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Abstract—In this work, Cu/LLDPE composites are obtained and it is shown that the method of combining the formation of the composite and the nanodispersed phase in the viscous-flow state of the polymer makes it possible to achieve a uniform distribution of nanoparticles in the matrix. The dielectric properties of the composites have been investigated. A change in the mechanism of electrical conductivity was revealed when the concentration of the nanodispersed phase was varied. At low frequencies, nanocomposites of this type exhibit through conductivity. It was found that no pronounced relaxation peaks are observed in the studied polymer nanocomposites. However, at high concentrations of the added additive > 20%, two weakly pronounced peaks appear in the region of low and high temperatures, which, with increasing temperature, shift towards higher frequencies.

Keywords—Composites, conductivity, nanoparticles, polymers.

#### I. INTRODUCTION

T HE development of polymer composite materials is one of the priority directions in the development of modern technologies and technics [1]-[14]. The unique combination of valuable properties of polymer matrices such as film formation, mechanical strength, chemical and corrosion resistance, on the one hand, and the functional properties of nanoscale fillers, on the other, makes it possible to consider such materials as promising for use in aerospace engineering, instrumentation, microelectronics, medicine and other high-tech industries [15]-[28].

The term "nanocomposites" appeared relatively recently,

but natural nanocomposites have been known for a long time [29]-[35]. Clay mineral nanoparticles are widely used to control the viscosity properties of polymer solutions and to stabilize gels. The nature of the effect of nanoparticles on the properties of composite nanomaterials and the directions of their use largely depend on the matrix (the medium where the nanoparticles are dispersed) [36]-[42].

The development of technologies for creating nanocomposite systems is currently mainly moving towards the development of filled composites with the introduction of functionally active compounds into an inorganic or polymer matrix, which makes it possible to create fundamentally new materials [43]-[73].

Analysis of the nanocomposites market showed that the production and use of nanosystems is growing up to 18% annually, where 77% is the thermoplastic market [8]. Progress can be attributed to the availability of raw materials and a large sales market.

Comparing the statistical data based on publications over the past 6 years, it can be concluded that polymer-matrix composites and coatings based on polymer-matrix composites are of primary interest. In recent years, interest has increased in the development of thin-film coatings based on composite nanomaterials.

The main purpose of the polymer binder is to bind the filler together, to ensure the joint operation of all monofilaments (or particles, if a dispersed filler is used), to ensure the solidity of the material and the transfer (distribution) of stresses. The properties of the binder almost completely depend on: heat and heat resistance, resistance to the action of various working media (water, steam, fuel, oils, etc.), impact strength, impact strength, resistance to prolonged exposure to alternating loads, creep, stress relaxation. After curing (for thermosetting materials) or hardening (for thermoplastic materials), the binder turns into a matrix. The matrix is a continuous phase, the layer thickness of which can vary from 1 to 1000  $\mu$ m [19].

In the "ideal" case, the binder should have the following properties: the deformation properties of the matrix should be no lower than that of the filler  $\epsilon m > \epsilon n$ ; the binder should have a relatively high modulus of elasticity (E> 2000 MPa); the binder should have good adhesion to the filler ( $\tau sd > 20$  MPa).

The matrix and filler must necessarily have good compatibility, however, must not dissolve in each other. Based on the factors of matrix selection: molding technology; type of production; geometric features of the resulting product; technological and operational properties in this study will be studied linear low density polyethylene.

The ability of metal nanoparticles (Au, Ag, Cu, etc.) to absorb and/or scatter radiation with a given wavelength, due to the effect of localized surface plasmon resonance, makes them an interesting object for study and opens up wide possibilities for the practical application of such particles. The main attention is attracted by nanoparticles of noble metals - gold and silver, for which there is a possibility of "tuning" localized surface plasmon resonance in a wide wavelength range by changing the shape and/or structure of particles, while copper is less "popular" among them. This is primarily due to the fact that it is easily oxidized. To reduce the oxidation state of copper nanoparticles, their synthesis is carried out in an inert atmosphere (in nitrogen or argon) and/or in non-aqueous media. The so-called "polyol" method is often used [20-22]. Its essence consists in the reduction of Cu<sup>2+</sup> ions using a strong reducing agent (hydrazine, sodium phosphinate, etc.) in a polar organic solvent (ethylene glycol, dimethylformamide (DMF), etc.) in the presence of polyvinylpyrrolidone (PVP). This polymer, firstly, due to the lone pair of electrons of the oxygen of the carbonyl group coordinates the Cu<sup>2+</sup>, ions, which are then reduced to Cu<sup>0</sup>. Second, PVP serves as a stabilizer for the dispersion of the forming copper nanoparticles, and, in addition, the adsorption layer of PVP to a certain extent prevents the oxidation of nanoparticles. Analysis of literature data allows us to draw the following conclusions. First, regardless of the method for preparing colloidal solutions of copper, when they come into contact with air, the oxidation of nanoparticles usually occurs very quickly. The situation is complicated by the fact that it is very difficult to visually detect this fact, since the salts of copper and its oxides are often close in color. As a consequence, in a number of cases, researchers could have dealt, rather, with nanoparticles of copper oxides of one composition or another. Indeed, for copper nanoparticles, the region of interband transitions overlaps with the region of localized surface plasmon resonance, which leads to a significant decrease in the intensity of the resonance absorption peak and causes the red-brown color of the Cu sol, which is observed in most cases (the color of the Cu<sub>2</sub>O sol changes from yellow to orange with increasing particle size). Secondly, to date, there is very little information

on the effect on the oxidation rate of copper nanoparticles of certain compounds present in a colloidal solution (reaction products, impurity ions, etc.). Only quite recently there have appeared several works devoted to the study of the regularities of the oxidation of Cu particles in non-polar solvents [23]-[25] (including in the presence of various stabilizers).

It is known that many polymers are characterized by low values dielectric constant. In recent years, considerable attention to the creation of polymer nanocomposite materials with high values of the dielectric constant while maintaining their acceptable mechanical properties and dielectric strength. Such materials are of interest for many technological problems [15]-[23]. However, for of the described composites of this type, the values of the dielectric constant relatively low (<300 at 1 kHz and room temperature) even at high filler content.

Interest in polymer nanocomposite materials is due to the fact that that due to the small size of the filler particles they find a tendency to manifest a relatively low value of the percolation threshold, making them good candidates for materials with the required electrical properties [24]-[36].

In this regard, in this work, it was of interest to study dielectric properties of the resulting nanocomposite materials.

#### II. SYNTHESIS OF POLYMER NANOCOMPOSITES

Modern composites have not only a wide range of physical and mechanical properties, but are also capable of directionally changing them, for example, increasing fracture toughness, regulating rigidity, strength, and other properties. These possibilities are expanded when fibers of different nature and geometry are used in composites, i.e., when creating hybrid composites. In addition, these materials are characterized by the appearance of a synergistic effect (coordinated joint action of several factors in one direction).

The properties of the interface or interfacial zone, first of all, the adhesive interaction between the fiber and the matrix, determine the level of properties of composites and their retention during operation. Local stresses in the composite reach their maximum values just near or directly at the interface, where material destruction usually begins. The interface must have certain properties to ensure efficient transfer of the mechanical load from the matrix to the fiber. The adhesion bond at the interface should not be destroyed under the action of thermal and shrinkage stresses arising from the difference in the temperature coefficients of linear expansion of the matrix and fiber or as a result of chemical shrinkage of the binder during its curing.

When creating nanocomposites, the key tasks are the development of efficient, reliable, and affordable production technologies for mass production, which make it possible to obtain materials with stable characteristics. The hand lay technique, also called wet lay, is the simplest and most widely used process for producing flat reinforced composites. The process consists of laying layers of a polymer in successive layering using an epoxy matrix. Wet-laying is a molding process that combines layers of reinforced carbon fiber with epoxy to create a high-quality laminate. Before starting the

installation process, you must prepare the appropriate form. This preparation consists of cleaning the table and applying a release agent to the surface. The manual laying process can be divided into four main steps: mold preparation, epoxy coating, laying and curing. Form preparation is one of the most important steps in the installation process. This process requires dry reinforcement layers and the application of a wet epoxy matrix. They are connected together - reinforcing material, impregnated with a matrix - epoxy resin.

Thermal decomposition of copper (II) precursor can be described in two stages as follows:

## 1. Cu(HCOO)2 Cu(CHOO)+1/2H2+CO2 (185-227°C),

2. Cu(CHOO) Cu+1/2H2+CO2 (227-247°C).

As a result of thermolysis at a temperature of 250 °C in an oven for analytical work and heat treatment with a constant temperature in vacuum ( $\pm$  3 mm Hg) for 1 hour, a powder of uniform consistency of light brown color was obtained. X-ray phase analysis showed that the average size of copper particles in this powder was 30-49 nm.

A thermoplastic polymer — linear low-density polyethylene — was used as a polymer matrix. Mark 3306 WC4, density -0.918g/cm<sup>3</sup>(Taiwan). To mix PE with copper nanoparticles, we used a twin-screw extruder HAAKE Minilab II with synchronous co-directional rotation of the screws. The mixture was carried out in the atmosphere of the region in order to avoid unnecessary contact with the air, which can lead to oxidation of components and deterioration of the characteristics of the resulting material.

The formation of metal nanoparticles occurs in situ, in a space limited by polymer chains, which prevents their agglomeration and promotes the formation of highly dispersed metal particles. XRF data confirm the formation of the nanocrystalline Cu phase. The considered method of obtaining polymer nanocomposites makes it possible to control the composition and size of copper nanoparticles: indeed, the average size of nanocrystallites of the metal phase, in accordance with the calculation of the coherent scattering regions according to XRF data, increases with an increase in the concentration of copper (II) formate in the composition of the initial product metal-containing precursor/polymer matrix.

The formation of nanoparticles involves a stepwise mechanism of nucleation, growth, and agglomeration. The change in the size of nanoparticles with an increase in the concentration of the metal-containing precursor in the copper (II) formiate/LLDPE mixture is apparently related to the fact that the number of forming nuclei is approximately the same in the considered range of reagent concentrations, and the growth of nanoparticles occurs due to the formed monomeric metal atoms, the amount which is determined by the content of the precursor.

In physical and mechanical tensile tests, the efficiency of filling the composite with Cu nanoparticles was monitored according to the following parameters: tensile strength (Rm, MPa), elastic modulus (E, GPa), relative deformation ( $\varepsilon$ , %).

The test included control samples not modified with copper and samples of nanocomposites with different contents of Cu nanoparticles. The size of the samples was  $10 \times 90$  mm, the thickness was 0,08 mm. The tensile test speed was 2 mm/min. The samples were securely fixed in rubberized clamps and a load was applied until complete destruction. Fracture occurred in the middle of the sample. After carrying out all the necessary tests and calculations, diagrams of the dependence of strength, modulus, and deformation on the concentration of Cu nanoparticles were obtained.

#### III. DIELECTRIC PROPERTIES OF POLYMER NANOCOMPOSITES

Dielectric measurements of Cu LLDPE samples were carried out on an automated broadband dielectric spectrometer Novocontrol, Novocontrol Technologies GmbH (Germany) based on a digital synchronous detector Stanford Research SR 810 with a measurement range: frequencies: 10-2-105 Hz, capacities: 5x10-13-10-3 F, resistances: 10 - 1014 Ohm, measurement accuracy tg( $\delta$ ) >10<sup>-4</sup>. It also includes a Quatro cryostat system, with a temperature range of measurements from -160 to 400 °C, and a temperature holding accuracy of up to 0.02 °C and WinDETA measurement control software, and dielectric processing - WinFIT 2.90.

Tests of nanocomposite samples were carried out in the frequency ranges  $f = 10^{-2} \cdot 10^5$  Hz and temperatures from -160 to 110 °C (113 - 383 K). The sample temperature during measurements was controlled with an accuracy of 0.5 °C. The measuring cell of the "sandwich" type consists of two stainless steel electrodes with a diameter of 20 mm, between which the sample was installed. The voltage between the electrodes did not exceed 1 V. Film samples, about 0,23 mm thick, cut along the diameter of the electrodes were measured.

The ability of a material to absorb electromagnetic radiation in the microwave range is described by the frequency dependence of the complex dielectric constant:  $\epsilon(f)=\epsilon'(f)-i\cdot\epsilon''(f)$ , where i – imaginary unit,  $\epsilon'(f)$ - real part,  $\epsilon''(f)$  – imaginary part. The real and imaginary parts characterize the dielectric polarization and loss in the material, respectively. The measurement error  $\epsilon'$  is 5%.

In this work, the dielectric properties of polymer composites Cu-LLDPE are investigated with varying the filler content from 1 to 20 wt.% In the frequency ranges f = 10-2-105 Hz and temperatures of -160...110 °C.

It was found that no pronounced relaxation peaks are observed in the studied polymer nanocomposites. However, at high concentrations of the added additive > 20%, two weakly pronounced peaks appear in the region of low and high temperatures, which, with increasing temperature, shift towards higher frequencies.

Fig. 1a shows the dependence of the imaginary part of the complex dielectric constant  $\varepsilon *= \varepsilon$  '-j  $\varepsilon$  " the frequency in Cu LLDPE with different copper content, measured at -50°C.

It can be seen from the figure that the dependence  $\varepsilon$  " (f) shows a weakly pronounced peak P1 (in the frequency range 1-104 Hz), which, with increasing concentration, shifts

towards higher frequencies. Peak P1 has a large half-width, which may indicate a wide distribution of relaxators in frequency. In addition, a high-frequency peak P0 is observed, which turned out to be at the edge of the frequency range of the device.

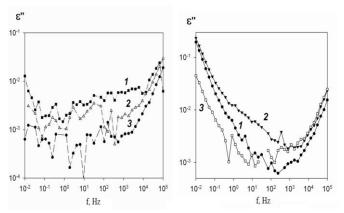


Fig. 1 Frequency dependences of ε" at -50°C (A) and 10°C (B) in nanocomposite films of Cu LLDPE with different filler contents: 8 (1), 20 (2), 3 - 30 wt. % (3)

It was found that the P0 peak does not depend on the measurement temperature or on the concentration of the introduced impurity. Consider now the region of high temperatures. Fig. 1b shows the dependences of the imaginary  $\varepsilon$ " part of the complex dielectric constant on frequency in composite Cu LLDPE films with different copper concentrations measured at 10°C.

It can be seen that, in addition to the P0 peak, at the edge of the frequency range, in the high-frequency region, there is a weakly pronounced P2 relaxation peak, which is superimposed by an increase in  $\varepsilon$ ", due to the presence of through conduction in this frequency/temperature range. Peak P2 is well observed at a concentration of 20% of the administered copper (see Fig. 1, curve 2). It was found that the P2 peak shifts towards higher frequencies with an increase in the impurity concentration.

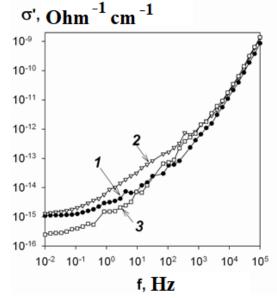


Fig. 2 Dependence of σ' on the logarithm of frequency in composite films based on polyethylene with different Cu concentrations: 1 - 8; 2 - 20; 3- 30%. Temperature of measurement was 10°C.

Fig. 2 shows the dependence of the real part of the complex electrical conductivity on the frequency in composite films based on polyethylene with different Cu concentrations measured at a measurement temperature of 10°C. It can be seen that the graph  $\log (\sigma) = \phi (\log (f))$  has two components, one of which shows a dependence on frequency, and the other does not. The pronounced plateau in the low-frequency region, which spreads to the high-frequency region with increasing temperature, can be attributed to the transport of free charges. The transition from the plateau region to the dependence on the frequency (at the frequency f \*) corresponds to a change in the mechanism of electrical conductivity. In this case, the plateau area on the left side of the dependence reflects the movement of charges over long distances, on the right side  $\sigma$ /ac increases with increasing frequency, and the movement of charges is limited by the space of their potential wells. To explain this, it is referred to the Fig. 2. Indeed, it can be seen from the figure that with an increase in the concentration of introduced Cu, it practically does not change. However, at a high concentration (30%), the through conduction decreases.

### IV. CONCLUSION

Thus, the study of the dielectric properties of the obtained polymer Cu / LLDPE nanocomposites showed that a feature of their dielectric properties is a change in the mechanism of electrical conductivity when the concentration of the nanodispersed phase is varied: at low filler concentrations at low frequencies, nanocomposites of this type exhibit through conduction. The pronounced plateau in the low-frequency region, which spreads to the high-frequency region with increasing temperature, can be attributed to the transport of free charges. It was found that no pronounced relaxation peaks are observed in the studied polymer nanocomposites. However, at high concentrations of the added additive > 20%, two weakly pronounced peaks appear in the region of low and high temperatures, which, with increasing temperature, shift towards higher frequencies. The results obtained open the prospective for tailored fabrication of nanocomposites with desired dielectric properties for their subsequent practical application as construction materials.

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