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RESEARCH ARTICLE

Features of changes in the electrical resistance of mixtures of crystallizing polymers with carbon black upon heating

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Abstract

Objectives. The effects of positive and negative temperature coefficients (PTC and NTC, respectively) in carbon black-filled conductive polymer composites based on high-density polyethylene grade 277-73 and polypropylene grade 01050 were investigated. Carbon black electrically conducting grade OMCARB C-140 (UM-76) was used as the filler.

Methods. To study the electrical characteristics of the compositions, plates were pressed with brass contact electrodes at the plate ends to simulate polymer heaters. The electrical resistance of the samples was evaluated using an ohmmeter DT9208A (*RESANTA*, Latvia). Tests at elevated temperatures were carried out in an SNOL 3.5 heat chamber (*NPF TherMIX*, Russia) with a heating rate of ~3°C/min. The crystallinity of the samples during heating was assessed by differential scanning calorimetry on a DSC 204F1 Phoenix device (*NETZSCH*, Germany) with a heating rate of 3°C/min.

Results. The complex PTC and NTC mechanisms in mixed polymer compositions are not solely related to thermal expansion and melting of the polymer. While changes in the electrical resistance of carbon-filled polymer composites are associated with the presence of crystalline regions with defects, the destruction of the conductive channels occurs at the earliest stages of polymer melting due to the formation of expanding amorphous “microdroplets” of the hot melt. For a carbon-filled, electrically conductive mixture of polyethylene and polypropylene, the magnitude and nature of the change in the peak temperature of the PTC depends on the melting onset temperature of the lowest-melting phase of polyethylene. At the same time, the heterogeneity of the mixtures of crystallizing polymers with technical carbon increases the thermal stability of the material by expanding the PTC zone into the melting region of the higher-melting phase of polypropylene. When comparing electrically conductive compositions of polymers with different melting points and carbon black, the low-melting polymer determines the temperature of self-regulation and the nature of PTC, while the high-melting polymer shifts the jump in electrical conductivity to the region of elevated temperatures.

Conclusions. The activation energies of carbon-filled mixtures of polyethylene and polypropylene, which are weakly dependent on the mixing method, are approximately 44 ± 3 kJ/mol. The obtained values are consistent with the activation energy values for the viscous melt flow process. The method of mixing the components in mixtures of carbon-filled compositions based on crystallizing polymers was found to have little effect on PTC. The use of carbon-filled polymer compositions with a mixed matrix of polyethylene and polypropylene allows for the regulation of the intensity of PTC and NTC.

Keywords

conductive polymer composites, positive and negative temperature coefficients, polyethylene, polypropylene, carbon black, degree of crystallinity, specific electrical resistance

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НАУЧНАЯ СТАТЬЯ

Особенности изменения электрического сопротивления смесей кристаллизующихся полимеров с техническим углеродом при нагревании

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Аннотация

Цели. Исследовать эффекты положительного и отрицательного температурных коэффициентов (ПТК и ОТК соответственно) саженаполненных электропроводных полимерных композиционных материалов на основе полиэтилена высокой плотности марки 277-73 и полипропилена марки 01050, где в качестве наполнителя использовали технический углерод специальной электропроводной марки Omcarb C-140 (УМ-76).

Методы. Для исследования электрических характеристик композиций были отпрессованы пластины с запрессованными на концах контактными электродами из обезжиренной латунной сетки, моделирующие полимерные нагреватели. Электрическое сопротивление образцов оценивали с помощью омметра DT9208A (*РЕСАНТА*, Латвия). Испытания при повышенных температурах проводили в термошкафу СНОЛ 3.5 (*НПФ ТермИКС*, Россия) со скоростью нагревания ~3°С/мин. Степень кристалличности образцов при нагревании оценивали методом дифференциальной сканирующей калориметрии на приборе DSC 204F1 Phoenix (*NETZSCH*, Германия) со скоростью нагревания 3°С/мин.

Результаты. Показано, что механизмы ПТК и ОТК в смесевых полимерных композициях носят комплексный характер и не связаны только с тепловым расширением и плавлением полимера. Изменение электрического сопротивления саженаполненных полимерных композиций происходит из-за наличия дефектных кристаллических участков. На ранних стадиях начала плавления полимера токопроводящие каналы разрушаются за счет появления расширяющихся аморфных «микрокапель» его расплава. Для саженаполненной электропроводной смесевой композиции полиэтилена и полипропилена величина и характер изменения пика ПТК зависят от температуры начала плавления наиболее низкоплавкой фазы полиэтилена. При этом гетерогенность смесей кристаллизующихся полимеров с техническим углеродом повышает термическую устойчивость материала за счет расширения зоны ПТК в область плавления более высокоплавкой фазы полипропилена. Для электропроводных композиций двух полимеров с различной температурой плавления и технического углерода показано, что низкоплавкий полимер задает температуру «саморегулирования» и характер ПТК, в то время как высокоплавкий полимер смещает скачок электрической проводимости материала в область повышенных температур.

Выводы. Установлено, что энергии активации смесевых саженаполненных композиций полиэтилена с полипропиленом мало зависят от способов смешения и составляют 44 ± 3 кДж/моль. Полученные величины совпадают со значениями энергии активации процесса вязкого течения расплава. Установлено, что способ совмещения компонентов смесей саженаполненных композиций на основе кристаллизующихся полимеров мало влияет на эффект ПТК. Установлено, что использование саженаполненных полимерных композиций со смесевой матрицей полиэтилена и полипропилена позволяет регулировать интенсивность эффектов ПТК и ОТК.

Ключевые слова

электропроводные полимерные композиционные материалы, положительный и отрицательный температурные коэффициенты, полиэтилен, полипропилен, технический углерод, степень кристалличности, удельное электрическое сопротивление

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INTRODUCTION

Although the effects of abnormally high positive and negative temperature coefficients (PTC and NTC, respectively) in polymer composites (PCs) have been known since the mid-20th century [1–3], researchers have yet to reach a consensus on the causes of these phenomena. At the same time, there are many patents for the application of carbon black-filled conductive polymer composites (CPCs) exhibiting PTC and/or NTC effects as various heating and thermostatic devices, heating elements with thermoregulation, self-regulating cables, etc. [4–16]. Such an abundance of patents and scientific publications, which indicates the promising industrial application of such materials, stimulates new research in the field of CPCs in order to understand the mechanism of PTC and NTC effects.

The selections of special grades of carbon black (CB) typically used as an electrically conductive filler in the production of CPCs is due to the peculiarity of the CB structure, in which the conductivity reaches 2.4 S/cm [17]. The electrical conductivity of CB polymer composites is achieved through the formation of conductive channels within the matrix structure, through which electrical charges flow [18–21]. The flow of electrons through interconnected carbon particles and their aggregates can occur through the mechanism of electronic conductivity [18, 22]. In polymer composites filled with CB, electron tunneling through thin dielectric layers (polymer matrix) at distances of ~5–10 nm also becomes possible [18, 22, 23].

In the case of crystallizing polymers, whose spherulites consist of dense crystallites and amorphous regions between them, local agglomeration of carbon particles is observed [24]. The observed concentration of carbon particles between crystallites during the growth of spherulites in polyethylene (PE) [24–27] permits the achievement of higher electrical conductivity values as compared to PCs based on amorphous polymers [28].

A significant increase in the electrical conductivity of polymer composites is achieved at a certain CB content, which is referred to as the percolation threshold, while the corresponding content at which the transition of CPCs from a dielectric to a semiconductor and further to a conductor occurs is called the percolation transition [29]. However, additional electrical conductivity with further increases in the CB content of the CPCs is impeded due to the formation of a stable electrically conductive cluster whose predominantly contact conductivity occurs in the volume of the polymer composite. The instability exhibited by the system of current-conducting channels at lower CB contents prior to the appearance of such clusters, which are stable and resistant to external influences, responds to thermal and deformation effects with a sharp increase in electrical resistance, giving rise to the PTC phenomenon in the studied polymer composites with CB.

The electrical characteristics of blended polymer compositions depend on the distribution of electrically conductive filler between polymer phases. In a study of high-density polyethylene (HDPE) compositions with polypropylene (PP) filled with CB with a primary particle diameter of 27 nm, CB was found to concentrate in the matrix of lower-melting PE [30]. The electrical conductivity of the CPCs can be increased by concentrating electrically conductive filler at the interface between the two polymers [30, 31]. The example of PE/PP mixtures shows that, despite the fact that carbon is concentrated in the PE phase, the percolation threshold of the composition remains virtually unchanged compared to filled PE [30, 32]. The distribution of CB along the phase boundary forms electrically conductive channels with a lower filler content [30, 31].

Analysis of scientific and technical information indicates accelerated annual growth in the number of publications and new developments in this field over the last two decades. However, the proportion of publications revealing the mechanism of the PTC phenomenon in polymer mixtures is relatively small. This work is devoted to the study of the peculiarities of the mechanism of the PTC phenomenon in mixtures of crystallizing polymers (PE and PP) prepared by various methods.

EXPERIMENTAL

The objects of the study were HDPE 277-73 Stavrolen (GOST 16338-85[1]) and PP 01050 Balen (CB 2211-074-05766563-2015[2]). CB grade OMCARB C-140 (UM-76)

[1] GOST 16338-85. Interstate Standard. Low-pressure polyethylene. Specifications. Moscow: Standartinform; 2005 (in Russ.).

[2] https://polimermsk.ru/image/catalog/product/passport/TU%202211-074-05766563-2015.pdf. Accessed January 21, 2026 (in Russ.).

(*Carbon*, Russia, CB 38-10001-94) was used as an electrically conductive filler. The preparation of the composite mixtures was carried out under the same conditions as in [33] using several methods to combine CB with PE and PP. The total content of CB in all composites was 20 wt % (11.7 vol %). The components were combined in two stages: following the preparation of two-component mixtures of PE/CB, PP/CB, or PE/PP, either a third component (PE, PP, or CB) was introduced or two mixtures were combined. Thus, various mixed composites (PE/CB)/PP, (PP/CB)/PE, (PE/PP)/CB, and (PP/CB)/(PE/CB) were obtained with PE and PP ratios equal to 1 to 1.

Test specimens having a length of $L = 120 \pm 2$ mm, width $b = 10 \pm 0.5$ mm, and thickness $\delta = 1.0 \pm 0.05$ mm were pressed with contact electrodes made of L-80 brass mesh (GOST 6613-86[3]) onto the ends at 180°C for 3 min and cooled in the press to 50°C. This helped to stabilize the crystal structure of the samples to prevent the appearance of the "calender" effect.

The electrical resistance of the samples was measured using a DT9208A ohmmeter (*RESANTA*, Latvia). Tests at elevated temperatures were carried out in a SNOL 3.5 oven (*ThermIKS*, Russia) at a heating rate of ~3°C/min. The temperature coefficient of electrical resistance α (1/°C) was calculated using Eq. (1):

$$\alpha = \frac{\Delta\rho}{\rho_0 \Delta T}, \quad (1)$$

where ρ is the measured specific volume electrical resistance (Ohm·cm); Δρ is the change in specific volume electrical resistance (Ohm·cm) with a change in temperature ΔT (°C); ρ_0 is the specific volume electrical resistance of samples (Ohm·cm) under normal conditions: PE/CB = 32.5 Ohm·cm and PP/CB = 11.5 Ohm·cm.

The change in the degree of crystallinity of polymers upon heating was studied using differential scanning calorimetry on a DSC 204F1 Phoenix device (*NETZSCH*, Germany) at a heating rate of 3°C/min. The degree of crystallinity D (%) was calculated using Eq. (2):

$$D = 100 \frac{\Delta H_m}{\Delta H_{cr}}, \quad (2)$$

where ΔH_m is enthalpy of melting of the crystalline phase of the sample, calculated taking into account the CB mass fraction (kJ/mol); ΔH_{cr} is the melting enthalpy of the crystalline phase of the polymer (kJ/mol). Values of D under normal conditions: PE/CB = 70.5%, PP/CB = 48.5%.

RESULTS AND DISCUSSION

Figure 1 shows the dependencies of the specific volume electrical resistances of the initial PE and PP compositions with CB during heating. The nature of the change in these dependencies corresponds to that described in the introduction. The presence of peaks on the curves ensures self-regulation of the heaters' power when the external temperature rises. The mechanism of this phenomenon was described in detail by in an earlier study [33]. The difference in the course of crystallization processes leads to a shift of the peak of the PTC of PP relative to the peak of PE to a higher temperature range, as well as to an expansion of the temperature range of the NTC of PP.

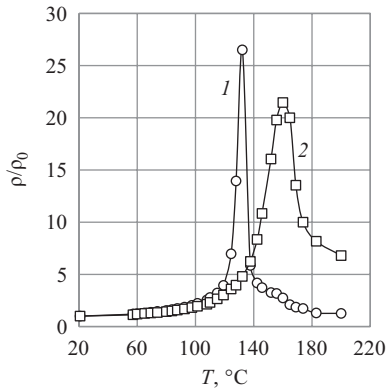

Fig. 1. Change in the specific volume electrical resistances (ρ/ρ_0) for PE/CB (*1*) and PP/CB (*2*) compositions when heated [33]

The phenomenon of NTC (decrease in electrical resistance) can result in a sharp increase in the power of the heater and its subsequent failure or even a fire. Currently, such phenomena are eliminated by radiation or chemical cross-linking of the polymer [28].

For greater clarity of these features and understanding of the mechanism of the PTC phenomenon, Fig. 2 shows graphs of the dependencies of the reduced degree of crystallinity D/D_0 (*1*) and reduced electrical conductivity σ/σ_0 (*2*) of PE (a) and PP (b) compositions with CB during heating. It should be noted that the CPCs samples were heated at a constant heating rate of ~3°C/min. For this reason, the data are considered to be kinetic, since the changes in time and temperature are proportional. The change in these dependencies for PE and PP compositions is similar, but here the electrical conductivity indicators decrease with increasing temperature. In PP CPCs, due to the higher melting point, this dependence is shifted to the higher temperature range. This indicates a connection between PTC and the

[3] GOST 6613-86. Interstate Standard. Square meshed woven wire cloths. Specifications. Moscow: Standartinform; 2005 (in Russ.).

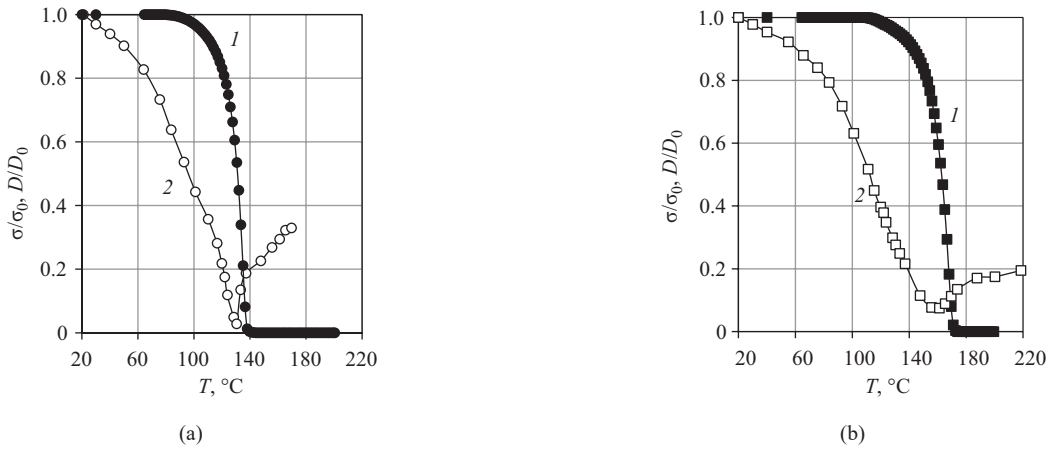

Fig. 2. Comparison of the dependence of the reduced degree of crystallinity D/D_0 (*1*) and reduced electrical conductivity σ/σ_0 (*2*) for (a) PE/CB and (b) PP/CB compositions when heated

process of decreasing crystallinity, which runs parallel to the decrease in their electrical conductivity until the onset of the NTC effect.

At the studied temperatures, the degree of crystallinity is characterized by a period of slow decline, followed by an accelerating decline until the polymer becomes completely amorphous. Unlike the degree of crystallinity, the electrical conductivity of CPCs begins to decrease significantly at much lower temperatures. This slow decrease in the electrical conductivity of CPCs can be partially attributed to thermal expansion during the heating of polymers, which is associated with the intensification of molecular thermal motion. But further in the experiment, as the temperature approaches the melting point of the crystalline formations that appeared in the final stages of polymer crystallization at low temperatures, this decline significantly outpaces the process of decreasing crystallinity. In contrast to the degree of crystallinity, which decreases to zero (Fig. 2), the PTC stage of the accelerated decrease in electrical conductivity of CPCs transitions to the NTC stage, in which electrical conductivity begins to gradually increase.

The above results of the study of the effect of heating on the properties of CPCs confirm the conclusions [28] that the mechanisms of PTC and NTC are not only related to thermal expansion and melting of CPCs. To clarify the described features of changes in electrical resistance in mixed CPCs, the activation energies of the described processes in the PTC temperature zone were determined. Figure 3 shows the dependence of the temperature coefficients of electrical resistance α on temperature in coordinates corresponding to the Arrhenius equation (the PTC temperature range of mixed compositions is highlighted with a dotted line).

The calculated activation energies of the studied mixed compositions, which are only weakly dependent on the methods of mixing the components, amount to 44 ± 3 kJ/mol. These values differ from the activation energies of their melting, but coincide with the values of the activation energy of the viscous flow of melts (E_{vf}) and the energy of destruction of conductive channels in the PTC zone (E_{el}) of the studied HDPE and PP [28]. For this reason, we may speak of their common mechanism.

Thus, the rate of increase in electrical resistance in polymer mixtures in the PTC temperature range depends on the presence of defective crystalline regions in the polymers. Due to the concentration of CB particles in the least heat-resistant interspherulite regions of crystallizing polymers and their mixtures, the destruction of conductive channels occurs at the earliest stages of polymer melting with the appearance of expanding amorphous microdroplets of its melt.

The effects of heating on changes in the electrical resistance of the CPCs under study are not limited to this phenomenon. Figure 4 shows the generalized results of electrical tests conducted on all of the above-described mixture samples. From Fig. 4 onwards, the dotted lines

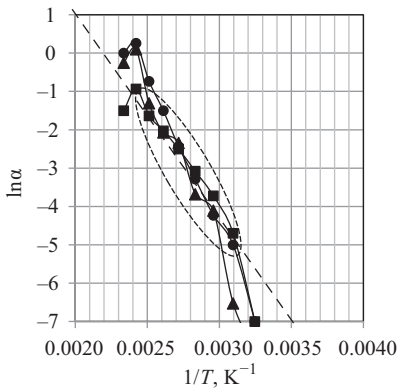

Fig. 3. Dependence of ln α on the reverse temperature ($1/T$, K^{-1}) for CPCs: (PE/CB)/PP (■), (PP/CB)/PE (▲), and (PE/PP/CB) (●)

highlight the detail previously studied [33] dependencies of two-component composites (PE/CB, PP/CB) used for comparison. Solid lines are used for three-component composites: (PE/CB)/PP, (PP/CB)/PE, (PE/PP)/CB, (PP/CB)/(PE/CB).

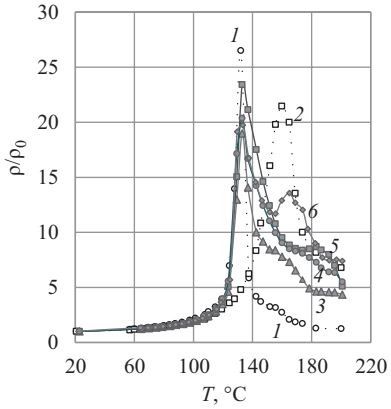

Fig. 4. Change in the specific volume electrical resistances (ρ/ρ_0) when CPCs are heated: PE/CB (*1*), PP/CB (*2*), (PE/CB)/PP (*3*), PP/CB/(PE/CB) (*4*), (PE/PP)/CB (*5*), and (PP/CB)/PE (*6*)

The first thing that catches the eye is the coincidence of all lines of mixed compositions in the PTC temperature range with the dotted line PE/CB, as well as the appearance of additional peaks of varying heights in the NTC temperature range of mixed PP/CB compositions. It should be noted that, unlike the PTC phenomenon in polymer heating devices, the NTC phenomenon is undesirable, since, at high operating temperatures of CPC heating elements, a drop in resistance can cause a sharp jump in power and destruction of the heater. As mentioned above, polymer heaters made of CPCs are currently subjected to radiation or chemical cross-linking to eliminate the NTC effect [28]. This is a technologically complex process that requires special equipment and reagents. This feature of electrical resistance change in mixed CPCs indicates the possibility of reducing the NTC effect. However, in Fig. 4 in the NTC temperature ranges, all lines diverge and intertwine, which makes it difficult to analyze the effect of the composition of mixed composites on the change in their specific volume electrical resistances (ρ/ρ_0). Therefore, the dependencies shown in the generalized Fig. 4 will be divided into three groups to take into account the intensity of the influence of the PP phase (the numbering of the lines is preserved in all figures).

The least influence on the nature of the change in specific volume electrical resistance is expected to be observed in the (PE/CB)/PP composite, in which CB is initially mixed with PE before mixing the concentrated PE/CB composition with PP. Due to the increased content of CB in the PE phase, the height of the PTC peak decreases.

Figure 5 shows the results of a study of the change in electrical resistance during heating of CPCs (PE/CB)/PP.

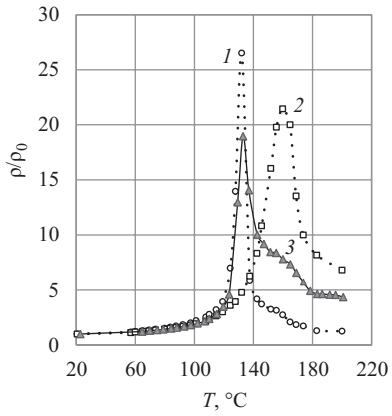

Fig. 5. Change in the specific volume electrical resistances (ρ/ρ_0) when CPCs are heated: PE/CB (*1*), PP/CB (*2*), and (PE/CB)/PP (*3*)

It should be noted that when melts with similar PE and PP contents are mixed, heterophase systems in which two continuous microfibrous phases are formed[4] [34]. The concentration of CB particles in the low-melting phase (PE) of the mixture leads to an increase in the stability of the system of conductive channels in its fibers and a consequent decrease in the peak height of the material's PTC. The NTC temperature zone of this mixture expands into the higher temperature region to partially capture the NTC zone of the PP/CB mixture. The appearance of PP traces in mixtures is commonly explained by the migration of CB particles from the PE phase into its phase [31, 32]. Although this factor cannot be ruled out, a more likely explanation may be the above-described beginning of the appearance of expanding microregions of amorphous PP melt. However, this increase (ρ increased approximately 2 times) is not sufficient for completely eliminating the undesirable NTC phenomenon in this composition.

Figure 6 shows the results of a study of changes in the electrical resistance of two CPCs with similar behavior when heated: (PE/CB)/(PP/CB) and (PE/PP)/CB.

The increased CB content in PP in these compositions results in the trace of PP in the temperature zone of its PTC becoming more noticeable and distinct to increase the resistance of the CPC data to overheating.

[4] Markov A.V. Technology of oriented multicomponent polymer films. Dr. Sci. Thesis (Eng.). Moscow: Lomonosov Moscow State Academy of Fine Chemical Technology; 2006 (in Russ.).

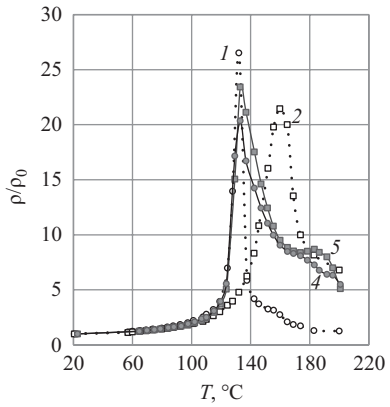

Fig. 6. Change in the specific volume electrical resistances (ρ/ρ_0) when CPCs are heated: PE/CB (*1*), PP/CB (*2*), (PE/CB)/(PP/CB) (*4*), and (PE/PP)/CB (*5*)

From the point of view of stable operation of CPCs at elevated temperatures, the best composition is (PP/CB)/PE (Fig. 7). The graph of the change in specific volume electrical resistance (ρ/ρ_0) during heating of the CPCs actually consists of two adjacent PTC peaks: the PE peak and the PP peak, which cover a wide temperature range from 120 to 160°C. This makes the latter CPCs the most resistant to overheating.

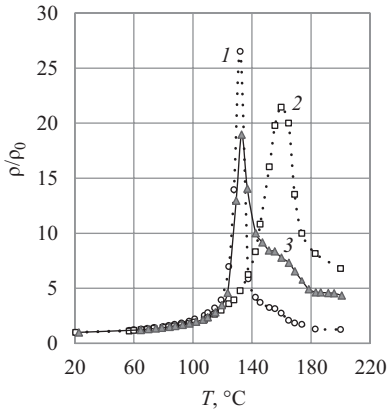

Fig. 7. Change in the specific volume electrical resistances (ρ/ρ_0) when CPCs are heated: PE/CB (*1*), PP/CB (*2*), and (PP/CB)/PE (*6*)

Thus, the method of combining components of crystallizing polymer mixtures with CB has little effect on the position of the PTC peak. The magnitude and nature of the change in the PTC peak of crystallizing polymer mixtures with CB is determined by the melting start temperature of the lowest melting phase (PE). Meanwhile, the heterogeneity of mixtures of crystallizing polymers with CB increases their thermal stability by expanding the PTC zone into the melting region of the higher-melting phase (PP).

CONCLUSIONS

The application of CPCs with a mixed polymer matrix consisting of two polymers having different melting points (e.g., PE and PP) permits adjustments to the intensity of the PTC and NTC effects. In this case, it is the low-melting polymer (PE) component of CPCs that determines the nature of PTC and the self-regulating temperature of the heater, while the higher-melting polymer with higher PTC and NTC (PP) shifts the jump in electrical conductivity of the mixed CPCs to the region of elevated temperatures, thus disrupting the operation of the heater. The results of the work made it possible to establish a number of phenomena occurring in CB-filled CPCs with PTC and NTC effects. For further research, it is necessary to consider PCs with PTC and NTC effects from the point of view of the filler, in particular, the geometric parameters of the technical specifications of special grades.

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Authors' contributions

A.V. Markov—conceptualization of the study, administration of the study, discussion and analysis of the results, writing the text of the article.

A.E. Zverev—setting up the experiment, studying the properties of the samples, collecting analytical data, processing, analyzing and visualizing the results, writing the text of the article.

E.V. Kalugina—consultation, discussion and analysis of the results obtained.

V.A. Markov—development of the methodology for the experimental section.

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