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Thermophysical and Magnetic Properties of Magnetite – Polyethylene Composite

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Abstract—In this work, it is shown that the advantage of using matrix-stabilized magnetic nanoparticles to obtain polymer nanocomposites based on them is that such nanoparticles retain their dispersion and stability of size and shape in the technological modes of obtaining polymer nanocomposite materials, and thus ensured stable ferro- and superparamagnetic properties of the obtained target products.

For the production of films by the method of hot pressing from blanks obtained in an injection molding machine or a mechanochemical mixture, a manual electrically heated hydraulic press was used. The magnetic properties of nanocomposite samples (about 50 mg on average) were studied using a vibration magnetometer.

The character of the dependence of the magnetization on the magnitude of the magnetic field confirms the ferromagnetic character of the behavior of the obtained nanocomposites. The resulting film nanocomposites exhibit ferromagnetic properties at room temperature.

Keywords—Composites, polymers, thermophysical properties, magnetite.

I. Introduction

Among the wide range of investigated nanoscale materials for various environmental and biomedical applications, magnetic nanoparticles have received considerable attention due to their intrinsic magnetic properties, making them successful as magnetically reduced catalysts, drug delivery agents, anti-cancer materials, magnetic resonance imaging, etc. This class of nanomaterials includes metallic, bimetallic nanoparticles, metal oxides, ferrites, and superparamagnetic iron oxide nanoparticles. Magnetic nanoparticles and nanocomposites have generated significant scientific and technological interest due to their potential applications in biomedicine, information technology, magnetic resonance imaging, catalysis, telecommunications, and environmental restoration [1]-[14]. Magnetic nanocomposites usually contain magnetic nanoparticles embedded in a non-magnetic or magnetic matrix. However, magnetic nanoparticles dispersed in composites usually have a strong tendency to form agglomerates to reduce the energy associated with the high surface area to volume ratio of nanosized particles [15-14]. To avoid aggregation of magnetic nanoparticles, protection strategies have been developed to chemically stabilize unprotected magnetic nanoparticles by grafting or coating with organic species, including surfactants or polymers, or coating with an inorganic layer such as silicon dioxide or carbon. Combining these functionalized magnetic nanoparticles in a polymer or other matrices to develop magnetic nanocomposite materials has proven to be more efficient [25]-[32]. There are mainly four types of magnetic nanocomposites, i.e. inorganic core nanocomposites, self-assembled nanocomposites, silica-based magnetic nanocomposites, and organic-inorganic nanocomposites [33]-[50]. Among them, organic inorganic magnetic nanocomposites have become more interesting due to the combination of the unique properties of the organic and inorganic components in one material. Hybrid organic inorganic magnetic nanocomposite materials can be obtained in situ, ex situ, microwave exposure, coprecipitation, melt mixing, ceramic glass treatment, and plasma polymerization methods [51]-[66].

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.

Magnetic nanoparticles can act as a new class of non-toxic and effective flame retardants. Fe_3O_4 enhanced both thermal stability and flame retardant properties of polyvinyl alcohol. Nanoparticles were synthesized by a simple precipitation reaction without using an inert atmosphere at room temperature [33]-[50]. The nanoparticles exhibited ferromagnetic behavior at room temperature. To obtain a magnetic nanocomposite, Fe_3O_4 nanoparticles were added to polyvinyl alcohol. Dispersed nanoparticles play the role of a magnetic barrier layer, which slows down the volatilization of the product and prevents the penetration of oxygen into the sample during polymer decomposition. However, these effects for polymer-nanoparticles composites often remain unclear and additional studies are needed for better elucidation of the phenomena observed.

The aim of this study was to synthesize the composite materials based on iron oxide nanoparticles and polyethylene and characterize their magnetic properties.

II. Synthesis and Study of the Properties of Nanocomposites

Iron oxide nanoparticles (II, III) (10 g) were synthesized by the reaction of coprecipitation of aqueous solutions of iron (II) and (III) chlorides in the presence of alkali in an argon atmosphere according to the following procedure: To a solution of 8.9 g (0.033 mol) $FeCl_3 \cdot 6H_2O$ and 2.1 g (0.0165 mol) $FeCl_2 \cdot 4H_2O$ (>97%, Aldrich), in water at40 °C and vigorous stirring, concentrated NH_4OH (25%) was added for 10-15 min., the reaction mixture was kept for 30 min. The formed black color precipitate was washed with water until neutral reaction, separated with a static magnet or centrifugation for 15 min, washed with additional ethyl alcohol and stored in dry benzene. A thermoplastic polymer was used as a matrix: Linear Low-Density Polyethylene (LLDPE).

For the production of films by the method of hot pressing from blanks obtained in an injection molding machine or a mechanochemical mixture, a manual electrically heated hydraulic press was used. The calculated amount of the composite material was loaded into a flat brass mold within the bounding frame of 100x100 mm with a thickness of 0,2 mm - for the manufacture of samples for measurements on the dielectric constant and physical and mechanical tests. Sample weight (m), in grams, was calculated by the formula: m = V (x * ρpe + y *ρme) *100%,

where V – shape volume, cm^3; x and y – fractions of sample components, %; ρ_{pe} and ρ_{me} - density of polymer and metal filler, g/cm^3.

To assess the interphase layer at the interface between the particles of iron oxide and a matrix containing linear low-density polyethylene, the above relation is used:

$$h(t) = K_0 \exp\left[-\frac{E \pm \kappa\sigma_{kk}}{2RT}\right]\sqrt{t}, \quad h(t) \equiv \delta(t) \quad (*)$$

where K_0 – this is a preexponential parameter determined experimentally, E – activation energy of the growth process of the reaction zone, R=8,314 – universal gas constant, T – process temperature on the Kelvin scale, σ_{kk} - ball tensor. The parameter k is determined according to the experimental data.

After determining the value k you can establish the dependence of the thickness of the interphase layer on the holding time.

In Figure 1, such a dependence is plotted:

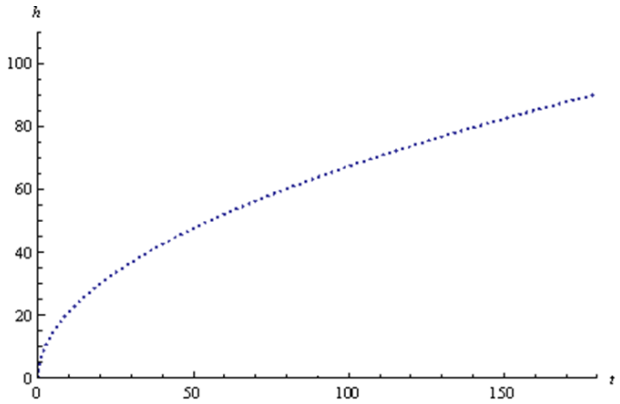

Fig. 1 Dependence of the interfacial layer thickness in nanometers.

Further the influence of pressure and temperature on the rate of formation of the interphase layer is investigated.

It was found that in the specified range of these characteristics, the growth of the interphase layer does not depend on them. This is demonstrated in Figure 2, which shows the dependences for the interfacial layer thicknesses when the parameter k is fixed:

The upper curve is plotted for temperature T=1020 K, and the lower one is for T=720 K.

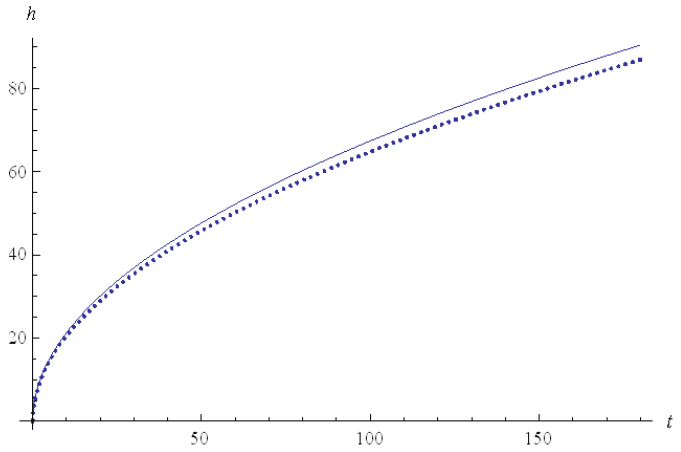

Fig. 2 Assessment of the effect of temperature on the thickness of the interfacial layer (in nanometers).

Let us find out whether the accuracy of determining the activation energy affects the estimates of the interfacial layer thickness. To do this, first the parameter k is determined again, according to the experimental test, and then the dependence of the layer thickness on the holding time is plotted.

After that, the time dependences for the interphase layer were constructed. The results are shown in the Figure 3:

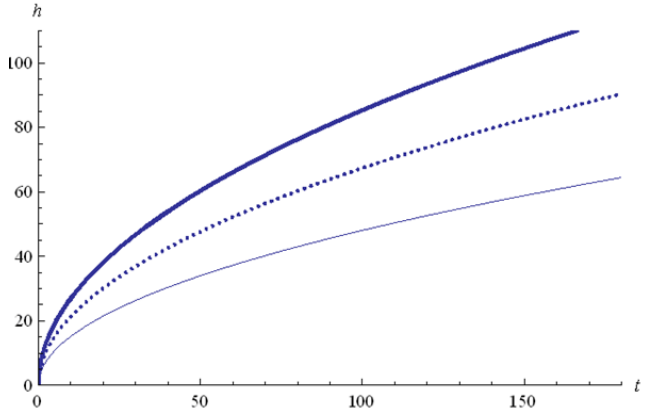

Fig. 3 Influence of the accuracy of determining the parameter K_0 on the prediction of the interfacial layer thickness.

It is important to note that the accuracy of the forecast is affected by the accuracy of determining the parameters in the formula (*). Therefore, it is very important to expand the experimental data, which can be used to determine (and refine) the parameters included in (*). Having these data and solving the problem of identifying the parameters of the model (*) by minimizing the target function (error of the theoretical dependence) in the rate selected after testing, we can significantly refine the forecast data for the thickness of the interphase layer.

According to some sources, the growth of the spinel interphase zone can begin only after the passage of the "incubation" period, which for temperatures amounts to 1000 K - 2000 s and for temperatures above 1000 K decreases to 500 s. Thus, the studied processing time is 3 min. may not be enough to start the growth of interfacial zones in the composite. Figure 4 shows the refined dependence of the thickness of the interfacial zone, depending on the temperature of the process and taking into account the incubation period. Here it was assumed that the incubation time decreases linearly with an increase in temperature from 970 to 1020 K from an initial value of 2000 s to a value of 500 s.

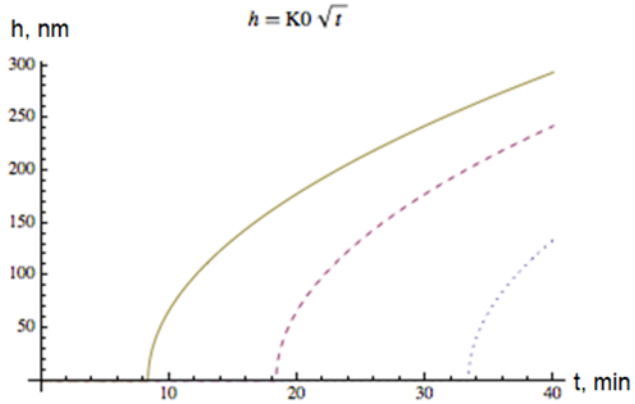

Fig. 4 Dependence of the thickness of the interphase zone on time, taking into account the incubation period (solid line T = 1020K, dashed line - T = 1000 K, dotted line - T = 970K).

Modeling shows that in a given range of temperature and pressure variation, an interfacial layer thickness of 60-120 nm can be realized. At the same time, neither temperature nor pressure (in the specified ranges) has a significant effect on the thickness. Optimum thickness can only be achieved by changing the holding time. Based on the preliminary calculations (which may require clarification during the experiments), it is possible to recommend carrying out the technological process for obtaining samples at a temperature of 750 K to ensure the shortest time of the incubation period of growth. The pressure should be minimal in the selected range - 30 atm., to increase the growth rate of the interphase zone. The process time should be 10-20 minutes to obtain an interface thickness of 80-150 nm. If the phenomenon of the incubation period of the beginning of the growth of the interfacial zone is not confirmed, the holding time should be 3-5 minutes.

III. Results and Discussion

The magnetic properties of nanocomposite samples (about 50 mg on average) were studied using a vibration magnetometer (VSM) M4500 EG & G PARC, which was calibrated using a standard pure nickel sample (90 mg) with a relative accuracy of 1 x 10^3, at room temperature. During the experiments, the magnetic field was varied from 0 to 10 kOe at room temperature, which made it possible to measure the saturation magnetization (Ms), remanent magnetization (Mr), and coercive force (Hc) for each sample.

The introduction of magnetite nanoparticles is reflected in the structural state of nanocomposites.

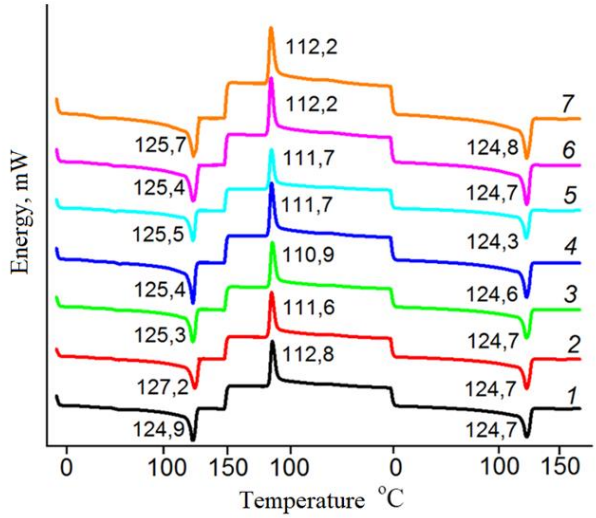

Fig. 5 DSC curves of LLDPE (1) and nanocomposites Fe3O4/LLDPE with filler concentration (wt%): 0.5 (2), 1 (3), 3 (4), 5 (5), 10 (6), 20 (7).

DSC data (Fig. 5) indicate a systematic decrease in the enthalpy of melting ΔH with an increase in the content of magnetite nanoparticles in the LLDPE polymer matrix, a similar character of the dependence is observed in the values of the degree of crystallinity of nanocomposites calculated by the formula:

$$\chi = H_m / H_{100} * 100\% ,$$

where Hm – heat released during the melting of the sample, H100 – heat released during melting of 100% crystalline polymer (for LLDPE H100 = 285 J/g). The decrease in the degree of crystallinity of the filled polyethylene in comparison with LLDPE (Table 1) is probably associated with the formation of a less ordered crystal structure.

Table 1. Temperature, heat of fusion and degree of crystallinity nanocomposite based on Fe_3O_4 / LLDPE.

Sample	Fe_3O_4 / LLDPE		
	tm, °C	ΔHpl, J/g	χ, %
LLDPE	124.7	119	42
0. PE	124.7	132	46
$1Fe_3O_4PE$	124.7	115	40
$3Fe_3O_4PE$	124.6	109	38
$5Fe_3O_4PE$	124.3	107	37
$10Fe_3O_4PE$	124.7	105	36
$20Fe_3O_4PE$	124.8	95	33

Magnetoactive polymer films obtained in the Fe_3O_4/LLDPE system. Magnetic characteristics (saturation magnetization (*Ms*), remanent magnetization (*Mr*) and coercive force (*Hc*) of the obtained nanocomposites are presented in Table 2.

Table 2. Magnetic properties of polymer nanocomposite materials.

Sample	Ms, emu/g	Mr, emu/g	Hc, Oe
$1Fe_3O_4PE$	0.32±0.04	0.025±0.001	44±3
$3Fe_3O_4PE$	0.15±0.03	0.007±0.0014	29±2
$10Fe_3O_4PE$	4.43±0.4	0.28±0.03	37±3
$20Fe_3O_4PE$	8.84±0.9	0.54±0.02	36±2

For comparison, note that at room temperature bulk magnetite is a soft magnetic material with a coercive force Hc ~ 200-400 Oe and a saturation magnetization of 92 emu/g. As can be seen from the data in the table, the magnetic properties are determined by the content of the filler, which can be clearly seen from the graphical data of the dependence of the magnetization on the strength of the applied magnetic field. The relatively small coercive force is due to the fact that the small size of nanoparticles (7-15 nm), which have superparamagnetic properties, make a significant contribution to the magnetic properties. It can also be seen that the saturation magnetization increases as the nanofiller is added.

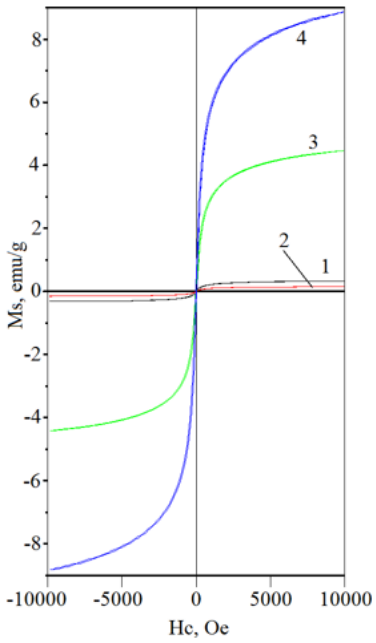

Fig. 6 Dependence of magnetization on the magnetic field, measured at 300 K, for nanocomposite films Fe_3O_4/LLDPE on the nanofiller content: 1 (*1*), 3 (*2*), 10 (3), 20 wt.% (*4*).

The character of the dependence of the magnetization on the magnitude of the magnetic field confirms the ferromagnetic character of the behavior of the obtained nanocomposites at room temperature. During the experiments, the magnetic field was varied from 0 to 10 kOe at room temperature, which made it possible to measure the saturation magnetization (Ms),

remanent magnetization (Mr), and coercive force (Hc) for each sample.

IV. Conclusion

Thus, the advantage of using matrix-stabilized magnetic nanoparticles to obtain polymer nanocomposites based on them is that such nanoparticles retain their dispersion and stability of size and shape in the technological modes of obtaining polymer nanocomposite materials, and thus stable ferro- and superparamagnetic properties of the obtained target products are provided. The resulting film nanocomposites exhibit ferromagnetic properties at room temperature. As can be seen from the data obtained, the magnetic properties are determined by the content of the filler, which can be clearly seen from the graphical data of the dependence of the magnetization on the strength of the applied magnetic field. The relatively small coercive force is due to the fact that the small size of nanoparticles (7-15 nm), which have superparamagnetic properties, make a significant contribution to the magnetic properties. It can also be seen that the saturation magnetization increases as the nanofiller is added.

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