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REGULAR ARTICLE

Electrical and Electrodynamic Properties of Polymer Composites with Nanocarbon Filler

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The paper presents the results of investigation of electrical and electrodynamic properties of polymer composites based on polychlorotrifluoroethylene with different nanocarbon fillers under heat load. As nanocarbon fillers thermoexfoliated graphite, thermoexfoliated graphite modified by silicon oxide and multiwalled carbon nanotubes have been used. Modification of TEG with silica was carried out from a colloidal 20 % solution of hydrosol (silicic acid). For investigations the bulk polymer composites based on polychlorotrifluoroethylene with different content of nanocarbon filler have been obtained by thermal pressing. The dielectric permittivity have been studied at room temperature using an ultra-high-frequency interferometer at a frequence υ = 10 GHz. Temperature dependence of electrical conductivity $\sigma(T)$ have been measured by two- and four-probe methods on direct and alternating current in temperature interval (293-425) K. Temperature dependence of the electrodynamic parameters has been studied with use panoramic meters of standing wave ratio and electromagnetic radiation attenuation in the temperature interval (293-373) K.

It is shown that use of thermoexfoliated graphite, thermoexfoliated graphite modified with SiO_2 Carbon nanotubes as fillers allows to obtain electrically conductive polymer composites with low percolation threshold ~ 0.955 % mass. At filler concentrations lower than the percolation threshold, the main contribution to the electrical conductivity of the polymer composite is made by the relaxation component of the conductivity, which is determined by the processes of interphase polarization at the polymer-filler interface. At filler concentration in the polymer composite is slightly higher than the percolation limit, the increase of the contribution of direct electrical conductivity due to direct contacts and contacts through thin polymer layers between fillers particles occurs.

It is revealed that heat of polymer composites up to 373 K leads to slight improvement in shielding characteristics mainly due to an increase in the electromagnetic radiation absorption coefficient. Changes in the effective dielectric permittivity of polymer composites in the temperature range of (293-373) K do not dramatically affect the shielding characteristics of investigated polymer composites.

Keywords: Polychlorotrifluoroethylene, Thermoexfoliated graphite, Multiwall carbon nanotubes, Conductivity, Dielectric permittivity.

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1. INTRODUCTION

In the development of new polymer composite materials (PCM) with the required level of specified properties, one of the tasks is to ensure the stability of the characteristics of the CM under the influence of various external factors, in particular, mechanical, thermal, radiation load [1-4]. The use of nano-sized conductive carbon fillers makes it possible to create polymer composites with high electrical and electrodynamic characteristics at a sufficiently low filler content and, accordingly, to preserve the functional characteristics of the polymer matrix [5, 6]. Since polymer materials are characterized by a fairly high coefficient of thermal expansion, microstructural transformations of polymer composites based on such matrices are possible upon heating, and in some cases these changes may be irreversible [5, 7-9]. In this regard, it is important to establish temperature intervals for the reproducibility of polymer composite's characteristics. This is especially important in the case of electrical and electrodynamic characteristics of polymer composites, which are very sensitive to the microstructure of composite, especially at filler concentrations near the percolation threshold [10-13].

The purpose of the presented work was to study the electrical and electrodynamic properties of polymer composites based on polychlorotrifluoroethylene with various types of nanocarbon filler and to establish the level of their stability under thermal load up to 373 K.

2. OBJECTS AND METHODS

In the work, polymer composites based on polychlorotrifluoroethylene (PCTFE, trade mark fluoroplast-3) with nanocarbon filler have been studied. PCTFE is a typical representative of the class of crystalline amorphous poly-

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mers. Macromolecules of PCTFE are built from linear flexible chains capable of crystallization. The PCTFE monomer link is asymmetric and has a constant dipole moment. That is why its dielectric properties in a wide range of temperatures and frequencies are highlighted in the literature better than other polymers [14]. Thermally exfoliated graphite (TEG) and multiwalled carbon nanotubes (MWCNTs), as well as TEG and MWCNTs modified with silicon oxide SiO_2, have been used as nanocarbon fillers. TEG has been produced by thermal shock of natural dispersed graphite (GAK 1) of the Zavalivskiy deposit intercalated with sulfuric acid. MWCNTs with a diameter of up to 40 nm and a length of up to 10 mcm have been obtained by the decomposition of hydrocarbon in the presence of a nickel catalyst and then purified by treatment with acid solutions. Modification of TEG with silica was carried out from a colloidal 20 % solution of hydrosol (silicic acid). Mixture of TEG particles, alcohol and hydrosol was sonicated (frequency 22 kHz) for 1.5 min. Next, the mixture was heated to 90 °C and, with periodic stirring, dried until the alcohol was completely removed. As a result, a powdered composite containing 7 mass parts of 7 nanocarbon and 3 mass parts g of SiO_2 was obtained.

The polymer composites with PCTFE have been obtained in two stages. On the first stage nanocarbon particles sonicated in an alcoholic medium (ethyl alcohol). On the second stage mixture of powdered PCTFE (spherical aggregates with a diameter of $d \sim 200$ nm) with the dispersed in the ethyl alcohol nanocarbon particles heating with constant stirring up to 640 K. The bulk polymer composites based on PCTFE with different content of nanocarbon filler were obtained by method of thermal pressing.

The real (ε') and imaginary (ε'') components of the complex dielectric permittivity have been studied at room temperature using an ultra-high-frequency interferometer at a frequence $\nu = 10$ GHz. Temperature dependence of electrical conductivity $\sigma(T)$ have been measured by two- and four-probe methods on direct and alternating current in temperature interval (293-425) K. Temperature dependence of electromagnetic radiation (EMR) reflection and absorption spectra and dielectric constant have been studied with use panoramic meters of standing wave ratio and EMR attenuation (P2-65). Measurements were performed at a frequency (26-37.5) GHz in the temperature interval $T = (293\text{-}373)$ K.

3. EXPERIMENT AND DISCUSSION

3.1 Concentration Dependences of Electrical Conductivity of Polymer Composites

The concentration dependences of the logarithm of the electrical conductivity of PCTFE-based polymer composites with TEG, MWCNTs, and silicon oxide-modified TEG at a frequency of 0.1 Hz and at a constant current are presented in the Figure 1.

As can be seen from the Figure 1, a sharp increase in conductivity on both alternating and direct current for polymer composites with TEG filler occurs in the concentration range of (1-1.3) %. At the same time, the increase in conductivity for composites with modified by silicon oxide TEG as a filler is somewhat greater than for composites with pure TEG. For composites with MWCNTs as a filler, the increase in conductivity is not of a percolation nature.

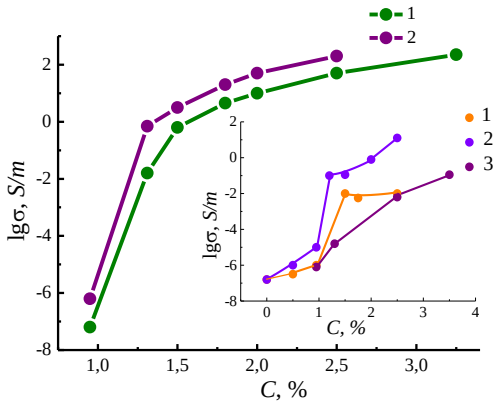


Fig. 1 – Dependences of the logarithm of the electrical conductivity lg(σ) of PCTFE-based polymer composites on the filler concentration at a frequence 0.1 Hz and at constant current (inset), $T = 295$ K, fillers are TEG (1), TEG modified with SiO_2 (2), MWCNTs (3)

As is known [15], the electrical conductivity of polymer composites with a dispersed filler σ_{PC} in alternating electromagnetic fields is described by the following expression:

$$\sigma_{PC} = \sigma_d + \sigma_\tau , \qquad (1)$$

where σ_d is the through electrical conductivity, which coincides with electrical conductivity at direct current and σ_τ is the relaxation component of electrical conductivity:

$$\sigma_\tau = \varepsilon_0 \omega_0 \varepsilon'' , \qquad (2)$$

where ω_0 is the cyclic frequency of the electromagnetic field, ε_0 is the absolute dielectric permittivity of vacuum, ε'' is the imaginary component of the complex dielectric permittivity. The magnitude of the conductivity σ_d depends on the electrical conductivity of the separate filler's particles and the contact electrical resistance between them. The relaxation component σ_τ is determined by the sum of relaxation processes in the polymer, filler, and interfacial layer [1]. Among them, as a rule, the dominant process that determines the conductivity σ_τ for composites with an electrically conductive dispersed filler is the process of interfacial polarization [2]. This is especially evident under the condition of a developed interfacial surface, that is, under the small size of dispersed inclusions. The electrical conductivity of the polymer composite with a low filler content is mainly determined by the relaxation component σ_τ ($\sigma_\tau >> \sigma_d$). A slight increase in the electrical conductivity of polymer composites with a small content of filler (up to 0.95 % mass) in comparison with the electrical conductivity of pure polymer is due to the occurrence of polarization in the interphase layer of polymer-dispersed TEG [16]. A more intense increase in electrical conductivity due to an increase in filler content both before and after the percolation threshold in PCTFE-TEG/SiO_2 composites compared to PCTFE-TEG composites occurs due to additional interfacial polarization in the TEG/SiO_2 interfacial layer. A similar effect of increasing the electrical conductivity of composites with oxide fillers in comparison with the electrical conductivity of a conductive filler was found in systems Al_2O_3/AgI, Al_2O_3/SiO_2, AgI/SiO_2, pentaplusT/AgI, CuI/SiO_2 [17].

As the filler content increases, the electrical conductivity of both systems increases due to the formation of direct electrical contacts between dispersed filler particles.The Figure 2 presents the temperature dependences of conductivity at constant current of polymer composites based on PCTFE with different contain of TEG and TEG modified with SiO_2.

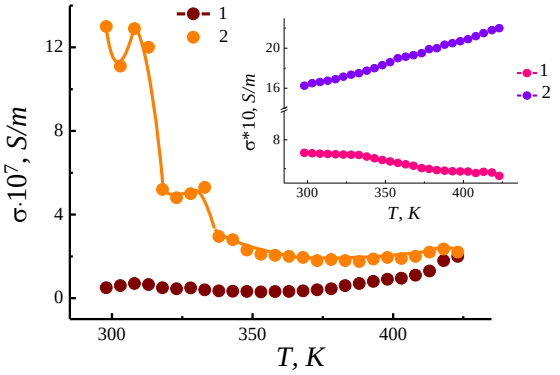


Fig. 2 – Temperature dependences of conductivity $\sigma(T)$ at constant current of polymer composites based on PCTFE with 0.95 % mass and 1.5 % mass (inset) of TEG (1) and TEG/SiO_2 (2)

As it follows from Figure 2, for composite PCTFE-TEG (0.95 % mass) in the temperature range up to 355 K, the conductivity decreases slightly. For composite PCTFE-TEG/SiO_2 (0.95 % mass), the decrease in conductivity in *a* analogous temperature range is significantly pronounced and amounts to ~ 6 times. Such a drop in conductivity occurs due to a decrease in the relaxation conductiviy component σ_τ and is associated with additional ordering of the supramolecular structure of the polymer during heating in the interphase layers of PCTFE–TEG and PCTFE-TEG/SiO_2. With a further increase in temperature, the electrical conductivity of both polymer composites increases, which is explained by the defrosting of the segmental mobility of the amorphous component of the polymer above the glass transition temperature. For polymer composites with a filler concentration of 1.5 % mass a monotonic change in conductivity is observed in the temperature range of (293-450) K. For composite PCTFE-TEG, there is a decrease in electrical conductivity by ~ 1.5 times, while for composite PCTFE-TEG/SiO_2, conductivity increases by ~ 1.3 times. Such differences in the temperature dependence of the electrical conductivity of polymer composites are due to the fact that in composite with TEG as filler the role of contact electrical resistance between the filler particles is quite large, which increases with increasing temperature due to the linear expansion of the polymer and the deterioration of the contact between the filler particles. In the case of a polymer composite with TEG/SiO_2 as a filler, the increase in the electrical conductivity of the filler particles themselves as the temperature increases prevails over the increase in contact resistance between the TEG particles, which leads to an increase in the electrical conductivity of the composite PCTFE-TEG/SiO_2 as a whole.

3.2 Concentration Dependences of Dielectric Permittivity of Polymer Composites

Polymer composites PCTFE-TEG and PCTFE-TRG/SiO_2 which are on concentration dependences up to the percolation threshold, behave as dielectrics, since a continuous conductive cluster of filler particles has not yet formed, individual particles or agglomerated formations of filler particles are isolated and there are quite thick polymer layers between it. Under the influence of an electric field, charge carriers of the filler are localized at the polymer-filler interphase boundaries and the process of interphase polarization occurs. When the filler content in CM increases, the area of interphase boundaries grows and, accordingly, the manifestation of interphase polarization intensifies, the dielectric constant increases. At the moment when percolation occurs, in the CM there is already through-flow electrical conductivity along continuous chains from the filler, but the dielectric constant still remains high, since a large number of isolated particles of the filler are still grounded.

The Figure 3 shows the concentration dependences of the components of the complex dielectric permittivity ε' and ε'' for polymer composites PCTFE-TEG and PCTFE-TEG/SiO_2.

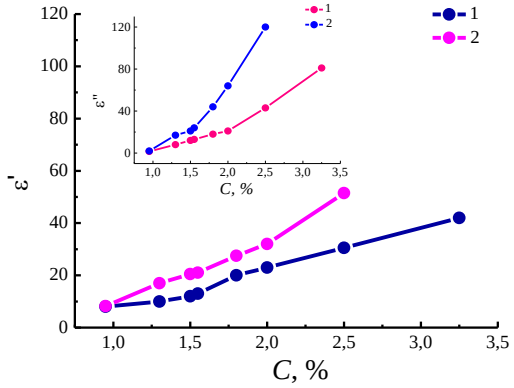


Fig 3 – Concentration dependences of real ε' and imaginary ε'' (inset) components of dielectric permittivity, fillers: TEG (1), TEG modified with SiO_2 (2)

As can be seen from Figure 3, a significant increase in the values of ε' and ε'' is observed for the polymer PCTFE–TRG when the filler content is increased up to 3.5 % mass. Modification of the developed surface of the TEG particles with SiO_2 leads to significant quantitative changes in the dielectric properties. In particular, the character of the concentration dependences of ε' and ε'' for both polymers is qualitatively repeated, however, the values of the components of the complex dielectric constant ε' and ε'' for composite containing silicon dioxide exceed the values of these constants for polymer PCTFE–TEG after the percolation threshold by ~ 1.5 and 3 times, respectively. This effect can be related to the strengthening of the relaxation component of the composite σ_τ due to the manifestation of interphase interaction at the interface between the filler TEG/SiO_2 and polymer PCTFE/SiO_2.

3.3 Temperature Dependences of the Spectra of Reflection and Absorption of Electromagnetic Radiation and Dielectric Permittivity

Figure 4 presents the frequency dependences of EMR attenuation (a) and standing wave ratio (SWR) (b) for a PCTFE-based polymer composite containing 2.5% mass of dispersed TEG at different temperatures.

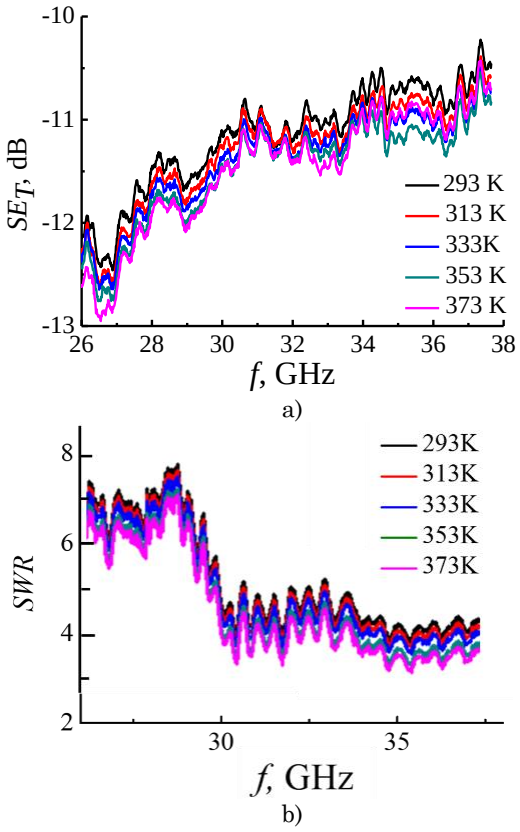


Fig. 4 – The frequency dependences of EMR attenuation (a) and standing wave ratio SWR (b) at different temperatures for a PCTFE-based polymer with 2.5 % mass of dispersed TEG. The temperature is indicated on the graphs

As can be seen from the Figures, heating the polymer composite to 373 K leads to a slight weakening of microwave radiation (up to ~ 0.5 dB) in almost the entire frequency range except for the (34-38) GHz range. At the same time, the SWR also slightly decreases in the investigated frequency range. Therefore, at the heating of this polymer composite up to 373 K, there is a slight increase in electromagnetic wave attenuation. For the polymer composite based on PCTFE with carbon nanotubes as filler, the value of the attenuation coefficient in the frequency range (34-38) GHz (Fig. 5a) practically does not depend on the temperature at the heating to 373 K, that is, the EMR absorption and reflection characteristics for this polymer composite are not affected by temperature. At the same time, the SWR (Fig. 5b) decreases with increasing frequency, and heating only slightly changes the value of the reflection coefficient.

Figure 6 shows the frequency dependences of EMR attenuation (a) and SWR (b) for a PCTFE-based polymer composite containing 2.5 % mass of TEG modified with SiO_2.

As can be seen from the Figures, the frequency dependence of the attenuation coefficient is weakly expressed for this polymer composite. However, the use of TEG modified with silicon oxide as a filler in the polymer composite leads to a significant ~ 2.5 times increase in the attenuation coefficient compared to the polymer composite with pure TEG as a filler (see Fig. 4). In addition, for this polymer composite, the influence of heating on the magnitude of the damping coefficient is pronounced. Thus, heating to 373 K leads to an increase in the attenuation coefficient by 1.3 times compared to the value of the attenuation coefficient at room temperature. The SWR for this composite increases by ~ two times in the entire frequency range compared to the polymer composite with TEG as filler. At the same time, as follows from Figure 6b, heating does not significantly affect the value of the SWR. Thus, the use of TEG modified with silicon oxide as a filler in polymer composite based on PCTFE results in to an increase in the absorption of radiation energy. Similar results of improving EMR absorption characteristics have been obtained for composites with FeSiAlCr and Ni-Co-P microcapsules covered with a layer of SiO_2 [18, 19].

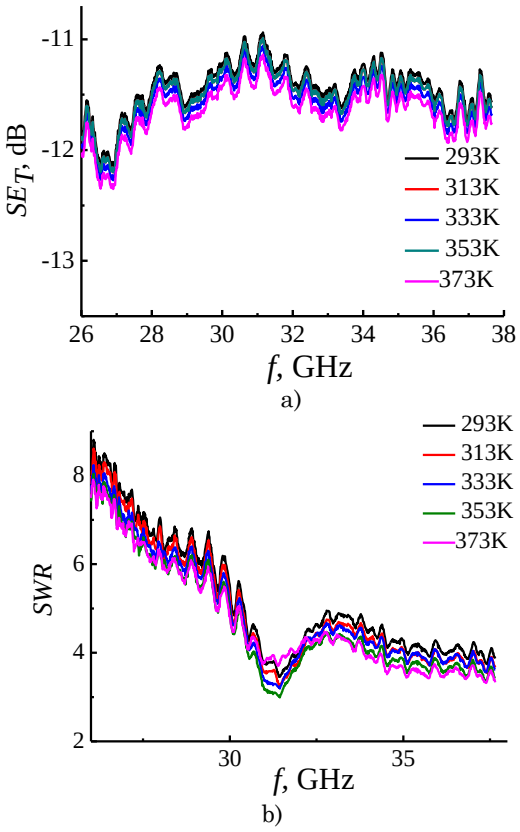
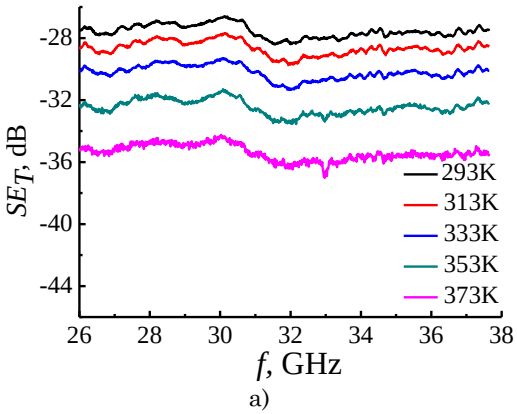


Fig. 5 – The frequency dependences of EMR attenuation (a) and SWR (b) at different temperatures for a PCTFE-based polymer with 3.5 % mass of MWCNTs



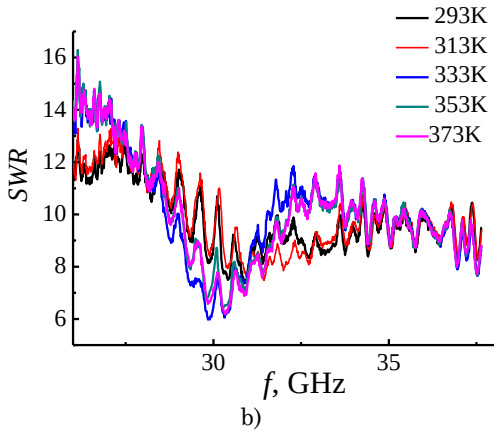


Fig. 6 – The frequency dependences of EMR attenuation (a) and SWR (b) at different temperatures for a PCTFE-based polymer with 2.5 % mass of TEG modified with SiO_2.

For a series of specimens of polymer composites based on PCTFE with various nanocarbon fillers, studies of the temperature dependence of the dielectric permittivity at a frequency of 31 GHz were carried out. In Figure 7 the results of the conducted research are presented.

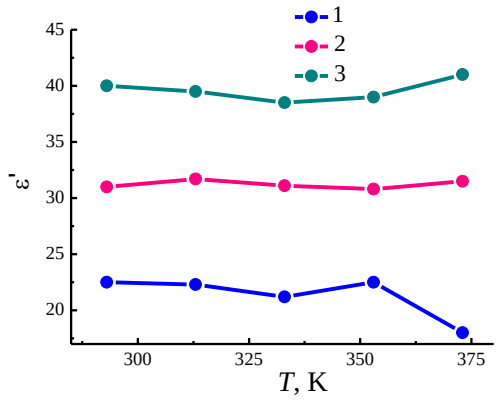


Fig. 7 – Temperature dependence of the dielectric permittivity at a frequency of 31 GHz for polymer composites based on PCTFE with fillers: TEG (1), TEG/SiO_2(2), MWCNTs (3)

As it is follows from Figure 7, the dielectric permittivity for all investigated polymer composites varies slightly in the studied temperature range. So, when specimens of polymer composites are heated from 293 K to 353 K, changes in dielectric permittivity (decrease or increase) amount to no more than (10-15) %. An exception is the polymer composite with a filler of 2.5 % mass TEG/SiO_2, for which, at heat from 353 K to 373 K, a decrease in dielectric constant by ~ 20 % is observed.

The main factors that determine the change in dielectric constant of polymer composite upon heating are the mobility of polymer chains, their kinetic energy, and thermal expansion of the polymer matrix. As the temperature increases, the mobility of polymer chains and, accordingly, dipoles becomes higher and, thus, a larger number of dipoles can be formed and oriented in response to the action of an electric field. On the other hand, due to the high kinetic energy of the dipoles at high temperature, the alignment and orientation of the dipoles along the electric field becomes increasingly difficult. Depending on which of the processes is predominant, either an increase or a decrease in dielectric constant is observed [20-22]. In addition, the thermal expansion of the polymer matrix can partially change the position of the isolated particles of the conductive filler polarized by electric field due to the interphase polarization. This process will also affect the value of the effective dielectric constant of the polymer composite. Note that changes in the electrodynamic characteristics of the studied polymer composites in the investigated temperature range are not critical.

CONCLUSIONS

Thus, the carried-out research made it possible to reveal the peculiarities of changes in the electrical and electrodynamic properties of polymer composites based on PCTFE with various types of nanocarbon filler under thermal load. The use of TEG, modified with SiO_2 TEG and MWCNTs as fillers allows to obtain electrically conductive polymer composites with high indicators of electrical properties and a low percolation threshold (Cp ~ 0.955 % mass), that preserves the unique properties of the polymer (chemical, thermal and radiation resistance, physical and mechanical - shock strength, plasticity, lightness, manufacturability, etc.). At filler concentrations lower than the percolation threshold, the main contribution to the electrical conductivity of the polymer composite is made by the relaxation component of the conductivity σ_l, which is determined by the processes of interphase polarization at the polymer-filler interface. The role of the relaxation component of conductivity is enhanced in polymer composites, in which TEG modified with silicon oxide is used as a filler due to the additional manifestation of interphase interaction at the interface between dispersed TEG/SiO_2 and PCTFE/SiO_2. At filler concentration in the polymer composite is slightly higher than the percolation limit, continuous chain structures of filler particles are formed in the composite, and electrical transport occurs both due to direct contacts and when the filler particles are in contact through thin polymer layers. This results in an increase in the contribution of through electrical conductivity σ_d. The temperature dependence of the electrical conductivity in this case will be determined both by the change with temperature of the intrinsic electrical conductivity of the filler particles and by the temperature dependence of the contact electrical resistance between the filler particles. Thermal expansion of the PCTFE matrix at heat can affect both the number of continuous conductive chains from the filler and the amount of contact electrical resistance between the filler particles due to a decrease in the contact area or an increase in the polymer layer between the filler particles.

When all investigated polymer composites were heated to a temperature of 373 K, a slight improvement in shielding characteristics was found, mainly due to an increase in the EMR absorption coefficient. The largest changes in the EMR attenuation coefficient at heating up to 373 K have been observed for the polymer composite with modified by silicon oxide TEG. Changes in the effective dielectric permittivity of polymer composites in the temperature range of (293-373) K are no more than

(10-20) %, which does not dramatically affect the shielding characteristics of investigated polymer composites. Therefore, the investigated polymer composites based on PCTFE with various types of carbon fillers have stable electrical and electrodynamic properties under thermal load conditions up to 373 K.

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Електричні та електродинамічні властивості полімерних композитів з нановуглецевим наповнювачем

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У статті наведено результати дослідження електричних та електродинамічних властивостей полімерних композитів на основі поліхлортрифторетилену з різними нановуглецевими наповнювачами за теплового навантаження. В якості нановуглецевих наповнювачів використано терморозширений графіт, модифікований оксидом кремнію, і багатошарові вуглецеві нанотрубки. Модифікацію ТРГ кремнеземом проводили з колоїдного 20 % розчину гідрозолю (кремнієвої кислоти). Для досліджень методом термічного пресування отримано масивні полімерні композити на основі поліхлортрифторетилену з різним вмістом нановуглецевого наповнювача. Досліджено діелектричну проникність при кімнатній температурі за допомогою ультрависокочастотного інтерферометра на частоті $\nu = 10$ ГГц. Температурну залежність електропровідності $\sigma(T)$ вимірювали дво- та чотиризондовим методами на постійному та змінному струмі в інтервалі температур (293-425) К. Температурну залежність електродинамічних параметрів досліджували за допомогою панорамних вимірювачів стояння. коефіцієнт хвиль і ослаблення електромагнітного випромінювання в інтервалі температур (293-373) К. Показано, що використання в якості наповнювачів терморозширеного графіту, модифікованого вуглецевими нанотрубками SiO_2, дозволяє отримати електропровідні полімерні композити з низьким порогом перколяції ~ 0,955 % мас. При концентраціях наповнювача, менших за поріг перколяції, основний внесок в електропровідність полімерного композиту вносить релаксаційна складова провідності, яка визначається процесами міжфазної поляризації на межі розділу полімер-наповнювач. При концентрації наповнювача в полімерному композиті трохи вище межі перколяції відбувається збільшення внеску прямої електропровідності за рахунок прямих контактів і контактів через тонкі шари полімеру між частинками наповнювача. Виявлено, що нагрівання полімерних композитів до 373 К призводить до незначного поліпшення характеристик екранування в основному за рахунок збільшення коефіцієнта поглинання електромагнітного випромінювання. Зміни ефективної діелектричної проникності полімерних композитів в інтервалі температур (293-373) К не впливають суттєво на екрануючі характеристики досліджуваних полімерних композитів.

Ключові слова: Поліхлортрифторетилен, Терморозширенний графіт, Багатошарові вуглецеві нанотрубки, Електропровідність, Діелектрична проникність.