

Dynamics of the conductance temperature dependence for composite based on linear polyethylene with impurity of soot and calcite

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Abstract. Within the temperature 293 to 425 K and frequency 10^{-3} to 10^6 Hz ranges by using the direct and alternating currents, the dynamics of electrical conductance σ of linear polyethylene with the impurity of 20 wt.% soot and 20 wt.% CaCO_3 (calcite) has been investigated. It has been shown that for the solid state of polyethylene (below 380 K), the dependence of electrical conductance on the temperature T on both the direct (σ_{DC}) and alternating (σ_{AC}) currents can be described by the power dependence on $T/(T - T_0)$ (where T_0 is the temperature of the phase transition for polyethylene). It has been shown that when being repeatedly measured, the σ_{DC} and σ_{AC} values increase, and the power indexes of temperature dependence decrease. The measured values are stable after the fourth measurement. The greatest changes in the conductance, depending on the first and second measurements (almost three orders of magnitude), were observed at a temperature close to T_0 . It has been assumed that the dynamics of electrical conductance, depending on the number of measurements, is caused by the influence of the electric field on the ordering of impurity in polymer. It has been shown that for $T > 380$ K, the typical for liquids Arrhenius dependence of σ_{DC} and σ_{AC} on temperature is observed. It has been found that at the first measurement, the temperature dependence of σ_{DC} and σ_{AC} can be described by two activation energies, while for a stable state (starting from the fourth measurement) – by one activation energy (within the measurement error of the same for σ_{DC} and σ_{AC} and equal to 1 eV).

Keywords: electrical conductance, temperature dependence, linear polyethylene, calcite, carbon soot.

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1. Introduction

Composite materials based on linear polyethylene and main impurities of different types can be used for manufacturing the heating elements in different types of premises where air should be heated. In this case, in the presence of a large area of a heater, it is not necessary to heat up to high temperatures. It will allow to eliminate

the effect of reducing the concentration of oxygen in the rooms, which is typical for heaters with a high temperature. Due to the design of the surface of such materials, they can be used as wallpapers. Therefore, an important scientific task is to study the electrical properties of these composite materials at those concentrations of impurities, at which they have the most suitable parameters for practical use.

Electrical properties of polymers with different type conductive impurities are investigated for a relatively long time [1-9]. In the recent papers, nanoparticles [10-17], in particular nanotubes [18-21], were used to a greater extent as impurities. In the vast majority of studies on the influence of impurities on the electrical properties of polymers, the study was performed in a wide range of concentrations. In this case, the greatest interest was focused on the study of percolation phenomena [22]. However, in these works little attention was paid to the mechanisms of charge transfer and the dynamics of the properties of these systems in the process of exploitation.

Another important task is the features of the electrical characteristics of composites with two or more impurities with different properties. In polymer, the impurities are bound to the matrix with weak forces. Therefore, also an important task is to study the dynamics of change in the electrical conductance, depending on the number of measurements.

Therefore, the aim of the work was to study the dynamics of electrical conductance (from the first measurement) for composites suitable for use as heating elements, namely for the following composite: linear polyethylene + 20 wt.% soot + 20 wt.% CaCO_3 . As it follows from the analysis of the published data, the effect of carbon soot [23-26] and calcite [27] on the electrical properties of polymers were previously investigated. At the beginning of our studies, the results of other investigations where the above mentioned substances could be introduced into the polymer simultaneously were not known for us.

2. Materials and methods

As in [24], for this research we used linear low-density polyethylene. The polymer hybrid composite was prepared of linear low-density polyethylene Sabic LLDPE 318B, calcite filler Omiacarb 2t-tn (median particle size is 2.7 μm) on soot N220 (Kremenchug Plant of Technical Carbon). Formation of the composite material was carried out by preliminary crushing polyethylene into a powder (200...300 μm). The polyethylene powder was mixed with soot and calcite by using a high-speed mixer of the Henschel type. The powder mixture was processed using a twin screw extruder with the diameter 22 mm in length of 40 diameters cyclically at the temperatures 160, 190 and 210 $^{\circ}\text{C}$ and rotation speed of the screw 250 rpm. The composite was obtained in the form of granules that, after drying from the surface moisture, were processed into a belt sample on a single-screw laboratory extruder equipped with a slit head, cooling bath and pulling device.

A microphotography of the sample obtained using a scanning electron microscope at the voltage 20 kV is shown in Fig. 1. As it follows from this figure, after the technological operations, the impurities are homogeneously distributed in the polymer.

We studied electrical properties of the samples with the geometric sizes 20x20x0.71 mm. We used silver

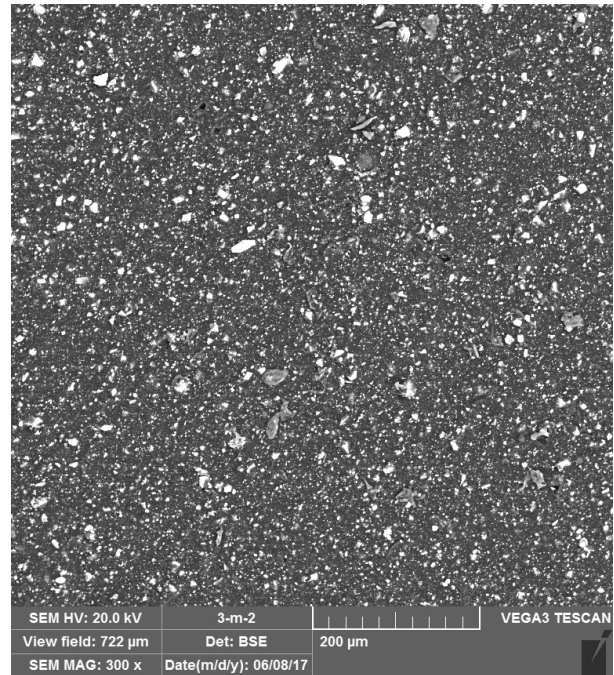


Fig. 1. Microphotograph of linear polyethylene with impurity of 20% carbon soot and 20% calcite obtained using a scanning electron microscope at the voltage 20 kV.

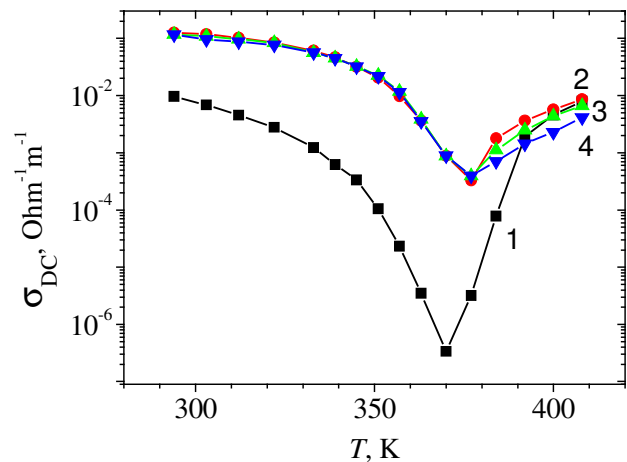


Fig. 2. Temperature dependence of conductance for linear polyethylene with the impurity of 20 wt.% soot and 20 wt.% calcite for the first (1), second (2), third (3) and fourth (4) measurements. The voltage 1 V was used when measuring.

paste as electrodes. The measurements were carried out within the temperature range 293...425 K. The temperature stabilizer allowed to maintain the chosen T -value with an error not worse than 0.5 K.

The conductance on direct current (DC) was determined using a source of DC voltage P4108 and an electrometer DC amplifier U5-11. The voltage, at which the measurements were made, varied within the range of 0.5 to 20 V. The predominant number of measurements was made at the voltage 1 V.

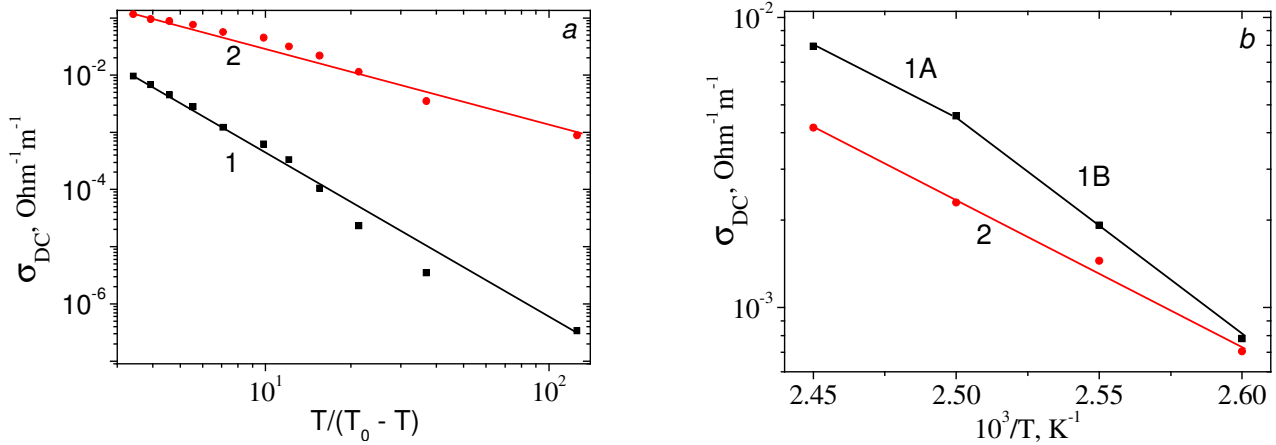


Fig. 3. Temperature dependences of σ_{DC} for the composite based on linear low-density polyethylene with soot and calcite impurities for $T < T_0$ (a) and $T > T_0$ (b) at the first (1) and fourth (2) measurements. Continuous lines on both (a) and (b) plots show the approximation of these dependences, respectively, by using the relations (1) and (2) with the values of parameters listed in Table 1.

The conductance and capacitance of the samples on alternating (AC) current were determined using the oscilloscopic method [28] within the frequency range $10^{-3} \dots 10^6$ Hz. To do this, we used the generator G6-36. For frequencies above 1 Hz, the resistance and capacitance at a certain frequency were determined from the oscillogram of the oscilloscope C1-83 that operated in the oscilloscope mode. At the frequencies less than 1 Hz, the oscillogram was recorded using a two-coordinate recorder. To determine the resistance and capacitance of DC current, the voltage of the measuring signal varied within 0.5 to 5 V. In most cases, measurements were made at the voltage 1 V (*i.e.*, at the same voltage like to that used in measurements on DC current).

3. Results and discussion

3.1. Dynamics of the conductance temperature dependence for linear polyethylene with impurity of soot and calcite at DC current

Fig. 1 shows the dynamics of temperature dependence of conductance of linear polyethylene with impurity of soot and calcite at DC current within the range of temperatures 293 to 425 K.

From the analysis of the dynamics observed in the measurement processes, it follows that the largest changes are observed between the first and second measurements. In addition, from Fig. 2 it can be concluded that the largest changes in conductance, depending on the measurement cycles, are observed at temperatures close to the melting point of linear polyethylene. As it follows from Fig. 2, the greatest change in conductance between the first and second measurements at the temperatures close to T_0 is almost three orders of magnitude.

At subsequent measurements (after the second one), the difference between the values of conductance is much smaller. It is higher than the error of measurements in the liquid state of polyethylene. But in this case, after the

fourth measurement, the changes in data did not exceed the measurement error in subsequent measurements. Therefore, in the future, the change in the properties of the samples, depending on the cycle of measurement, will be compared with the results of the first and fourth measurements.

From the analysis of Fig. 2, it follows that the temperature dependences of the conductance of linear polyethylene with the impurity of soot and calcite on DC current are different in solid (at $T < T_0$) and liquid (at $T > T_0$) phases. Therefore, they will be analyzed separately. In the solid state of polyethylene (Fig. 2), the conductance decreases with increasing the temperature. In general, this tendency is typical for metal conduction. For purely metal conduction, the value of σ_{DC} should decrease linearly with temperature. Our analysis showed that it is impossible to describe our obtained data by such a dependence. From the analysis of experimental data, it follows that the temperature dependence of conductance for $T < T_0$ can be described quite well by two power dependences, but it was difficult to agree the presence of temperature (of the order of 360 K), at which the power index varied, with existing perceptions about the conductance of polymers.

A detailed analysis of the temperature dependence of σ_{DC} for the composite based on linear polyethylene with impurity of soot and calcite (Fig. 3a) for $T < T_0$ showed that it can be described by the following equation

$$\sigma = \sigma_0 [T/(T - T_0)]^{-n}, \quad (1)$$

where σ_0 is the conductance at $T = \infty$, n – power index. The performed by us analysis of publications showed that in none of the articles known to us the relationship (1) was reported.

Fig. 3a shows the dependence of $\sigma(T/(T - T_0))$ in double logarithmic coordinates for σ_{DC} at $T_0 = 380$ K. It follows from this that the experimental data for both the first measurement (curve 1) and the fourth measurement (curve 2) are in good agreement with the relation (1).

Table 1. The values of parameters for σ_{DC} , at which the experimental data can be described with the smallest deviations by the relations (1) and (2).

T_0 , K	n_1	n_4	W_{1A} , eV	W_{1B} , eV	W_4 , eV
380	2.9 ± 0.3	1.3 ± 0.3	1.00 ± 0.05	1.52 ± 0.05	1.05 ± 0.05

Table 1 shows the values of parameters, at which the temperature dependences of σ_{DC} for the composite based on linear low-density polyethylene with the impurity of soot and calcite can be described using the relations (1) and (2) with the least deviations from the experimental results.

As it follows from our estimations, the power index in the relation (1) decreases from 2.9 ± 0.3 for the first measurement (n_1) down to 1.3 ± 0.3 for the fourth (n_4) and subsequent measurements. That is, the changes in conductance on the temperature, which are the most typical for the first and second measurements, are reduced with each subsequent measurement, reaching practically identical (for subsequent measurements) values after the fourth measurement.

These changes are apparently caused by formation under the influence of the electric field of bonds between the impurities introduced into the polymer (soot and calcite) that do not break when heating to the temperature 425 K. Because for these complexes, mainly, the charge transfer occurs through the composite.

Fig. 3b shows the temperature dependence at $T > T_0$. In contrast to the data for $T < T_0$, the shown in Fig. 3b temperature dependence of conductance can be described by the Arrhenius law inherent to the conductance of the most liquids. In this case, the formula for the temperature dependence of the conductivity has the form

$$\sigma = \sigma_0 \exp(-W/kT), \quad (2)$$

where, as in the relation (1), σ_0 is the conductance at $T = \infty$, and W – activation energy of the conductivity.

It is important to note that the temperature dependence of σ_{DC} for the first measurement can be described by not one activation energy, but as shown in Fig. 3b and Table 1, by two activation energies of W_{1A} and W_{1B} . It may be caused by participation of at least two types of ions different in their sizes, when the charge is transferred through molten polyethylene. In the stationary state (beginning with the fourth measurement), only ions of one type are obviously involved in the charge transfer.

Comparing Figs. 3a and 3b, the important regularity should be noted. If in the solid state of polymer (Fig. 2a), the conductance increases with each measurement (due to formation of new bonds between the impurities under the action of the electric field), then the conductance in the liquid state of polymer decreases with each measurement (Fig. 3b). That is, the influence of soot and calcite impurity on the dynamics of conductance of linear low-density polyethylene for solid and liquid states of polymer is fundamentally different. If for a solid state the

conductance with each subsequent step of measurements increases due to formation of additional to existing bonds between impurities, then for the liquid state, it falls due to the polymer purification apparently stimulated by electric field because of precipitation of a certain number of ions on the surface of the electrodes.

3.2. Dynamics of the conductance temperature dependence for linear polyethylene with impurity of soot and calcite on AC current

When measuring on AC current, an important parameter is the frequency of the measuring signal. Therefore, at the first stage of research, it was important to find a frequency range, in which conductance does not depend on frequency. Since by using the device we had the ability to investigate in a fairly wide range of frequencies ($10^{-3} \dots 10^6$ Hz), it was important for at least one temperature to perform these measurements to estimate the most important frequency range for analyzing the influence of impurities on the conductance of this polymer.

The frequency dependence of conductance of linear low-density polyethylene with impurity of soot and calcite at the temperature 293 K is shown in Fig. 4. As it follows from the data obtained for this temperature, the conductance is not frequency dependent within the measurement error. Moreover, our studies at the temperatures higher than 293 K showed that the conductance does not depend on the frequency within the range $10^{-3} \dots 10^{-2}$ Hz. Therefore, for higher (than 293 K) temperatures, we performed the research starting from the frequency 10^{-2} Hz. Since for the studied frequencies

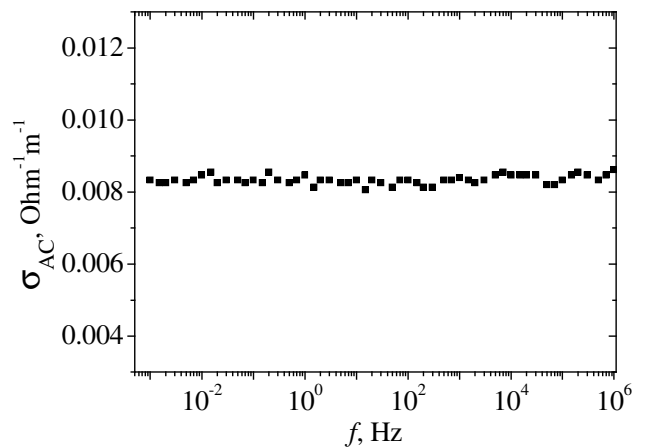


Fig. 4. Frequency dependence of conductivity on alternating current σ_{AC} of linear low-density polyethylene with impurity of 20 wt.% soot and 20 wt.% calcite at the temperature 293 K.

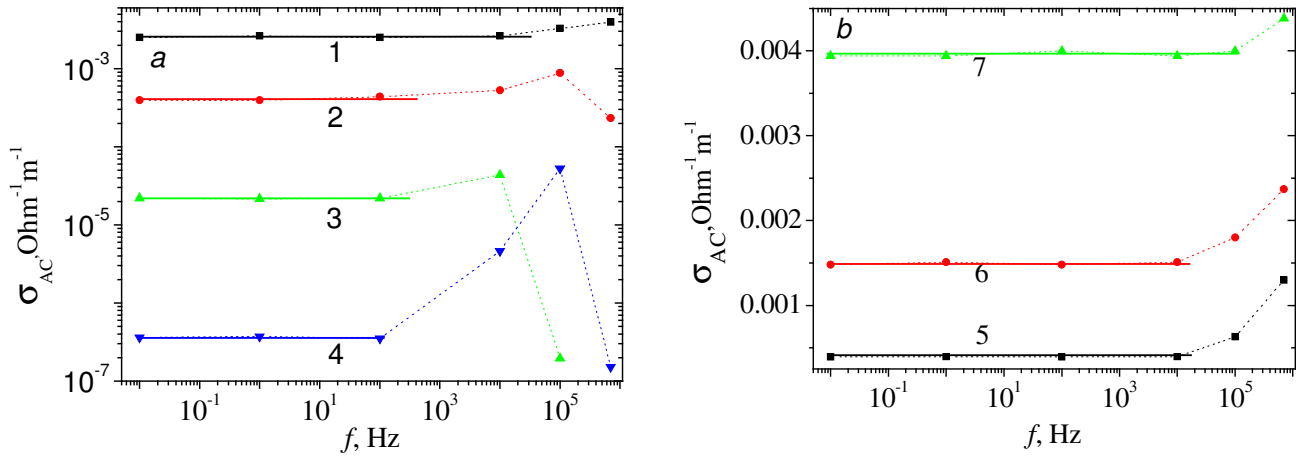


Fig. 5. Frequency dependences of conductance at alternating current σ_{AC} of linear low-density polyethylene with the impurity of 20 wt.% soot and 20 wt.% calcite for $T < T_0$ (a) and $T > T_0$ (b). Measurement at the temperatures T : 322 (1), 345 (2), 357 (3), 370 (4), 377 (5), 392 (6), and 408 K (7).

the significant dependence of conductance on the frequency was not observed, then we increased the interval between the individual frequencies in the measurement.

Fig. 5a shows the frequency dependences of σ_{AC} for linear low-density polyethylene with the impurity 20 wt.% soot and 20 wt.% calcite for some temperatures at $T < T_0$. As it follows from this figure, the conductance does not depend on frequency within the frequency range $10^{-2} \dots 10^2$ Hz. It is for this frequency interval that the temperature dependence of σ_{AC} will be further analyzed. One of the main reasons for the absence of dependence of conductance on the frequency can be formation of a grid between the impurities on which the main charge transfer occurs at the frequencies $f < 10^2$ Hz.

An increase in the conductance of linear polyethylene with the frequency $f > 10^2$ Hz can be explained on the basis of that hopping charge transfer between impurities that are at a short distance from each other. As it was shown in [29], for such a hopping charge transfer, the power dependence of the conductance on the frequency is typical. Therefore, the contribution of conductance due to the hopping charge transfer between impurities is manifested exactly at high ($f > 10^2$ Hz) frequencies.

As it follows from Fig. 5, the frequency dependence of σ_{AC} in the liquid state of polymer (Fig. 5b) is by no means fundamentally different from the conductance in the solid state of the polymer (Fig. 5a). Obviously, in the liquid state of polymer the grids of the impurities, on which the charge transfer occurs, remain. When analyzing the temperature dependence of σ_{DC} for $T < T_0$,

we are able to show that it can not be described by any of the known relations. On the basis of the analysis of the data obtained by us, the relation (1) was proposed. Therefore, it was important to check whether the temperature dependence of σ_{AC} is also described by the relation (1).

Fig. 6a shows the dependence of $\sigma(T/(T - T_0))$ in double logarithmic coordinates for σ_{AC} at $T_0 = 373$ K. It follows from this that the experimental data for both the first measurement (curve 1) and the fourth one (curve 2) are in good agreement with the relation (1).

Table 2 shows the values of the parameters, at which the temperature dependences of σ_{AC} for the composite based on linear low-density polyethylene with impurity of soot and calcite can be described with the least deviation from the experimental results by the relations (1) and (2).

As it follows from our estimates, the power index of n in the relation (1) for the σ_{AC} value decreases from 3.0 ± 0.3 for the first measurement (n_1) to 1.5 ± 0.3 for the fourth (n_4) and subsequent measurements. As for σ_{DC} , the changes in σ_{AC} on the temperature, which are most characteristic between the first and second measurements, are reduced with each subsequent measurement, reaching practically the same values (for subsequent measurements) starting from the fourth measurement.

From the comparison of Tables 1 and 2, it follows that within the measurement error, the power indexes (n_1 and n_4) in the relation (1) practically coincide for σ_{AC} and σ_{DC} . We consider that the difference between the T_0 value for σ_{DC} (380 K) and σ_{AC} (373 K) is most significant to measure in a solid phase. From our viewpoint, it may be

Table 2. The values of the parameters for σ_{AC} , at which the experimental data with the least deviations can be described by the relations (1) and (2).

T_0 , K	n_1	n_4	W_{1A} , eV	W_{1B} , eV	W_4 , eV
373	3.0 ± 0.3	1.5 ± 0.3	1.20 ± 0.05	2.50 ± 0.05	1.00 ± 0.05

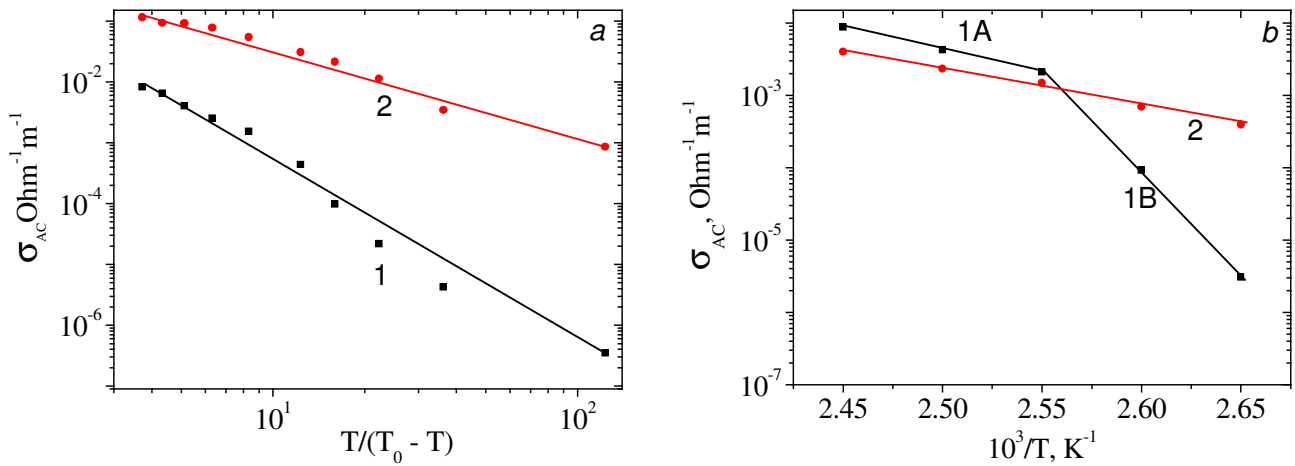


Fig. 6. Temperature dependences of σ_{AC} for the composite based on linear low-density polyethylene with impurity of soot and calcite for $T < T_0$ (a) and for $T > T_0$ (b) at the first (1) and fourth (2) measurements. Continuous lines in (a) and (b) shows the approximation of these dependences, respectively, with the relations (1) and (2) with the values of the parameters listed in Table 2.

due to the fact that AC measurements are more sensitive to the presence of certain heterogeneities of the sample structure, which take place when the temperature changes. That is, they need to be carried out with a greater duration of time between each measurement as compared with the DC one. But to provide equal conditions, we measured the temperature dependences of σ_{DC} and σ_{AC} with the same time of sample relaxation in the transition from one temperature to another.

From the comparison of Fig. 6b with Fig. 3b, one can conclude that the difference between the temperature dependences of σ_{DC} and σ_{AC} in the liquid phase of linear polyethylene (for $T > T_0$) is more significant than that for the polyethylene solid phase. To a greater extent, it relates to the first measurement. As it follows from the comparison of Tables 1 and 2, the difference between the W_{1A} and W_{1B} values for σ_{DC} and σ_{AC} significantly exceeds the error of the experiment. Moreover, if for σ_{DC} (Fig. 3b) the conductivity at the fourth measurement is higher for the entire temperature range, then in the case of σ_{AC} (Fig. 6b) at the temperatures close to T_0 , the conductivity at the first measurement is less than that in the fourth measurement.

It should also be noted that there is a very significant difference between the W_{1B} values for σ_{DC} and σ_{AC} . From our viewpoint, all this can be caused by the inhomogeneities of the sample structure, effect of which on the measurement of σ_{AC} is more significant than that of σ_{DC} and σ_{AC} . This hypothesis is confirmed by comparing the value of the activation energy for the fourth measurement.

As it follows from the comparison of Tables 1 and 2, the value of the activation energy for σ_{DC} and σ_{AC} in the liquid phase of linear polyethylene within the experimental error is practically the same. The reason for this may be precisely the ordering of impurity of soot and calcite under the action of electric field. Our estimates show that the σ_0 values are also the same for measuring on direct and alternating currents.

4. Conclusions

On direct and alternating currents within the frequency range $10^{-3} \dots 10^6$ Hz and the temperature range 293 to 425 K, the dynamics of conductivity of linear polyethylene with the impurity of 20 wt.% soot and 20 wt.% calcite has been studied.

1. It has been shown that the temperature dependence of conductivity for both alternating and direct currents in the solid phase of linear polyethylene cannot be described by known dependences. The empirically found dependence has been proposed $\sigma = \sigma_0 [T/(T - T_0)]^{-n}$ (where σ_0 is the conductance at $T = \infty$, T_0 – phase transition temperature for linear polyethylene, n – power index), which describes the experimental data obtained throughout the temperature range.

2. It has been shown that starting from the first measurement, the temperature dependences of σ_{DC} and σ_{AC} with each subsequent measurement are changed. The largest changes occur between the first and second measurements, and the maximum change in conductance is observed at the melting point of polyethylene T_0 . With each subsequent measurement, the σ_0 -value increases and the power index n decreases. Beginning with the fourth measurement, the change in electrical conductance does not exceed the measurement error.

3. It has been assumed that the dynamics of conductance of linear polyethylene with a content of 20 wt.% soot and 20 wt.% calcite is due to the ordering under the action of the electric field of impurity (through which charge transfer occurs).

4. It has been shown that after the fourth measurement, the parameters describing the temperature dependence of the conductance in the solid state are the same for conductance on direct and alternating currents.

5. It has been shown that the temperature dependence of the conductance of linear polyethylene with a content of 20 wt.% soot and 20 wt.% calcite above the melting point of polymer can be described in the

Arrhenius coordinates. The greatest differences between the data for conductance on direct and alternating currents are observed for the first measurement. It is for this measurement that the temperature dependence can be described by two exponential dependences with different activation energies.

6. It has been found that starting from the fourth measurement, the temperature dependence of conductance on the direct and alternating currents can be described by one exponential dependence with the activation energy 1.00 ± 0.05 eV.

7. Reducing the conductance of linear polyethylene with the impurity of carbon soot and calcite with each subsequent measurement in the liquid state of polymer can be explained by the purification of polymer under the action of electric field due to adsorption of ions carrying elective charge on the surface of the electrodes.

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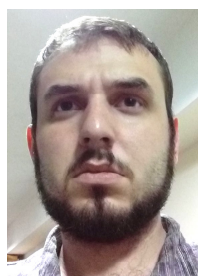
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