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Influence of Carbon Fibres on Properties of Composites Based on Sulfaryl-BSP-7 Copolymer

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The paper considers the effect of low- (Uglen-9) and high-modulus (Ural-N-24/320) carbon fibres (CF) on the physical and technical characteristics of copolymer BSP-7/CF carbon plastics. All materials, namely, CF, BSP-7, and BSP-7/CF composites, demonstrate electron paramagnetic resonance (EPR) signals with different characteristics. The whole composite and its individual components' properties, as well as the interaction of the matrix with fillers are studied. The shape of EPR spectrum of CF Ural is controlled by the conduction electrons in dependence on the sample and skin-layer thicknesses for microwaves. The EPR spectra of CF Ural and Uglen are isotropic showing that these are carbonized rather than graphitized fibres. CF Uglen is characterized by a significant specific surface area, which causes a strong (by ≅ 25 times) broadening of the EPR line due to the interaction of CF electron spins with spins of air oxygen. The technology for preparing the composites, which includes magnetic mixing of the components inevitably leads to the ferromagnetic (FM) and superparamagnetic absorption signals within both BSP-7/Ural and BSP-7/Uglen. The magnetic resonance characteristics of composites depend on porosity of the samples, nature and concentration of fillers and FM impurities. The study of the tribological properties of composites shows that addition of Uglen-9 and Ural-N-24/320 fibres leads to a significant positive effect: a decrease in the friction coefficient, linear wear intensity and heat release over the restored surface of the original polymer by 1.6–2.6, 18.9–81.3 and 1.65–2.6 times, respectively.

В даній роботі розглянуто вплив низько- (Углен-9) та високомодульних (Урал-Н-24/320) вуглецевих волокон (ВВ) на фізико-технічні характеристики вуглепластиків БСП-7/ВВ. Встановлено, що всі складові, —

БСП-7, вуглецеві волокна, композити БСП-7/ВВ, — демонструють наявність парамагнетних сиґналів, характеристики яких визначено зі спектрів електронного парамагнетного резонансу (ЕПР), в тому числі виявлено роль взаємодії матриці з наповнювачами. Форма спектру ЕПР ВВ Урал визначається електронами провідности в залежності від співвідношення товщини зразка та товщини скін-шару для мікрохвиль. Ізотропність ЕПР-спектрів досліджених ВВ встановлює їхню природу як карбонізованих матеріялів. ВВ Углен мають значну питому поверхню, що приводить до сильного (в ≅ 25 разів) розширення лінії ЕПР завдяки взаємодії електронних спінів Углену зі спінами молекулярного кисню повітря. Технологія синтези композитів, яка включає магнетне перемішування компонентів, приводить до появи сиґналів феромагнетного (ФМ) та суперпарамагнетного вбирання у БСП-7/Урал і БСП-7/Углен. Магнетно-резонансні характеристики композитів залежать від пористости зразків, природи та концентрації наповнювачів і ФМ-домішок. Трибологічні дослідження показали, що додавання волокон Углен-9 і Урал-Н-24/320 в БСП-7 приводить до значного позитивного ефекту: зменшення коефіцієнта тертя, інтенсивности лінійного зношування та тепловиділення по відновлювальній поверхні вихідного полімера у 1,6–2,6, 18,9–81,3 й 1,65–2,6 разів відповідно.

Key words: carbon fibres, polyarylatesulfone BSP-7, electron paramagnetic and ferromagnetic resonances, intensity of linear wear, friction coefficient.

Ключові слова: вуглецеві волокна, поліарилатсульфон БСП-7, електронний парамагнетний і феромагнетний резонанси, інтенсивність лінійного зношування, коефіцієнт тертя.

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1. INTRODUCTION

Polymer composite materials (PCM) with fillers of various natures are widely used in many fields of science and technology, as they are characterized by an improved set of tribological and physical-mechanical properties compared to the original polymers, metals and ceramics [1, 2]. Purposeful creation of new PCM with the set characteristics, it is impossible without knowledge of their structure, electric and magnetic properties at the atomic and molecular level. It is important to control both the electronic properties of the base polymers and fillers, the degree of their dispersion in the composite and the mechanisms of interaction with the polymer matrix. Among the many PCM, one of the most common is carbon plastic (CP), which contains as a filler low- and high-modulus carbon fibres (CF). The scope of these data is very diverse: thermal protection of spacecraft and satellite components, automotive, friction units of various mechanisms, including in agricultural engineering, the use of activated CF as effective sorbents and more.

One of the features of explosives is their turbostratic structure, in which adjacent graphite layers are rotated at an angle relative to the normal to the layer, which is also accompanied by an increase in the distance between the individual layers [4]. The magnitude of these effects depends on many factors, including the type of raw material and the processing temperature of CF. As a result, the structure of CF consists of layers several microns long and about a nanometer-thick [4, 5]. Accordingly, the electronic properties of CF are determined by the presence of both conduction electrons (CE) and a significant concentration of localized centres—defects such as broken carbon bonds [6, 7]. It is likely that the features of the electronic structure of CF will be reflected in their mechanical properties as well as the properties of composites with their participation. One of the effective methods of studying the electronic structure of solids is the method of electron paramagnetic resonance (EPR). This applies to studies of both dielectrics and conductive materials, the theory of spin resonance of conduction electrons (CESR) which is developed in detail [8–11].

Among the polymer matrices of considerable interest is polyarylatesulfone BSP-7. This material surpasses the known serial polymers of tribotechnical purpose in terms of performance (strength and toughness): caprolon B, polyamide PA 6 210/310, polyamide 12-L. due to the presence of sulfonic groups in the chemical composition. However, low wear resistance hinders its widespread use in tribological compounds. It is known from the literature [12, 13] that low- and high-modulus CF improve the tribological properties of aliphatic and aromatic polyamides, because of which the developed PCM is actively used in the agricultural, metallurgical and automotive industries instead of serial materials.

The aim of this work was to develop high quality composites for tribological applications based on BSP-7 with CF as fillers. The influence of low- (Uglen-9) and high-modulus (Ural-N-24/320) CF on the features of para- and ferromagnetic responses of polymer, nanofillers and composites, processes of polymer–filler interaction, and tribological properties of the obtained composites was studied.

2. EXPERIMENTAL/THEORETICAL DETAILS

As a polymer matrix for the creation of CP was chosen polyarylatesulfone block copolymer sulfaryl BSP-7. This polymer was synthesized under the conditions of acceptor-catalytic polycondensation in an organic solvent based on 2.2-di (4-oxyphenyl) propane, phenolphthalein, 4,4′-dichlorodiphenylsulfone and dichloranhydrides of tere- and isophthalic acids. As filler, we chose carbon fibres: low- (Uglen-9, further Uglen) and high-modulus (Ural-N-24/320, further

TABLE. Technical characteristics of carbon fibres [3].

Parameter	Carbon fibre	
	Ural-N-24/320	Uglen-9
Length of fibre, mm	3–4	
Density, g/cm^3	1.5	1.6
Tensile strength, H/tex	1.5–2.0	3.5–5.0
Breaking elongation, %	1	—
Elasticity modulus, GPa	60–80	15–18

Ural) produced by PC 'Svetlogorsk Khimvolokno', the main properties of which are given in Table. The choice of CF was determined, on the one hand, their technical characteristics and, on the other hand, cost. The PCM based on copolymer BSP-7 containing CF Uglen (10–30 mass.%) and Ural (10–30 mass.%) was made by compression moulding according to the method described in [14].

The EPR is an effective method for studying the electronic and magnetic properties of matter, including the mechanisms of interaction between the polymer matrix and fillers in composites. Since both the polymer matrix BSP-7 and all the fillers used in this work had paramagnetic or ferromagnetic properties, EPR studies have become an important component of the development of CP based on BSP-7. EPR measurements were performed at room temperature using an X-band Radiopan X-2244 spectrometer (microwave frequency $\nu \cong 9.4$ GHz) with 100 kHz magnetic field modulation. The estimated accuracy in determining the g-factor was of $\pm 2 \cdot 10^{-4}$. The absolute accuracy of the spin concentration (N_s) was of ±50%. Measurements were performed on both source and evacuated samples. To do this, the latter was placed in a quartz tube and pumped out for 1 hour at $T = 413$ K. After that, the sample was moved to the resonator of the spectrometer, and the EPR spectrum was recorded without changing the pumping out conditions.

Tribological properties (coefficient of friction (f), intensity of linear wear (I_h) and heat dissipation (q)) were studied under friction conditions without lubrication on a friction disk machine [16], steel 45 (45–48 HRC, $R_a = 0.32$ μm) at a load of 0.6 MPa and a sliding speed of 1 m/s, the friction path was of 1000 m.

3. RESULTS AND DISCUSSION

Magnetic Resonance Properties. Samples for EPR measurements of carbon fibres in the form of bundles were placed in a quartz tube, so that the axis of the carbon fibre was directed perpendicular or parallel to the external magnetic field. In the case of crushed and diluted in paraffin explosives, chaotic distribution of CF in all directions was realized. Chaotic particle distribution was naturally realized in powder samples of BSP-7/CF composites.

Figure 1 shows the EPR spectrum of the powder sample BSP-7.

Theoretical analysis of the spectrum (dotted curve in Fig. 1) showed that it consists of an intense signal with the following parameters: $N_s \cong 4\cdot10^{14}$ g^{-1}, $g = 2.0028$, line width $\Delta B_{pp} = 0.66$ mT, and a weaker signal with parameters as follow: $N_{loc.} \cong 4.8\cdot10^{13}$ g^{-1}, $g = 1.993$, $\Delta B_{pp} = 0.58$ mT.

Figure 2 shows the EPR spectrum of the CF bundles Ural.

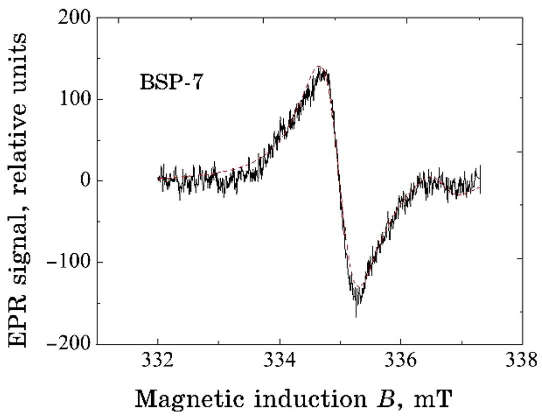

Fig. 1. EPR spectrum of polymer BSP-7 powder sample.

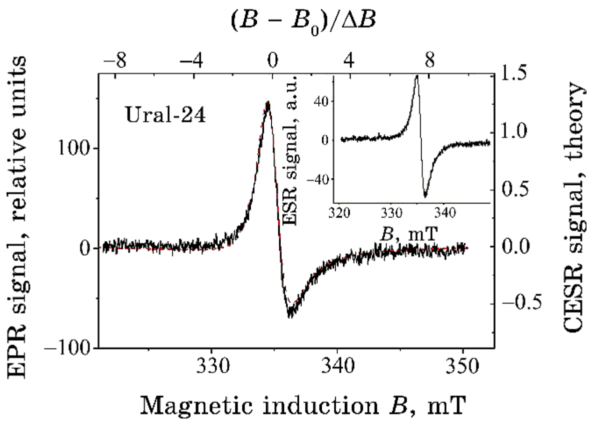

Fig. 2. Spin resonance of conduction-electrons' signal of the CF bundles Ural. Dashed line—simulated spectrum in dimensionless units calculated according to the theory [7–10]. The insert shows a spectrum of fibres dispersed in a paraffin matrix. $\nu = 9406$ MHz.

The following parameters of the spectrum were determined: $N_s \cong 5\cdot10^{17}$ g^{-1}, $g = 2.0030$, $\Delta B_{pp} = 1.7$ mT. These data refer to the symmetric line with the Lorentz shape shown in the insert. The data for the asymmetric CESR line with the Dyson shape, calculated on the basis of the spin-resonance theory of conduction electrons [7–10], as expected, coincide with those obtained for the dispersed sample. It is established that the orientation of CF relative to the direc-

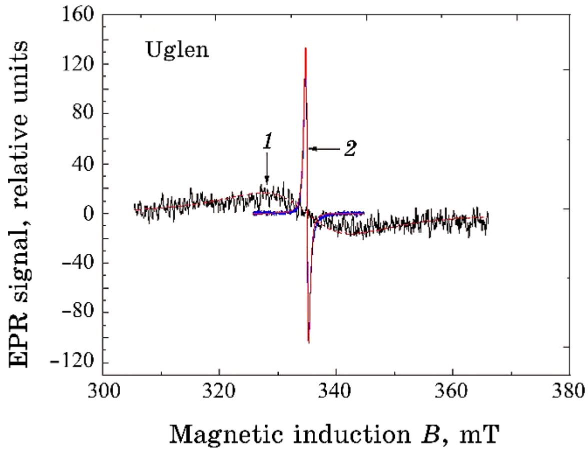

Fig. 3. EPR spectrum of the CF bundles Uglen. *1*—sample in the air; *2*—pumped out for 1 hour at $T = 413$ K. Modulation amplitude were chosen optimal for each case.

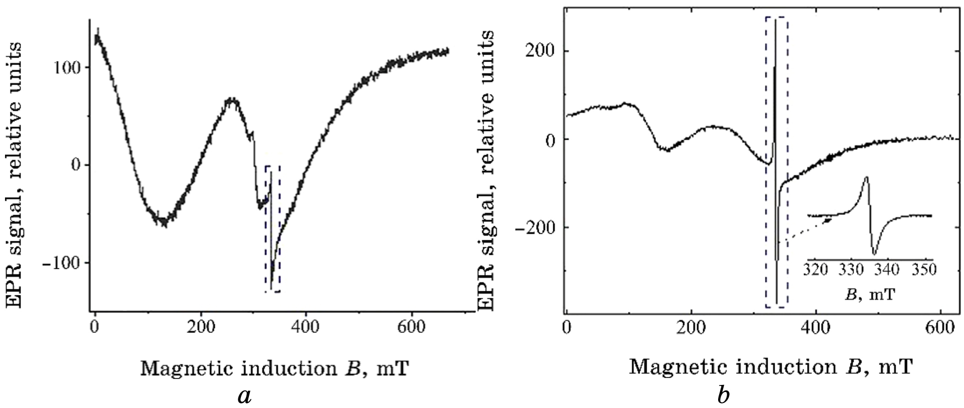

Fig. 4. Spectra of magnetic resonance signals for samples of composite BSP-7/Ural with the content of CF 10 (*a*) and 30 mass.% (*b*). Broad intense signals are due to the FMR and SPM resonances. Narrow paramagnetic signals (marked by dashed frame) belong to CF. Spectra were recorded under pumping out conditions at $T = 413$ K.

tion of the magnetic field does not affect the value of the g-factor.

Figure 3 presents the EPR spectra of low-modulus CF Uglen.

The parameters of the spectra in Fig. 3 were determined after comparing the experimental spectra with those theoretically calculated according to the theory [8, 11]. The results are as follow: $N_s \cong 7 \cdot 10^{17}$ g^{-1}, $g = 2.0030$, $\Delta B_{pp} = 12$ mT and 0.45 mT for lines *1* and *2*, respectively. It is seen that the EPR line dramatically expands in air due to the presence of paramagnetic molecular oxygen.

In the BSP-7-based composites with CF Ural and Uglen fillers, the situation changes significantly. Signals from fillers are still present, but their characteristics are changing. In addition, new ferromagnetic (FMR) and superparamagnetic (SPM) signals are emerging, the nature of which is related to the technology of obtaining CP, which includes the process of magnetic mixing of the mixture using ferromagnetic particles [11]. The magnetic resonance spectra of the composite BSP-7/Ural with a CF content of 10 and 30 mass.% are given in Fig. 4, *a*, *b*, respectively. The pumping out of the samples does not affect the FMR and SPM signals; at the same time, the EPR signals from CF are narrowed during pumping with a corresponding increase in their amplitude. It can also be seen that the amplitude of FMR and SPM signals is ≅ 2 times larger for the composite with 10 mass.% CF (Fig. 4, *a*) compared to the composite with 30 mass.% CF (Fig. 4, *b*). The behaviour of the EPR line width of samples 1–4 with different content of CF Ural turned out unexpected (Fig. 5).

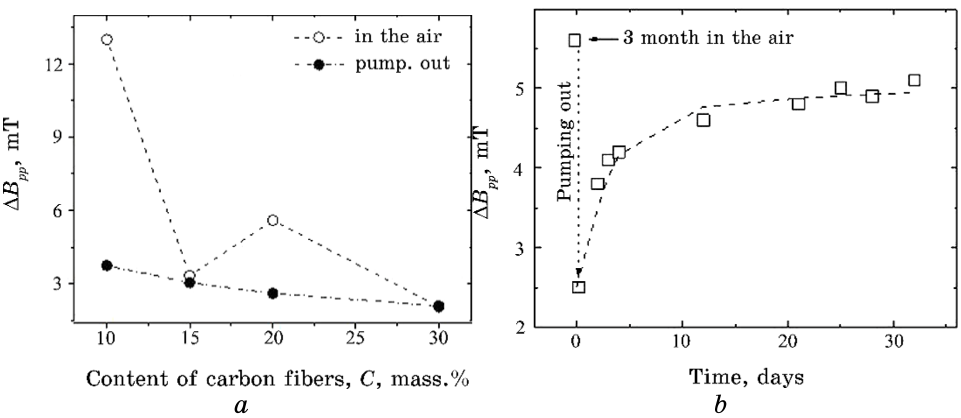

Fig. 5. (*a*) Dependence of the EPR line width of BSP-7/Ural composite samples with different fillers' content in the air and when pumping out for 1 hour at $T = 413$ K; dashed lines are an eye guide. (*b*) An increase in the width of the EPR line for the composite BSP-7 samples with 20 mass.% CF Ural in the air after pre-pumping the sample; the dashed curve is constructed according to formula (1).

Figure 5, *a* shows that, in air, the width of the EPR line of the filler Ural can significantly exceed its width outside the composite content. The effect is strong for samples 1 and 3, and almost imperceptible for samples 2 and 4. Figure 5, *b* also shows that the gas permeability of the composite occurs at room temperature too, but the speed of the processes is much lower. According to the work [14] on the study of absorption processes in porous carbon, the curve in Fig. 5, *b* describes the rate of diffusion of oxygen entry and filling of the immediate environment of paramagnetic centres (PC), because ΔB_{pp} is proportional to the oxygen concentration in the PC environment. The dashed curve is calculated theoretically according to the law [14]:

$$\Delta B_{pp}(t) = \Delta B_{pp,\max} \frac{\exp\left(\frac{t}{\tau_1}\right)}{1+\exp\left(\frac{t}{\tau_1}\right)}, \quad (1)$$

where $\tau_1 = 14.4$ hours is the time of oxygen diffusion in the sample.

A significantly different situation is observed for BSP-7/Uglen composites. The FMR and SPM resonance signals are also observed in them (Fig. 6). However, the EPR signals from Uglen fillers have a different dynamics in them, when pumping out samples.

Under the conditions of pumping out samples of this composite at $T = 413$ K, the dynamics of the signals is such that regardless of the

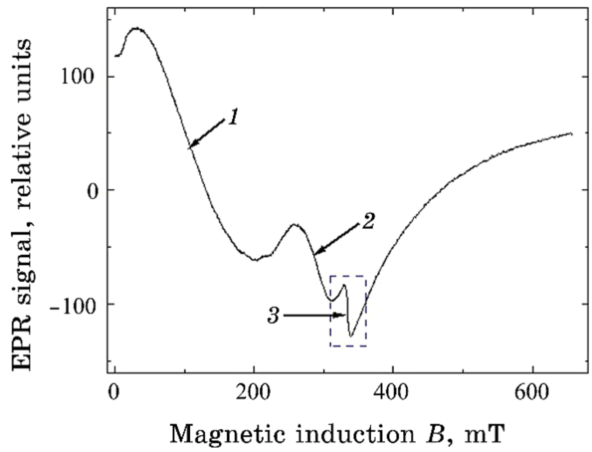

Fig. 6. Signals of FMR (*1*), SPM (*2*) and EPR (*3*) in the composite BSP-7/Uglen with a content of the CF Uglen 30 mass.%. The spectrum is recorded in the air. The EPR line width from CF Uglen (line *3*, marked by dashed frame) $\Delta B_{pp} \cong 9$ mT.

content of Uglen filler (samples 5–8), the EPR signals from it are narrowed in all samples approximately equally, namely, to the value of $\Delta B_{pp} \cong 0.5$–0.6 mT, inherent to that in CF Uglen.

Analysis of EPR Results and Discussion. It follows from Fig. 1 that the polymer BSP-7 has paramagnetic properties. The concentration of defects is small: $N_s \cong 4 \cdot 10^{14}$ g^{-1}. The value of their g-factor $g = 2.0028$ and EPR line width $\Delta B_{pp} = 0.6$ mT are very similar to the data for dangle bonds in polymer chains of other polymers, for example, in aromatic polyamide phenylene [2]. In bulk samples, the concentration of dangling bonds, as expected, is approximately an order of magnitude lower.

The Ural filler EPR spectra (Fig. 2) show an asymmetric Dyson shape line for CF bundles and a symmetric line for dispersed samples. The spectral parameters for both types of samples are almost identical, indicating that the nature of paramagnetism does not change.

The Dyson shape line is typical for the EPR of the conductive samples. The parameters of such spectra are described with the involvement of the theory of spin resonance of conduction electrons, developed in [7–10]. CESR theory takes into account the processes of electron diffusion in the volume of the skin layer into which the radio frequency field penetrates. If the thickness of the conductive sample significantly exceeds the thickness of the skin layer, the Dyson shape characterizes the CESR line, which is a consequence of the simultaneous manifestation of the contributions of dispersion and absorption [7–9]. In our case, the calculated CESR spectrum (dashed curve in Fig. 2) well describes the experiment with the following parameters: $g = 2.0030 \pm 2 \cdot 10^{-4}$, $\Delta B_{pp} = 2$ mT, $R = 50$, where $R = (T_D/T_2)^2$, T_D is the time of diffusion of the electron through the skin layer, T_2 is the time of spin relaxation of the electron.

It is also known [7–10] that, when the thickness of the conductive sample is less than the depth of the skin layer for a given microwave frequency, the CESR line becomes symmetrical one, and its shape becomes of Lorentz's type. Indeed, the CESR line of the same CF bundles, but crushed and dispersed in paraffin, acquires the Lorentz's shape (see insert in Fig. 2). It is important to note that the value of g-factor is the same for all fibre orientations, *i.e.*, there is no spectral anisotropy. This directly indicates that Ural CF are carbonized, not graphitized.

Another type of fibre is low-modulus Uglen fibre, which has different properties. Due to the peculiarities of its structure, high conductivity of samples [3], and a small length of individual fibres as well as because of a larger specific surface area of the samples, the EPR line is symmetrical, and its width is determined by the interaction of electronic spins of Uglen with molecular oxygen spins. In our case, the effect of broadening the EPR line of CF Uglen due to oxygen is strong, approximately 25 times (see Fig. 3).

Magnetic resonance signals in BSP-7/Ural and BSP-7/Uglen composites are much more complex. First, new signals of ferromagnetic and superparamagnetic resonances are registered. The intensity of the FMR and SPM signals, and their ratios in samples with different content of fillers, changes. The reason for this behaviour is related to the technology of obtaining these composites, where the process of magnetic mixing of the initial mixture plays an important role. This process is accompanied by the friction of strong fibres with uniaxial FM particles—mixing activators, because of which micro- and nanoparticles of FM material are cleaved, which are responsible for the FM and SPM properties of the prepared composites [11]. A characteristic feature of these composites is the dependence of the intensity of the FMR and SPM signals on the nature of the fillers (Ural, Uglen) and their percentage amount in the composite (see Fig. 4 and Fig. 6).

The behaviour of EPR signals in BSP-7/Ural composites with different filler concentrations turned out to be unexpected. Thus, evacuated samples 1–4 show a gradual decrease in the width of the EPR line ΔB_{pp} from 3.75 mT to 2.1 mT with increasing filler concentration from 10 to 30 mass.%. At the same time, these samples, which were in contact with air for 1–2 months (paramagnetic oxygen!), show a nonmonotonic change in ΔB_{pp} from 12 to 2.1 mT, which does not correlate with the change in filler concentration (see Fig. 5). The analysis showed that the reason for this behaviour is the difference in the porosity (density) of the composite samples. It can be expected that the best tribotechnical characteristics of the composite will be characteristic of the densest samples 2 and 4. At the same time, in the composites BSP-7/Uglen, it was found that regardless of the change in the concentration of Uglen CF from 10 to 40 mass.%, the pumping out of samples at $T = 413$ K always leads to a decrease in the EPR line width of Uglen from 9–12 mT to 0.45–0.6 mT, *i.e.*, the value of the line width inherent in pure pumped powdered samples of Uglen.

Tribological Properties of Composites. Analysis of the results of tribological properties of carbon plastics showed that the use as a filler of low-modulus fibre Uglen (Fig. 7) reduces the intensity of linear wear and friction coefficient of the original polymer in 6.7–18.9 and 1.1–1.6 times, respectively.

The introduction of high-modulus CF fillers Ural (Fig. 8) into the BSP-7 block copolymer proved to be more effective compared to Uglen, the intensity of linear wear and the friction coefficient is decreased in 39.2–81.3 and 2.2–2.6 times. This is because this filler is characterized by high strength characteristics (see Table) and, as a result, strengthens the polymer matrix more, because of which CP

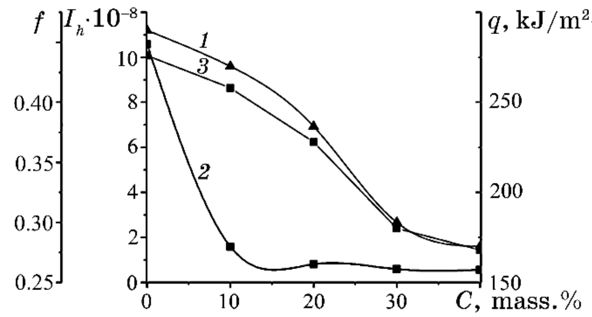

Fig. 7. Influence of CF Uglen content (C, mass.%) on friction coefficient (f, *1*), intensity of linear wear (I_h, *2*), and heat release (q, *3*) of block copolymer sulfaryl BSP-7.

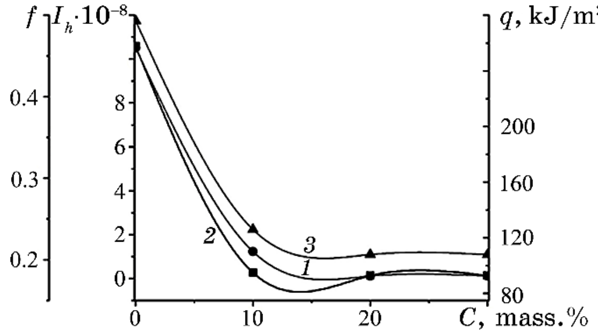

Fig. 8. The effect of the percentage of CF Ural (C, mass.%) on coefficient of friction (f, *1*), intensity of linear wear (I_h, *2*), and heat (q, *3*) block copolymer sulfaryl BSP-7.

has a stronger resistance to abrasion and external deformations.

The decrease of heat intensity in 1.65 (Fig. 7) and 2.6 (Fig. 8) times in the contact zone between the counter body and the sample is probably [15] due to an increase in the thermal conductivity of the CP, which provides heat removal from the friction zone. In general, the improvement of the tribotechnical characteristics of the original polymer can be explained by the fact that, in the process of friction on the steel counter–friction transfer, film is formed, resulting in friction occurring according to the 'polymer–polymer' scheme [12].

4. CONCLUSION

The magnetic resonance properties of fillers and composites BSP-7/CF at the atomic level reflect the relationship between the characteristics of fillers and composites with their participation. In particular, this concerns to the porosity of composites, gas permeability at different temperatures, and ferromagnetic properties as a side effect of the technological process of synthesis of composites. Analysis of the results of operational properties of the developed CP showed that the use of Uglen-9 and Ural-N-24/320 as a filler for block copolymer BSP-7 is a promising way to improve its tribotechnical characteristics: reducing friction, intensity of linear wear and heat on the reducing surface of the original polymer in 1.6–2.6, 18.9–81.3 and 1.65–2.6 times, respectively. It is established that more effective improvement of these properties takes place for Ural-N-24/320 filler.

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