High strength metallurgical graphene as an additional reinforcing phase for carbon fibre composites

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Abstract

The main objective of the present study is an elaboration of a method for a transfer of high strength metallurgical graphene (HSMG) and its introduction into the structure of fibre laminates. In this work, two transfer procedures have been established: a transfer onto structural adhesive film (SAF) and a direct transfer onto two types of carbon fibre pre-impregnates (uniaxial and biaxial cloth). An important novelty of the presented experimental work is a use of scanning electron microscopy (SEM) operating in the absorbed electrons (AE) mode. This technique enables the evaluation of graphene continuity on a large area within a relatively short time. The SEM/AE technique made it possible to ascertain that the transferred graphene was characterised by a low level of defects, indicating that it could be used as a reinforcing phase in composites. Both transfer procedures, one onto SAF and the other directly onto the pre-impregnates were used for manufacturing flat laminates as well as tubular profiles. The flat samples, containing 0.68 ppm HSMG by weight, exhibited a 9% increase of low cycle fatigue strength. The tubular profiles (bicycle components) with approximately 0.22 ppm of HSMG exhibited a significant increase of fatigue wear during initial tests. In addition, no fatigue delamination was observed at interfaces between the pre-impregnates separated with HSMG graphene.

Keywords Composites · Graphene · Mechanical properties · Resins · Fibers

1 Introduction

A number of reports in the literature describe an application of various forms of graphene flakes as a reinforcing phase in polymer matrix composites [1–15]. A number of reports in the literature describe the application of various forms of graphene flakes as a reinforcing phase for polymer matrix composites [1–15] inter alia polyethylene (PE) [1, 2, 8, 12], poly(vinyl alcohol) (PVA) [3, 10, 13], polymethylmethacrylate (PMMA) [4], poly(vinyl chloride) (PVC) [6], polypropylene [7], polyimide (PI) [9], polyamide (PA) [5, 11] or epoxy resins [14, 15]. An addition of 6 wt% of graphene nanosheet (GNS) to a PE matrix resulted in an

increase of its elastic modulus from 520 to 580 MPa [1]. Also in the case of a composite based on ultra-high molecular weight polyethylene (UHMWPE), the addition of 1 wt% of graphene oxide (GO) stimulates an increase of its tensile modulus from 864 to 1236 MPa [8]. As Li and Xie report 0.2 wt% of GO blended with high-density polyethylene (HDPE) composite results in an increase of stress at break from 18.1 to 24.2 MPa and of strain at break from 850 to 1941% [12]. The effect of reinforcing the PI matrix by the addition of 2 wt% of functionalized graphene sheet (FGS) was also demonstrated, by an increase of tensile strength from 88.4 to 118.5 MPa [9]. The addition of 4 wt% of chemically modified graphene (CMG) to the PMMA showed an increase of the modulus from 3.43 to 4.53 GPa [4]. In turn, the addition of 0.6 wt% of functionalized graphene (FG) to a composite based on PA improved tensile strength by 35%, and elongation at break by 200% [5]. PP based composite with the addition of 1.9 vol% of FG showed an increase of modulus from 0.6 to 1.16 GPa [7]. The addition of 0.1 wt% of reduced graphene oxide (rGO) to the PA composite causes a increase of strength by 210% and of modulus by 240% [11]. Composite manufactured as a blend of 2 wt%

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of graphene nanoflakes with PVC matrix improves tensile strength from 24 to 55 MPa, as well as increases modulus from 0.8 to 2 GPa [6]. The addition of 0.8 wt% of partially reduced graphene oxide (prGO) to the PVA polymeric base improves tensile strength from 32 to 69 MPa [3]. In turn, 3.5 wt% of GO added to the PVA resulted in an increase of its tensile strength from 22 to 29 MPa [13]. Songs group manufactured a composite material as a graphene/PVA sandwich [10]. It is also possible to make a composite based on epoxy as a matrix with the addition of various graphene fillers [14]. For example, 0.2 wt% of thermally reduced graphene oxide used as filler in epoxy composite improves its elastic modulus from 2.9 to 3.1 GPa [15].

In addition, both graphene flakes and large-surface graphene produced by chemical vapour deposition (CVD) have been applied as an auxiliary reinforcing phase for carbon fibre reinforced polymer (CFRP) composites [16]. The reports in the literature regarding composites of this type discuss only reinforcement of the matrix with graphene flakes, and the results are neither unambiguous nor consistent.

In Ref. [17], an increase of tensile strength of epoxy resin from 55 to 66 MPa was obtained through the application of 0.25 wt% of graphene nanoplatelets (GNPs). In Ref. [18], the reinforcement of an epoxy resin polymer matrix with 0.54 vol% of graphene oxide (GO) resulted in an increase of its tensile strength from 72.6 to 79 MPa. Similarly, an increase from 77.52 to 82.31 MPa was obtained in Ref. [19] by adding 1 wt% of GNPs to an epoxy resin polymer matrix that was additionally reinforced with a single layer of carbon fibre (CF).

For multi-layer fibre composites, reinforcement effects were observed through significantly higher relative values of their mechanical properties. Mannov et al. [20] demonstrated a 53% increase of critical stress (from 170 MPa at 0 wt% GO to 260 MPa at 0.3 wt% GO) for a pre-impregnated composite consisting of an epoxy matrix filled with GO and additionally reinforced with glass fibre reinforced polymer (GFRP). For similar pre-impregnated composites reinforced with CFRP, a 19% increase of compressive strength was obtained (from 150 to 170 MPa with the addition of 0.3 wt% GO). Moreover, Shen et al. [17] created a 13-layer pre-impregnate using carbon fibre reinforced plastic (CFR plastic) to reinforce an epoxy matrix filled with GNPs. As a result, its strength was increased from 419 MPa for 0 wt% GNPs to 446 MPa for 0.25 wt% GNPs. In Ref. [21], a hybrid epoxy composite was fabricated, filled with different wt% of GO, and reinforced with CF. An increase by 66% of bending critical strength (420 MPa for 0 wt% GO to 700 MPa for 0.3 wt% GO), 72% in the bending modulus (20,000 MPa for 0 wt% GO to 330,000 MPa for 0.3 wt% GO), and by 25% of shear strength between layers (32 MPa for 0 wt% GO to 41 MPa for 0.3 wt% GO) was observed in that study.

In all the cases described above, graphene flakes constitute a random geometric filling of the polymer matrix. On the other hand, the directional impact of an ordered (laminar) reinforcement on the mechanical properties of composite materials is well known. A laminar arrangement of the reinforcement can be achieved by introducing CVD large-surface graphene to the composites; however, there are no reports on that subject in the literature. The transfer technology is a likely problem in this case. One possible solution is the application of high strength metallurgical graphene (HSMG), characterised by high strength and a potential for effective transfer [22–28]. This original 2D material is grown on the surface of liquid copper in the form of quasimonocrystalline monolayer [22, 23, 28]. It exhibits semi-conductive behaviour [27, 28], in a consumption useful properties of various new sensors [24]. It also offers high strength and a affinity to gaseous hydrogen, which may be used in a manufacture nanocomposites for reversible storage of hydrogen [25, 26].

This study describes the direct transfer of large-surface graphene from metal process substrate onto pre-polymerised epoxy resin and two different types of pre-impregnates (unidirectional and bidirectional). An attempt to create a CFRP composite reinforced with HSMG using the two intermediate products is also described.

2 Materials and methods

2.1 Materials

In this study, the following materials were used: HSMG grown on a Cu/Ni bimetallic substrate (according to patent no. EP2865646 A1), structural adhesive film (SAF) of 3 M™ Scotch-Weld™ AF 163-2 60 µm (USA), unidirectional pre-impregnate [unidirectional carbon fabric with a resin content of 33% and grammage of 125 g/m² (SGL Group)], bidirectional pre-impregnate [bidirectional carbon fabric, plain, grammage of 200 g/m² with a resin content of 42% and thickness of 0.2 mm (Hexcel-HexPly)], anhydrous FeCl₃ (Sigma-Aldrich), 40% HF solution (Chempur), and H₂O.

2.2 Identification of graphene morphology and structure on the forming matrix

Preliminary inspection of the graphene continuity consisted of submerging the substrate in pure water for 3–5 min to reveal major discontinuities in the HSMG. A more detailed inspection was conducted within small areas by applying droplets of a strongly oxidising 0.1 M aqueous solution of FeCl₃ or through oxidation with a 30% solution of H₂O₂. This allowed one to obtain an information on the nature of

defects in the form of growth shortages and cracks in the graphene layer, as well as on the graphene growth mechanism. An evaluation with the droplet oxidation method was performed at several points on the growth substrate. A drop of FeCl_3 remained in contact with the substrate for approximately 5 s, while a drop of H_2O_2 was in contact for 20 s. Following oxidation time, the sheet of metal was rinsed thoroughly with deionised water. Changes in the growth surface of graphene before and after the droplet test were recorded using a Nikon Eclipse MA 200 optical microscope. Morphology of graphene layers was characterised using a Hitachi scanning electron microscope, model S-3000M, working in secondary electrons (SE) mode at an accelerating voltage of 5 kV.

The bond structure of the HSMG was examined with the help of Raman spectroscopy. An inVia Renishaw Raman spectrometer working at a wavelength of 785 nm was used for that purpose, with the measurements being carried out at several sites of samples. The laser power on the surface of the samples was 20 mW, and the laser spot size was 800 nm. All the measurements were performed under ambient conditions.

2.3 Technological concepts

Two technological concepts were tested: a transfer of graphene onto SAF as an intermediate phase during the creation of the composite, and a direct transfer onto pre-impregnates (prepregs) with unidirectional and bidirectional arrangements of CF. A schematic representation of graphene transfer on SAF/prepreg is shown in Fig. 1. Structural adhesive film (SAF)/prepreg was applied on a bimetallic growth substrate covered with HSMG.

Using a roller made of high-density foam, the SAF/prepreg was pressed on the substrate surface, and the system was then passed through a set of 4 pairs of rollers. Subsequently, it was subjected to a pressure of 400 N for 60 min. Pressing was performed using a smooth intermediate surface. After manual stocking and preliminary lamination, a green compact was placed into a vacuum bag made of a diaphragm membrane and sealed using sealing tape. The process was carried out under an atmosphere of -0.7 bar over 60 min to achieve better binding of the substrate with graphene to the SAF/prepreg. The operating system enabled air to be removed from the laminate during simultaneous pressing of individual layers.

2.4 Separation of intermediate product from the graphene forming matrix

Separation of the HSMG–(SAF/prepreg) from the forming matrix was achieved with the help of chemical etching. The metallurgical substrate was etched out within 24 h by

an application of 1 M aqueous solution of FeCl_3 at room temperature.

2.5 Identification of metallurgical graphene morphology and structure on the SAF/prepreg

Initially, an evaluation of the HSMG transfer was performed by testing electrical resistance of graphene on the SAF substrate at multiple points within a square using a UNI-T70b multimeter. In the case of carbon prepregs, such a simple evaluation with a multimeter is not possible because the prepreg conducts electricity (a small content of epoxy resin causes exposure of the carbon fibres). A Hitachi S3000N scanning electron microscope working in AE mode at an accelerating voltage of 5 kV was used for precise evaluation of the efficiency of the transfer of graphene onto the SAF and prepregs. In the AE mode, only the current of the incoming beam and the table current are compared, therefore, enabling a comparison between materials with substantially different electrical conductivities.

2.6 Fatigue tests

2.6.1 Flat sample tests by three-point bending

Fatigue strength analysis of composite samples, both graphene containing (PPG) and graphene free (PP), were carried out by the three-point bending test. For that purpose, a two-column strength tester LVF-50kN servo-hydraulic for static and dynamic tests by Walter + Bai was used. The load range for static and dynamic tests is taken $F = -50$ kN. However, the working accuracy according to PN-EN ISO 7500-1 corresponded to the 0.5% class.

2.6.2 Fatigue tests of tubular profiles

The testing station is presented in Fig. 2. It consists of a rigid support, holder, loading grip, and blade joint through which the force from the main actuator of the machine is transferred to the sample. The system assures that the direction of the force is always perpendicular to the sample axis, enabling free deformation of the sample in the deformation plane. The tests were carried out through one side bending. First, the sample was subjected to an initial force to apply a preloading, σ_m . Next, cyclic tension, σ_a , was applied using a constant deformation amplitude. The number of cycles required for a destruction of the composite or for the maximum force in the test to reach the value of the initial force, σ_m , was recorded. An identification of the character and the formation mechanism of wear cracks was performed on cross-sections of the profiles using SEM analysis.

Fig. 1 Schematic representation of graphene transfer; (1) bimetallic growth substrate covered with HSMG (each layers are presented in the enlarged fragment); (2) HSMG covered bimetallic growth substrate coated with SAF or prepreg (each layers are presented in the enlarged fragment); (3) manual roller pressing of SAF or prepreg; (4) machine roller pressing of SAF or prepreg; (5) pressing of the stack

under force of 400 N for 60 min.; (6) trapped air evacuation using vacuum bag; (7) Separation of the HSMG-(SAF/prepreg) from the forming matrix by FeCl_3 etching of metallic components; (8) washing graphene-(SAF/prepreg) with dH_2O ; (9) set of graphene-(SAF/prepreg) ready for application in a composite structure (layers of graphene and SAF/prepreg are presented in the enlarged fragment)

3 Results

3.1 Qualitative evaluation of HSMG continuity on the forming matrix

In the course of preliminary evaluation of the graphene continuity on the forming matrix, various macroscopic (Fig. 3a) and microscopic (Fig. 3b,c) defects were identified. After the water test, only those areas of the substrate that were characterised by a lack of significant macroscopic defects (discontinuities) when in contact with water (Fig. 3a-B) were selected. The droplet test revealed two HSMG growth mechanisms on the forming matrix: one dendritic (Fig. 3b) and one cellular (Fig. 3c). In our previous studies, cellular graphene was characterised

using SEM in SE mode [22, 23], while dendritic graphene was characterised in [28]. However, HSMG with structural discontinuities or formed by the dendritic mechanism were excluded from further investigations. This means that for the type of graphene characterised by a quasi-monocrystalline structure resulting from cellular growth on a liquid matrix was utilised subsequent tests. The HSMG formed by this mechanism exhibited a continuous structure in microscopic areas without extensive macroscopic defects, and it contained only a small amount of point defects or growth shortages (Fig. 3c-A). To confirm these observations the Raman spectrum of HSMG created by cellular growth was analysed. The main Raman band of the first order, also known as the G band ($\sim 1580 \text{ cm}^{-1}$) characteristic of all the sp^2 hybridised

Fig. 2 Fatigue tests station with a sample mounted: (1) rigid support; (2) sample; (3) loading grip; (4) blade joint

carbon atoms, was observed (Fig. 4). The presence of the strongest 2D (or G') band ($\sim 2700 \text{ cm}^{-1}$), which is a second-order Raman process originating from the in-plane

breathing-like mode of carbon rings [29], and the D band (around 1320 cm^{-1}) indicate the presence of point and linear defects.

3.2 Evaluation of HSMG quality transferred onto SAF

Compared to that of the base substrate an additional 2D band ($\sim 2700 \text{ cm}^{-1}$) was observed in the Raman spectrum of HSMG transferred onto SAF (Fig. 5). This confirms the presence of graphene on the SAF. Following the transfer process, a satisfactory continuity of coverage with graphene was achieved, as revealed by SEM/AE observations and electric resistance testing. After the transfer process onto the SAF, the HSMG was characterised by layer continuity with only a few microscopic defects (Fig. 6b-A). On a non-conductive adhesive film, every discontinuity (shortage) of graphene is visible as a dark area (Fig. 6). Additionally, after the transfer process, HSMG on the SAF substrate exhibited a satisfactorily low resistance of 2–9 k Ω /square see Table 1.

3.3 Evaluation of HSMG graphene continuity after transfer onto pre-impregnated carbon fibres

HSMG was transferred onto unidirectional (Fig. 7a, b) and bidirectional (Fig. 7c, d) prepreps. In Fig. 7a, c SEM/AE images of the bare CF are presented. Conductive areas (i.e., the CF) appear bright, while the non-conductive resin is

Fig. 3 Graphene grown on the Ni/Cu substrate: **a** macroscopic observations—water test, **b** HSMG grown through the dendritic mechanism after etching in 30% H_2O_2 , **c** hexagonal (cellular) growth of HSMG after etching in 30% H_2O_2 (A—graphene discontinuities, B—area free of any macroscopic defects, chosen for further transfer)

Fig. 4 Raman spectrum of HSMG grown through the cellular mechanism on the Ni/Cu substrate

Fig. 5 Raman spectra of the SAF and HSMG on the SAF

Fig. 6 SEM/AE images of HSMG on SAF: **a** dark area at the bottom of the image—SAF without graphene, bright area—graphene on SAF; **b** point defects in the HSMG on SAF—A

represented as dark areas. The SEM/AE images (Fig. 7b, d) show that the HSMG transferred onto both types of preregs is of good quality, with only a few defects. Thus, SEM/AE is a good method for determination of the graphene transfer quality onto conductive as well as non-conductive substrates. In the images presented in Fig. 7d, only rare microscopic discontinuities of the graphene layer on the biaxial prepreg substrate are observed. More discontinuities can be seen on the unidirectional prepreg (Fig. 7b).

3.4 Technological test

The technological test consisted of manufacturing flat composite samples composed of 17 prepreg layers and a tubular profile composed of 4 prepreg layers. For manufacturing the flat composite samples, 17 preregs with a biaxial fibre arrangement were used with three layers of SAF containing HSMG. Bench samples did contain SAF without HSMG. The composite was prepared in a quasi-isotropic $0^\circ/45^\circ/90^\circ/-45^\circ/0^\circ$ system. SAF with and without graphene was applied between the 4th and 5th, 8th and 9th, and 13th and 14th prepreg layers. A vacuum bag method was used to create the composite.

The low cycle fatigue test were performed for three different loads for each sample. Figure 8 shows the diagram of low cycle fatigue test results.

An analysis of the fatigue strength characteristics indicate that PPG samples show not only higher fatigue strength compared to PP samples, but also higher values of maximum stresses. For example: taking the values of maximum stress equal 630 MPa, the number of cycles to reach that value for PPG samples was approximately 5 times greater than for PP samples. Considering the same number of cycles' equal 5000 of the difference between stress values is about 9% in favor of PPG samples. The increase of the fatigue strength of PPG samples can be explained, among others by its failure mechanism observed by analysing fatigue fracture for both cases. Where graphene is used, there is a stronger inter-phase between preregs. This phenomenon contributes to the reduction of the scatter of obtained results of the fatigue

Table 1 Resistance of HSMG on SAF

Graphene on SAF (kΩ/square)			
5	7	2	3
7	3	4	9

characteristics discussed. As seen in Fig. 8, the scatter of results for areas more loaded for the graphene reinforced composite (PPG) is much lower than for the regular composite (PP).

The technological test consisted of manufacturing a tubular profile. It was constructed of four prepreps: two unidirectional prepreps with graphene transferred directly onto them, as shown in Fig. 9b, and two bidirectional prepreps outside and inside of the tube. The above procedure produced the tubular profile presented in Fig. 9a. This profile was fixed in steel sockets using epoxy resin to create the sample (Fig. 9c) for testing of low-cycle fatigue with one-side bending.

In the preliminary fatigue tests, an evident positive impact of HSMG on the fatigue strength of the composite was observed. However, the fatigue data were widely divergent. Several samples reinforced with HSMG graphene successfully withstood one million cycles without

any damage. Under the same testing conditions, the standard composite samples reached the initial σ_m after only 589 840 cycles. The damage to the samples never caused total fracture, but rather resulted in a local delamination of the polymer matrix. Microstructural examinations using SEM revealed two different fatigue mechanisms. Samples with HSMG were destroyed by delamination of the bidirectional prepreg, while samples without graphene failed through delamination within the unidirectional prepreg following a comparable number of cycles. For the overloaded samples, it was confirmed that delamination never occurred at the sites of HSMG presence (Fig. 10A), but it rather took place at the border between non-reinforced layers (Fig. 10B) and inside the original structure of the bidirectional prepreg (Fig. 10C).

4 Discussion

Typically, graphene is transferred onto target substrates from the growth substrate on a supporting film made of polymer, commonly poly(methyl methacrylate) (PMMA) [23, 24, 28, 30–33]. However, this standard procedure was revealed to be inappropriate for creating composites

Fig. 7 SEM/AE images of prepreps before and after HSMG transfer: **a** unidirectional prepreg; **b** unidirectional prepreg with HSMG; **c** bidirectional prepreg; **d** bidirectional prepreg with HSMG; (A—defects in the HSMG after transfer onto the prepreg)

Fig. 8 Fatigue test results S–N for three-point bending, a comparison of carbon fibre composites reinforced with HSMG graphene (PPG) introduced into the composite structure on SAF and carbon fibre composite (PP) with bare SAF sheet

Fig. 9 A sample of carbon fibre composite for low-cycle one-side bending fatigue test: **a** real tubular profile used in bicycle frame construction; **b** prepreg pattern used for tubular profile formation with indicated site of graphene sheet introduction (hatched area); **c** final sample for the fatigue tests (red dashed lines show indicative reinforced section of tubular profiles)

from pre-impregnated carbon fibre. Employing a PMMA film with graphene in the composite production resulted in delamination. In this study, a procedure for transferring graphene onto adhesive film or directly onto a resin-based pre-impregnate was elaborated for the purpose of production of carbon fibre composites. The analysis of SEM/AE images clearly demonstrate that it is possible to transfer graphene while maintaining a low level of mechanical defects resulting from the transfer. Satisfactory results were obtained both with an adhesive film having a relatively smooth surface of contact with graphene and with the prepregs. Biaxial and monoaxial pre-impregnates were characterised by a higher level of graphene defects compared to the adhesive film. The degree and nature of graphene defects on the unidirectional and bidirectional prepregs were similar.

An introduction of graphene into a composite with an epoxy matrix containing CH groups, aromatic rings, and hydroxyl groups should hinge about interactions between these functional groups and its π -electrons [34] delocalized throughout the entire surface of the large-format graphene piece placed between the resin layers. Though these interactions are weak when occurring in great numbers they may improve the composite properties [35], including, indirectly, those related to mechanical strength or adhesion.

In the studies of flat samples, a significant improvement of mechanical properties was observed in the fatigue tests. The 9% increase of low cycle fatigue strength has been achieved for PPG specimens which contained as little as 0.68 ppm graphene by weight. More impressive was an increase of the cycle number at failure that was obtained for PPG samples maintained at constant stress (up to 200%).

The initial fatigue strength tests of bicycle tubular profiles confirmed the observed reinforcing effect. The PPG tubular sample reached 1,000,000 cycles whereas the PP tubular sample has been destroyed after 589,840 cycles using the same pulsing load. It should be emphasised that the amount of graphene introduced into the structure of the composite was only 0.22 ppm in this case.

The presence of CH– π , π – π , or OH– π interactions may lead to an improvement of the adhesion between individual prepregs within the composite, which is strongly suggested by the lack of delamination observed at the HSMG surfaces (SEM observations). Additionally, the reinforced polymer matrix makes it difficult for the fibres to move within the composite at the compressed part of the profile, therefore, preventing fibre micro-buckling, which is one of the main causes of destruction of fibre-reinforced polymers [36]. Reinforcement of composites with large-format graphene has one advantage compared to the more commonly used solutions employing graphene flakes [37–39]. The presented method results in a uniform reinforcement on the entire surface between the prepregs, rather than acting locally in the vicinity of individual flakes. Uneven dispersion of graphene flakes within the matrix may lead to local reductions of strength at conglomerate locations, thus having a negative impact on the strength of the whole composite [39].

The presented solution requires further systematic studies on the impact of large-format HSMG on the properties of composites. Perhaps a hybrid solution, i.e. a combination of large format graphene and a polymer matrix reinforced with graphene flakes, would result in a superposition of the properties of both of these solutions and eliminate the disadvantages of each.

Fig. 10 Fatigue crack nucleation (SEM images): A—location of graphene introduction, B—delamination between preregs without graphene, C—delamination of fibres from the epoxy matrix

5 Conclusions

- Two effective methods for transferring large-format HSMG onto SAF and directly onto uni- and bidirectional preregs were elaborated.
- The effectiveness of SEM/AE observations for the assessment of the transfer quality of large areas of HSMG onto both conductive substrates (prepreg) and non-conductive substrates (SAF) was demonstrated.
- Preliminary results confirmed a significantly positive effect of the introduction of HSMG on the fatigue strength of CFRP composites.
- In the fatigue tests, no delamination between preregs was observed in those sites where the HSMG had been introduced.
- The increase at fatigue durability of HSMG-reinforced composites offers an added value to the performance of extremely strong CFRP composites.
- A significant growth (9%) of low cycle fatigue strength was recorded as a result of an introduction of 0.68 ppm by weight of HSMG graphene into the CFRP.
- The similar reinforcing effect was observed at the initial fatigue tests of industrial tubular CFRP profiles containing 0.22 ppm by weight of HSMG graphene.

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