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Hetero- and low-dimensional structures

Dynamics of temperature dependence of the dielectric properties of a nanocomposite material based on linear polyethylene in the vicinity of the percolation transition

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Abstract. Within the frequency range of 30 to 10^5 Hz and temperatures of 18 to 80 °C, the dielectric properties of linear low-density polyethylene with impurities of flame retardant (20 wt.%) and multilayer carbon nanotubes (1.5 wt.%) were investigated using the oscilloscopic method. This concentration value of carbon nanotubes slightly exceeds the concentration of nanotubes (1%), at which the percolation transition begins for the dependence of the polymer electrical conductivity on the content of nanotubes. It has been shown that the frequency dependence of electrical conductivity can be approximated by two exponential dependences. It has been found that when the sample is cooled, changes in the values of dielectric permittivity and electrical conductivity do not occur in the same way as when heated. The temperature dependences of conductivity for low (10^2 Hz) and high (10^4 Hz) frequencies have been obtained.

Keywords: carbon nanotubes, impurities of flame retardant, linear polyethylene, electrical conductivity, dielectric properties.

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1. Introduction

Due to the transformation of the power system of Ukraine into the European one, the transition to more economical usage of the thermal energy is important. Electric energy refers to energy that can be not only relatively easily transmitted over a distance, but also effectively transformed into other types of energy, in particular, into the thermal one. Thermal energy losses can be drastically reduced during transition to individual heating systems. But even in this mode of heating, it is possible to make a much more dynamic (than water heating) system, where the release of heat does not occur due to warm water, but due to the passage of electric current through various types of heating elements.

One of the effective methods for creating such heating elements can be application of linear polyethylene with conductive impurities, in particular, carbon nanotubes [1]. In this case, the heating element with a thermal protective film (that prevents heating the wall itself) can be pasted on the wall similarly to ordinary wallpapers. The large area of these heating wallpapers can provide effective air heating even with a small temperature difference between the heater and air.

With the presence of these heating elements, it is much easier and more efficient to control the temperature in the room. Since both linear polyethylene and nanotubes can burn at relatively low temperatures, one of the main elements of the heating device should be a substance that would counteract this process. That is, in addition to linear polyethylene as a matrix, at least two components must be used in the heating element: nanotubes to increase conductivity and an impurity that prevents the ignition of the heating element itself.

As shown in [2], when nanotubes are introduced into a liquid crystal, a sharp increase in conductivity can occur due to the percolation transition from the state when the electrical conductivity is provided by the liquid crystal to the state when the charge transfer mainly occurs through the nanotubes. The parameters of such a transition depend significantly on the chemical composition and concentration of the impurity.

As for the effect of carbon nanotubes on the conductivity of linear polyethylene, a significant part of research has already been published, which shows that indeed at certain concentrations of nanotubes (of the order of several percents) there is a transition from the semiconductor type of conductivity at “subthreshold” concentrations of nanotubes to the percolation transition, after which the conductivity of polyethylene with nanotube impurities is caused by charge transfer through the nanotubes [3–7]. But these studies were mainly laboratory, in which there was no need to add a flame retardant substance (FRS) to the mixture. Certainly, the presence of this FRS at sufficiently high concentrations (tens of weight percents) can affect the parameters of the percolation transition.

In contrast to linear polyethylene with only carbon nanotube impurities, the influence of carbon nanotube concentration on the conductivity of linear polyethylene with a FRS admixture has been studied much less, and on the basis of these studies, much fewer works have been published than in the absence of such admixture in the studied samples. Among them, the work [8] should be noted. As it follows from their analysis, only insufficient attention was focused on the analysis of possible changes in the morphology of the films during the measurement process at different temperatures. As shown in [9], the co-authors of which are several authors of this paper, this process occurs even at small voltages of the measuring signal. This process is especially significant, when the measurements were carried out in a sufficiently wide range of temperatures, including those higher than the melting point of polymer.

Therefore, to obtain stable results over time, several cycles (of the order of 4) of studies were performed in the work [9]. This work presented the results of experimental studies for a conductive impurity (carbon black) at the concentration close to 20%, which significantly exceeded the threshold concentration for the percolation transition. Therefore, it was difficult to assess the extent to which the parameters of the samples can change during the measurement process precisely in the vicinity of the percolation transition. It is certainly impossible to investigate all the possible features of the percolation transition for the concentration dependence of the conductivity of linear polyethylene with the FRS impurity on the content of CNTs in one paper.

Therefore, the purpose of this work was to solve a somewhat simpler problem, namely to investigate the dynamics of the dielectric properties of linear polyethylene with the impurity of multilayer carbon nanotubes and a flame retardant substance as the temperature increases and decreases. For this, the samples with the concentration 1.5% of carbon nanotubes were studied, taking into account the fact that the percolation transition for the concentration dependence of the conductivity of linear polyethylene on the content of carbon nanotubes begins at their concentration close to 1%.

2. Materials and methods

The studied film was made from a pre-prepared compound in the form of granules. We used a twin-screw extruder to make these granules. The ring head of the extruder had a diameter of 40 mm and a forming hole of 1.2 mm.

The composition of these raw materials was as follows: 1) low-density polyethylene 16802-70 in powder form, 2) multi-layer carbon nanotubes Nanocyl NC7000 (in this work, the concentration of nanotubes was 1.5%) and 3) flame retardant substance 20%. As a percentage of the volume of the entire mixture, FRS consisted of 18% flame retardant decabromodiphenylether and 2% antimony trioxide.

The initial components in powder form were mixed in a laboratory mixer for 3 minutes at the speed 3000 rpm. The subsequent mixing of the powder mixture by using a twin-screw extruder took place under the following conditions: screw speed 350 rpm, temperature in the extruder zones 160–220–190–200–190 °C, productivity 2.7 kg/h. The ring head of the extruder had a diameter of 40 mm and a forming hole of 1.2 mm.

After mixing in a twin-screw extruder, the melt was cooled in a water bath in the form of strands and cut into granules. The granules were dried at the temperature close to 80 °C and used to make a film.

The film for electrical conduction measurements was produced using the sleeve blowing method in a laboratory film line based on a single-screw extruder. The ring head of the extruder had the diameter 40 mm and the forming hole close to 1.2 mm. The degree of film expansion was 2.6 times in width and 4.2 times in length. The temperature profile of the single-screw extruder was 180–210–190–190–195 °C.

To measure the dielectric properties, we used parts cut in different places from the tape that is formed at the exit of the extruder. The area of such samples, roughly square-shaped, was of the order of 2 cm^2. It was found that the difference between the electrical parameters of the tape formed by the extruder cut in different places did not exceed the experimental error when measuring the electrical parameters of the samples. Therefore, it was concluded that the distribution of impurities in the polymer is sufficiently homogeneous.

The surface of the films obtained after the extruder was not smooth enough to obtain a reliable electrical contact. Therefore, before the measurement, the samples were polished using a diamond paste with a particle size of 60 μm. The sample thickness measured with a micrometer after polishing and cleaning from the remains of diamond paste was 500 ± 10 μm.

Polished copper electrodes were used for measurement. To ensure reliable electrical contact and at the same time thermal contact, a very thin layer of suspension based on grade C0 graphite (Zavalev graphite) was applied to the surface of the polymer.

The stabilization of temperature *t* within the range 18 to 80 °C with an error of no more than 0.1 °C was achieved using an experimental setup consisting of the temperature stabilizer Solid State Module FOTEX (produced in Taiwan) and the temperature meter and regulator REX-C100FK02 (produced in Japan). Electrical properties of the samples were measured at the temperatures of 18, 35, 50, 65, and 80 °C during heating and cooling of the samples.

Because the morphology of similar samples was reported in the work [9], we did not study the morphology of the obtained films. The identical devices and technology for obtaining the studied films in this work and in [9] give every reason to assert that the samples with a homogeneous distribution of components were analyzed. This is confirmed by comparison of the data for films cut in different places of the film obtained at the exit of the extruder. The difference between the conductivity and dielectric permittivity of these films did not exceed the experimental errors.

To measure resistance and capacitance within the frequency range 30 to 10^5 Hz, we used the oscilloscopic method for measurements [10]. In addition to the temperature stabilizer, the measuring device is consisted of a low-frequency signal generator G3-112 and oscillograph C1-93. The oscillograph operated in oscilloscopic mode. The amplitude value for the sinusoidal voltage of the measuring signal was 2.5 V. We measured the resistance *R* and capacitance *C* of the samples according to the method described in [9] at the voltage 1 V.

Based on the known geometric parameters of the samples and the values of *R* and capacitance *C*, we determined the conductivity σ and dielectric permittivity ε of the samples at the certain frequencies, respectively. Since the frequency was varied in a rather wide interval (30 to 10^5 Hz), we chose the frequencies in such a manner that the interval between them was practically the same in a logarithmic scale. Conclusions about the electrical properties of the studied samples were obtained on the basis of the analysis of frequency and temperature dependences of the values σ and ε, when the temperature increases and decreases.

Using the method described above for determining the resistance and capacitance of the samples, the conductivity and dielectric permittivity were estimated. Based on this, we obtained the data with an error that did not exceed 5%.

3. Experimental results and their analysis

Fig. 1 shows the frequency dependences of the dielectric permittivity ε of the studied samples at the temperatures 18 °C (curves *1* and *2*) and 80 °C (curves *1* and *3*) with increasing (curves *1* and *3*) and decreasing (curves *2* and *4*) the temperature.

The following conclusions can be drawn from the analysis of the data presented in Fig. 1. First, each of these curves can be separated into two regions, in which the ε value of the samples changes linearly *vs* the logarithm of frequency. Both when the temperature increases and when the electric field is applied for a sufficiently long time (for measurements when the temperature decreases), the transition from a low-frequency region with a stronger dependence ε(*f*) to a high-frequency region with a weaker dependence ε(*f*) shifts toward higher frequencies. Second, at the temperature 80 °C, changes in the value of ε(*f*) in the region of low frequencies are significantly greater than at 18 °C. While at higher frequencies there is practically no difference between the dependences of ε(*f*) for the temperatures 18 and 80 °C. To explain the reason for such a difference and the presence of two regions with different dependence ε(*f*), it is also important to analyze the dependences σ(*f*). These data are shown in Figs 2a and 2b for the same temperatures 18 °C (Fig. 2a) and 80 °C (Fig. 2b).

Based on the analysis of the data shown in Figs 2a and 2b, it is possible to draw practically the same conclusions as when analyzing the frequency dependences of ε. As in the case of the frequency dependence of the ε value, two regions are observed in the frequency dependence of the σ value. The frequency, at which there is a transition from one region to another in the frequency dependence of the σ value, shifts toward higher frequencies as the temperature increases. Since in the case of frequency dependences of σ, a coordinate system was used, where the frequency, like the value of σ, changed according to a logarithmic law, then taking

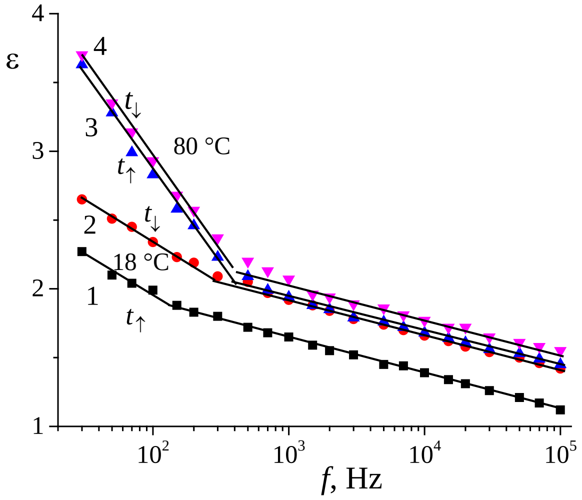

Fig. 1. Frequency dependences of the dielectric permittivity ε of linear low-density polyethylene with an admixture of a flame retardant substance (20 wt.%) and multilayer carbon nanotubes (1.5 wt.%) at the temperatures 18 °C (curves *1* and *2*) and 80 °C (curves *1* and *3*) with increasing (curves *1* and *3*) and decreasing (curves *2* and *4*) the temperature.

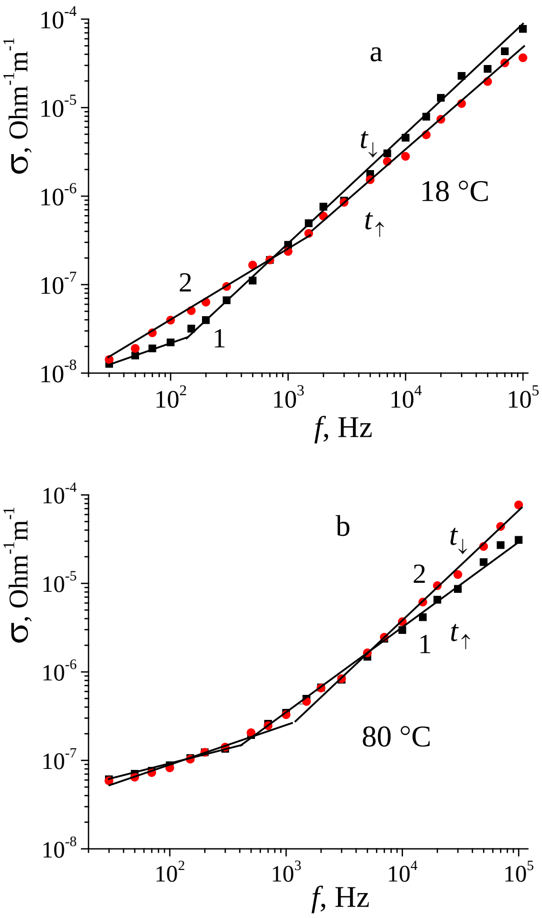

Fig. 2. Frequency dependences of the electrical conductivity σ of linear low-density polyethylene with the impurity of a flame retardant substance (20 wt.%) and multilayer carbon nanotubes (1.5 wt.%) at the temperature 18 °C (a) and 80 °C (b). The curve *1* corresponds to the increasing temperature, and the curve *2* – to the decreasing one.

into account the presence of two regions, such a law of changes in electrical conductivity with frequency can be described by two power-law dependences. That is, in general, for the entire frequency range, this dependence can be described by the following relation

$$\sigma = a f^m + b f^n . \qquad (1)$$

In the relation (1), parameters *a* and *m* characterize the low-frequency dependence of electrical conductivity, and parameters *b* and *n* – the high-frequency dependence of electrical conductivity.

Our data analysis showed that the parameters *a*, *m*, *b*, and *n* change insignificantly with a change in temperature. Therefore, there was no point in citing their values for analysis.

Before proceeding to the analysis of the temperature dependence of the conductivity, attention should be paid to the difference between the change in the σ value during cooling of the samples. As follows from the comparison of Fig. 2a and Fig. 2b, in the case of measurement at the temperature 18 °C, the σ value decreases. This is characteristic of dielectric liquids and liquid crystals in particular [11] and can be explained using the electrolytic purification [12].

In the case of measurement at the temperature 80 °C (Fig. 2b), on the contrary, the long-term action of the electric field leads to an increase in the value of electrical conductivity. To explain such a difference in the dynamics of changes in electrical conductivity at different temperatures, additional studies will be performed, the conclusions of which will be formulated in subsequent (after this) publications.

It was already mentioned above that the conductivity of the studied samples did not change significantly with increasing the temperature. Therefore, it is necessary to analyze this dependence in greater detail. But before doing this analysis, it is important to consider the temperature dependence of the ε value.

The temperature dependences of ε for the frequencies 10^2 Hz (curves *1* and *2*) and 10^4 Hz (curves *3* and *4*) are shown in Fig. 3.

Unlike the electrical conductivity, there are no simple theoretical models that could be used to explain the temperature dependence of ε even for pure substances. In this work, we investigated a three-component mixture (without taking into account the composition of FRS). Therefore, we have given only the dependences ε(*t*) without their analysis. For this work, it is important that the ε value increases with prolonged action of the electric field.

Therefore, when the temperature decreases (Fig. 3, curves *2* and *4*), the dielectric permittivity increases in relation to that when the temperature increases (Fig. 3, curves *1* and *3*). Since the polymers themselves, components of FRS and nanotubes are polar, this can be explained by the rotation of the dipoles in the direction

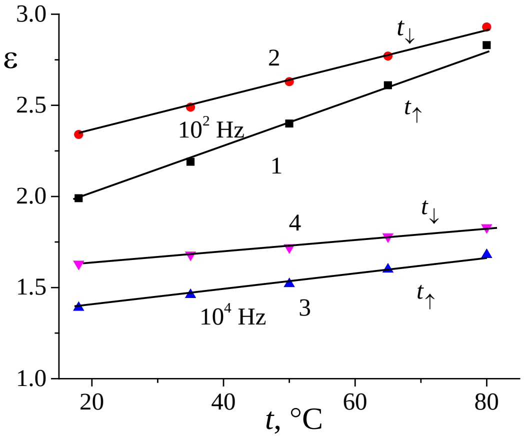

Fig. 3. Temperature dependences of the dielectric permittivity ε of linear low-density polyethylene with the impurity of a flame retardant substance (20 wt.%) and multilayer carbon nanotubes (1.5 wt.%) at the temperatures 18 °C (*1* and *2*) and 80 °C (*1* and *3*) with increasing (*1* and *3*) and decreasing (*2* and *4*) the temperature.

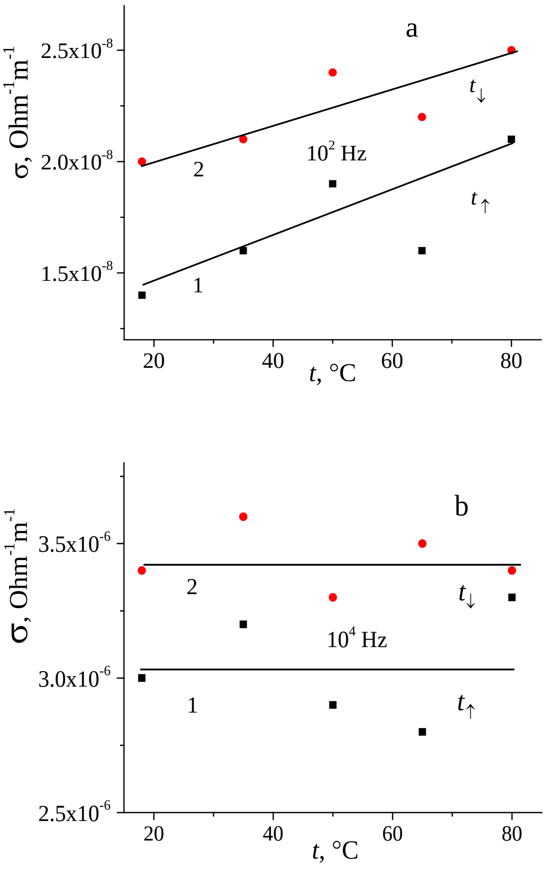

Fig. 4. Temperature dependences of the electrical conductivity of linear low-density polyethylene with FRS (20 wt.%) and multilayer carbon nanotubes (1.5 wt.%) for the measuring signal frequency 10^2 Hz (a) and 10^4 Hz (b). Curve *1* corresponds to an increase in temperature, and curve *2* – to a decrease in temperature.

of the electric field. This is confirmed by larger changes in the ε value for increasing and decreasing the temperature at the frequency close to 10^2 Hz (Fig. 3, curves *1* and *2*) than at the frequency 10^4 Hz (Fig. 3, curves *3* and *4*).

Shown in Fig. 4a is the temperature dependence of the electrical conductivity inherent to linear low-density polyethylene with FRS (20 wt.%) and multilayer carbon nanotubes (1.5 wt.%) for a measuring signal frequency close to 10^2 Hz. From the analysis of this dependence, it follows that, with increasing the temperature, there is a slight increase in conductivity. Since in the dependence on the temperature the conductivity changes were quite small and did not much exceed the experimental error, it is quite difficult to ascertain the ratio by which the conductivity changes.

Considering that in this range of frequencies the dependence σ(*f*) differs from that for higher frequencies, it can be assumed that for these frequencies the main mechanism of charge transfer is ionic-electronic (ionic through the polymer and electronic mainly through the nanotubes). Previously, when studying the conductivity of linear polyethylene only with FRS, we found that this impurity also leads to an increase in electrical conductivity, but by more than an order of magnitude less than the nanotubes themselves alone. Based on our research, it is difficult to estimate the contribution to the electrical conductivity of the studied mixture of each of the impurities separately (FRS and nanotubes). This will be done in the next work, where samples with different concentrations of nanotubes will be investigated.

Since the mobility of ions is significantly lower than that of electrons, in this case, at higher frequencies, the conductivity of the studied samples should be determined only by the electronic component through the nanotubes. Therefore, the temperature dependence of the σ value for the frequency 10^4 Hz was analyzed, too.

The temperature dependence of the σ value of linear low-density polyethylene with FRS (20 wt.%) and multilayer carbon nanotubes (1.5 wt.%) for the frequency 10^2 Hz is shown in Fig. 4a.

From the analysis of this figure, it follows that as the *t* value increases, the σ value increases slightly, which in principle is characteristic of both substances with a semiconductor type of conductivity and structurally disordered systems. A larger value of ε for these frequencies, as in the liquids [12], may be caused by the non-uniform distribution of the electric field due to the partial blocking of the transfer of charge carriers by the near-electrode layers. In the case of the studied samples, this mechanism can be significant, since we used "clamping electrodes". At the same time, at the electrode-polymer interface, an electric double layer can form, which will prevent charge transfer like to the case of liquids [12].

The temperature dependence of the σ value of linear low-density polyethylene with RFS (20 wt.%) and multilayer carbon nanotubes (1.5 wt.%) is shown in Fig. 4b.

The analysis of this dependence allows to conclude that with a small error it can be assumed that the conductivity does not depend on temperature. This may be a confirmation that even at these low concentrations of nanotubes (1.5 wt.%) the charge transfer mainly occurs through the nanotubes. But at the same time, there is also observed the charge transfer through FRS.

4. Conclusions

Within the temperature range of 18 to 80 °C and frequencies 30 to 10^5 Hz by using the oscilloscopic method, the frequency and temperature dependences of the dielectric permittivity ε and electrical conductivity σ of linear low-density polyethylene with the impurity of a flame retardant substance (20 wt.%) and multilayer carbon nanotubes (1.5 wt.%) were studied.

1. It has been shown that for all the temperatures, the decrease in ε with increasing the frequency *f* can be approximated by straight lines in the coordinates ε(lg*f*) within the limits of experimental errors. Moreover, depending on the frequency, two regions are observed.

The frequency, at which there is a transition from one frequency dependence ε(*f*) to another within the range of 10^2 to 10^3 Hz, increases with increasing the temperature. The difference between the frequency dependences of ε in each of these regions is that in the low-frequency region, the slope of the frequency dependence increases with increasing the temperature, and for the high-frequency dependence, it does not depend on temperature.

2. It has been found that when measuring the frequency dependences of ε when the temperature decreases, an increase in the value of the dielectric permittivity is observed without any change in the slope of the dependences ε(*f*). The largest hysteresis for ε(*f*) when measured with increasing and then decreasing the temperature value is observed at 18 °C (increase of ε by 1.17 times). At temperatures near 80 °C, the changes in the ε value with increasing and decreasing the temperature are much smaller and close to the experimental errors.

3. It has been shown that the frequency dependences of the electrical conductivity value can also be described by two straight lines, but in contrast to the frequency dependences of the ε values in double-logarithmic coordinates. At the same time, it should be noted that unlike the dependence ε(*f*), the value of conductivity increases with increasing the frequency, and the slope of the straight line for the dependence σ(*f*) for higher frequencies is greater than that for the lower ones.

4. As in the case of the frequency dependence of ε, there is a difference between the σ values for the data obtained when the temperature increases and decreases. However, it is much smaller (especially for the temperature 18 °C) than in the case of a change in the ε value. It is also important to note that at the temperature 18 °C, the long-term effect of an electric field leads to a decrease in the value of electrical conductivity. This can be explained by the fact that the change in electrical conductivity during prolonged exposure to an electric field can be caused by two competing processes: an increase in conductivity due to the arrangement of sections through which charge transfer occurs, and a decrease in conductivity due to electrolytic cleaning of impurities that cause this conductivity. The difference between the values of conductivity when heating and cooling the samples can also be caused by the presence of FRS.

5. The temperature dependences of electrical conductivity for low-frequency and higher-frequency regions differ. If for the low-frequency region there is a slight increase in electrical conductivity with increasing the temperature, then in the higher-frequency region the value of electrical conductivity does not depend on temperature within the experimental errors.

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All authors discussed the results and commented on the manuscript.

Динаміка температурної залежності діелектричних властивостей нанокомпозитного матеріалу на основі лінійного поліетилену в околі перколяційного переходу

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Анотація. У діапазоні частот 30…10^5 Гц і температур 18…80 °C за допомогою осцилоскопічного методу досліджено діелектричні властивості лінійного поліетилену низької щільності з домішками протидіючої горінню речовини (20 мас.%) та багатошарових вуглецевих нанотрубок (1,5 мас.%). Це значення концентрації вуглецевих нанотрубок дещо перевищує ту концентрацію нанотрубок (1%), при якій починається перколяційний перехід для залежності електропровідності полімеру від вмісту нанотрубок. Показано, що частотну залежність електропровідності можна апроксимувати двома експоненціальними залежностями. Знайдено, що при охолодженні зразка зміни величин діелектричної проникності та електропровідності відбуваються не так, як при нагріванні. Отримано різні за характером змін температурні залежності провідності для низьких (10^2 Гц) та високих (10^4 Гц) частот.

Ключові слова: вуглецеві нанотрубки, домішки протидіючої горінню речовини, лінійний поліетилен, електропровідність, діелектричні властивості.