It is usually expressed in decibels (dB) and is described by the formula:

$$E = 20 \log_{10} \frac{E_{ref}}{E_{mat}}$$

RESULTS AND DISCUSSION

Macrodispersion of SWCNTs/MWCNTs

Obtaining a uniform dispersion of the nanofiller in the polymer matrix is one of the challenges during the fabrication of nanocomposites and affects the obtained properties of both the nanocomposites and the CFRPs, especially the mechanical and electrical properties. To this purpose, the macrodispersion of SWCNTs and MWCNTs in the Elium® thermoplastic acrylic resin was studied before the polymerization process and the fabrication of CFRPs. The results, which were obtained for the blends produced with the same mixing parameters, are presented in Figure 1. In the case of the Elium/SWCNT mixture (Figure 1a), a more uniform distribution of SWCNTs in the polymer matrix was observed than in the case of the MWCNTs. This could be caused by the fact that SWCNTs have lower intermolecular energy than MWCNTs since there are no interlayer interactions (as in MWCNTs). Also, the multilayer nature of MWCNTs could result in difficulties in uniform wetting of the nanotubes by the resin, leading to agglomeration.

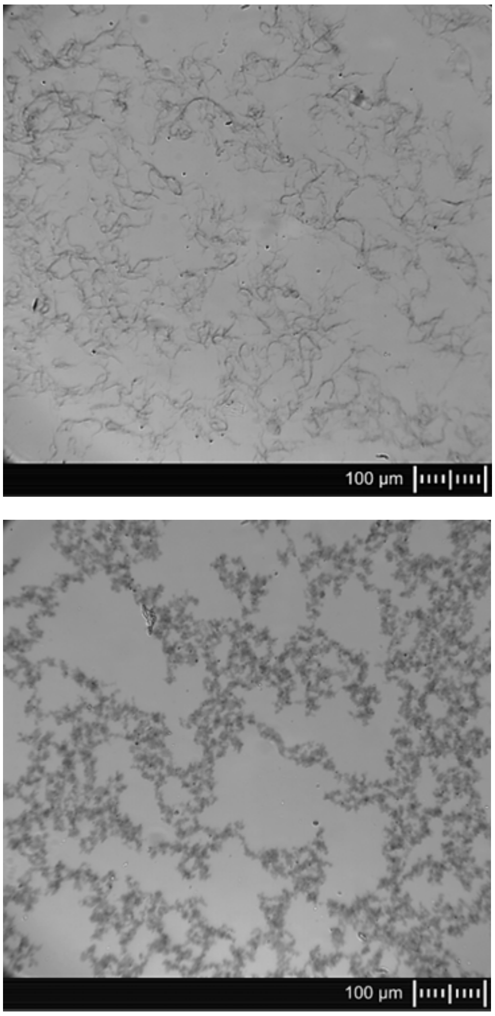

Fig. 1. Macrodispersion in thermoplastic acrylic resin a) SWCNTs, b) MWCNTs

Microstructure observations

In order to evaluate the quality of the fabricated laminates and the infusion process conducted using the modified acrylic resin, microstructure observations were performed by means of SEM, and the obtained micrographs are presented in Figure 2a-2c. The L_REF and L_SWCNT laminates were defect-free; no voids or air bubbles were observed, and also in the case of the SWCNT addition, all the carbon fabric layers were very well infiltrated. In the case where MWCNTs were applied to modify the Elium® acrylic resin, single voids were noticed in the laminate structure (Figure 2c). This was likely due to the less uniform distribution of the MWCNTs in the polymer matrix and visible agglomerates.

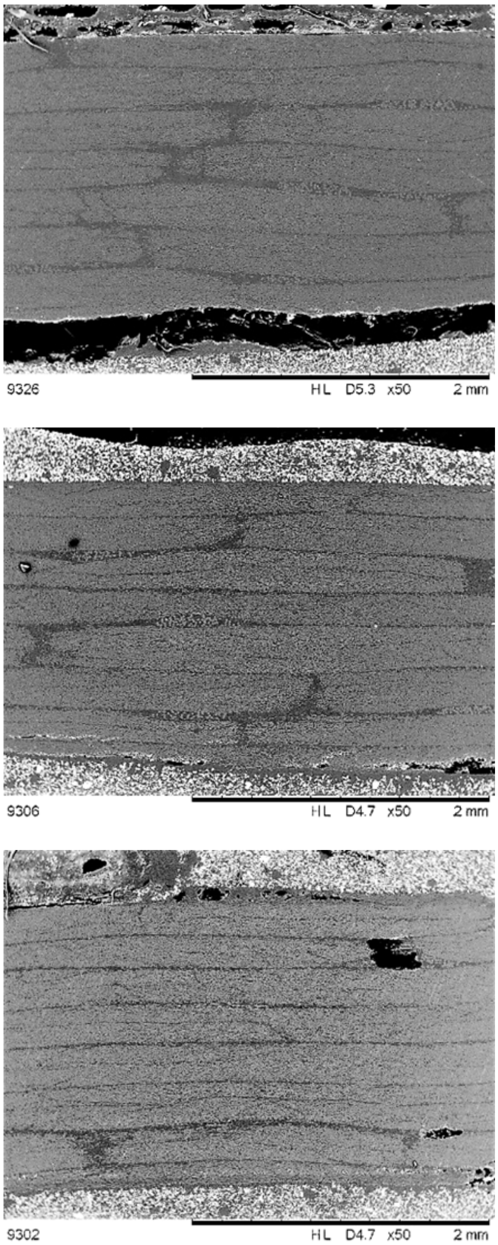

Fig. 2. Microstructure observations of a) L_REF, b) L_SWCNT, c) L_MWCNT

Electrical and mechanical properties

The effect of modification of Elium® thermoplastic resin by adding SWCNTs or MWCNTs on the electrical and mechanical properties of the CFRPs was investigated, and the results are presented in Figure 3. Starting with electrical conductivity, the lowest value was noted for L_REF and was 0.76 ± 0.08 S/m. Adding SWCNTs or MWCNTs to the resin improved the electrical conductivity of the CFRPs. For L_SWCNTs, a value of 2.72 ± 0.34 S/m was obtained, and for L_MWCNTs, the conductivity was 1.41 ± 0.11 S/m, respectively. The introduction of CNTs into Elium® resulted in the formation of conductive networks in the layers between the carbon fabrics [27-29], and this resulted in an increase in electrical conductivity. Comparing the electrical conductivity values between L_SWCNT and L_MWCNT, higher values were obtained for L_SWCNT, which was attributed to better dispersion of the nanoparticles in the polymer matrix. As for the impact resistance, as in the case of electrical conductivity, the lowest value (138 ± 12.5 kJ/m2) was obtained for L_REF. The addition of SWCNTs to the resin resulted in an improvement of 11.5% and a value of 154 ± 14.6 kJ/m2 was obtained. The addition of MWCNTs to the resin also resulted in an increase, but not as noticeable as for SWCNTs; the improvement was 7.2% and the impact value was 148 ± 14.6 kJ/m2. The enhancement in impact resistance following the incorporation of SWCNTs or MWCNTs was attributed to mechanisms like the pullout and bridging of CNTs, which played a significant role in improving the fracture toughness of the CFRPs [30]. Based on the obtained results, a statistical analysis was also carried out employing one-way ANOVA and then a Tukey post-hoc test to compare the impact resistance values of L_REF, L_SWCNT and L_MWCNT. The analysis was performed with a significance level of $p < 0.05$. The results show that the difference between L_REF and L_SWCNT is statistically significant ($p < 0.05$), confirming the effectiveness of SWCNT reinforcement in improving impact resistance. Nevertheless, the difference between L_REF and L_MWCNT, as well as between L_SWCNT and L_MWCNT, proved to be statistically insignificant ($p > 0.05$), which suggests that the improvement observed for the MWCNTs may be within the range of measurement variability, possibly resulting from the weaker dispersion of CNTs in the acrylic matrix for L_MWCNT than L_SWCNT.

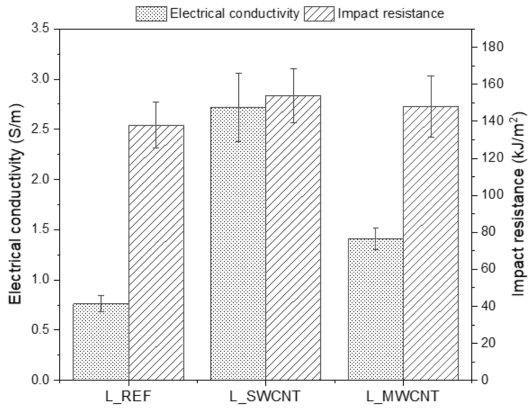

Fig. 3. Obtained properties of manufactured laminates

Shielding effectiveness

The achievement by the CFRPs of SE values above 10 dB for frequencies above 450 MHz is the basis for considering it as an electromagnetic radiation absorbing material and having justification for use in many industrial applications. Literature reports indicate that materials with an electromagnetic field shielding efficiency of 10 dB can prevent up to 90% of the delivered energy from passing through, and up to 99% of the energy in the case of a shielding efficiency of 20 dB [31, 32]. Analyzing the results presented in Figure 4, it could be concluded that all the fabricated laminates meet this requirement. The highest average value of electromagnetic field shielding was obtained for L_SWCNT and was 24.6 dB, which represented a 30% improvement over L_REF, where the average value was 18.8 dB. The increase in the obtained SE can be attributed to the much higher electrical conductivity of the L_SWCNT laminate compared to L_REF, which is one of the factors affecting the level of shielding efficiency [33], as well as the absence of defects in the structure of the L_SWCNT laminate. In the case of the L_MWCNT composite, despite the slightly higher electrical conductivity, a lower SE efficiency was obtained. This may be attributed to the much less uniform dispersion of the MWCNTs in the polymer matrix, as well as visible defects in the microstructure.

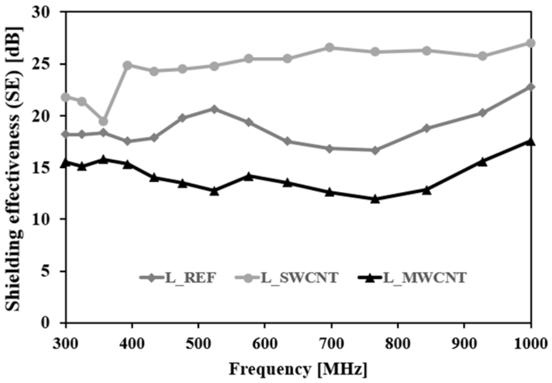

Fig. 4. SE results for analyzed laminates

SUMMARY

The study focused on the incorporation of SWCNTs and MWCNTs in CFRPs based on Elium® thermoplastic acrylic resin. The work was aimed at facing the challenges of increasing the electromagnetic interference (EMI) shielding effectiveness in addition to achieving excellent mechanical and electrical properties, which are crucial for advanced aerospace, electronics and industrial applications, as well as examining the correlation between the two. The research involved the preparation of three types of CFRP laminates: a reference laminate (L_REF) without nanofillers, and two modified variants containing SWCNTs (L_SWCNTs) and MWCNTs (L_MWCNTs) at a concentration of 0.02 wt%. Dispersion of the nanofillers was achieved by ultrasonication, using precisely controlled parameters to ensure optimal distribution of the nanotubes in the resin matrix. The laminates were then fabricated by means of a vacuum-assisted infusion technique, ensuring a uniform structure. A detailed analysis of the macro- and microstructural properties of the laminates was carried out utilizing light microscopy and SEM. The SWCNTs showed better dispersion compared to the MWCNTs, as evidenced by the absence of voids and agglomerates in the L_SWCNT laminates. In contrast, the MWCNT-modified laminates exhibited localized defects and uneven dispersion, which negatively affected their performance. Mechanical characterization using Charpy impact tests revealed notable improvements in the impact resistance for L_SWCNT (154 ± 14.6 kJ/m²) compared to L_REF (138 ± 12.5 kJ/m²) and L_MWCNT (148 ± 16.6 kJ/m²). These improvements were attributed to mechanisms such as nanotube pullout and bridging, which increased the fracture toughness of the composite. The electrical conductivity measurements showed significant improvements for the SWCNT-modified laminates (2.72 ± 0.34 S/m) compared to both L_REF (0.76 ± 0.08 S/m) and L_MWCNT (1.41 ± 0.11 S/m), correlating with the improved SWCNT dispersion and the formation of conductive networks in the resin matrix. The electromagnetic shielding (SE) performance tests confirmed that all the laminates exceeded the industrially relevant threshold of 10 dB at frequencies above 450 MHz. Nonetheless, L_SWCNT achieved a maximum SE of 24.6 dB, a 30% improvement over L_REF, while L_MWCNT performed worse because of microstructural defects and reduced conductivity. The observed shielding performance was mainly attributed to the electrical conductivity of the composite, as well as the uniformity of the nanofiller distribution. This study underscores the use of SWCNTs as effective nanofillers to improve the multifunctional properties of CFRPs based on thermoplastic acrylic resins.

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