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Investigation of the stability of polymer composites based on epoxy matrix and astralenes under exposure to high temperatures

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Introduction. The paper considers one of the ways to improve performance characteristics of products based on polymer composites with epoxy matrix by improving their thermal stability and durability by introducing modifiers.
Problem Statement. The objective of this study is to compare the heat resistance indicators of epoxy matrices of classical design with compositions improved by modification with carbon nanostructures.
Theoretical Part. For basic information, the selection of modifying materials, the selection of the optimal composition of the binder based on epoxy resin, low-molecular hardener, plasticizer and filler was carried out. The technology of introducing modifiers into the structure of the epoxy matrix was developed. Thermogravimetric and differential thermal studies were used to analyze changes in the temperature of the beginning and the end of the thermal effect, the temperature of the maximum thermal effect, the amplitude value and width of the peak effect, the index of its shape, and the mass loss of heated samples depending on their formulation.
Conclusion. The results of the study indicate the possibility of using epoxy resins filled with powdered carbon nanostructures in various areas of production due to the positive effect of additives on thermal stability indicators.

Keywords: epoxy resin, polymer composite material, thermal analysis, carbon nanostructures, astralen, fire safety, thermal stability.

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Introduction. In modern conditions, composite materials (composites) are considered one of the most promising and most popular materials. Their wide distribution is largely due to the ability to predict the required properties even at the development stage by modifying the composition and manufacturing technology of products. Polymer composite materials (PCM) occupy a separate niche of composites. In their structure, an organic polymer is used as at least one continuous phase. This can be, for example, polyester, epoxy, or organosilicon resin [1].

The structure of structural PCM includes a matrix (polymer binder) and one or more reinforcing fillers that give the material high strength. The composite components are selected in such a way that their mutual dissolution or absorption does not occur. Due to the specified adhesive bond between the matrix and the filler, some essential qualities of the PCM are provided, including solidity. You can change the adhesive properties by selecting and modifying the matrix composition.

One of the most common binders is epoxy. The basis of the epoxy matrix is epoxy resin (ER). The epoxy matrix of the composite is obtained by introducing functional additives into the resin: hardeners, thickeners, etc. ER is a liquid and viscous or solid transparent thermoplastic structure that has a shade from light to dark brown. Cured epoxy polymers are quite fragile matrices with low fire resistance, so various methods of their modification are widely used. When hardeners are added to epoxy resins, thermosetting materials are formed that have high adhesion and cohesion; good dielectric properties; increased mechanical strength, as well as resistance to water and many chemical compounds [2]. However, significant disadvantages that limit the use of such single-phase materials are low thermal stability and stability when exposed to elevated temperatures [3].

To increase the thermal stability, it is proposed to reinforce the epoxy matrix with astralenes (Astr), which are carbon nanostructures. The paper analyzes the formulation of the modified composition and the method of complex introduction of a low-molecular hardener and nanoparticles into the ER.

Due to the binding of the molecules of the components of epoxy matrices with carbon nanostructures and due to their antioxidant properties, the strength characteristics and thermal stability of materials are increased. This, in turn, leads to an increase in the energy required for the destruction of the structured sample, which makes it possible to slow down the destruction processes in the matrix. Obtaining experimental confirmation of increasing the thermal stability of PCM by introducing Astr will allow us to draw conclusions about the possibility of using this filler as a nanomodifier in practice.

Problem Statement. The purpose of this study is to compare the heat resistance of epoxy matrices with compositions improved by modification with carbon nanostructures. At the same time, it is necessary to evaluate the possibility of using epoxy resins in various areas of production due to the positive effect of additives on thermal stability indicators. It is necessary to select modifying materials, as well as the optimal composition of the binder based on epoxy resin, while it is necessary to develop the technology for introducing modifiers into the matrix structure. Based on these results, several criteria identified during the study should be analyzed.

Theoretical Part. The objects of the study were the characteristics of heat resistance of samples of a binder consisting of ED-20 epoxy resin [4], a hardener — triethylenetetramine (TETA) [5] and a plasticizer — diglycidyl ether of diethylene glycol DEG-1 [6], as well as samples of PCM based on this binder and powdered nanoscale fillers — astralens. According to [7], astralenes are polyhedral structures made of carbon atoms in the form of multi-layer particles with dimensions of 15-150 nm. The advantages of innovative Astr additives in comparison with nanoparticles of other elements are associated with high characteristics of conductivity, heat resistance, stability, and strength [8].

The mass concentration of the material components should be carefully selected taking into account the recommendations [9], since otherwise the performance characteristics may decrease, as well as the migration of molecules to the surface of the cured sample may be observed. Table 1 shows the formulation of materials selected according to the recommendations. Further preparation of samples of the modified material was carried out in several stages. It is known [10] that it is possible to ensure the quality of the fillers introduced into the matrix volume due to their dispersion in a low-molecular hardener using an ultrasonic bath followed by the addition of resin.

Table1

Formulation of the studied materials

Ingredients	Content by weight, %					
Epoxy resin ED-20	90	90	90	85	85	85
Hardener TETA	10	9	8	10	9	8
Pasticizer DEG-1	–	–	–	5	5	5
Filler Astr	–	1	2	–	1	2

At the first stage of the modified samples creation, the suspensions were prepared based on the hardener with the addition of carbon nanostructures and their subsequent dispersion at a temperature of 20.0±0.2 ºC and normal atmospheric pressure under the influence of an ultrasound source of 1 kW with a frequency of 60 kHz for 10 minutes. At the same time, homogeneous suspensions of epoxy resin with a plasticizer were prepared to obtain plasticized samples. Further, the modified hardener solutions were added to the pre-suspended resin to obtain compositions with element concentrations corresponding to table 1. After that, the mixture was stirred for 5 minutes until it became homogeneous. At the next stage, the compositions were cured at room temperature. Fig. 1 shows the obtained samples of the studied materials.

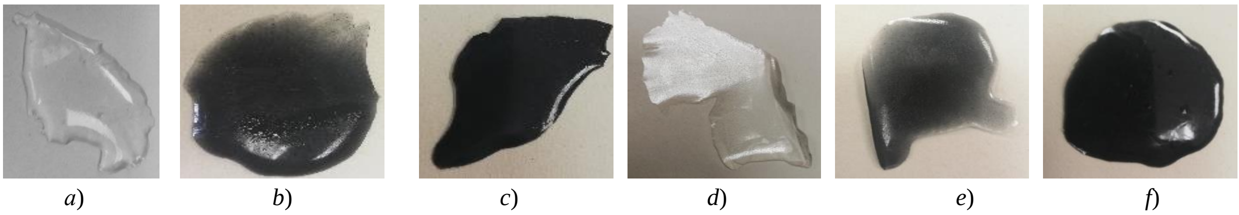

Fig. 1. Photos of samples with the formulations indicated in table 1:
a — without plasticizer and Astr; b — without plasticizer and with 1 % of Astr;
c — without plasticizer and with 2 % of Astr; d — with plasticizer and without Astr; e — with plasticizer and 1% of Astr;
f — with plasticizer and 2 % of Astr

To evaluate the behavior of the materials under study under thermal influence, their thermal effect indicators were determined at the "Termoscan-2" installation. The method of thermal analysis [11] is used, based on the study of the properties of substances and the processes occurring in them when the temperature changes. Two variants of this analysis are traditionally used: thermogravimetric (TGA) and differential thermal (DTA).

The essence of differential thermal analysis is to register the thermal effects that accompany physical transformations and chemical reactions under the influence of high temperatures [12]. The method is based on changing the fundamental features of substances related to their chemical composition and crystal structure.

The analysis of the heating process of the material under study on thermograms of differential thermal analysis, called DTG curves (Fig. 1, 2), indicates the presence of several peaks corresponding to the thermal effect (TE) of the components of materials with the formulation corresponding to table 1. All samples have two characteristic peaks indicating intensive oxidation followed by dissociation of Triethylenetetramine — the first (low-temperature) peak and epichlorohydrin — the second (high-temperature) peak.

The main indicators of TE in differential thermal analysis are:

— start temperature — the temperature at which intensive oxidation of the substance begins;

— maximum temperature — the temperature at which self-ignition occurs, and the oxidation process is replaced by dissociation processes — chemical and physical decomposition of the oxidized component;

— end temperature — the end temperature of the component dissociation process;

— amplitude value — temperature change (Delta) from the beginning of the TE to its maximum value;

— peak width — the temperature range during which the TE was observed;

— shape index — describes the ratio of the duration of dissociation and oxidation processes.

Table 2 shows the values of indicators describing the processes of burnout of the components of the studied samples without a plasticizer.

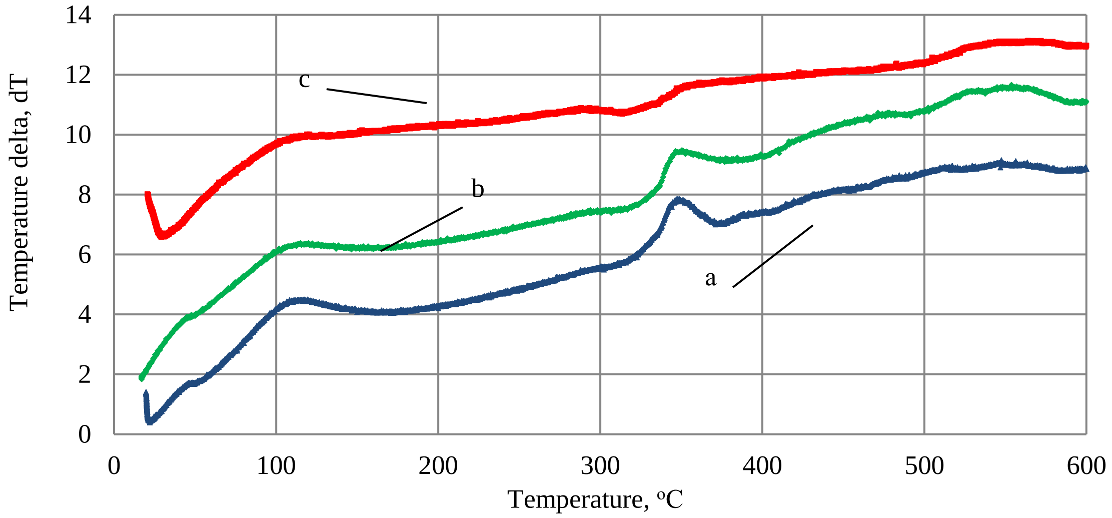

Fig. 2. Differential-thermal curves of materials without plasticizer:
a — without Astr; b — with 1% of Astr; c — with 2% of Astr

Table 2

Parameters of differential-thermal curves of materials without plasticizer [1]

Temperature indicators, °C, and TE shape index	Parameter values for material formulations					
	without Astr		with 1% of Astr		with 2% of Astr	
	Peak 1	Peak 2	Peak 1	Peak 2	Peak 1	Peak 2
Start temperature	51	315	48	313	not observed	317
Maximum temperature	115	351	118	353		562
End temperature	170	375	151	377		*
Amplitude value	2.78	2.2	2.37	1.88		2.33
Peak width	119	60	103	64		>317
Shape index	0.86	0.67	0.47	0.6		-

[1] Note — no exothermic reactions are observed before 600 °C

The comparison of the indicators of thermal analysis of materials without plasticizer allows us to draw the following conclusions:

1. For a sample with an Astr filler at a concentration of 2 %, the 1st TE peak is not observed during heating, indicating intensive destruction of the hardener components. The beginning of the peak of the other component begins at a temperature similar to the temperature of the beginning of the TE of the sample without fillers, but the ignition temperature shifts by about 200 °C, and when the sample is further heated to 600 °C, exothermic reactions do not stop.

2. The rate of oxidation of the modified sample is lower than the rate of volatilization of thermal decomposition products, which indicates an increase in the thermal stability of the material by increasing the oxidation time from the beginning to its transition to the self-ignition process.

3. Peak value of the TE material with the addition of 1 % of Astr is lower than the values for the sample without filler (control sample), indicating the decrease of intensity of the exothermic reactions and is also the proof of the increasing thermal stability of the modified composition compared with the unmodified one.

4. The presence of bright peaks with a gentle deviation of the descending branch on the thermogram indicates the effects of crystal lattice destruction occurring in the structure, which, however, is not observed for a sample with a 2% of Astr content.

The materials with the addition of a plasticizer behave somewhat differently (fig. 3). The results of these studies are shown in figure 3 and table 3.

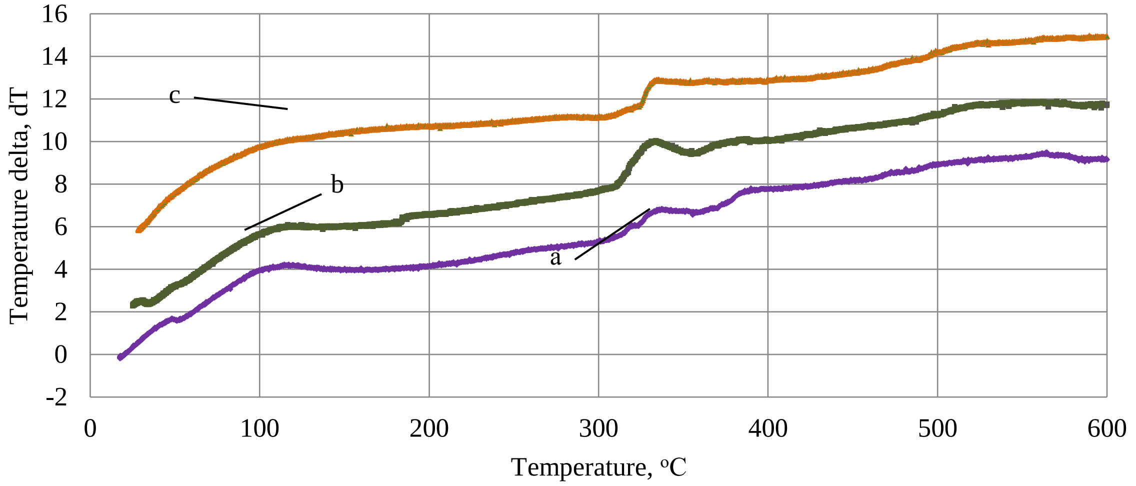

Fig. 3. Differential-thermal curves of materials with plasticizer: a — without Astr;
b — with 1 % of Astr; c — with 2 % of Astr

Table 3

Parameters of the differential-thermal curves of the materials with plasticizer

Temperature indicators, °C, and TE shape index	Parameter values for material formulations					
	without Astr		with 1 % of Astr		with 2 % ofnAstr	
	Peak1	Peak 2	Peak 1	Peak 2	Peak 1	Peak 2
Start temperature	50	305	56	311	not observed	*
Maximum temperature	111	336	116	334		
End temperature	136	355	138	357		
Amplitude value	2.48	1.41	2.73	2.08		
Peak width	86	50	82	46		
Shape index	0.41	0.61	0.37	1.00		

* — there is only the beginning of the thermal effect at 321 °C with a slight inflection when the temperature reaches 332 °C..

When comparing the results of the analysis of the control sample and the sample with the addition of plasticizer, a decrease in all 6 indicators of DTA, indicating the increase of flammability, increasing the rate of oxidation and burn-out material, and, as a consequence, the decrease in the combustion stability when included in the resin composition of the plasticizer on the basis of diglycidylether ether of diethylene glycol. This phenomenon is primarily due to the dilution of epichlorohydrin, the self-ignition temperature of which exceeds 400 °C, with more combustible diethylene glycol with a self-ignition temperature of 210 °C [13]. This problem can be solved by using plasticizing compounds with higher thermal protection properties [14]. The comparison of the DTA indicators of materials with the plasticizer allows us to draw the following conclusions:

1. For a plasticized material sample with 2% of Astr, the TE peak is not observed, as in the case of non-plasticized material.

2. There is a significant shift and smoothing of the second characteristic peak of the TE. So, for it, only the beginning of the thermal effect is observed at 321 °C with a slight inflection when the temperature reaches 332 °C. The temperature of the maximum TE, as well as the temperature of its end, is observed when heated above 600 °C. This flat shape and elongation of exothermic effects is characteristic of slow-moving oxidation processes, i.e. the formation of a coke layer with subsequent burning and volatilization of coke.

3. There were no significant changes for the better affecting the flammability of the material with the plasticizer when adding 1% of Astr.

The data obtained during the DTA indicate a significant improvement in the thermal stability of the epoxy resin, in the structure of which there are Astr nanoparticles of 2% by weight. This effect is observed due to the antioxidant properties of modifiers, which inhibit the destructive processes of oxidation by increasing the energy required for the destruction of the sample structured by nanomodifiers.

Study of samples thermolysis by thermogravimetric analysis method. Thermogravimetric (thermal weight) analysis is based on obtaining and studying the regularities of changes in the weight of a substance during heating [15]. Thermogravimetric (TG) curve shows the change in the absolute or relative mass of a substance from the beginning to the end of heating. It is also called a normal or integral thermal weight curve.

The processes occurring in a substance during heating can be interpreted most fully only in a combination of TGA and DTA data by obtaining the corresponding curves. For example, physical and chemical reactions of dissociation and dehydration, such as evaporation of moisture, volatilization of carbon dioxide and other volatile components are accompanied by mass loss. The reverse process is an increase in mass; it is caused by oxidation and the formation of a carbonized carbon layer on the surface of products. During heating, a number of oxidation and carbonation transitions in the dissociation reaction can occur in the sample. Also, there may be several peaks on the TG curves, which indicate the destruction of the moisture contained in the sample, the surface fat layer, unpolymerized molecules, and other impurities in the material.

The main characteristics of the TG curve are the decomposition start temperature and the reaction end temperature corresponding to the maximum mass change. The pronounced peaks of the TG curves mean acceleration of thermal decomposition processes, and, as a result, an increase in the rate of mass loss. In practice, we can trace the relationship between TG curves and DTG curves due to the fact that strong TE leads to the accelerated growth of the carbon layer with its further intensive decomposition (fig. 4-9).

When analyzing the process of mass loss of samples of the materials under consideration, the following is observed:

1. There is a gradual oxidation of the control sample material when heated up to 375 °C, but when this temperature is reached, equal to the temperature of the end of the TE, the continuous destruction of the sample occurs.

2. The loss of mass of the plasticized sample without adding fillers has several differences. First, the curve is more uniform, which indicates a stable rhythm of the processes of mass change. Secondly, at 180 °C, there is a transition from mass build-up to its volatilization. On the DTG curves at this temperature, a smooth increase in TE is observed. A slight loss of mass during this process, observed on the TG curve, allows us to judge the volatilization of the burnt surface layer, which is accompanied by a decrease in the thermal insulation capacity of the sample surface undergoing destruction. The end of thermal decomposition of the material is observed at 355 °C, which correlates with the temperature of the end of the TE obtained by analyzing the DTG curve.

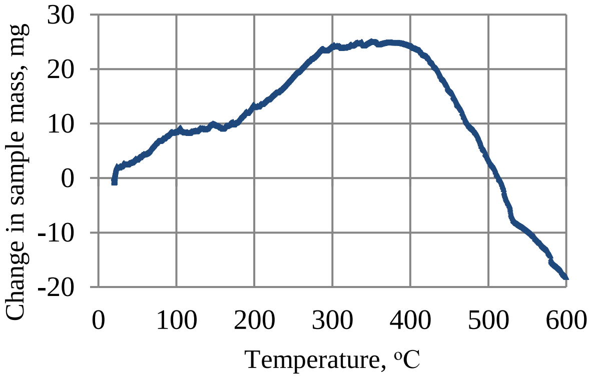

Fig. 4. Thermogravimetric curve of the control sample

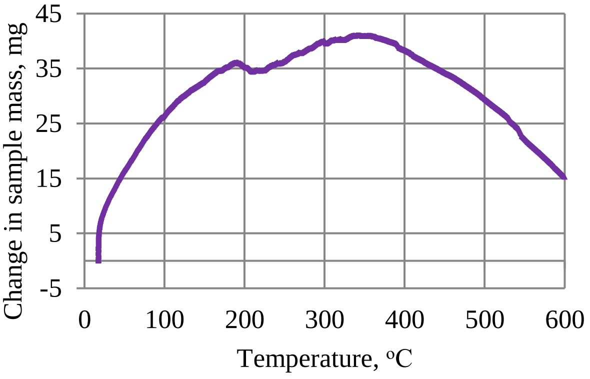

Fig. 5. Thermogravimetric curve of the sample without Astr and with plasticizer

3. For a nanofilled sample with 1 % of Astr, the first transition from mass gain to mass loss is observed at 237 °C, but in the temperature range of 289-332 °C, the change in the mass of the sample is not observed, or is insignificant. When 332 °C is reached, the process of material destruction begins. However, comparing the TG and DTG curves, it is possible to notice that the mass loss of the sample begins before the maximum TE is reached. This is also due to the loss of the heat-insulating layer due to the volatilization of compounds that have been oxidized during heating.

4. The mass gain of a nanofilled plasticized sample containing 1% of Astr occurs when heated above 50 °C up to a temperature of 600 °C. Otherwise, the TG curve is close to a linear one with a slight change in the angle of inclination, corresponding to a change in the rate of mass gain, when heated to temperatures of 180, 354 and 442 °C. In this case, the temperature at which the maximum deviation from the previously recorded angle of inclination is observed coincides with the temperature of the end of the TE. This indicates an increase in the duration of volatilization and the decrease in the intensity of dissociation.

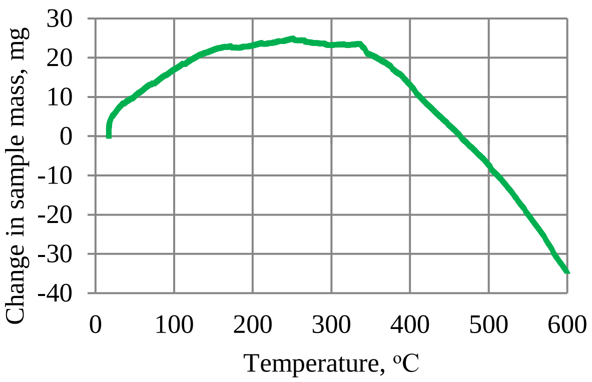

Fig. 6. Thermogravimetric curve of the sample with 1% of Astr

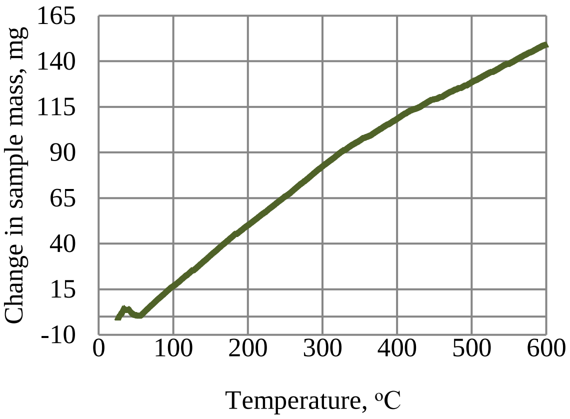

Fig. 7. Thermogravimetric curve of the sample with thea plasticizer and 1% of Astr

5. A sample with 2 % of Astr is characterized by the presence of small peaks corresponding to short dissociation sections with a heating of no more than 10-15°C. The beginning of such sections is observed at temperatures of 56, 140, 281, 305, 458 and 545°C. In addition, the heating of the sample is not accompanied by the end of the destruction due to the fact that the change in mass is in the nature of an increase. These observations indicate that the thermal decomposition of the sample does not occur when heated to 600 °C. A large number of small peaks indicates an uneven loss of volatile components, and the short duration of their dissociation indicates the ability of the material to build up a heat-insulating carbon layer.

6. The plasticized material containing 2% of Astr also shows only the process of mass gain and confirms the absence of TE when the sample is heated up to 600 °C. Moreover, when heated above 245 °C, the increase in mass takes a linear character, that is, it becomes as stable as possible.

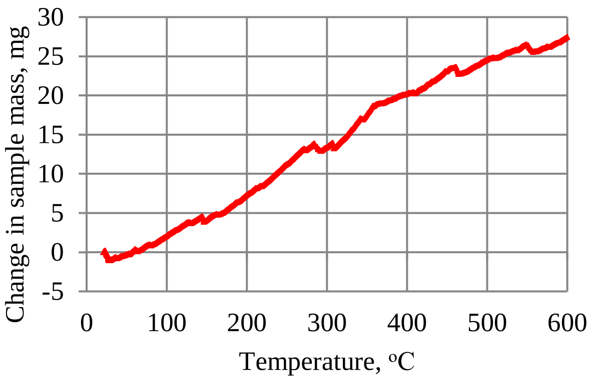

Fig. 8. Thermogravimetric curve of the sample with 2% of Astr

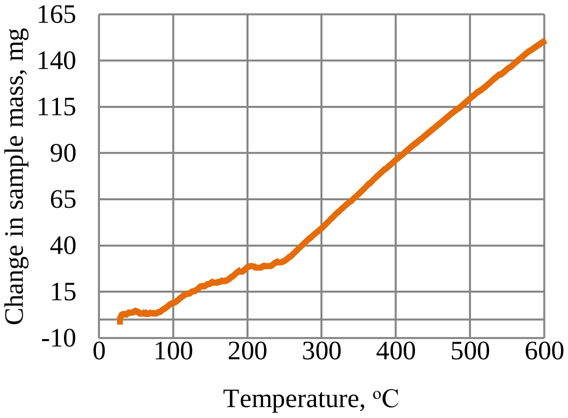

Fig. 9. Thermogravimetric curve of the sample with a plasticizer and 2% of Astr

Thus, it can be observed that for samples that include a plasticizing agent, the TG curves are smoothed, which indicates the stabilization of the heating process. Samples containing 2% of Astr nanoparticles are characterized by the absence of thermal decomposition, which is accompanied by the mass loss. This fact is also associated with the build-up of the carbonized layer on the sample surface. This makes it possible to strengthen the thermal insulation of the material and limit the access of the oxidizer, which is an accompanying protective mechanism during the combustion of polymers. It is also worth noting that the nature of the mass loss of the plasticized samples is close to linear. This is primarily due to the fact that the plasticizer reduces the probability of cracking and reduces the number of defects in the sample volume, which also has a positive effect on the material's resistance to high temperatures.

Conclusion.

1. Due to the strength characteristics and reinforcing properties of Astr, which make it possible to cross-link structural defects of the modified material, for samples with 2% of Astr, there are no peaks of thermal effect on the DTG curves, that is, when the material is heated up to 600 °C, the crystal lattice is not destroyed.

2. When the Astr modifier with the pronounced antioxidant properties is introduced into the material structure, the oxidation process is inhibited. The increase in the duration of oxidation contributes to the formation of a carbonized layer that contributes to thermal protection and insulation.

3. Due to their small size, Astr particles have a large specific surface area, which determines their high sorption properties. This reduces the amplitude value of the thermal effect and, consequently, the intensity of exothermic reactions for samples containing 1 % of Astr.

4. The addition of Astr with the plasticizer makes it possible to achieve a uniform, close to linear change in the mass of the sample, which indicates the stable nature of heating and oxidation processes. This also contributes to the stable formation of a heat-protective and insulating carbonized layer.

5. A large number of small peaks of mass change on the TG curves of the sample without a plasticizer indicate uneven dissociation of components, which also contributes to the build-up of the carbon layer with regular updating.

The results obtained make it possible to specify and supplement theoretical data on the behavior of epoxy materials modified with carbon nanostructures at elevated temperatures, and also to demonstrate the positive effect of modification on thermal characteristics of the PCM of the proposed composition. The synergistic properties of the modifiers lead to an increase in the thermal stability of the material compared to the base polymer composition. The use of modified material will improve the performance of heat resistance and strength of composite products under the influence of elevated temperatures during a fire.

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Contribution of the authors:

V. A. Borisova — formulation of the basic concepts, goals and objectives of the study, scientific supervision, calculations, formulation of conclusions; A. A. Egorov — analysis of research results, preparation and finalization of the text, correction of conclusions.