Preparation of Low-Density Polyethylene Composite with Copper Nanoparticles

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Abstract—In this work, devoted to the preparation and study of the properties of copper-containing nanocomposites based on linear low density polyethylene, it is shown that the composition of the resulting nanocomposite is not complicated by phase transformations during its synthesis. Varying the concentration of copper (II) formate (1.2÷50.8 wt%) makes it possible to control the size of the formed nanoparticles (10.2±2.0÷ 21.3±1,5 nm). It has been shown that the presence of Cu nanoparticles in a polymer matrix leads to a decrease in its degree of crystallinity - the value decreases from 42% for polyethylene to 37% for 3Cu / LLDPE, which is due to the restriction of the free movement of polymer chain segments by metal nanoparticles and, accordingly, the formation of a less ordered crystal structure.

Keywords—Polymers, composites, nanoparticles, copper.

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1. Introduction

It is known that the symbiosis of materials of different nature can lead to the formation of a new material with new characteristics in comparison with individual components of this material [1],[2],[3],[4],[5],[6],[7],[8],[9],[10],[11]. The filler affects the properties of the matrix depending on the concentration, particle size, and the nature of the interaction with the polymer [12],[13],[14],[15],[16],[17],[18],[19],[20], [21],[22],[23],[24],[25],[26],[27],[28]. Recently, nanocomposites have become widespread - multiphase (multicomponent) materials in which at least one phase (component) has an average crystallite (grain) parameter in the nanoscale (up to 100, nm).

The term "nanocomposites" appeared relatively recently, but natural nanocomposites have been known for a long time [29],[30],[31],[32],[33],[34],[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],[37],[38],[39],[40],[41],[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],[44],[45],[46],[47],[48],[49],[50],[51],[52],[53], [54],[55],[56],[57],[58],[59],[60],[61],[62],[63],[64],[65],[66], [67],[68],[69],[70][71].

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.

2. The structure of metal-polymer nanocomposites

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 μm [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 εm> εn; the binder should have a relatively high modulus of elasticity (E> 2000 MPa); the binder should have good adhesion to the filler (τsd> 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], [21],[22]. Its essence consists in the reduction of Cu^{2+} 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^{2+}, ions, which are then reduced to Cu^0. 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 sols 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_2O 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],[24], [25] (including in the presence of various stabilizers).

3. Synthesis of Cu nanoparticles and metal-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.

The synthetic procedure used in this study was the following. 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 (± 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^3(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. For further details of the synthetic procedure it is referred to the literature [23],[24], [25].

4. Properties of synthesized nanocomposites

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 (Fig. 1). The considered method of obtaining polymer nanocomposites makes it possible to control the composition and size of copper nanoparticles. Thus, the average size of nanocrystallites of the metal phase, in accordance with the calculation of the coherent scattering regions according to XRF data (Fig. 1), increases with an increase in the concentration of copper (II) formate in the composition of the initial product metal-containing precursor / polymer matrix.

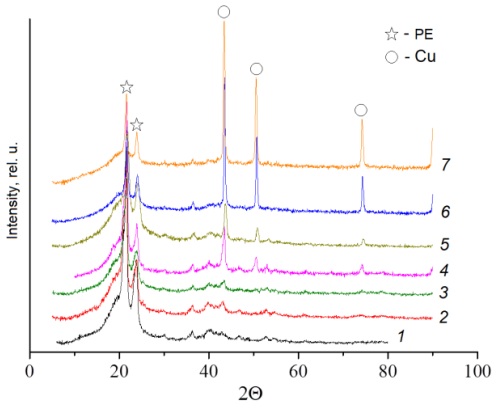

Fig. 1 x-ray diffraction patterns of nanocomposites 0.5Cu/LLDPE (1), 1Cu/LLDPE (2), 3Cu/LLDPE (3), 8Cu/LLDPE (4), 20Cu/LLDPE (5), 30Cu/LLDPE (6).

From the XRD data it is clearly seen, that formation of Cu-PE nanocomposite is observed for all samples, but distinguished Cu peaks are observed starting from the concentration of Cu nanoparticles of 8%.

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. This effect is ascribed to the in situ method of formation of the nanoparticles.

Tests for examination of the glass transition temperature value of the obtained epoxy nanocomposites were carried out by the method of differential scanning calorimetry with standard procedure with an increase in temperature from 20 to 200 °C and a test speed of 5 °C/min in a nitrogen atmosphere. These tests gave the result that the glass transition temperature of the obtained epoxy nanocomposites weakly depends on the Cu concentration, however, there is a tendency to a slight decrease: at concentrations less than 1 wt. % the glass transition temperature decreases to 167 °C, and at concentrations up to 5 wt. % rises and reaches 173 °C. Such a decrease in the glass transition temperature may indicate a change in the viscosity and elasticity of the system, an increase in the flexibility of molecules and their mobility, and a further increase in the concentration of nanoparticles leads to an increase in the rigidity and, consequently, to an increase in the glass transition temperature of the system. These results are presented in the Fig. 2.

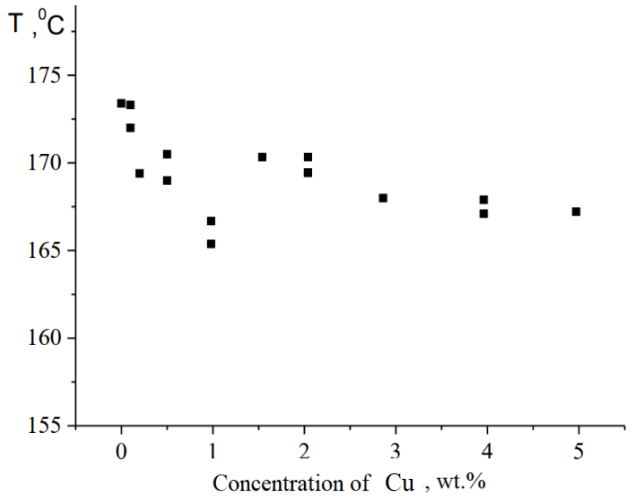

Fig. 2 Dependence of the glass transition temperature of Cu/EP nanocomposites on the concentration of Cu.

Based on the results obtained, it can be concluded that the presence of titanium dioxide nanoparticles in the system leads to a slight decrease in the glass transition temperature at a concentration of 1% of Cu nanoparticles.

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 (ε, %). 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 10×90 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. As it was shown, tensile strength drops sharply at Cu concentrations greater than 3 wt. %, which is possibly associated with secondary processes of agglomeration of Cu (Fig. 3). Therefore, the working concentration should not exceed 3 mass. % Cu.

At concentrations above 1 wt. % increase in the modulus of elasticity is 20-25%, which indicates the effect of antiplasticization of the nanocomposite (Fig. 4). The change in the relative deformation with varying nanofiller content is complex: the deformation increases at Cu concentrations less than 1 wt. %, and decreases at Cu concentrations above 1 wt. % (Fig. 5).

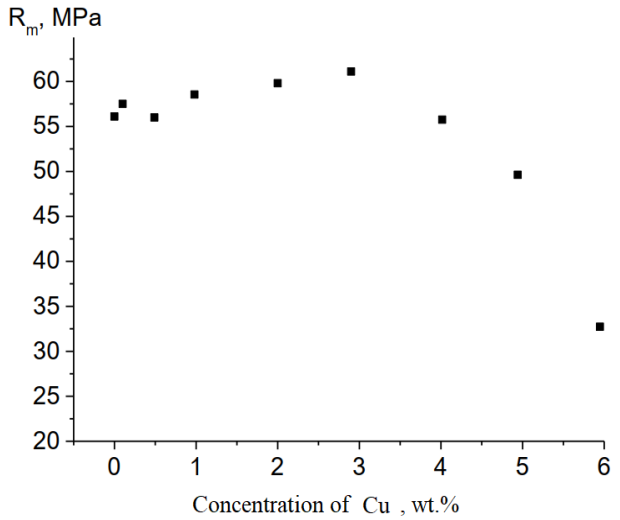

Fig. 3 Dependence of the tensile strength of Cu/EP nanocomposite on the concentration of Cu.

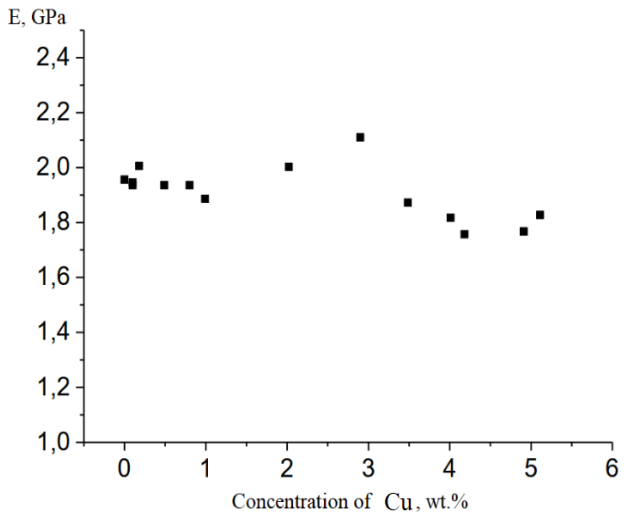

Fig. 4 Dependence of the modulus of elasticity of Cu/EP nanocomposite on the concentration of Cu.

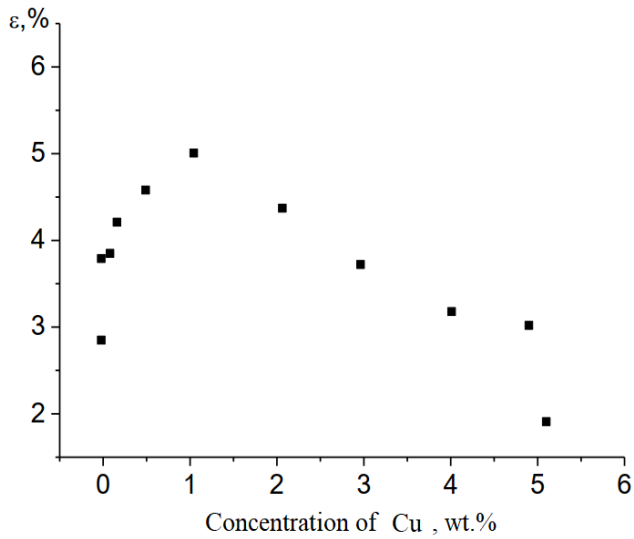

Fig. 5 Dependence of the relative deformation of the Cu/EP nanocomposite on the Cu concentration.

Thus, the mechanical properties depend on the concentration of Cu nanoparticles. The values of the modulus of elasticity and tensile strength are maximum at a nanoparticle concentration of 3 wt. %. The relative deformation increases with increasing concentration up to 1 wt. %.

The technical result is to simplify the production technology of film composite materials by combining the production of a composite material and a nanodispersed metal phase in one stage, as well as obtaining homogeneous (with uniformly distributed nanoparticles in a polymer matrix) film composite materials based on thermoplastic polymer matrices and copper nanoparticles with adjustable size and concentration of the dispersed phase.

The advantage of the proposed method is its technological simplicity, the availability of starting materials and thermoplastic polymers for large-scale production, the use of traditional technological cycles for processing polymeric

materials (extrusion, mixing in micro-mixers, pressing) to obtain a film nanocomposite material. This opens new prospective for tailored fabrication of polymer nanocomposites with desired structure and properties. Further research can be aimed toward the development of nanocomposites with advanced mechanical properties.

5. Conclusion

In the case of Cu / LLDPE nanocomposites obtained by the in situ method, metal nanoparticles are formed during the thermal decomposition of the precursor at the moment of its mixing with the polymer melt during extrusion. The process of obtaining a nanocomposite material is carried out under conditions of coincidence of the temperature ranges of the decomposition of metal formate with the temperature range of the presence of polymers in a viscous-fluid state.

To determine the structure of the obtained nanocomposite, the method of IR spectroscopy was used. IR spectroscopy allows first to get evidences of the interaction between nanoparticles and polymer matrix and, second, get the information about the structure of the resulting nanocomposite. For the details of the IR characterization of the polymer nanocomposites, it is referred to the literature [53],[54],[55].

Nanocomposites based on epoxy resin and copper nanoparticles have been obtained by introducing finished nanoparticles into epoxy resin at the stage of its curing, and the effect of nanoparticle concentration on physical and mechanical properties has been investigated. The physical and mechanical properties of epoxy nanocomposites have been investigated. It is shown that the introduction of 1 mass. % Cu nanoparticles leads to an increase in the relative deformation by 35-40%. The maximum tensile strength values are achieved at a concentration of 3 wt. % Cu. The increase in the elastic modulus is 20-25% at Cu concentrations above 1%. Improved mechanical properties indicate the formation of strong interfacial interactions.

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