Journal of Radiation Researches, vol.2, No.1, 2015, Baku

PACS: 61.80.Ed; 72.80.Tm; 77.22.Ej

FREQUENCY DISPERSION OF ELECTROCONDUCTIVITY AND DIELECTRIC CHARACTERISTICS OF COMPOSITES OF POLYETHYLENE / TiO_2

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Abstract: HDPE/TiO_2 composite was obtained from the homogeneous mixture of powders of high density polyethylene (HDPE) and titanium oxide (TiO_2) by the method of hot pressing. It was studied their specific electroconductivity γ_{ac} on alternating current and dielectric properties (ε / and tgδ) depending on the volume content of TiO_2 and frequency (25-10^6 Hz) of measuring electrical field. In a double logarithmic scale, the dependence $\ln\gamma_{ac} = f(\ln\nu)$ shows two linear region that indicates the degree of dependence of conductivity on frequency. It is assumed that in the studied composite samples it was carried out barrier mechanism of hopping conduction.

It is shown that with increasing concentrations of TiO_2 in the matrix, the decrease $\varepsilon^{/}$ and tgδ depending on the frequency corresponds to an exponential law and explained by the presence of a number of dipole elements.

Cole-Cole diagrams were drawn for HDPE and HDPE + TiO_2 composites on the basis of experimental data. It was revealed the changes of this dependence, resulting in the stabilization of TiO_2 particles in the matrix and showed that the studied samples are characteristic for the whole spectrum of relaxation times.

Keywords: HDPE + TiO_2 composite, dielectric loss, dielectric permittivity, electroconductivity, frequency.

1. Introduction

The development of modern technology requires new multifunctional polymer micro - and nano-composite materials, used in the development of electroactive components of solid-state micro - and optoelectronics. Therefore, in recent years, research in the field of development of materials with special and practically important electrophysical properties based on polymeric composites, containing micro- and nanoparticles of metal oxides, greatly enhanced. The complex of methods, allowing to form the nanoparticles in the polymer matrix, has been developed [1-7]. Such composites have a number of unusual and promising application in relation to electrophysical and optical properties that make them suitable for use as base materials when developing new sensors [8], photoelectric converters, various types of sensors, piezo materials, etc. [6,9]. It should be noted that, dielectric permittivity is sufficiently high, in the polymer composite materials containing dispersed particles of metal oxide, to enable the use of materials with high specific energy (over 10^5Dj/m^3) for high-voltage impulse and microwave technology [10, 11]. However, the expansion of the practical application of polymeric composite materials (PCM) is limited by the lack of detailed information about the adhesion between the particles and the matrix and other possible interactions between the phases, the formation of interface near the micro- and nanoparticle filler, carrier transport mechanism, the role of the phase boundary, the electron ionic and polarization processes in the formation of their active properties. In this context, in the development of new PCM, it is necessary to have information about the temperature and frequency dispersion of the real (ε') and imaginary (ε'') part of the complex permittivity (ε), dielectric loss tangent (tgδ), conductivity constant (γ_{dc}) and alternating (γ_{ac}) and the basic laws of variation of parameters of the spectrum of dielectric relaxation when introduced into the polymer matrix of micro - and nanoparticles of inorganic filler [10]. Such studies are attractive in terms of miniaturization of electronic components and the transition to technology at the molecular level, as well as the performance of actual electronic devices (their performance is largely determined by the rate of relaxation processes). Despite the increase in the number of experimental studies, dedicated to the study of above mentioned processes in polymer micro - and nanocomposites [11-16] the question still remains insufficiently known. In this regard, the study of the dielectric properties and electrical conductivity of the composite medium of HDPE + TiO_2 in a wide temperature and frequency range seems topical. The results of these studies will help better understanding of important, but not fully studied issues. This field, which includes the mechanisms of interaction among the components, electroconductivity, dielectric relaxation, dielectric loss and the nature of charge transfer processes and energy in the composite system of polymeric metal oxide. In addition, the improvement of properties of polymer composites is interesting from the point of view of commercial use and future development of the micro and nano dielectronics.

In this work, it was carried out experimental study of frequency behavior of dielectric properties and electroconductivity in order to find opportunities to control the properties of composite materials based on TiO_2 micro particles in a matrix of high density polyethylene.

2. Experimental

As a dielectric (matrix) it has been chosen powdered high density polyethylene (HDPE) of brand 20806-024, with average molecular weight 95000, crystallinity degree – 52%, melting point - 130˚C and density – 958kg/m^3. Selection of HDPE as a matrix imposed good dielectric properties and processability of the material. Semiconductor TiO_2 served as filler.

While preparing film samples of unfilled HDPE and HDPE+TiO_2 composites, it has been used process flowsheet, using the operations:

- obtain of TiO_2 powders with particle size less than 50 microns;
- drying within 24 h. at T=100˚C and admixture of powdery TiO_2 with HDPE powder in a porcelain mortar;
- compressing homogenous mixture of component powders in a hydraulic press with heated plates under pressure 15 MPa with soaking at temperature 150˚C for 5 min. and obtaining samples of the composites in the form of discs with a diameter of 15mm and thickness of 140÷270microns.
- During obtain process, the concentration of initial components varied.

All things shown in the work are volumetric. Preparation mode of the composite provides repetitive electrophysical parameters (electrical resistance, capacitance, dielectric loss, Q-factor) for the main quantity of samples at the same concentration. Individual samples, which had the parameters different from the parameters of the main group (their number was smallish), were not considered within the analysis. It is assumed that, the filler is distributed fairly evenly in the composite.

The study of the materials were carried out in flat capacitors according to the two-electrode system in equivalent circuit of parallel connected resistor and capacitor in the frequency range 25-10^6 Hz at the temperature of 20C and amplitude of measuring voltage U=1V using a special screened and earthed heated measuring cell of “sandwich” type, having measuring system and potential electrodes with diameters of 10 and 15 mm, respectively. The samples were placed in measuring cell with clamping stainless steel electrodes. The temperature of the sample was controlled using thermocouple copper-constantan. Alignment of electrodes was provided by special mandrel in heated chamber. The thickness of test samples determined the distance between electrodes. Measurement of capacitance C, tangent of dielectric loss angle tgδ, ac – conductivity γ_{ac} in variable fields were conducted by standard methods in perpendicular direction to plane of pressing of samples using a broadband precision immitance meter of type E7-20. Then on the base of measured values of the parameters and geometric dimensions of the samples it was determined the values of real ε' ($\varepsilon' = C \cdot d / \varepsilon_0 S$, where S – area of flat sample, d – thickness of the film) and imaginary ε''($\varepsilon'' = \varepsilon' tg\delta$) parts of complex permittivity of measuring cell with the sample.

Study of electrical (specific conductivity **γ**) and dielectric properties (real part of dielectric constant ε' and dielectric dissipation tgδ) was conducted on a two-electrode system in frequency range 25Hz-10^6Hz at the temperature 20˚C and amplitude of measuring voltage U=1W using special screened and grounded heated measuring cell of “sandwich” type, having the system of measurement and potential electrodes with diameters 10 and 15mm, respectively. The samples were placed in measuring cell with clamping stainless steel electrodes. The temperature of the sample was controlled using thermocouple copper-constantan. Measurement of capacitance C, and tgδ of the samples was carried out by broadband precision immitance meter of type E7-20, and bulk resistance R was measured using teraohmmeter E6-13A. Then on the base of measured values of the parameters and geometric dimensions of the samples it was determined the values ε' ($\varepsilon' = C \cdot d / \varepsilon_0 S$, where S – area of flat sample, d – thickness of the film), ε''($\varepsilon'' = \varepsilon' tg\delta$) and the value of specific conductivity γ_{ac} ($\gamma_{ac} = d/R \cdot S$, where R(Ohm)- sample resistance) of the measuring cell with sample by standard methods.

3. Results and Discussion

The main results of studied frequency dispersion of $\boldsymbol{\gamma_{ac}}$ conductivity, ε/ and tgδ, and Cole-Cole diagrams of ε″=*f*(ε′) HDPE and composite materials on its basis are provided in Figure 1-3.

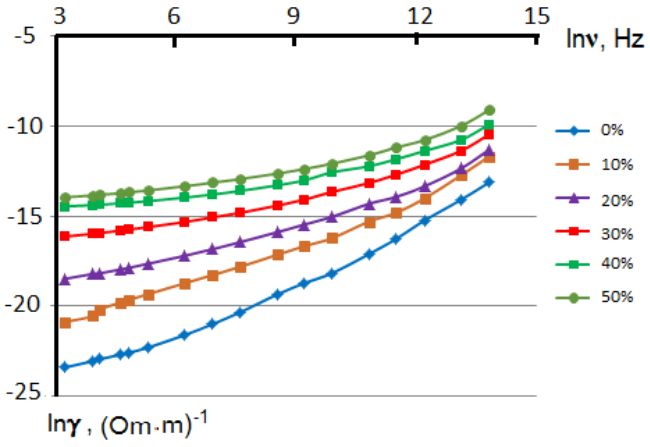


Fig.1 The dependence of conductivity on the frequency of AC of HDPE + TiO_2.composite.

Research of the dependence of electroconductivity of $\boldsymbol{\gamma_{ac}}$ composite medium of HDPE+TiO_2 (the value of $\boldsymbol{\gamma_{ac}}$ for unfilled HDPE at ν = 1 kHz is ~7,5·10^{-}10(Om·m) $^{-1}$) on the content of TiO2 microparticles have shown that there are two regions for conductivity: relatively rapid increase $\boldsymbol{\gamma_{ac}}$ with increasing concentration of TiO_2 up to 20% or more slopping in the region of increase of filler up to 50%. From the linear approximation of the region of curve it has been defined threshold of electric current flow, which is 25-30%. Decrease in the degree of increase of $\boldsymbol{\gamma_{ac}}$ composites after the formation of a percolation cluster, according to [9], means that contribution to the conductivity of additional channels in a cluster of particles, forming at the increase of concentration of the latter above percolation threshold, is substantially less than the total conduction of channel, formed during the formation of a percolation cluster at the percolation threshold. When injection microparticle of TiO_2 up to 50%, $\boldsymbol{\gamma_{ac}}$ HDPE increases up to 1,96·10^{-6}(Om·m)$^{-1}$, ie more than three orders. We assume that the increase of the electroconductivity of the investigated composite materials is conditioned by the increase in the concentration of charge carriers.

Fig.1 shows the change of γ_{ac} depending on the frequency at room temperature for HDPE and composites of HDPE + TiO_2 in the double logarithmic scale.

All dependence characterizes the general property of the increase of conductivity with increasing frequency. It is seen that, the dependence of **$\gamma_{ac}(\nu)$** in logarithmic coordinates shows two linear regions for all samples. This demonstrates the power dependence of conductivity on frequency. According to [10-12], electroconductivity of composites on alternating current is represented as the cumulative effect of dc - conductivity (ν = 0 Hz) due to the migration of the charge and frequency-induced dielectric dispersion and described by the expression

$$\sigma_{ac}(\nu) \approx \sigma_{dc} + A\omega^{s} \qquad (1)$$

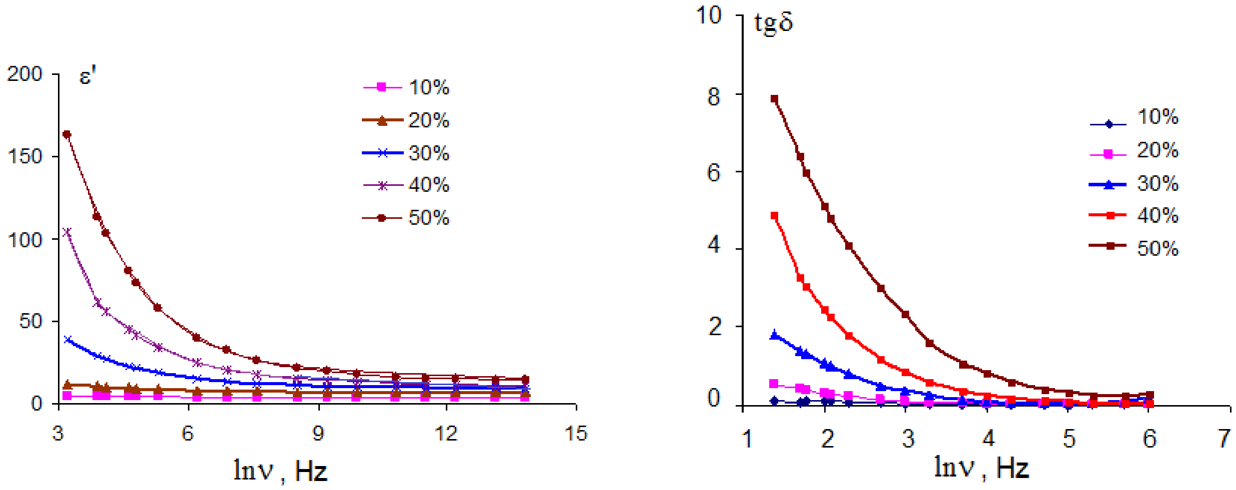
Where σ_{dc}- the conductivity in direct current (dc), $\omega=2\pi\nu$- angular frequency A- constant, temperature dependent on the temperature T, s-parameter, determined by the nature of the mechanism of conductivity and temperature-dependent and frequency of the applied electric field. As a rule [13], 0< s< 1 is relevant for many disordered materials, which include HDPE +TiO_2 composites investigated by us. The presence of linear regions in dependence of $\ln\sigma_{ac}=f(\ln\nu)$ according to [10], shows hopping mechanism of charge transfer. It is seen from figure 1 that, for HDPE and all composites in both regions the parameter s<1, which indicates the existence of nonohmic charge transfer. For all samples of above mentioned certain critical frequency ν_c the region of relatively weak dependence **γ_{ac}** (I region) moves to a stronger region (II region). The corresponding values of ν_c and parameter of s, calculated from the slope of the linear regions of **$\gamma_{ac}(\ln\nu)$**$=f(\ln\nu)$ show that, the parameter of s depends on volume fraction of filler and with the increase of TiO_2 concentration in the polyethylene matrix, it decreases from $s_1 \approx 0{,}50$ up to $s_1 \approx 0{,}14$ for I region, but for II region it decreases from $s_2 \approx 0{,}73$ up to $s_2 \approx 0{,}57$, which is consistent with the introduction of [9]. In addition, the figure shows that the value of critical frequency of ν_c shifted to higher frequencies (from 1·10^4 Hz up to 1·10^5 Гц Hz), ie, I region is expanding. Founded value of parameter of s (from 0,14 up to 0,73) indicates a barrier mechanism of hopping conductivity [15]. According to this model, **$\gamma_{ac}(\nu)$** can be explained by jumping of charge carriers over the states, localized near the Fermi level and for the calculation of s it was given the formula:

$$S = 1 - \frac{6k_{\hat{a}}\grave{O}}{W_{i} - k_{\hat{a}}\grave{O}\ln(1/\omega\tau_{0})} \qquad (2)$$

here, w_M - the maximum height of the barrier and τ_0- the characteristic time of relaxation of order of the vibrational period of atom $\tau_0 \sim 10^{-13}$ c.

At the II region (high frequency region) behavior of **γ_{ac}** - conductivity of composites similar to γ_{ac} of pure polymer and is typical of the multiplet hopping transport of charge carriers. Decrease of **$\gamma_{ac}(\nu)$** at low frequencies is conditioned by interfacial polarization (Maxwell-Wagner polarization), caused by the capture of the volume charge at the interface and polar elements, formed by thermal oxidation of the samples during the obtaining by the method of thermo-compression. In this region padded electric field moves current carriers to long distances and electrical conductivity of γ_{dc} prevails [14]. The increase of frequency leads to reduction of average displacement of charge carriers and the real part of electroconductivity, ν_c changes under the law of **$\gamma_{ac}(\nu)$** $\sim \nu^{0,73}$ after reaching a certain critical frequency. Relatively rapid increase of **$\gamma_{ac}(\nu)$** at the II section with the increase of frequency is attributed to the electron polarization [15].

Results of the study of frequency dependences of ε′ and ε″ of matrix of HDPE (it is not shown in a separate figure) showed that in the range of studied frequencies of ε′ practically remains constant, and ε″ finds a maximum at $\nu=2\cdot10^4$ Hz, then decreases at $\nu=5\cdot10^4$ Hz, passes through a minimum and $\nu=2\cdot10^5$ Hz reaches a maximum. A further increase of frequency leads to a reduction of ε″ up to $\nu=7{,}5\cdot10^5$ Hz, then there is observed a tendency to increase of ε″.
Results of the study of frequency dependency of ε′ and tgδ composites of HDPE + TiO_2 with different volume content of TiO_2 filler at room temperature are shown in Fig. 2 (a, b).

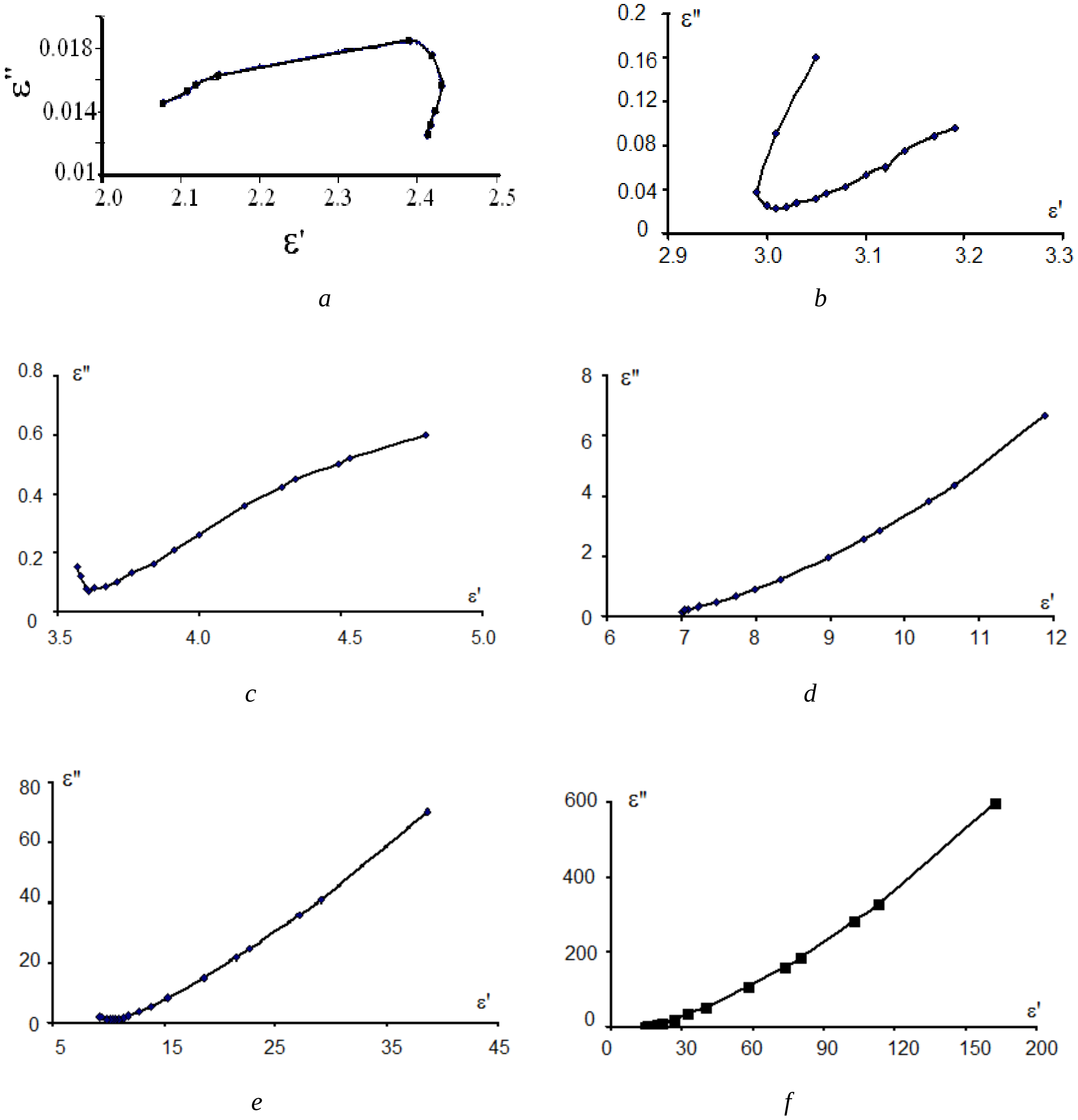


a *b*

Fig.2 The frequency dependence of $\varepsilon^{/}$*(a) tgδ (b) of HDPE +* TiO_2 *composite.*

It is seen that with increase of filler concentration (with growth of electroconductivity) up to 50%, ε′ increases in frequency ranges 10^2 and 10^3 Hz, respectively 35 and 14 times in comparison with polymer matrix. With concentration increasing up to 50%, the rate of change (reduction) in ε′ and tgδ increases and for composite HDPE+50%TiO_2 the rate is maximal. The values ε′ and ε″ recede 5,6 and 153 times, accordingly and the parameter $\Delta\varepsilon=(\varepsilon_c-\varepsilon_\infty)$, where ε_c and ε_∞ - are respectively, low-frequency and high-frequency dielectric constants, describing full width of dispersion, is equal to 66. More than 20% initial reduction in ε′ and ε″, characteristic for composites with TiO_2 concentration, with an increase in frequency up to $2\cdot10^4$ Hz corresponds to exponential law. The authors [18] explain such behavior of ε′ and ε″ of the composites by the presence of a number of dipole elements (eg., in this case the group CO), which appear during obtaining by thermal-pressing method and processing of the material by partial thermal oxidation and their ability to be oriented under electric field. Besides, the authors [18] note that molecular groups located perpendicular to the cross-section of the polymer, usually make their own contribution to the mechanism of dielectric relaxation. At low frequencies of impressed voltage all free dipole elements in the composite are easily oriented through the field (orientation polarization) and form high value of dielectric permittivity at these frequencies. At high frequencies the dipole elements do not have time for the field, their orientation becomes more difficult, oscillation decelerates and orientation polarization disappears which leads to a decrease in dielectric constant. On the other hand [19], dielectric constant of TiO_2 particles also decreases with increase of the frequency of electric field. These two factors substantially lead to decrease in dielectric constant of HDPE+TiO_2 composites with increase of the frequency of electric field. So, practically the constant value of dielectric constant at high frequencies is conditioned by only interfacial Maxwell-Wagner polarization [20]. In this field the kinetics of the process is characterized by long-term relaxation.

On the basis of the obtained experimental data it has been drawn Cole-Cole graphs for HDPE and HDPE+TiO_2 composite medium (Fig.3 a-e) in the case of changes in frequency of applied exterior electric field. It has been revealed the changes in the shown dependence arising within stabilization of TiO_2 particles in HDPE matrix.



a b

c d

e f

Fig. 3 Cole-Cole diagrams $\varepsilon^{//}(\varepsilon^{/})$ for HDPE (a) and samples of composite of HDPE + TiO_2(b-e), b-5%; v-10%; g-20%; d-30%; e-50% TiO_2.

In the case of Debye dispersion in equilibrium state, diagrams $\varepsilon''(\varepsilon')$ have proper semicircle (Cole-Cole diagram). The power of dispersion $\Delta\varepsilon=\varepsilon^{\infty}-\varepsilon^{0}$ remains unchanged from the highest to the lowest frequency [21]. From fig.3 it is seen the existence of non-Debye mechanism of relaxation with a broad distribution of relaxation time, i.e. their range is characteristic to the studied samples. The reason for non-Debye character of the spectrum is obvious: physical constants of effective medium in which, randomly distributed particles of the first and second components of blend are immersed, depend on frequency ν of electric field. The character of the obtained dependence shows that the polymer matrix and composites have a set of polar elements. This indicates that non-polar polymer HDPE material initially acquires polar properties within obtaining (as a result of partial thermal oxidation) and after stabilization there appears TiO_2 particles in it. We assume that polar elements in composites appear at phase boundary of particle-matrix.

It is seen from Fig.3 that diagrams $\varepsilon''(\varepsilon')$ of HDPE+TiO_2 composite medium greatly differ from the diagrams $\varepsilon''(\varepsilon')$ of pure HDPE. With an increase in TiO_2 concentration in diagrams of the composites there appears a linear dielectric dispersion range. The authors [21] associate the linear dispersion with the response to the external relaxation field of polar elements, which are included in dielectric response as a result of reducing orientation polarization. Since the system is non-equilibrium, dielectric response depends on preceding state, whereby the system could not come to the equilibrium state during the time between two states. Migratory defects “freeze” dipoles and include them in instant reorientation of the complex permittivity (non-Markov relaxation) [21]. Non-Markov relaxation is a common problem of imperfect crystals and many polymers, including the composites based on them, which are far from equilibrium state.

4. Conclusion

1. It is shown that in the double logarithmic scale, the dependence $\ln\gamma_{ac}=f(\ln\nu)$ shows two linear area, that indicates the power dependence of conductivity on the frequency. Founded values of parameter s (from 0.14 up to 0.73) indicate that barrier mechanism of hopping conduction is carried out in the investigated composite samples.
2. It is shown that with increasing concentrations of TiO_2 in the matrix, the decrease of ε' and tgδ depending on the frequency corresponds to an exponential law and explained by the presence of a number of dipole elements that appear during the addition of TiO_2 and obtain by method of thermocompression and processing the material by partial thermal oxidation and their ability to be orientated under the influence of electric field.
3. Diagrams of the Cole-Cole was drawn for HDPE and HDPE + TiO_2 composites, the change of this dependence, resulting in the stabilization of TiO_2 particles in a matrix was revealed and it was showed that the investigated samples are characteristic for whole spectrum of relaxation times.

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ЧАСТОТНАЯ ДИСПЕРСИЯ ЭЛЕКТРОПРОВОДНОСТИ И ДИЭЛЕКТРИЧЕСКИХ ХАРАКТЕРИСТИК КОМПОЗИТОВ ПОЛИЭТИЛЕН /TiO_2

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Резюме: Из гомогенной смеси порошков полиэтилена высокой плотности (ПЭВП) и окиси титана (TiO_2) методом горячего прессования получены композиты ПЭВП/TiO_2. Изучены их удельная электропроводность γ_{ac} на переменном токе и диэлектрические характеристики (ε и tgδ) в зависимости от объемного содержания TiO_2 и частоты (25-10^6 Гц) измерительного электрического поля. В двойном логарифмическом масштабе зависимость $\ln\gamma_{ac} = f(\ln\nu)$ обнаруживает два линейных участка, что свидетельствует о степенной зависимость проводимости от частоты. Предполагается, что в исследованных композитных образцах осуществляется барьерный механизм прыжковой проводимости.

Показано, что с увеличением концентрации TiO_2 в матрице уменьшение ε и tgδ в зависимости от частоты отвечает экспоненциальному закону и объясняется присутствием ряд дипольных элементов.

На основании экспериментальных данных построены диаграммы Коула-Коула для ПЭВП и композитов ПЭВП+ TiO_2. Выявлены изменения данной зависимости, возникающие при стабилизации частиц TiO_2 в матрице и показаны, что исследованным образцам свойствен целый спектр времен релаксации.

Ключевые слова: Композит ПЭВП+ TiO_2, диэлектрические потери, диэлектрическая проницаемость, электропроводность, частота.

POLİETİLEN/TiO_2 KOMPOZİTLƏRİNİN ELEKTRİK KEÇİRİCİLİYİ VƏ DİELEKTRİK XARAKTERİSTİKALARININ TEZLİK DİSPERSİYASI

M.M. Guliyev, N.Ş. Əliyev, A.M. Məhərrəmov, R.S. Ismayilova, M.N. Bayramov

Xülasə: Termik presləmə üsulu ilə yüksək sıxlıqlı polietilen (YSPE) və titan oksidinin (TiO_2) homogen qarışığından YSPE /TiO_2 kompozitləri alınmışdır. Dəyişən elektrik sahəsində TiO_2 həcmi faizindən və tezlikdən (25-10^6 Hz) asılı olaraq xüsusi keçiricilik (γ_{ac}) və dielektrik xarakteristikaları (ε və tgδ) öyrənilmişdir. İkiqat loqarifmik $\ln\gamma_{ac} = f(\ln\nu)$ əyrilərində iki xətti hissə alınır ki, bu da keçiriciliyin tezlikdən üstlü asılılığını göstərir. Güman edilir ki, tədqiq edilmiş kompozit nümunələrində sıçrayışlı keçiriciliyin baryer mexanizmi baş verir.

Göstərilmişdir ki, matrisada TiO_2 konsentrasiyasının artıması ilə ε və tgδ tezlikdən asılı olaraq azalması eksponensial qanuna tabe olur və bir sıra dipol elementlərinin olması ilə izah olunur.

Eksperimental məlumatlara əsasən YSPE və YSPE /TiO_2 kompozitləri üçün Koula-Koula diaqramları qurulmuşdur. Matrisada TiO_2 hissəciklərinin stabilləşməsi zamanı bu asılılıqlardakı dəyişikliklər aşkar edilmişdir və göstərilmişdir ki, tədqiq edilmiş nümunələr relaksasiya müddətlərinin bütöv spektri ilə xarakterikdir.

Açar sözlər: YSPE +TiO_2 kompoziti, dielektrik itkisi, dielektrik nüfuzluğu, elektrik keçiriciliyi, tezlik.