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SUBMICROMETRIC GLASSY CARBON AS FILLER FOR HDPE-BASED CONDUCTIVE POLYMER COMPOSITES FOR ESD AND EMI APPLICATIONS

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Glassy carbon (GC) is a unique form of carbon that possesses a wide range of useful properties, including high thermal stability, low thermal expansion, and excellent electrical conductivity. This makes it a promising candidate for reinforcement in thermoplastic composites. In this work, micrometric GC powder (μ GC) and submicrometric GC powder (μ GC) were used to make a high-density polyethylene (HDPE) base composite. The GC reinforcement was introduced to the polymer matrix by two different methods to form random and segregated reinforcement distribution. The influence of the GC volume content (φ) and composite structure on electrical conductivity was examined. It was demonstrated that while glassy carbon can enhance the conductivity of HDPE more effectively than graphite, it falls short of matching the exceptional performance of carbon nanotubes, which bridge the gap between them. The research showed that the addition of GC increases the conductivity of HDPE, and achievement of the percolation threshold (φ_c) is possible at $\varphi \approx 4\%$. The segregated distribution of GC leads to lower values of the percolation threshold ($\varphi_c \approx 1\%$) than the random distribution.

Keywords: glassy carbon, submicrometric particles, electric conductivity, polymer matrix composites, percolation threshold

INTRODUCTION

Glassy carbon (GC) has found many applications in industries, including electrochemistry, electronics, and biomedicine [1, 2] since its discovery in the 1940s. It is characterized by many beneficial features, namely a high Young's modulus [3, 4], chemical stability [5], good temperature resistance, a favorable thermal expansion coefficient ($5 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$ in the range of $20 \text{ }^\circ\text{C} - 1000 \text{ }^\circ\text{C}$) and thermal conductivity ($6 \text{ Wm}^{-1} \text{ K}^{-1}$) [6], bio- [7] and hemocompatibility [8] as well as anti-thrombogenicity [9]. The literature also reports its beneficial electrical properties, with a conductivity of about $3 \cdot 10^2 \text{ S cm}^{-1}$ [10]. Despite the fact the conductivity of glassy carbon is lower than that of other conductive carbon forms (i.e. graphite, graphene, or carbon nanotubes), its temperature resistance, thermal properties, and the possibility of the direct fabrication of GC via photolithography followed by pyrolysis, make this material beneficial in the field of microelectronic device fabrication [11]. One of the greatest advantages of glassy carbon is the fact that it can be easily manufac-

ured in various forms including bulk, thin films, foams, and micrometric particles [1, 2]. Moreover, the extraordinary chemical stability of GC in various solutions makes it a common material for electrodes in electrochemical cells [11-15].

Another possibility is to use glassy carbon particles in polymer matrix composites to obtain conductive polymer composites (CPC). An important factor in such materials is the percolation threshold, which indicates the volume content of conductive filler (i.e. metal or carbon particles) necessary to significantly lower the polymer resistivity. This is achieved by forming a conductive network in the polymer matrix and might be produced via random distribution [16] or a more structured (segregated) approach [17]. CPCs may find use in various applications such as electromagnetic interference (EMI) shielding [18], electrostatic discharge (ESD) protection [19], or triboelectric nanogenerators for wearable electronics [20]. Additionally, such materials can be potentially manufactured via 3D printing

[21]. An example of using GC in antistatic packing for electronic devices was presented by Santos et al. [22]. Additionally, the influence of the GC particle size on the mechanical and electrical properties of the polymer composite was studied [23]. It was found that a filler with a particle size greater than 75 μm is more suitable for antistatic materials due to the lower percolation threshold compared to smaller GC particles ($< 45 \mu\text{m}$). Despite the relatively low electrical conductivity of GC [24], the possibility of antistatic applications of low-density polyethylene (LDPE) filled with GC was also investigated [25]. Additionally, the wide range of possible applications of GC was reviewed [26]. For instance, owing to the specific structure of GC, it can be used as a material for water decontamination, sensing elements, as well as an electrode in energy storage devices. It is important to highlight the significant potential in the field of EMI shielding, where the cellular structure of the filler naturally captures electromagnetic waves [24]. Additionally, GC was also investigated as a filler for 3D printing filaments [27], demonstrating its potential for use in this field.

In this work, submicrometric GC powder (μGC) was employed to produce an HDPE-matrix CPC. Composites with two different types of distribution of conductive particles in the material were investigated. The performance of CPC with μGC was compared to that of composites with micrometric powder (μGC) to observe the influence of the GC grain size on electrical conductance and the percolation threshold.

MATERIALS AND METHODS

The μGC powder was obtained by milling GC foams produced in-house. The process of manufacturing the foams was described in previous papers [28, 29]. Pre-crushed carbon foams underwent a two-step milling process (Table 1). First, GC was milled for 8 hours in a Szegvari Attritor System with a Teflon chamber in the presence of Si_3N_4 balls and water. Then the mixture was dried and moved to a high-energy planetary ball mill (Fritsch Pulverisette 7 premium line) with ZrO_2 balls and ethyl alcohol. Milling was carried out for 1 hour, and during the process, the alcohol was refilled every 20 minutes to mitigate its evaporation. The powder underwent dispersion in distilled water, followed by the measurement of grain size distributions and determination of the powder's mean diameter using the laser diffraction method, carried out with a Malvern MasterSizer 3000. Before the measurement, the GC powders were deagglomerated in water by an ultrasonic homogenizer for 15 min. The obtained submicrometric powder had $D_{4/3} = 0.58 \mu\text{m}$ and its grain size distribution is presented in Figure 1. The procedure of obtaining μGC was described in another work [30].

To prepare composite samples, GC powder was introduced to the polymer matrix in two different ways. In the first method, GC powder was mixed with HDPE

(Hivorex 2600J by Lotte Chemical, South Korea), in hot xylene (Chempur, Piekary Śląskie, Poland), followed by drying in ambient conditions overnight. In the second method, GC powder was mixed with HDPE powder in a mortar according to the procedure described by Kolisnyk et al. [31] and Maruzhenko et al. [32].

TABLE 1. Milling parameters of μGC

Mill	Planetary mill	Attritor system
RPM	1000	300
Balls	ZrO_2 1.75 mm	Si_3N_4 3 mm
Ball-to-powder-powder ratio	10:1	2:1
Chamber	Alumina	Teflon
Conditions	Ethyl alcohol (2 ml/5 g of GC powder)	Water

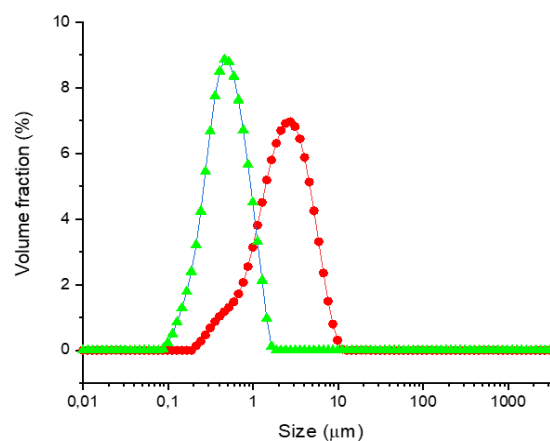


Fig. 1. Grain size distribution of micrometric glassy carbon (●) and submicrometric glassy carbon (▲). Lines are only to guide the eye

The polymer powder was self-produced using a laboratory grinding machine manufactured by Seo-tec (Sumy, Ukraine). The HDPE powder fraction of 200 μm - 400 μm was obtained by sieving. Using two methods of combining GC with HDPE allowed us to obtain composites with random distribution (the first method) and segregated distribution (the second method). From the prepared mixtures, samples for conductivity testing were produced by hot pressing in a steel mold. 0.5 grams of respective compositions were placed in a steel mold with a diameter of 20 mm and then transferred to a drier at 150 $^{\circ}\text{C}$. After two hours, when the leftover xylene had evaporated, the composite was pressed under a load of 30 MPa. Samples with different volume fractions were produced to determine the percolation threshold and the sample names are listed in Table 2.

The distribution of GC in the produced composites was investigated using a Zeiss Primo Star light microscope (Carl Zeiss, Germany) in transmission mode on slices with a thickness of 20 μm .

TABLE 2. List of produced CPC samples

GC [vol.%]	Randomized micro-sized GC	Randomized submicro-sized GC	Segregated micro-sized GC	Segregated submicro-sized GC
1	μ 1R	$s\mu$ 1R	μ 1S	$s\mu$ 1S
2	μ 2R	$s\mu$ 2R	μ 2S	$s\mu$ 2S
3	μ 3R	$s\mu$ 3R	μ 3S	$s\mu$ 3S
5	μ 5R	$s\mu$ 5R	μ 5S	$s\mu$ 5S
7	-	-	μ 7S	$s\mu$ 7S
10	μ 10R	$s\mu$ 10R	μ 10S	$s\mu$ 10S
12	μ 12R	$s\mu$ 12R	μ 12S	$s\mu$ 12S
14	μ 14R	$s\mu$ 14R	-	-
16	μ 16R	$s\mu$ 16R	-	-
18	μ 18R	$s\mu$ 18R	-	-
20	μ 20R	$s\mu$ 20R	-	-

The resistance of the samples was measured using a Keithley 6430 electrometer. Gold electrodes were deposited on the top and bottom surfaces of each sample. The electrodes were connected to the measurement setup with copper wires. The voltage-current characteristics were measured, and sample electrical resistances were calculated according to Ohm's law. The sample conductivity was determined by means of the following equation:

$$\sigma = \frac{L}{RA} \quad (1)$$

where: R – sample resistance, A – electrode area equal to 2.18 cm^2 , and L – sample thickness.

The percolation threshold was found by fitting conductivity to the percolation model:

$$\sigma = \sigma_0(\varphi - \varphi_c)^t \quad (2)$$

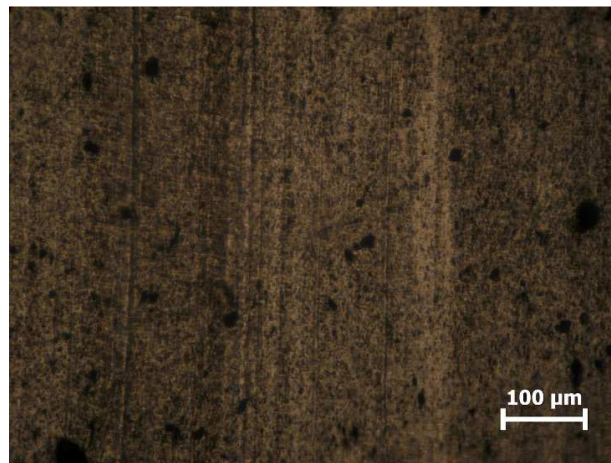
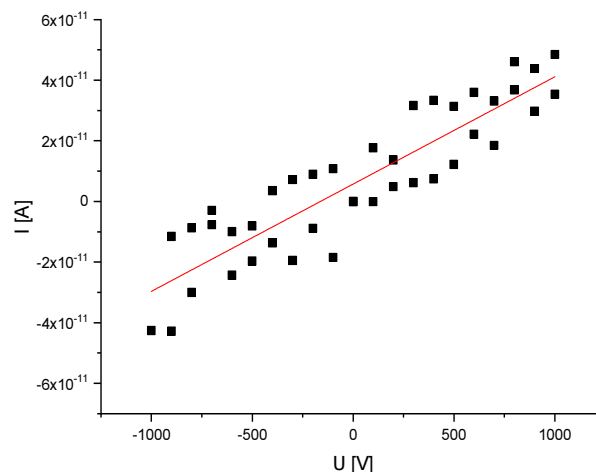
where: σ_0 – the adjustable parameter, describing the conductivity of the filler phase, φ – the volume fraction of the filler, φ_c – the percolation threshold, and t – the critical exponent.

RESULTS AND DISCUSSION

An evident difference in GC distribution is visible in the cross-sections of the produced composites (Figs. 2 and 3). In the case of the segregated distribution sample, a more organized, net-like, or skeleton-like structure is present. This suggests better conductivity according to percolation theory. In the sample with random distribution, some agglomerates are visible and the $s\mu$ GC particles are evenly distributed. This observation confirms the achievement of different structure types by the fabrication methods.

Electrical measurements were performed on the CPC samples. By applying different voltages, the current was measured, and the obtained values were fitted to determine sample conductivity. The representative results are presented in Figures 4-7. Unfortunately,

samples with random distribution and GC volume content $\varphi = 3 \%$ and below exhibit isolator-like properties and could not be properly measured as the current was low and out of the electrometer measurement range. Therefore, we excluded those samples from further analysis.

Fig. 2. Structure of composite with random $s\mu$ GC distribution (5 vol.%)Fig. 3. Structure of composite with segregated $s\mu$ GC distribution (2 vol.%)Fig. 4. Voltage-current characteristics for sample $s\mu$ 1S. Straight red line represents fitted Ohm's law dependence

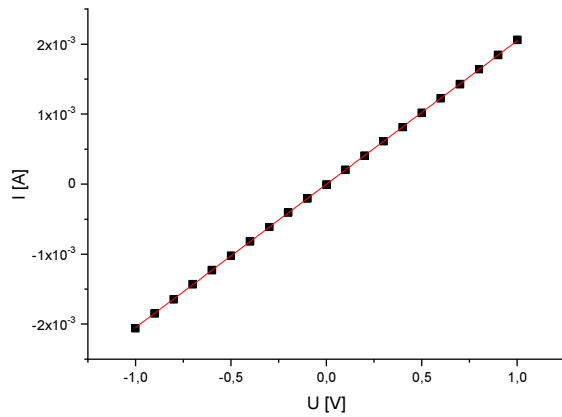


Fig. 5. Voltage-current characteristics for sample $\mu 18R$. Straight red line represents fitted Ohm's law dependence

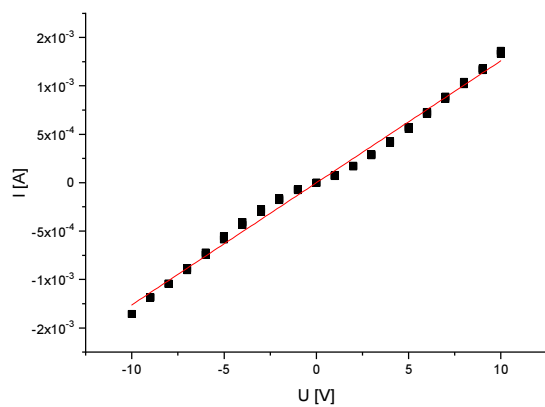


Fig. 6. Voltage-current characteristics for sample $\mu 5R$. Straight red line represents fitted Ohm's law dependence

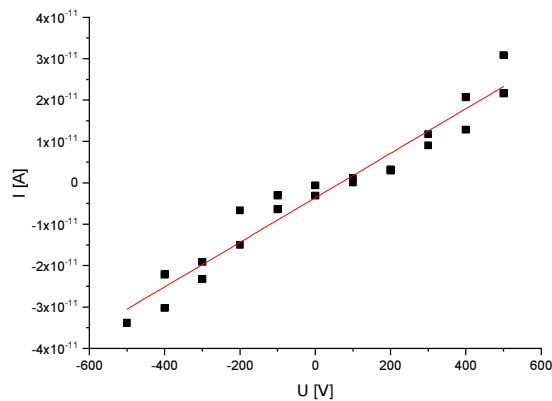


Fig. 7. Voltage-current characteristics for sample $\mu 3S$. Straight red line represents fitted Ohm's law dependence

Based on the registered voltage-current characteristics, the conductivity was calculated using equation (1). Figures 8 and 9 present the conductivity of the CPCs with random and segregated distribution of the GC particles, respectively. The results show (Fig. 8) that the φ_c for the random systems is achieved at a μGC content of around 4.0 % - 5.0 %, with μGC exhibiting a slightly lower value. Additionally, μGC resulted in higher conductivity than μGC . This can be explained by the better dispersion of submicrometric particles within

the polymer matrix compared to the micrometric particles. The use of smaller powder grains allowed a reduction in distance between the conductive fillers, which improved electrical conductivity, as expected. In similar random systems, Hoang et al. [33] and Valentino et al. [34] obtained a much lower percolation threshold by using nano-fillers such as MWCNT (0.55 %) and CNT (between 1.0 % - 2.5 %), respectively. However, Weng et al. [35] and Wu et al. [36] obtained much higher φ_c values, around 10 vol.% when using expanded graphite and graphite powder. This suggests that glassy carbon can be more effectively used to increase HDPE conductivity than graphite, but still cannot exceed the performance of carbon nanotubes.

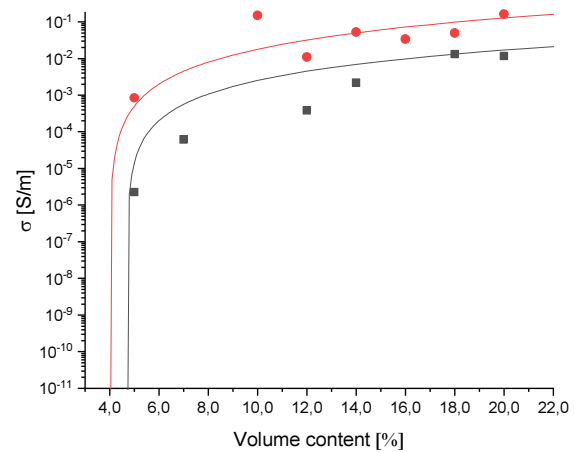


Fig. 8. Conductivity of CPC with a random distribution of μGC (■) and $\mu 5GC$ particles. The curves represent best-fitted theoretical dependences given by Eq. (2)

Moreover, segregation of the μGC particles can effectively lower the value of the percolation threshold to 1.0 % - 3.0 % (Fig. 9), filling the interval between GC and CNT. This was possible due to more localized concentrations of conductive powder in the HDPE matrix. The formation of a conductive path leads to more efficient particle distribution than a random one.

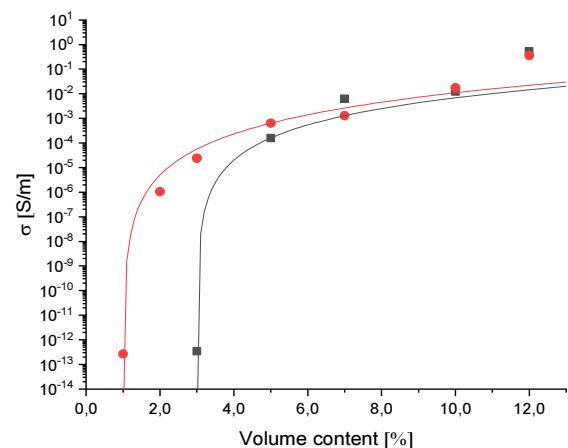


Fig. 9. Conductivity of CPC with segregated distribution of μGC (■) and $\mu 5GC$ particles. Curves represent best-fitted theoretical dependences given by Eq. (2)

In the case of a segregated distribution of GC, a lower percolation threshold was obtained with submicrometric GC compared to micrometric GC, similar to the results observed with a random distribution of GC.

The sample percolation thresholds (Table 3) were determined by fitting the obtained data to the percolation model using Equation (2).

TABLE 3. Results of conductivity modeling and percolation threshold values

Composite	φ_c [vol.%]	t	σ_0 [S/m]
random μ GC	4.7	1.8	0.5
random μ GC	4.0	2.0	5.0
segregated μ GC	3.0	3.0	20.0
segregated μ GC	1.0	3.5	50.0

σ_0 –adjustable parameter, describing conductivity of filler phase, φ – volume fraction of filler, φ_c – percolation threshold, and t – critical exponent.

The calculations revealed that for segregated distribution, the values of adjustable parameter σ_0 nearly reach the value of electrical conductivity of the pressed GC powder (60.3 S/m). This result is in good agreement with the previous explanation that segregated systems create a conductive "frame" inside the composite volume with direct contact between the filler particles. Furthermore, the size of the filler particles is significant as it enables an increase in the number of layers within the "frame" structure, potentially resulting in improved electrical conductivity of the composite.

In the case of the submicrometric GC composite, the increase in σ_0 from 5 S/m for random distribution to 50 S/m for segregated distribution of GC suggests that electrical charge encounters much less resistance in flowing through the formed path than between particles separated by a polymer matrix. Such an effect was described by Maruzhenko et al. for UHMWPE [32]. In that work, for micro-fillers in the form of thermally treated anthracite, the percolation threshold (φ_c) value was around 3.0 %, while a composite with a 1/3 mixture of graphene and anthracite exhibited a φ_c value of 0.5 %. Comparable results for the segregated system were obtained by Alaferdov et al. [37]. By using graphite nanobelts they obtained a conductive composite at a filler volume content of 0.4 %. Our results support the above concept and demonstrate the potential of μ GC as a valuable reinforcement for potential electrical applications such as ESD- and EMI-shielding, based on the correlation between good conductivity and EM shield efficiency [38]. For instance, Al-Saleh et al. presented in their work an increase in EM shielding efficiency for ABS-based composites with different carbon fillers (high structure carbon black, carbon nanotubes and multiwall carbon nanotubes). The reported increase was visible especially after reaching the percolation thresholds of the nanofillers [39].

CONCLUSIONS

The new submicrometric glassy carbon has been proven to be a valuable filler for conductive polymer composites. This is evidenced by the ability to achieve conductivity levels of an HDPE composite up to 0.16 S/m and 0.3 S/m in random and segregated systems, respectively. It was shown that while glassy carbon can enhance the conductivity of HDPE more effectively than graphite, it still falls short of matching the performance of carbon nanotubes. Additionally, it was found that the percolation threshold for HDPE CPC with submicrometric glassy carbon can range from 1 % to 4 %, depending on the reinforcement distribution type. The obtained results suggest that GC can be a good alternative for graphite and CNT for lightweight conductive composites in EMI-shielding applications.

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