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ELECTRICAL AND MECHANICAL PROPERTIES OF FLEXIBLE POLYMER COMPOSITE MATERIALS WITH VARIOUS CARBON FILLERS

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Abstract. We studied the conductive and mechanical properties of polymer composites based on styrene butadiene rubber as a matrix and three different carbon fillers, namely graphite, graphene and carbon nanotubes. Based on the analysis of the resistivity dependences on the concentrations of various fillers, the values of percolation thresholds were determined. The sensitivity of the resistivity of all synthesized composites to the degree of their uniaxial stretching was investigated. The maximum values of the gauge factor were observed in composites with graphene filler. It was also found that in composites with carbon nanotubes as a filler, the deformations arising under applied stresses have an elastic character. After removing the tensile stresses, these composites completely relaxed to their initial size. Contrary to this, in composites with graphite and graphene, even minimal stresses caused the appearance of plastic deformations, and their complete relaxation did not occur after stress was removed.

Keywords: polymer composite, styrene-butadiene rubber, carbon, resistivity, percolation threshold, gauge factor, deformation

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ЭЛЕКТРИЧЕСКИЕ И МЕХАНИЧЕСКИЕ СВОЙСТВА ГИБКИХ ПОЛИМЕРНЫХ КОМПОЗИЦИОННЫХ МАТЕРИАЛОВ С РАЗЛИЧНЫМИ УГЛЕРОДНЫМИ НАПОЛНИТЕЛЯМИ

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Аннотация. Исследованы проводящие и механические свойства полимерных композитов на основе бутадиен-стирольного каучука и трех различных углеродных наполнителей: графита, графена и углеродных нанотрубок (УНТ). На основании анализа концентрационных зависимостей удельного сопротивления определены значения порогов перколяции. Исследована чувствительность сопротивления композитов к степени их одноосного растяжения и обнаружено, что наибольшие значения коэффициента тензочувствительности наблюдаются в композитах с графеном. Показано, что в случае наполнителя из УНТ деформации, возникающие под действием приложенных напряжений, имеют упругий характер, а после снятия напряжения эти композиты полностью релаксируют к своему исходному размеру. В композитах с графитом и графеном возникают пластические деформации даже при минимальных напряжениях, а их полной релаксации не происходит.

Ключевые слова: полимерный композиционный материал, бутадиен-стирольный каучук, углеродный наполнитель, электропроводность, коэффициент тензочувствительности

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Introduction

Polymer nanocomposite film materials show promise for applications in sensor devices for wearable electronics. These materials are characterized by a wide variety of structures, making it possible to achieve the necessary properties such as lightness, flexibility, wear and corrosion resistance, as well as to obtain products in diverse shapes. Devices based on polymer nanocomposites are miniature, so, while they are in constant contact with the body, they should be barely noticeably to the user. Therefore, development of new sensor materials can contribute to rapid progress in many fields, for example, in medicine to monitor human health [1], in sports to track the performance of athletes [2], in hazardous workplace environments to monitor the health status of workers, in the gaming industry to simulate immersive scenarios and in the fashion industry to create smart fabrics capable of adapting to external stimuli [3].

One of the promising applications for polymer nanocomposites is in flexible stretchable strain sensors [4, 5]. For this purpose, a conductive filler is introduced into the polymer matrix. Its particles form a conductive network, which partially collapses under deformation, leading to an increase in the resistance of the material. Composites containing metal fillers are characterized by low flexibility and elasticity; they are sensitive to strain only in a limited range [6], while polymer composites based on carbon fillers have significantly better parameters.

In view of this, research is underway to determine the properties of composites based on various polymers using graphite [7], graphene [8] or carbon nanotubes (CNTs) [9] as fillers. The properties of the obtained materials significantly depend on the technology used for synthesis, the type of polymer matrix and its concentrations of filler particles.

We developed a technology for synthesis of polymer composites based on styrene-butadiene rubber (SBR) as a matrix, as well as graphite, graphene and CNTs as fillers.

This paper presents a comparative study on the influence of concentrations of various carbon fillers on the conductive, piezoresistive, and mechanical properties of synthesized SBR-based composites.

Materials and methods

Preparation of samples. We used Sigma-Aldrich styrene butadiene rubber as a non-conductive polymer matrix, Super P Conductive Carbon Black graphite powder, graphene powder, and TUBALL single-walled carbon nanotubes as fillers.

To obtain a composite film, a weighed portion of the filler powder (graphite, graphene, or CNTs) was mixed in carbon tetrachloride (solvent) using a magnetic stirrer; the procedure was carried out in a sealed flask to prevent evaporation of the solvent and the ingress of air bubbles into the solution. Next, SBR polymer granules were introduced into this dispersion system at a concentration of 5.9 wt.% (relative to the solvent) and mixing continued until the polymer was completely dissolved. The resulting solution was poured into a PTFE mold and dried for 1 hour under a fume hood until completely dry. The composite film was then removed from the PTFE mold with tweezers.

To determine the percolation threshold and measure the mechanical and electrical properties depending on filler concentration, we synthesized samples with graphite and graphene concentrations from 13 wt.% to 33 wt.% and CNT concentrations from 0.25 wt.% to 2.5 wt.% (relative to the finished composite). The filler concentration was selected taking into account the average size and shape of its particles, using a model estimating the percolation threshold in a material of a given thickness.

Measurement procedure. The resistivity of all films obtained from synthesized composites, as well as its dependence on the degree of stretching of the samples, were measured by the four-probe van der Pauw method. The experiment was conducted by the following steps: the film sample was fixed in a measuring cell with a movable crosshead, after which electrical contacts were connected to it. During the stepwise movement of the movable crosshead, the sample was stretched to a certain length; the resistivity was measured after each step.

The mechanical properties of composite polymer films were investigated with the same setup, but without connecting the contacts. To measure the strength and relaxation ability of the material, the samples were repeatedly stretched, each time increasing the tension in increments of 10 mm. At the final time step of each stretching test, the sample was held for 5 seconds, then the tensile stress was abruptly removed and the length of the sample was measured with a caliper after 25 seconds. To measure the relaxation time, the samples were stretched by 400% of the initial length, and after the load was removed, the length of the sample was measured every 20 seconds until the relaxation effect disappeared.

Results and discussion

Study of percolation properties. To analyze changes in the conductive properties of composites under deformation, it is necessary to take into account the percolation threshold for particles of a conductive filler placed in a dielectric polymer matrix. The structural parameter of the composite that characterizes these properties is the degree of overlap of the particles [10]. A continuous conductive cluster is formed at a high concentration of particles, with the particles directly touching each other. As the concentration decreases, the arrangement of the particles becomes more dispersed and the conduction mechanism changes to a hopping mechanism. A further decrease in particle concentration, taking into account their average size in one or more directions, significantly reduces the probability of charge carrier hopping and the conductivity of the composite abruptly tends to zero. The concentration of filler particles at which the composite stops conducting electric current is the percolation threshold.

Samples with filler concentrations at which hopping conductivity prevails are promising for subsequent use as active elements of sensing devices. The hopping probability (and hence the value of conductivity) exponentially depends on the distance between the conductive particles, which is why such composites have the greatest strain sensitivity.

For this reason, to obtain samples with high strain sensitivity, it is necessary to determine the value of the percolation threshold in synthesized composites for fillers with different structures and particle sizes. Fig. 1 shows experimentally obtained dependences of resistivity of the studied samples on the concentration of fillers: graphene, graphite and CNTs (at room temperature).

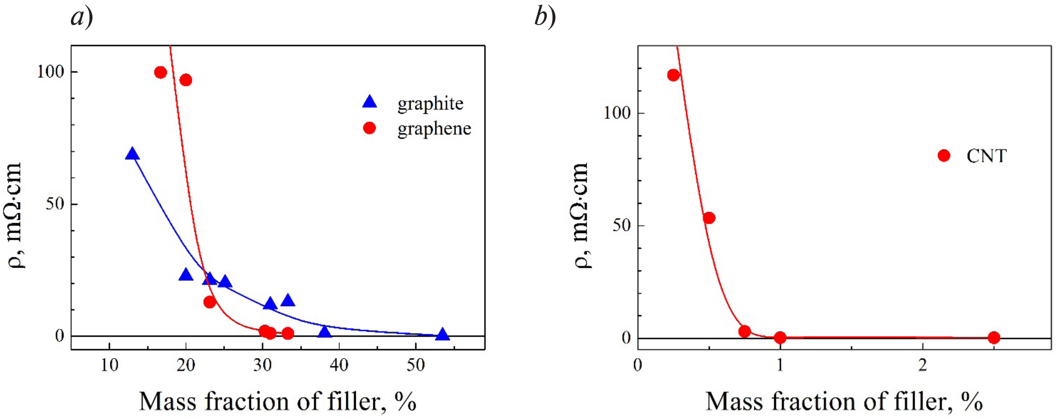

Fig. 1. Dependences of resistivity on filler concentration for composites containing graphene or graphite (*a*) and CNTs (*b*)

As can be seen from the above data, there is a sharp increase in resistivity for samples with graphite and graphene with about 37 wt.% and 30 wt.% concentrations, respectively. This may be due to the collapse of the percolating cluster and a transition of the cluster's conductivity to the hopping type. For a CNT-based composite, such a change in the conductive properties of the composite is observed at significantly lower concentrations of the filler (below 1 wt.%).

To clarify the conductivity mechanism, we plotted the dependences of ln(ρ) on $N^{-1/3}$ (where N is the concentration of particles) based on the obtained data. Filler particles in the matrix are considered as a set of nodes surrounded bv closed surfaces of the same shape, randomly distributed in space. When the surfaces intersect, a conductive chain of nodes is formed, with the electron tunneling occurring between these nodes (the Miller–Abrahams model for nearest-neighbor hopping). The localization length of the wave function (hopping distance) can be determined from the slope of the dependence of ln(p) on $N^{-1/3}$ (see Fig. 2 for composites with graphite and graphene). Based on the calculations, we established a hopping distance of 5.4 Å for the composite with graphite, 1.1 Å for the composite with graphene and 60 Å for the composite with CNTs. Note that a similar calculation within the framework of the two-dimensional problem (circle approximation) yielded localization lengths of the order of 10^{-11} cm, which is physically unreasonable.

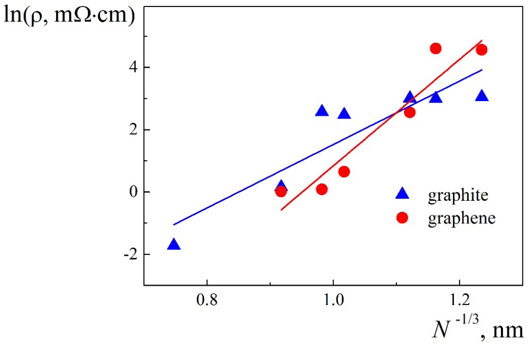

Fig. 2. Dependences of log resistivity for composites with graphite and graphene on $N^{-1/3}$ (N is the particle concentration)

Thus, to further calculate the percolation threshold, we use the three-dimensional model of the structure, although Monte Carlo simulations on the properties of a composite with graphene [11] considered graphene particles as two-dimensional circular nano-disks.

Our hypothesis is that flat graphene flakes coagulate, forming three-dimensional agglomerates during synthesis of the composite. For this reason, to interpret the results, we used the model proposed in [12] and schematically shown in Fig. 3. Here, spheres with small radius are agglomerates of conductive filler particles, spheres with large radius are non-conductive particles which are approximations of regions filled with a non-conductive matrix, and the circle in the inset connecting the filler particles is the tunneling space. As stated in [12], the percolation threshold depends on the ratio λ of the sizes of conductive and non-conductive particles:

$$\lambda = d_c / d_i,$$

where d_c, d_i are the sizes of conductive and non-conductive particles, respectively.

The higher the value of λ, the lower the percolation threshold. This model of the structure formed in the composite allows to explain the difference in the values of the percolation threshold subsequently found for composites with different fillers.

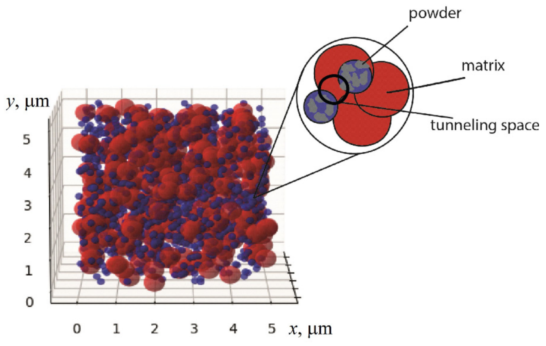

Fig. 3. Model of composite structure: spheres of small and large radii are, respectively, agglomerates of conductive and non-conductive particles (an approximation of regions filled with a non-conductive matrix). *Inset:* the tunneling space is shown by a circle with small radius connecting the filler particles (powder)

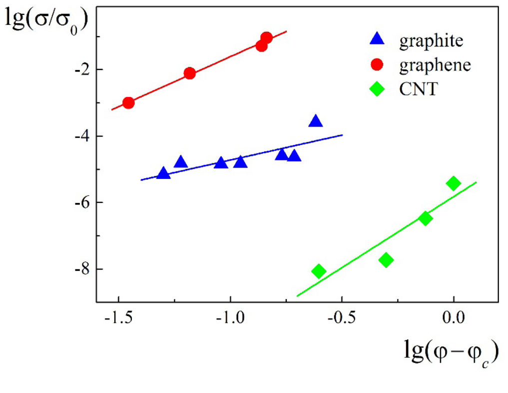

Fig. 4. Logarithmic dependences of normalized conductivity σ/σ_0 of composites with graphite, graphene and CNTs on logarithm of the difference in filler concentration φ and its value φ_c corresponding to the percolation threshold

To analyze the percolation model, we synthesized samples with different fillers with selected concentrations, considering their conductive properties in the absence of deformation.

The percolation threshold was determined graphically using the paired point method according to the well-known expression for conductivity σ (S):

$$\sigma = \sigma_0 \cdot (\varphi - \varphi_c)^t,$$

where φ, φ_c are the filler concentration and its value corresponding to the percolation threshold (wt.% relative to the final composite); *t* is the critical exponent (depending on the dimensionality of the structure); σ_0, S, is the conductivity of the filler [13].

Calculations were carried out based on data for the samples with filler concentrations at which a significant decrease in conductivities of composites is observed, i.e., a continuous cluster of conductive particles collapses.

Dependences of $\lg(\sigma/\sigma_0)$ on $\lg(\varphi - \varphi_c)$ were constructed to determine the critical exponent (Fig. 4).

Similar to [14], we simultaneously varied the values of φ_c and *t* to obtain an optimal linear relationship approximating the experimental data (see Fig. 4). The initially known range of possible values of the critical exponent *t* was used as a basis.

As a result, the critical exponent and the corresponding percolation threshold were determined for each of the composites. According to the calculation results, the values of the critical exponent were $t = 2.38$, 2.08 and 2.37 for composites with graphite, graphene and CNTs, respectively. These values generally correspond to the problem with spheres. Small deviations from the theoretical value may be associated with partial violation of dimensionality [13]. The obtained values of the critical exponent correspond to certain percolation thresholds, amounting to 12.0% and 16.5% for composites with graphite and graphene, respectively. The slight difference in these values can be explained, as mentioned above, by the difference in the values of λ for composites with different fillers in accordance with the model in [12]. The percolation threshold for the CNT-based composite was 0.1%, i.e., it turned out to be significantly lower than the values obtained for composites with graphite and graphene. Note that the value we obtained is in good agreement with the literature data for CNT-based composites [15, 16]. Such a small value of the percolation threshold is clearly due to the structural properties of the filler, namely, a very high aspect ratio of CNTs, making it possible to form a continuous conductive cluster even at their low content in the composite.

Study of strain–resistance relationship of samples Next, the main functional property of the obtained composites was investigated: the sensitivity of their electrical resistance to the degree of stretching. Fig. 5 shows the variation in the resistivity of composites under uniaxial stretching in the deformed regions where they are conductive. Here, the relative elongation of the sample is plotted as a percentage along the abscissa: $\delta = \Delta L/L_0 \cdot 100\%$ (L_0 is the initial length of the sample, ΔL is the variation in its length during stretching). The resistivity of the sample ρ, normalized to its value ρ_0 in the absence of applied stress, is plotted along the ordinate.

As expected, the lower the filler concentration in the matrix, the lower the elongation at which the resistivity in the sample starts to change. A significant increase in filler concentration leads to a decrease in stretching sensitivity of the composite: such samples had to be stretched to a much longer length for a noticeable change in resistivity. This effect of filler concentration on the sensitivity to tensile strain can be explained by an increase in the distance between its particles, which turns out to be equivalent to a decrease in filler concentration in the context of electrical conductivity.

Furthermore, as pointed out in [17], stretching of the composite leads to a change in the critical exponent t, which in turn indicates a change in the dimensionality of the structure, and consequently, the value of the percolation threshold, since the latter depends on both the dimensionality of the structure and the size ratio of conductive and non-conductive particles (as mentioned above).

As can be seen from Fig. 5, at the minimum concentration of the introduced filler, the graphite composite (13 wt.%) turned out to be the most sensitive to stretching, which begins to sharply change its resistivity when stretched by 2–3%. The resistivity of the graphene composite at its minimum concentration (16 wt.%) changed with a relative elongation of 12%, and that of the composite with CNTs (0.25 wt.%) with an elongation of more than 40%. In addition, an increase in concentration for composites with graphite and graphene expands the range of applied stresses where the composite preserves its resistivity. This effect is less pronounced for composites with CNTs, which may be due to the characteristics of the formed structure, for example, the curvature of nanotubes inside the composite [18].

The values of the gauge factor were calculated for different types of composites:

$$GF = (\Delta\rho_{max}/\rho_0)/(\Delta L_{max}/L_0),$$

where ρ_0, L_0 are the resistivity and length of the sample before stretching; $\Delta\rho_{max}$, ΔL_{max} are the maximum values of changes in its resistance and length, respectively.

This factor characterizes the strain sensitivity of the composite's resistance and can be used to compare data obtained for different composites [19].

Table 1 shows the values of the gauge factor for different types and concentrations of fillers. As can be seen from the data, composites with graphene filler are characterized by the highest sensitivity to stretching. The maximum sensitivity for all types of fillers is observed for composites with moderate concentrations (24 wt.% for graphene and graphite and 0.50 wt.% for CNTs). Analyzing the data in Table 1, we can conclude that the gauge factor first increases and then decreases with an increase in filler concentration. A likely explanation for this is that an increase in this concentration leads to two different effects. On the one hand, the range of strains where the composite preserves conductive properties is expanded (see Fig. 5), which should lead to a decrease in the GF value. On the other hand, the resistivity of the composite decreases in the absence of deformation (see Fig. 1), which should lead to an increase in GF. As a result of the competition between these two effects, an optimal concentration exists for each filler type, at which the maximum value of the gauge factor is achieved.

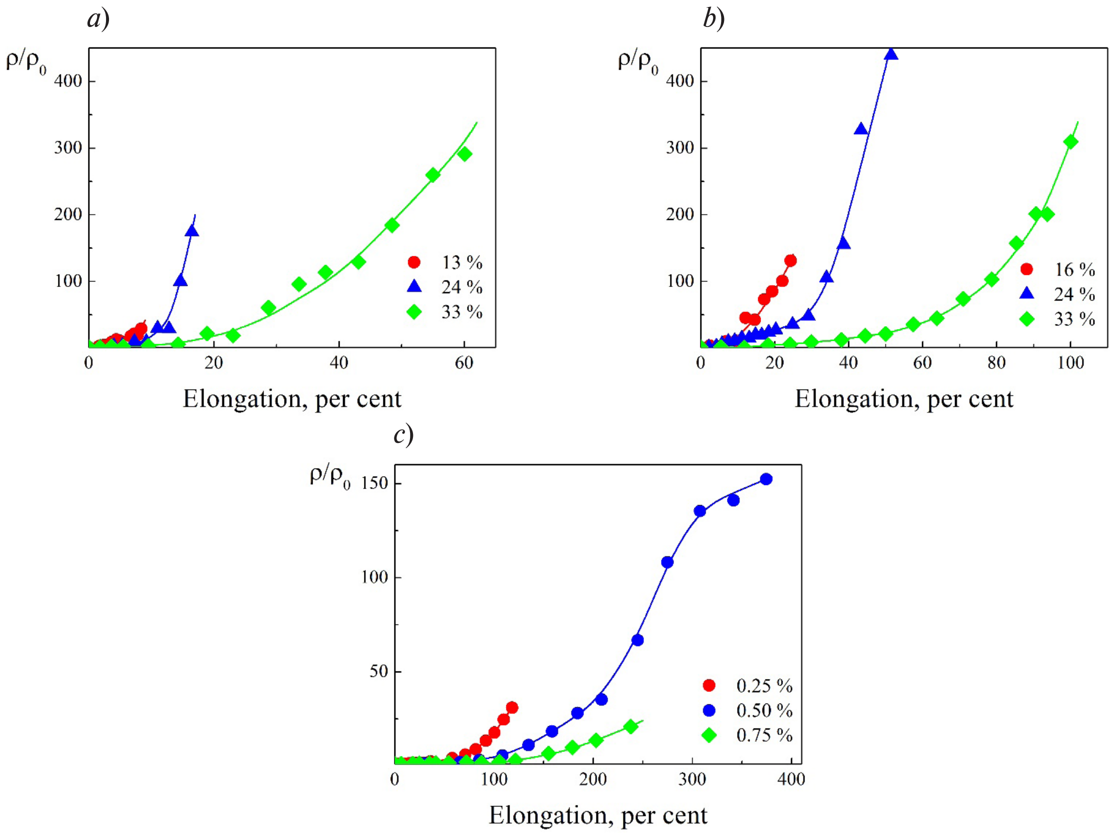

Fig. 5. Change in normalized resistivity of composite samples with graphite (*a*), graphene (*b*) and CNTs (*c*) in different concentrations, under stretching (elongation $\delta = \Delta L/L_0$)

Table 1

Gauge factor for samples of various composites subjected to stretching

Filler	Concentration, %	GF value
Graphene	16	534.7
	24	26661.0
	33	340.4
Graphite	13	224.3
	24	1005.0
	33	481.9
CNTs	0.25	25.3
	0.50	40.3
	0.75	8.4

Study of mechanical properties. As the first step in analysis of the mechanical properties of synthesized composites, we estimated their ultimate tensile strength, the nature of deformations developing in them under uniaxial static stretching and their relaxation properties.

First, the samples were repeatedly stretched in 10 mm increments until collapse. The length L of the sample was measured after each step (25 seconds after stress release). The obtained results are presented in Table 2 and in Fig. 6, showing the dependences of the ratio L/L_0 (L_0 is the initial length of the sample) on its relative elongation under stretching.

The data in Table 2 indicate that the maximum tensile strength in the case of the filler with CNTs is observed in the sample with the highest filler concentration, while the tensile strength in the graphene composite sample decreases gradually with increasing filler concentration. The ultimate tensile strength for the composite with graphite first increases with increasing filler concentration, then dropping abruptly.

An increase in filler concentration in the composite with CNTs expands the range of strain values at which the sample almost completely returns to its initial length (see Fig. 6,*c* and Table 2). This suggests that deformation within these strain ranges is purely elastic in nature. Increasing the CNT concentration in the composite improves its elastic properties, i.e., the higher the concentration, the fewer defects and residual strains the sample accumulates under uniaxial stretching.

The reason for this improvement may be related to both the three-dimensionality of the structure formed by introducing CNTs into the polymer matrix and the properties of individual particles of the filler itself. It is due to these characteristics that composites with CNTs can be considered the most promising for creating flexible sensor electronics devices. Residual strains arise in them only after reaching a certain critical strain value, which increases progressively with increasing filler concentration (see Fig. 6,*c* and the data in Table 2).

Table 2

Mechanical properties of polymer composite sample under stretching

Filler	Concentration	Elastic strain range	Ultimate tensile strength
	%		
Graphene	16	–	565
	24		542
	33		401
Graphite	13	–	485
	24		708
	33		380
CNTs	0.25	150	585
	0.50	250	556
	0.75	440	659

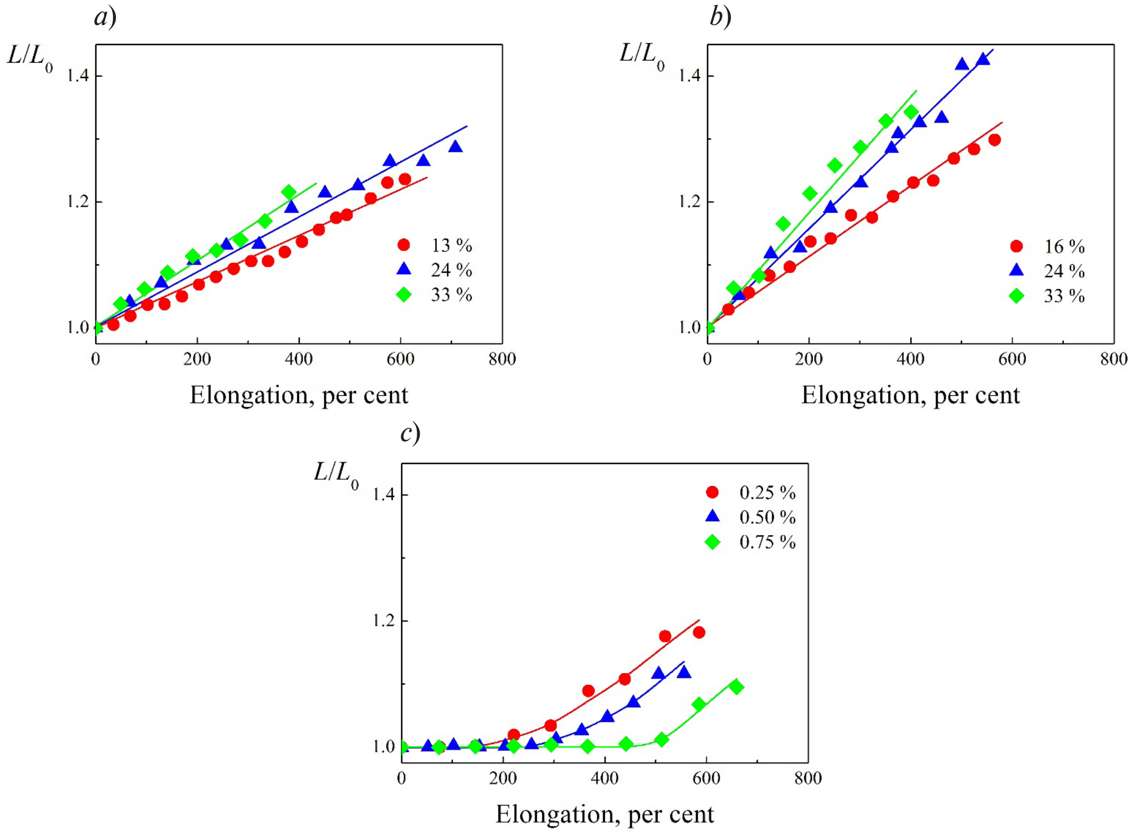

Fig. 6. Dependences of relative elongation L/L_0 of composite samples with graphite (*a*), graphene (*b*) and CNTs (*c*) in different concentrations on applied strain $\delta = \Delta L/L_0$ (measurements were carried out 25 seconds after each stress release)

In the case of composites with graphite or graphene fillers (see Fig. 6, *a*, *b*), the samples did not preserve their initial length even when minimal stresses were applied to them, which points to the emergence of irreversible plastic strains (along with elastic ones). Note that in both cases, the slope of the dependences in Fig. 6 increases progressively with increasing filler concentration, which indicates an increase in the proportion of residual strains, i.e., the effect of their accumulation during repeated stretching of the sample. There are two differences in the reaction of composites to repeated stretching for different types of fillers.

Firstly, the slope of the curves in Fig. 6 corresponding to composites with graphene is significantly greater than that for composites with graphite at the same (or similar) filler concentrations. This indicates that the proportion of residual strains is higher in the first case than in the second one. Secondly, a steeper increase in this slope is observed with increasing filler concentration for composites with graphene, i.e., the effect of accumulation of residual strains is more pronounced in this type of composites.

The results of the study on the relaxation properties of synthesized composites (for cases with CNT and graphene fillers) after 400% strain are shown in Fig. 7. Evidently, in the case of CNTs, samples that did not return to their initial length in 25 seconds after stress relief (at filler concentrations of 0.25 and 0.50%) exhibit almost complete relaxation within 60–80 seconds (see Fig. 7,*a*). In contrast, graphene composites do not return to their initial length (see Fig. 7,*b*) even 2–3 minutes after the load is removed. This result may be associated with jamming from flat filler particles inside polymer matrices during stretching. The sample with 33% graphene concentration undergoes virtually no relaxation; relaxation in the sample with 16% graphene concentration is weak and the sample with 24% graphene concentration exhibits the best relaxation properties, in terms of both the relaxation rate and degree.

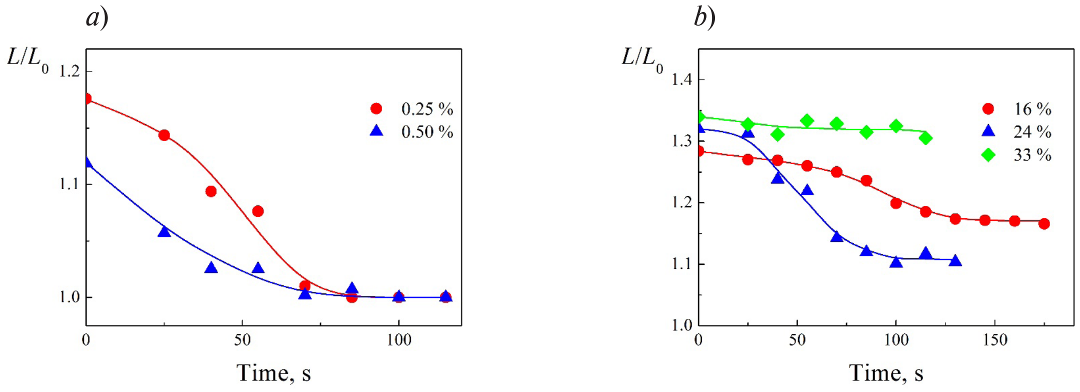

Fig. 7. Time evolution of relative elongation of composite samples with CNTs (*a*) and graphene (*b*) in different concentrations after stress release

Therefore, the filler concentration of 24% is close to critical for this material: once it is reached, the mechanical properties of the composite stop improving and start deteriorating. Note that this result indirectly corresponds to the data obtained in [20], analyzing the properties of composites based on polyvinyl acetate with graphene filler, considering samples in a narrower range of graphene concentrations than in our study (up to its possible critical value). However, the study found that its gradual increase initially leads to a sharp improvement in the mechanical properties of the composite, but this improvement subsequently slows down.

Conclusion

In this paper, we considered the electrical and mechanical properties of composites based on styrene butadiene rubber (SBR) with three different types of carbon fillers (graphite, graphene and carbon nanotubes), varying their concentrations. Analyzing the dependences of resistivity of composites on filler concentration, we determined the dimensionality of the filler particles formed during synthesis (which turned out to be close to three-dimensional in all cases), as well as the percolation thresholds, amounting to 0.1 wt.% for the composite with CNTs, 12.0 wt.% for the composite with graphite and 16.5 wt.% for the composite with graphene.

It was found that an increase in the concentration of all fillers in the SBR matrix broadens the range of the applied uniaxial stresses where the composite slightly changes its resistivity. However, this effect was less pronounced for the CNT-based composite compared with graphite and graphene-based composites. Sequentially increasing the concentration of all fillers in the SBR matrix led first to an increase in sensitivity of composite resistance to stretching and then to its decrease. Consequently, the greatest sensitivity was observed in composites containing about 24 wt.% of graphite or graphene filler and in composites with 0.5 wt.% CNT. The highest sensitivity of resistance to stretching was found in the composite with 24 wt.% of graphene filler; its gauge factor reached 2661, which is due to a high conductivity of this composite in an undeformed state and a narrow stress range where it preserves resistance.

Studies of mechanical properties of the obtained composites revealed that the ultimate tensile strength of graphene composites decreases with increasing filler concentration, that of CNT composites increases and that of graphite composites first increases and then sharply decreases. Furthermore, an increase in the CNT concentration in the composite expands the range of uniaxial strain over which the deformation remains elastic. In contrast, even the application of minimal stresses in composites with graphene and graphite leads to the emergence of plastic deformation. The proportion of this plastic deformation increases with repeated stretching of the sample as filler concentration is increased; this increase is more significant in the case of graphene compared with graphite. Composites with graphene exhibit the maximum values of the gauge factor, while composites with CNTs exhibit the best relaxation properties.

Thus, our studies and analysis indicate that composites with graphene are the most promising materials for further development of flexible sensor electronics devices from the standpoint of strain sensitivity of resistance, while CNT-based composites exhibit the best mechanical properties. Further research is necessary to optimize both the electrical and mechanical properties of polymer composites based on carbon fillers.

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