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The influence of temperature on electrophysical characteristics of carbon–containing polymer composites

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The influence of temperature on the electrophysical characteristics of polymer composites based on a matrix of ethylene vinyl acetate filled with carbon black (CB) is investigated. It is established that the most noticeable transformation of the temperature dependences of the resistivity $\rho(T)$ after the first heating–cooling cycle occurs in composite samples saturated with peroxide. Saturation of the composite with peroxide and the crosslinking of macromolecules leads to the N-shaped character of the dependence $\rho(T)$. The occurrence of hysteresis $\rho(T)$ and its character indicate that in the studied composites, the process of crystallization of the polymer matrix has significantly less effect on the destruction of the conductive mesh formed as a result of agglomeration of CB particles compared with the processes accompanying melting.

Keywords: polymer composites, carbon black, electrical conductivity, temperature dependence.

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Introduction

The electrical-conductive composite materials based on polymer matrices are used more and more in production of self-regulating heating elements, screen cladding of the high-voltage cables, electrodes of a various type, cladding of electromagnetic screening and many others [1–4]. Application of carbon black (CB) as a filler ensuring electrical conductivity despite a comparatively high percolation threshold ($C \approx 15\%$) does not complicate a mixing procedure to achieve the required homogeneity of the composites and provides required physical-mechanical and technological characteristics during production, for example, the screen cladding of the high-power cables. Using carbon nanotubes for the specifics of their geometry and the reduced content ($\sim 1.5-2\%$) does not exclude the probability of occurrence of local centers of discharge and breakdown in those places where the nanotubes come out to the surface of a conducting layer of the composite. That is why the polymer composite materials filled with carbon black continue to attract much attention of researchers and developers of new materials [5,6].

A large number of the studies is dedicated to the studies of the temperature dependences of these materials [7–10]. At the same time, one of the important tasks is to investigate factors affecting the temperature dependence of the specific volume resistance $\rho(T)$ and its stability in multiple increase and decrease of the temperature (thermal cycling), which is especially important for various applications of such composites, particularly, as self-regulating heating elements, and for functioning thereof. Most often, the polymer electrical-conductive composites have a pronounced temperature dependence of the specific volume resistance to demonstrate both its decrease with the temperature increase (resistance's negative temperature coefficient (RNTC)), and the increase in the values of this parameter (resistance's positive temperature coefficient (RPTC)). At this, the resistance's temperature coefficient is determined as $\frac{1}{\rho_{20}}\frac{d\rho}{dT}$, where ρ_{20} — the resistivity at $T = 20^\circ$C. Depending on the phase composition of the polymer matrix, the character of the temperature dependences of resistance can be fundamentally different. Thus, the non-crystal polymer composites have either RPTC or RNTC depending on the nature of the polymer and the filler, whereas at various portions of the temperature dependence $\rho(T)$ the composite with the polymer crystal phase can have both RPTC or RNTC [11,12]. There is evidently also ambiguous behavior of the values of resistivity at the room temperature ρ_{20} after heat treatment, i.e. its decrease or increase as a result of heating and cooling cycles.

Often, a path of the dependence $\rho(T)$ in cooling does not follow the path in heating and there is evident hysteresis [13,14]. The hysteresis of the electrical properties is found in the composites based on polyethylene with CB [15], fluoroplastic lacquer with carbon multi-wall nanotubes [16], polyethylene with vanadium dioxide [17]. At the same time, the hysteresis of the dependence $\rho(T)/\rho_{20}$ was not observed (almost absent) in the composites based on the epoxy matrix with CB [18].

The purpose of the present study was to investigate the influence of the temperature and thermal cycling as well as the content of CB and the peroxide cross-linking on the electrophysical characteristics of the carbon-containing polymer composites, in particular, on values and temperature dependences of the specific volume resistance in the samples with a large range of the values of the latter.

1. Samples and experimental procedure

The present study has investigated the samples of the composite material based on an ethylene-vinyl acetate matrix. The filler providing the electrical conductivity was carbon black (CB) C40 produced by „OMCARB“, whose content (C) varied from 15 to 35 wt. (%) to ensure passage through the percolation area. The composite samples were produced by mixing the polymer matrix in melt in the laboratory extruder EX30. Then, a part of the produced material was held for 30 minutes in the dicumylperoxide melt at the temperature of 55°C. The thickness of the samples produced by pressing was 2 mm. The measurements of the resistivity ρ within the interval $0-10^5\,\Omega\cdot$cm were performed by a four-probe method within higher values by means of the instrument Megger MIT 1025. The temperature dependences $\rho(T)$ were recorded by placing a measurement cell with the sample into the electric oven, and the temperature change step was 2−5°C depending on the portion, so was the accuracy of its installation and maintenance — ±1°C. At each measurement temperature the sample was pre-held for 3 minutes. The curves of differential scanning calorimetry (DCS) were recorded on the METTLER TOLEDO DSC823e unit within the temperature range 20−150°C. The rate of temperature change was 5° C/min. As with the temperature change, the endo- or exothermal processes in the studied composite samples are definitely correlated to changes in the state of the matrix (ethylene-vinyl acetate), then the DCS curves of the figures are given as calculated per unit mass thereof, which are corrected (multiplied) along the ordinate by the respective coefficient $k = 1/(1-C)$, where C — the CB content.

2. Experimental results

Figure 1 shows the dependences $\rho(T)$ for the three heating-cooling cycles of the initial (peroxide-free) composite samples containing 15, 25, 30 and 35% of carbon black. As follows from the obtained data, with the increase in the temperature from 20°C the resistivity of all the samples increases, and at the same time the temperature coefficient of resistance also increases. In the first cycle of heating the sample with CB $C = 15\%$ (Fig. 1, *a*), the sharp increase in the resistivity starts at the temperature ~ 72°C and goes to the maximum at $T = 86$°C. At this, the increase in the relative value $\rho(T)/\rho_{20}$ is $\sim 1.6\cdot10^5$. Then, there is sharp decrease in ρ with the temperature, while RNTC is higher than RPTC at the portion of increase in $\rho(T)$. The reverse (with the decrease in the temperature) dependence $\rho(T)$ appears like the curve in heating, but here the maximum $\rho(T)_{\max}$ is less and observed already at $T = 74$°C, i.e. it is shifted towards the smaller temperatures approximately by 10°C. As it is clear from the figure, in the second cycle of heating-cooling of this sample there is evidently no substantially qualitative transformation of the dependence $\rho(T)$, but it includes strong increase in the maximum to $\sim 8\cdot10^5$ on the dependence $\rho(T)/\rho_{20}$. Figure 1, *a* clearly shows some irregularity at the portion of decrease in $\rho(T)$, which specifically exhibits in the process of the second and subsequent cycles of heating. As follows from the corresponding DCS curve, a position of this irregularity corresponds to the melting point of the main polymer crystal phase. As follows from the obtained data, the second and subsequent cycles of heating-cooling result in substantially smaller transformation of the temperature dependence $\rho(T)$.

With increase in the CB content in the composite composition, there is significant decrease in the magnitude of the maximum relative values $\rho(T)_{\max}/\rho_{20}$ (Fig. 1, *b*–*d*).For the samples with $C = 30$ and 35% CB, the irregularity observed at the portion of decrease in the direct dependence $\rho(T)$, is caused by another factor. This factor means melting of not the main (dominant), but rather additional polymer crystal phase at somewhat higher temperature, which additional phase is initiated by carbon black particles [19].

Figure 2 shows temperature dependences of the relative values of the resistivity $\rho(T)_{\max}/\rho_{20}$ for three cycles of heating-cooling of the composites containing 25, 30 and 35% CB, which were saturated with peroxide. It should be noted that the sharp increase in the resistivity as a result of saturation of the samples with CB $C = 15\%$ did not allow recording the temperature dependence $\rho(T)$ with sufficient accuracy. It is clear from the presented results that the character of the temperature dependence $\rho(T)_{\max}/\rho_{20}$ of such composites is substantially different from the initial ones unsaturated with peroxide. Starting from ~ 64°C, there is evidently the sharp increase in ρ in the first and subsequent cycles of heating. As follows from the DCS curves, at this temperature a process of melting of the polymer crystal phase starts. The end of the melting process ($T \approx 95$°C) corresponds to a kink point of the initial dependence $\rho(T)$, whereas the temperature coefficient of resistance remains positive, but somewhat less in the value. The further increase in ρ continues up to 150°C, and at the same time its relative value $\rho(T)/\rho_{20}$ for the sample with $C = 35\%$ increases in about 88 times.

The reverse dependence $\rho(T)$ recorded at the temperature decrease is of an essentially different character. First, there is decrease in the resistivity, then starting from the temperature of ~ 102°C it increases. At some temperature (within the interval 74−85°C), whose value depends on C, there is evidently a local maximum with a subsequent sharp fall of $\rho(T)$. It is clear that the temperature corresponding to this maximum coincides with the temperature of the crystallization process start on the DCS reverse curve.

In the second cycles of heating, the direct dependence $\rho(T)$ for all the samples is fundamentally different from the dependence $\rho(T)$ for the first cycle and already has an N-like appearance similar to the dependence $\rho(T)$

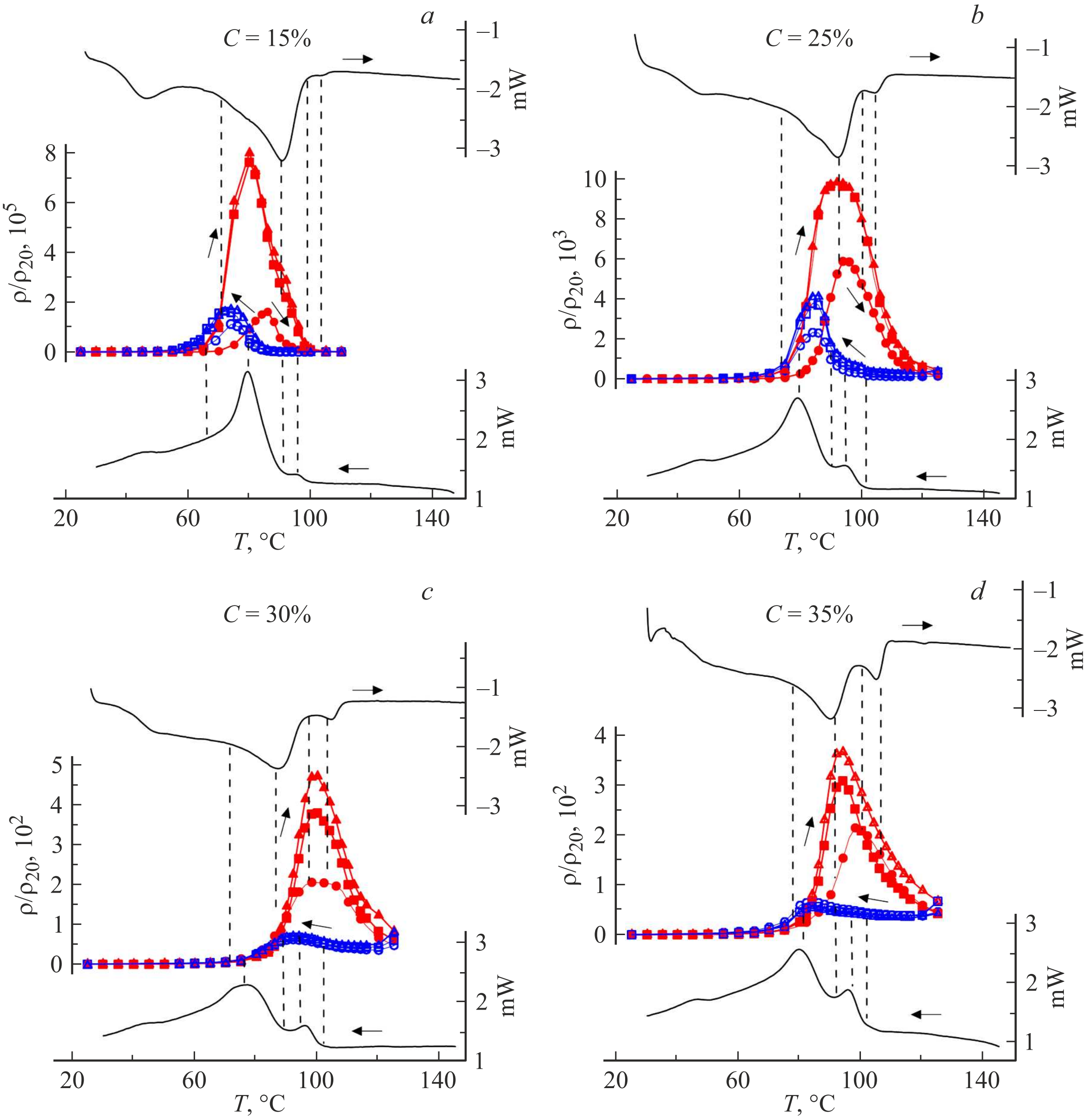

Figure 1. Temperature dependences of the resistivity $\rho(T)/\rho_{20}$ of the initial composite in heating (the dark symbols) and cooling (the light symbols). The circles — the first cycle, the squares — the second cycle, the triangles — the third cycle. The DCS curves are shown at top and bottom.

recorded during the cooling process in the first cycle. At this, there is evidently a local maximum and the RTC changes the sign two times. It is clear that the value of this maximum decreases with the increase in the CB content, and in the sample with CB $C = 35\%$ the extremum disappears, while the reverse dependences $\rho(T)$ exhibit a portion with the RTC value close to zero. In the third and subsequent cycles of heating-cooling, the change in the character of the direct and reverse dependences $\rho(T)$ was substantially less. As it is clear from Fig. 2, for all the concentrations and in all the cycles, in cooling the temperature corresponding to the local maximum is about by 10°C less than in heating. It is also clear that there is hysteresis of the dependences $\rho(T)$, but its value, especially, for the second and third cycles, is significantly less than for the initial composites unsaturated with peroxide.

The table shows the initial values of ρ_{20} as well as the values of this magnitude after the first and second cycles of heating-cooling. It is clear that after the first cycle there is significant increase in the values ρ_{20} both for the initial and peroxide-saturated samples. After the second cycle, the changes of ρ_{20} are not so obvious, and, besides, the character of the changes is different for the initial and peroxide-saturated samples. If the former continue to exhibit rise in ρ_{20}, the latter have some reduction of this parameter.

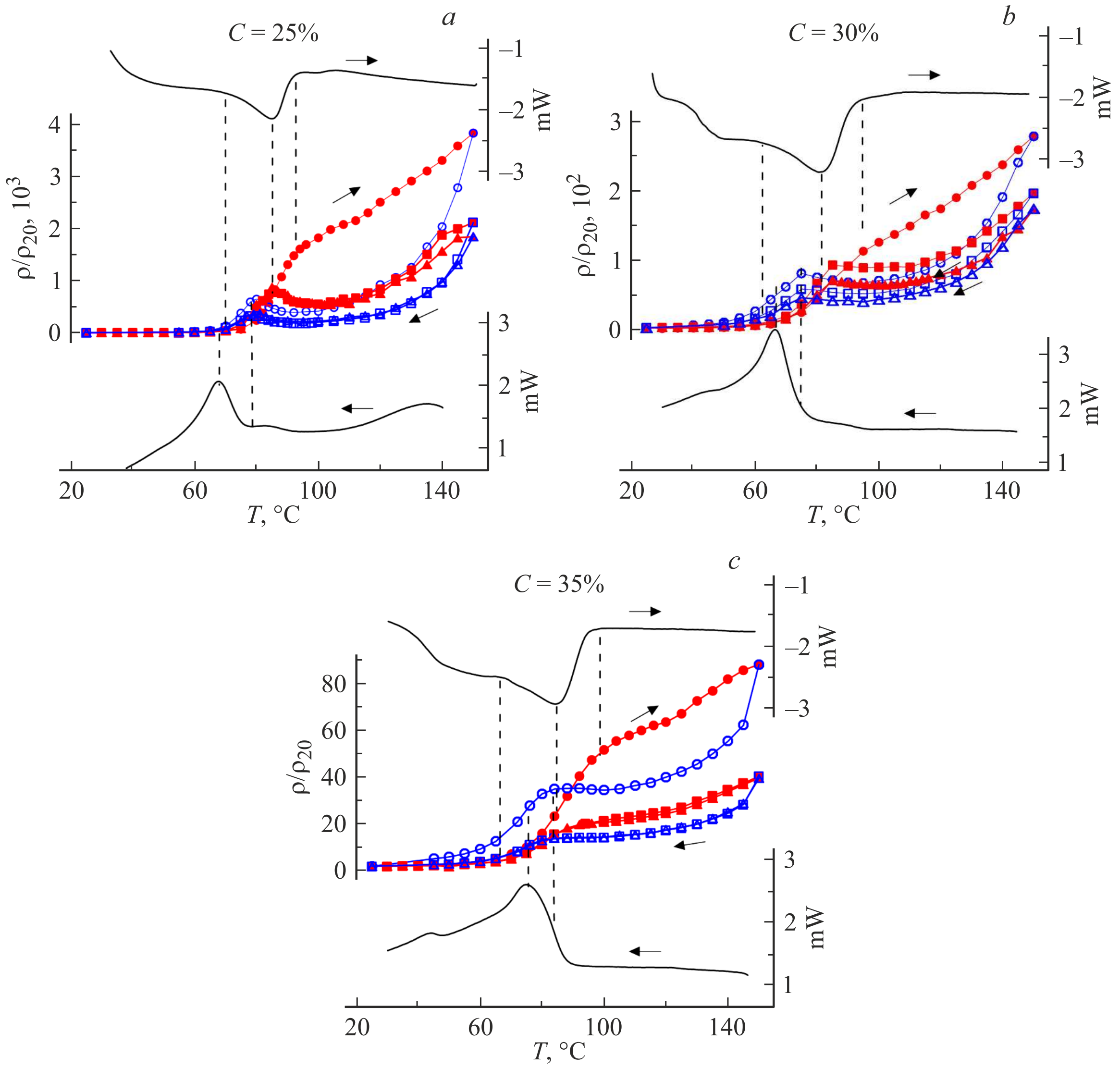

Figure 2. Temperature dependences of the resistivity $\rho(T)/\rho_{20}$ of the composite saturated with peroxide, in heating (the dark symbols) and cooling (the light symbols). The circles — the first cycle, the squares — the second cycle, the triangles — the third cycle. The DCS curves are shown at top and bottom.

Parameters of composite samples

Parameters	Initial samples			Samples saturated with peroxide		
	Initial value	After 1 cycle	After 2 cycle	Initial value	After 1 cycle	After 2 cycle
C, %	ρ_{20}, $\Omega \cdot$ cm	ρ_{20}, $\Omega \cdot$ cm	ρ_{20}, $\Omega \cdot$ cm	ρ_{20}, $\Omega \cdot$ cm	ρ_{20}, $\Omega \cdot$ cm	ρ_{20}, $\Omega \cdot$ cm
15	$1.59 \cdot 10^5$	$1.9 \cdot 10^6$	$2.3 \cdot 10^6$	–	–	–
25	68.5	408	506	$5 \cdot 10^4$	$8.5 \cdot 10^4$	$8.3 \cdot 10^4$
30	4.9	6.2	6.6	159	616	566
35	2	3.3	3.5	7.5	15.4	14

3. Discussion

In our opinion, the strong increase in ρ_{20} can be explained by the fact that except for rearrangement of the permolecular structure of the composite polymer matrix, the additional substantial influence is made by the changes of the sizes of current-conductive fragments of the CB particles, which results from their agglomeration at the

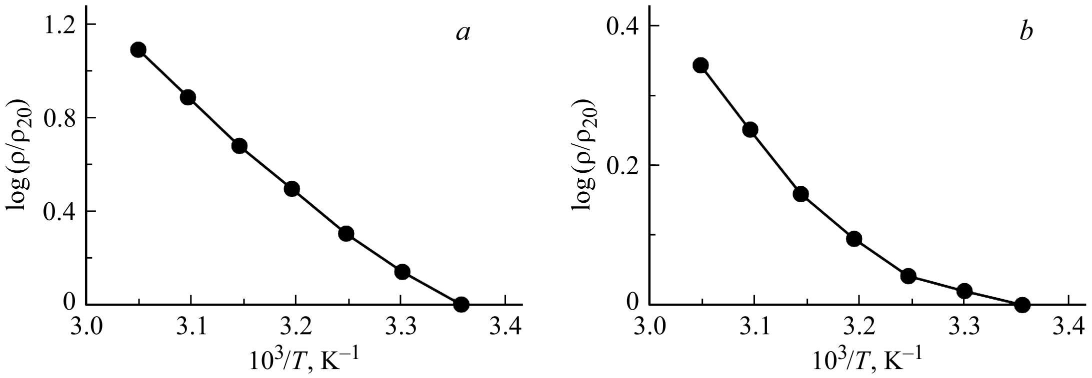

Figure 3. Dependences $\log(\rho/\rho_{20})$ on 1000/T for various CB concentrations (log — the decimal logarithm).

temperatures above the melting point of the polymer crystal phase.

The increase in $\rho(T)$ with the temperature at the initial portion (from 20°C) both for the initial and peroxide-saturated samples is explained by a substantially bigger value of the temperature expansion coefficient of the polymer matrix in comparison with CB [1]. As a result, with increase in the temperature there is opening of current-conductive circuits, thereby resulting in the rise of the resistivity. Starting from the temperature approximately equal to 60°C, the portion of the sharp increase in $\rho(T)$ is correlated to melting of the crystal phase of the matrix and more intense destruction of the current-conductive mesh formed by the CB particles. Upon the end of the melting process, with further increase in T, the initial samples unsaturated with peroxide exhibit reduction of the viscosity of the polymer matrix. As a result, the mobility of the CB particles increases, they agglomerate, new conductive paths are formed and, as a consequence, there is sharp decrease in ρ. One of the typical differences of the temperature dependence of the resistivity for the composite with the CB concentration of 15% is the fact that in comparison with other samples, the $\rho(T)_{\max}$ maximum on the direct dependence $\rho(T)$ is observed at the temperature, which is substantially lower than the melting point of the main polymer crystal phase (the temperature of $T \approx 91$°C, corresponding to the peak of the DCS curve). The position $\rho(T)_{\max}$ ($T \approx 86$°C) approximately corresponds to the melting start of this phase. The melting point has an evident sharp fall $\rho(T)/\rho_{20}$. The said difference can be explained by the specifics of the conductivity mechanism at such CB concentration. Based on the values ρ_{20}, it can be concluded that in the samples with $C = 15\%$ the conductive mesh of the CB particles is still unformed and the main conductivity mechanisms include the jump mechanism caused by jumps of the electrons having the energy near the Fermi level, and the mechanism correlated to tunneling of electrons through potential barriers between closely (less than 5 nm) located conductive CB particles [20]. It is indicated by the temperature dependence of the resistivity in the coordinates $\log(\rho/\rho_{20})$ on 1000/T, which for the samples with such a concentration was linear (Fig. 3, a). At high concentrations, such a dependence had a substantially non-linear character (Fig. 3, b). The availability of just such a conductivity mechanism in the samples with CB $C = 15\%$ is also confirmed by the fact that for them the dependence of the current density j on the field strength E (the current-voltage characteristic) was described with good accuracy by the Fowler–Nordheim relationship [21,22]:

$$j = AE^2 e^{-\frac{B}{E}},$$

where A and B — the constants. In the samples with $C = 25$, 30 and 35% the dependence $j(E)$ was linear. It follows from this that the jump conductivity mechanism and tunneling of the current carriers are affected by thermally-stimulated processes, which occur within the temperatures preceding the melting point of the polymer crystal phase of the composite.

As it is clear from Fig. 1, after the first cycle, the $\rho(T)/\rho_{20}$ maximum both at the direct and reverse dependences is shifted approximately by 6° towards the low temperatures, which is true for all the samples. There was no such shift after the second and subsequent cycles. It is correlated to the DCS data, after the first cycle the maximum at the corresponding curve was also shifted towards the low temperatures, but by a lesser value, about by 2−3°C (Fig. 4). The observed shifts can be explained by some rearrangement of the permolecular structure of the matrix as well as of the structure of the conductive component (CB agglomerates) of the composite, which results from the initial thermal impact. The conductivity and its temperature dependence turn out to be more sensitive to this rearrangement. It is clear from Fig. 1 that both the first and subsequent cycles have a pronounced hysteresis of the dependence $\rho(T)$, wherein the hysteresis value, which can be determined by an area limited by the direct and reverse dependences $\rho(T)$, noticeably increases after the first cycle.

In the samples saturated by peroxide, some decrease in ρ observed above 84°C (Fig. 2), i.e. after completion of the process of melting of the polymer crystal phase can also be explained by the agglomeration of carbon black particles becoming more mobile. But, no such a sharp decrease

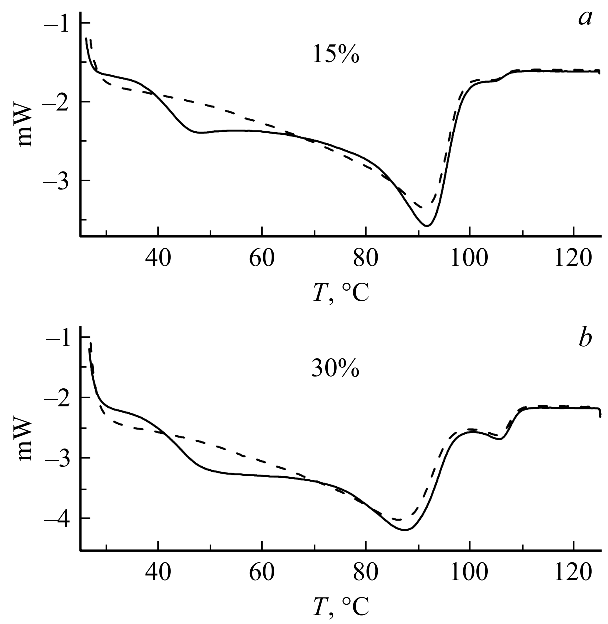

Figure 4. DCS curves of the initial samples, the solid line — the first cycle, the dashed line — the second cycle.

in $\rho(T)$ (as in the initial composites) and much smaller values $\rho(T)_{\max}/\rho_{20}$ apparently are correlated to influence of fragments of cross-linked matrix macromolecules. With further increase in the temperature, the effect of thermal oscillations starts prevailing, i.e. those oscillations which partially destroy ohmic contacts in the current-conductive CB circuits, thereby resulting in the increase in $\rho(T)$. The sharp transformation of $\rho(T)$ observed after the first cycle of heating can be explained by the undergone process of cross-linking of the polymer matrix macromolecules, which is caused by the peroxide molecules.

As already noted above, the dependences $\rho(T)$ have a hysteresis especially pronounced in the samples unsaturated by peroxide. Being the hysteresis, but not a result of „lagging " of the actual sample temperature from the given temperature or the result of some irreversible rearrangement of the structure, it is confirmed by the fact that the direct and reverse dependences are not just shifted along the temperature scale, but substantially differ in the values and their behavior is repeated in the second and subsequent cycles of heating-cooling. It is clear from Fig. 1 that the value of increase in $\rho(T)/\rho_{20}$ at the reverse dependence is much less than at the direct one, i.e. in cooling the conductivity becomes significantly much higher than in heating. In the second and third cycles of the increasing temperature, the value of $\rho(T)_{\max}$ not only reaches this value, but even exceeds this value of the first one. This behavior can be explained by the fact that the value of $\rho(T)$ depends on a prehistory of the state as a result of the specifics of the processes of destruction and formation of the current-conductive structure, which occur during heating and cooling the samples. In our opinion, the following fact can explain the occurrence of the hysteresis at the temperature dependences $\rho(T)$ studied in the present study, which manifests itself in the fact that in the reverse process of temperature reduction the resistivity of the composite becomes substantially much lower than in heating. Figure 5 schematically shows the changes of the composite structure, which occur in increasing and decreasing of the temperature. As noted above, the rise of $\rho(T)$ at the initial portion is correlated to a significantly higher value of the temperature expansion coefficient (TEC) of the polymer matrix and, in particular, the polymer crystal fragments, in comparison with the carbon black particles and their agglomerates correlated to these fragments, thereby resulting in segregation of the CB particles and, in turn, partial destruction of the conductive circuits. As a result of melting of the polymer crystal phase (PCP), the destruction process becomes quite intense, thereby resulting in sharp increase in $\rho(T)/\rho_{20}$. Upon the end of the melting process, with further increase in T, the viscosity of the polymer matrix decreases. As a result, the mobility of the CB particles increases, they agglomerate and new conductive paths are formed, which is accompanied by sharp decrease in $\rho(T)/\rho_{20}$. In the reverse process, with decrease in the temperature as a result of interaction of the CB agglomerates with forming crystal fragments of the matrix and of the thermal fluctuations, the initial conductive circuits are partially destroyed and the $\rho(T)$ increases, but to the values which are much less than in melting. The further decrease in the temperature results in reduction of $\rho(T)$ due to the above-mentioned difference of TECs of the polymer matrix and the CB particles. At the same time, there is substantially bigger reduction of sizes of the polymer crystal fragments in comparison with the distance between the CB particles bonded to these fragments, whereas in a part of them they touch each other, and, therefore, the difference between them can not change anymore. As a result, there is „slipping" of the polymer crystal fragment in relation to the CB particles bonded thereto and their agglomerates. The new process (second cycle) of heating results in the fact that as a result of accompanying increase in sizes of the crystal fragments due to the above-said difference of TECs there is segregation of the CB particles, disruption of the ohmic contacts and, as a consequence, destruction of the conductivity of the circuits. It also explains a much bigger value of $\rho(T)_{\max}$ in the second cycle of heating in comparison with the first one, which is observed for all the initial samples unsaturated with peroxide. The third and subsequent cycles of heating and cooling are already accompanied by much smaller changes of the dependence $\rho(T)$.

Thus, the process of formation of the polymer crystal structure of the composite substantially less affects the destruction of resulted agglomeration of the conductive mesh of the CB particles in comparison with the processes accompanying the melting. The peroxide saturation and cross-linking of the matrix macromolecules and, as a consequence, formation of a more rigid polymer frame

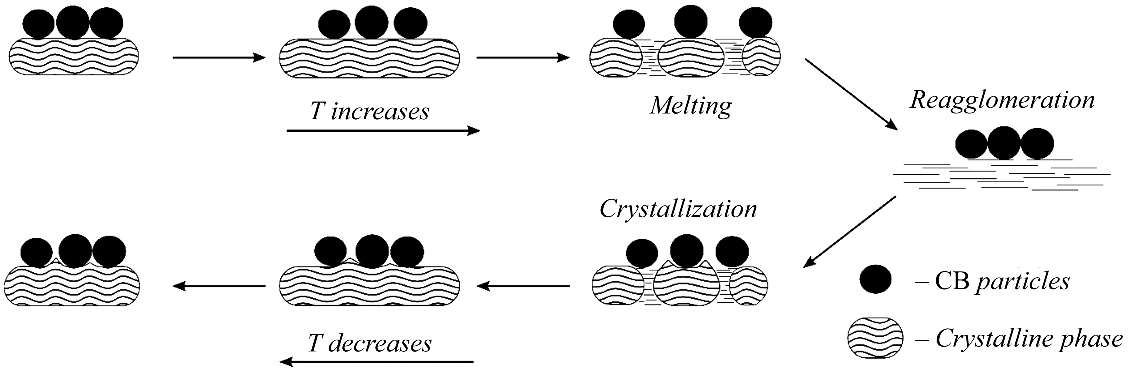

Figure 5. Transformation of the composite structure with increase and decrease in the temperature.

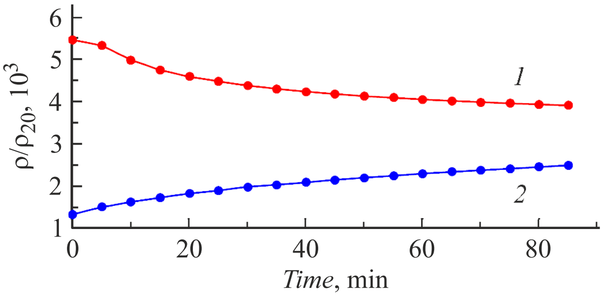

Figure 6. Dependences ρ/ρ_{20} for the sample $C = 25\%$ on the holding time at the temperature of 95°C in heating (the line *1*) and cooling (the line *2*).

results in significantly less hysteresis of the dependence $\rho(T)$ (Fig. 2).

The features of the above-described processes of transformation of the internal structure of the composite also explain no equivalence of the equilibrium states, in particular, the values of $\rho(T)$ corresponding to the equal values of the temperature, but which in one case are reached in transition from lower temperatures, and from higher temperatures in another. It is illustrated on Fig. 6, which represents the dependences $\rho(T)/\rho_{20}$ for the sample with CB $C = 25\%$ on the holding time of the fixed temperature, which corresponds to the maximum ρ on the direct dependence $\rho(T)$, i.e. at the temperature of 95°C. In one case, this temperature was reached as a result of heating, starting from the room one, while in the second case after cooling from $T = 125$°C. It is clear that despite quite long holding these two cases do not exhibit the same values of ρ. Such a character of the dependence $\rho(T)/\rho_{20}$ was also in the samples with another CB content.

Figure 7 shows the dependences $\rho(T)/\rho_{20}$ on the holding time t for the samples with $C = 15$ and 35% at some typical temperatures, i.e. at the temperature corresponding to the melting point of the dominant polymer phase (88°C) and

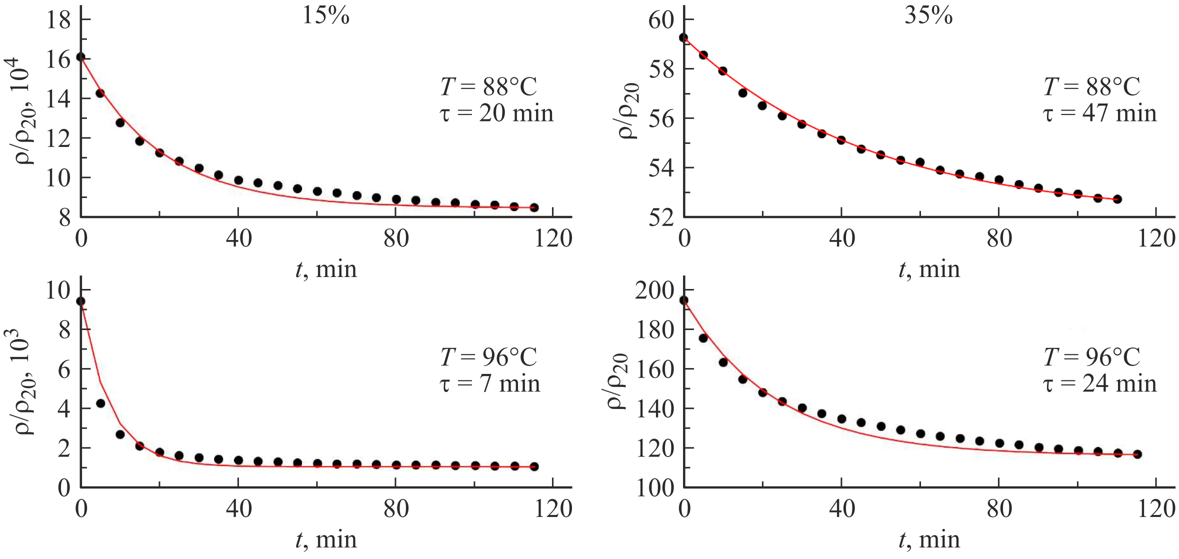

Figure 7. Dependences ρ/ρ_{20} on the holding time t for the samples with $C = 15$ and 35%. The circles — the experiment, the line — the calculation.

at the temperature of the end of the melting process of this phase (96°C). It is clear that the said dependences are of a relaxation character and with good accuracy are described by the expression $\rho = \rho_0 \exp(-t/\tau)$, where τ — the relaxation time. With the increase in the temperature T, the time τ of setting of the equilibrium state of the conductive structure is sharply reducing, while with the increase in the CB content this time increases. One of the typical moments consists in the fact that the samples with the various CB content, but at the temperatures corresponding to $\rho(T)_{\max}$ (88 and 96°C for $C = 15$ and 35%, respectively), the time τ turns out to be almost the same.

Conclusion

Thus, based on the results obtained in the present study, the following conclusions can be made.

It is found that in all the studied samples, after the first cycle of heating-cooling, ρ_{20} increases. In the samples unsaturated with peroxide, there is also significant increase in $\rho(T)_{\max}$ at the direct dependences $\rho(T)$. The value of $\rho(T)_{\max}$ at the reverse dependences changes in a significantly lesser degree as a result of thermal cycling.

The most noticeable transformation of the dependences $\rho(T)$ is in the composite samples saturated with peroxide after the first cycle of heating-cooling. In the subsequent cycles of heating-cooling, these samples exhibit higher thermal stability. It is explained by the cross-linking of the polymer molecules, which occurs under the high temperature.

It is found that saturation of the composite by peroxide and undergoing cross-linking of ethylene-vinyl acetate macromolecules leads to the N-shaped character of the temperature dependence $\rho(T)$ both in heating and in cooling.

The occurrence of the hysteresis $\rho(T)$ is caused by the fact that the process of formation of the polymer crystal structure of the composite substantially less affects the destruction of resulted agglomeration of the conductive mesh of the CB particles in comparison with the processes accompanying the melting. The peroxide cross-linking of the matrix macromolecules strongly reduces the hysteresis of the dependence $\rho(T)$.

It is demonstrated that the processes of setting the equilibrium states of the conductive structure of the carbon-containing composite are of a relaxation character. The relaxation time sharply decreased with the temperature and substantially increases with the increase in the CB content.

Conflict of interest

The authors declare that they have no conflict of interest.

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