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RESEARCH INTO PARAMETERS OF MAGNETIC TREATMENT TO MODIFY THE DISPERSE-FILLED EPOXY COMPOSITE MATERIALS

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Покращення експлуатаційних властивостей епоксидних композитів дозволяє розширити сферу застосування. Одним із способів підвищення міцності та довговічності епоксидних композитів є застосування магнітного оброблення.

Для модифікування полімерних композицій під впливом магнітного поля необхідно дотримуватись оптимальних режимів обробки та температурно-часових умов, визначення яких є метою досліджень.

В результаті проведення експериментальних досліджень встановлено вплив параметрів змінного магнітного поля на фізико-механічні характеристики модифікованих епоксикомпозитів. Досліджено закономірності впливу коефіцієнтів пропорційності, інтегрування та диференціювання алгоритму регулятора магнітного оброблення на ударну в'язкість та теплостійкість модифікованих епоксикомпозитів. Визначено оптимальні значення частоти змінного магнітного поля при обробці епоксикомпозитів. Досліджено залежності вмісту дрібнодисперсного феромагнітного наповнювача на залишкові напруження та температуру в зоні магнітного оброблення. При дослідженнях епоксидних композитів паралельно проводили випробування властивостей від впливу температури аналогічній при магнітному обробленні. Закон зміни температурних параметрів без магнітного оброблення задавали таким же, як і при магнітному обробленні. Наведено результати математичного планування експерименту та кореляційні залежності індукції магнітного поля та вмісту наповнювача на теплостійкість модифікованого епоксикомпозитного матеріалу. В результаті досліджень визначено граничні значення вмісту наповнювача та режимів обробки епоксикомпозитних матеріалів, що забезпечить підвищення експлуатаційних властивостей. Проведені дослідження важливі для вдосконалення технологічного процесу модифікування епоксидних композицій магнітним обробленням

Ключові слова: магнітна обробка, теплопровідність епоксидний олігомер, поліетиленполіамін, залишкові напруження, ударна в'язкість, індукція, ПІД-регулятор

1. Introduction

The development of modern industry implies the reduction of metal consumption in the production of mechanisms and machines. Resolving this task is possible when applying polymeric composites. The most effective are the composites based on epoxy dianeic resins. Such material is used in the form of coatings, including the surfaces with a complex profile, to protect metallic articles against corrosion and the influence of aggressive environments. In addition, epoxy composites are applied as structural materials for parts of mechanisms and machines. However, the strength indicators of polymeric composites that are offered from industrial production do not always match the set of operational requirements. Improving the strength indicators of epoxy-composite materials and coatings is a relevant task of modern materials science. In this field of research, it is interesting from a scientific and practical point of view to modify the epoxy-composite materials using force fields as the methods to improve operational characteristics. It should be noted that the effect of modification using the force fields on the strength indicators of epoxy composites has not been investigated sufficiently. Specifically, it relates to treating with magnetic fields. Particularly, the application of treatment with a variable magnetic field at solidifying epoxy compositions can improve strength indicators of the formed polymeric material at the expense of changing the degree of cross-linking in the material of a binder [1].

2. Literature review and problem statement

It should be noted that the modification of epoxy compositions using the magnetic treatment leads to a number of difficulties:

– selection of optimal magnetic treatment regimes. Study into duration of the treatment, the intensity of a magnetic field, temperature in the treatment zone [2];

– establishment of the optimal content of components in an epoxy composition (binder, hardener, filler) that are exposed to the action of a magnetic field [3].

In this case, selecting the optimal dispersion of a filler has an important role that is associated with a specific surface area of the filler's particles [4]. The specified factor affects the volume of outer surface layers (OSL) when forming an epoxy composite. A material with the OSL structure is different from the base material of a binder in its structure. A significant effect on the volume of OSL formation is exerted by the nature and activity of the filler relative to the components of the composition [5]. It is possible to intensify the process of structuring at the interface of phases "binder–filler" when using magnetic fields [6].

Given the complexity of studying the entire range of a filler, a hardener, as well as the concentration and modes of magnetic treatment, the preliminary stage implied mathematical modeling of results of experiments. It should be noted that at certain modes of magnetic treatment, when applying the ferromagnetic fillers, there are phenomena when a jump-like rise in temperature in the treatment zone was observed. In this case, when changing the content of a filler, they observed both a significant improvement of the physical- mechanical characteristics of a material and a change in the temperature conditions in the treatment environment. This relates to the fact that in this case the treated material with the ferrite filler is the electromagnet core, and the value of the filler's content affects the induction magnitude of the magnetic field. Theoretically, while applying a method of mathematical planning of the experiment, it is difficult to consider it. Therefore, experimental studies into the influence of optimal ratios of a filler and treatment modes must continue.

Treatment using a magnetic field is applied to polymeric composites of various magnetic nature [7]. Formation of a composite under the influence of a magnetic field remains insufficiently investigated. When applying such a type of treatment, there are problems associated with the difficulty, and often the impossibility, to form the shape of parts with surfaces that have a complex profile. This relates to the fact that it is necessary for different shapes of parts to fabricate a separate solenoid for magnetic treatment so that the influence of the magnetic field on a material remains the same throughout the entire part's volume.

At the same time, the formation of a polymeric composite material under the influence of a magnetic field typically employs a continuous mode of treatment with a certain specified duration [8, 9]. In this case, it remains to examine the influence of a magnetic field with a controlled intermittent intensity that could be derived based on the proportional-integral-differential (PID) algorithm.

An analysis of the scientific literature revealed [2–11] that most research in the field of magnetic treatment of polymeric composites addressed rendering or changing the dielectric and magnetic properties of a material. Changing the strength indicators of a material in the formation of articles in an alternating magnetic field remains insufficiently explored.

3. The aim and objectives of the study

The aim of this work is to establish the effect of the content of a ferromagnetic filler in an epoxy material and the modes of its magnetic treatment on the heat resistance, impact strength, and residual stresses of composites for materials for electrical purposes. That would make it possible to improve the technological process of magnetic treatment and enhance operational properties of articles.

To accomplish the aim, the following tasks have been set:

– to determine the optimal frequency of an alternating magnetic field when treating an epoxy composition in order to form a material with enhanced physical-mechanical characteristics;

– to establish the influence of coefficient of proportionality, integration and differentiation of the PID-algorithm for magnetic treatment on the physical-mechanical characteristics of an epoxy-composite material;

– to explore the effect of the content of a ferromagnetic filler and temperature in the zone of magnetic treatment on residual stresses of the modified epoxy-composite material.

4. Materials and methods of research

It is proven that the effect of magnetic field on the characteristics of composite materials (CM) strengthens with the use of ferromagnetic fillers owing to the larger value of magnetic perceptibility of environment [12]. The most effective for the magnetic treatment of epoxy compositions, and affordable at that, is to use the filler of ferrite grade 1500 NMZ that has high ferromagnetic properties. It was established at the previous stage that the optimal content of such a filler is q=30–40 mass share and the optimal dispersion is D=5–0 μm. We used as the epoxy matrix the epoxy dianeic oligomer ED-20 (GOST 1087-84), which is characterized by high specific indicators of strength, low shrinkage, high adhesive and cohesive strength. The macro molecules of this oligomer represent domains that are capable of orienting due to the effect of electromagnetic fields along its force lines. The magnetic field intensity must be sufficient to overcome the resistance of orientation, induced by the viscosity of the material, which constantly increases until the material is completely solidified. To cross link the binder, we applied the low-temperature hardener polyethylene polyamine (TU 6-05-241-202-78). Significant advantages of choosing the specified hardener is the possibility of forming a material at ambient temperatures, technological affordability, and the possibility of application onto the long-dimensional surfaces with a complex profile.

To determine the change in strength indicators of the modified composite material, we have chosen the impact strength. Tests of the samples were conducted in 24 hours after treatment with the magnetic field. This is explained by the need to stabilize the structural transformations in the material after its shrinkage as a result of hardening. Impact strength by Sharpe was determined according to GOST 4647-80. The value of residual stresses was defined using the console method. The heat resistance (by Martens) of epoxy-composite materials was determined according to GOST 21341-75. To mathematically plan the experiment, we employed the software STATISTICA 6.0.

Magnetic treatment was performed at a specially designed device [13]. The device includes a solenoid that generates an oscillating magnetic field. The examined composition was placed in the solenoid. To maintain the optimum temperature conditions in the treatment zone, we used a cooler and a microcontroller with a temperature sensor and the PID-controller. Given the nonlinearity and non-stationarity of the system, the values for the coefficients of proportionality, integration and differentiation of the PID-controller were determined experimentally. Temperature measurement was carried out using a thermocouple, positioned beyond an electrical magnet, in the region of air discharge from the cooling system. By adjusting the voltage in the solenoid winding, we purposefully changed the intensity of the magnetic field within the experimentally determined optimal range. At an excessive increase in temperature, a PWM regulator in the device decreased the value of voltage in the solenoid winding and thus lowered the intensity of the magnetic field. The optimal treatment modes that were established experimentally at the previous stages of research are: induction V=0.8...2 Tl, duration of the treatment τ=3 hours.

It should be noted that the principal technological objective was to maintain a stable temperature (within the operating temperature of the hardener) in the area of magnetic treatment. The introduction of an epoxy composition with a ferromagnetic filler leads to an increase in the value of magnetic induction in the device's electrical magnet. Eddy currents carry out additional heating of the composition. In addition, the hardening reaction of an epoxy polymer is, as a result of the introduction of the hardener, is exothermic, and varies over time. Without the use of treatment with force, the temperature at solidification smoothly increases initially and gradually reduces until the composite material is fully solidified.

5. Results of studying the physical-mechanical characteristics of epoxy composites modified by magnetic treatment

It was experimentally proven that the solidification of composites in an alternating magnetic field leads to the additional heating of compositions in the course of treatment, which enables a faster process of material cross-linking. A solidification that is accompanied by the exothermic effect leads to the increased values of residual stresses [14]. To study the influence of the temperature component on residual stresses in composite materials, the samples were made without treating them with force fields. The temperature-time law complied with the law of change in these characteristics when modifying with an alternating magnetic field. We investigated experimentally (Fig. 1) the dependences of residual stresses σ_3 of composite materials on temperature of the environment T, K in the region of cross-linking and concentration q of the ferromagnetic filler. The residual stresses in composite materials formed in an alternating magnetic field using the para- and diamagnetic fillers were not examined. Previous studies found that the use of such fillers does not significantly affect a change in the temperature of a composition in the process of cross-linking [15].

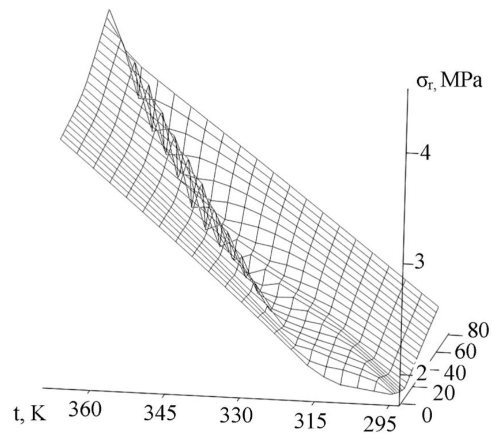

Fig. 1. Dependence of residual stresses σ_r in epoxy-composite materials on temperature T and content of filler q

It was established that an increase in temperature in the zone of treatment with an alternating magnetic field contributes to the growth of the absolute magnitude of residual stresses σ_r in the obtained material. Thus, for a non-modified epoxy matrix under normal conditions (t=295±2 K), the absolute magnitude of residual stresses is σ_r=1.8 MPa, and increases depending on the content of a ferromagnetic filler, σ_r=2.2 MPa at 80 mass shares. It was proven that cross-linking the compositions at additional heating reduces the time of composite hardening and leads to the growth in the absolute magnitude of residual stresses to σ_r=4.2 MPa. In this case (Fig. 1), σ_r grows much larger in the plane $\sigma_r=\varphi(t)$ rather than $\sigma_r=\varphi(q)$. Thus, it was proven that temperature exerts a more significant influence to the growth in values of σ_r. And in all cases, when the temperature increased above t>360 K, we observed destruction processes with the emergence of pores (Fig. 2).

When carrying out experimental research into the values of residual stresses, it was found that changing the content of a ferromagnetic filler changes the modes of treatment because the result is the changed magnetic perceptibility of the environment. Therefore, the next stage involved a mathematical modeling of the experiment in order to determine the influence of the entire range of values for the content of a filler and magnetic induction on the CM physical-mechanical characteristics. The chosen characteristic for an epoxy composite was heat resistance. In a correlation analysis, the independent variables were represented by the value of the frequency logarithm (ln ν), induction of the magnetic field (V, Tl) and the content of a ferromagnetic filler (*q*, mass share). Results of studying the correlation dependence are shown in Fig. 3.

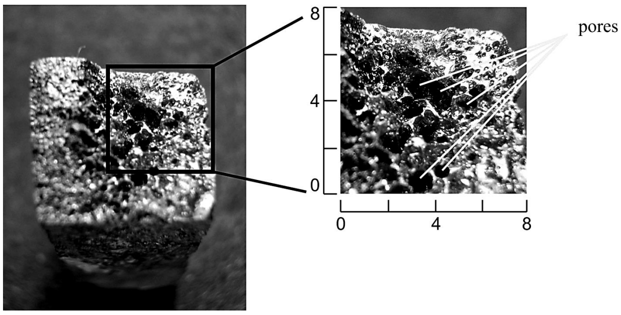

Fig. 2. Porous defects in a material

Variable	The marked correlations are significant at level p < 0.5000, N=180			
	Ln V	V, Tl	q, mass sh.	T, K
Ln V	1.0	-1.0	-0.00	0.27
V, Tl	-1.0	1.0	-0.00	-0.27
q, mass share	-0.0	-0.0	1.00	-0.03
T, K	0.3	-0.3	-0.03	1.00

Fig. 3. Correlation analysis of influence of independent variables on heat resistance

It was established experimentally that the heat resistance of the examined CM correlates with the frequency of the magnetic field (ν, kHz), since 0.25</*r*/≤0.75, and is 0.27. With an increase in the frequency of treatment from ν=0.02 kHz to ν=0.2 kHz, the heat resistance of the examined composites also increases from *T*=380 K to *T*=406 K. The content of a ferromagnetic dispersed filler does not significantly affect heat resistance, the correlation in this case is 0.25</*r*/ and is –0.03. When increasing the magnetic induction, we observed a decrease in heat resistance. It should be noted that the impact strength and bending resistance increase with an increase in the induction of the treated field. This can be explained by the fact that at the higher values of frequency of the alternating magnetic field the ferromagnetic particles in a filler interact more actively with an alternating magnetic field. Optical microscopy has proven that with an increase in the frequency of the field, particles of the filler are denser arranged in a CM. There is also an increase in the CM density.

Thus, it was established experimentally that increasing the frequency of an alternating magnetic field from ν=20 Hz to ν=10 kHz ensures a growth in heat resistance by Δ*T*=10 K. The presence of an alternating magnetic field leads to the activation of domains of macromolecules, which in turn alters conditions for cross-linking, and in the presence of dispersed particles of the filler affects in a greater extent the modulus of elasticity, and increases the creep of a polymer. Changing these characteristics leads to the increased heat resistance. With an increase in the content of a filler from *q*=20 mass shares to *q*=50 mass shares the heat resistance grows by 30–40 K compared with an unfilled composite, and at the content exceeding 50 mass shares – by 20–30 K. It was proven that at such a content of dispersed particles in the volume of a polymer, the material is formed with the insufficient particle wetting by oligomer, which leads to a decrease in the physical-mechanical characteristics, including heat resistance.

The result of processing the data obtained in the course of research is the polynomial regression (Fig. 4) and the derived regression equation.

N=180	Summary of regression for a dependent variable: T, K R=0,88289283 R2=0,77949974 Correct. R2=0,77052589 F(7,172)=86,863 p<0,0000 Estimation standard error:3,4333					
	BETA	St. Err. BETA	B	St. Err. B	t(172)	p-level
Free Term			391.2936	0.799949	489.1480	0.000000
LN V	0.0852	0.143029	0.4308	0.722850	0.5960	0.551954
q, mass share	6.2079	0.288036	0.9702	0.045017	21.5526	0.000000
V2**2	0.2981	0.210577	1.1357	0.802193	1.4157	0.158661
V2**3	0.5511	0.224219	0.6954	0.282914	2.4579	0.014967
V2**4	-0.8050	0.292717	-0.5486	0.199481	-2.7502	0.006593
V4**2	-14.6716	0.738413	-0.0151	0.000760	-19.8691	0.000000
V4**3	8.7172	0.486361	0.0001	0.000003	17.9234	0.000000

Fig. 4. Results of the polynomial regression of experimental data

The regression equation takes the form:

$$a(\ln \nu, q) = 391{,}29 + 0{,}97 \cdot q +$$
$$+0{,}69 \cdot \ln \nu^3 - 0{,}55 \cdot \ln \nu^4 - 0{,}01 \cdot q^2. \quad (1)$$

The resulting equation makes it possible to calculate the value of heat resistance at a predefined frequency of the magnetic field and the content of a ferromagnetic dispersed filler. An analysis of residues revealed that an error in the regression model when calculating the impact strength does not exceed 2.23 %.

Thus, it was experimentally proven that the dependence of heat resistance on the content of a ferromagnetic filler and frequency of an alternating magnetic field is extreme in character. The optimum content of disperse particles in a filler is *q*=30...40 mass shares, at a frequency of the alternating magnetic field of ν=1...10 kHz, which ensures the value of heat resistance *T*=415 K. An increase in the specified factors in the process of cross-linking the composite leads to the heating of the composition, which, as noted above, compromises the heat resistance. It was established that decreasing the content of disperse particles in a filler to *q*<30 mass shares and the frequency of an alternating magnetic field to ν<1 kHz does not lead to an increase in heat resistance.

6. Discussion of results of studying the physical-mechanical characteristics of the modified epoxy composites

It is established that the largest effect of magnetic treatment can be achieved by forming an epoxy composite containing a ferromagnetic filler. The mechanism of the influence of the magnetic field is as follows: sufficient induction enables the orientation along the force lines of the magnetic field of a polymer's macromolecules that constitute the domains. In addition, there occurs the uniform arrangement of the ferromagnetic particles of a filler in a material. In this case, a material acquires the anisotropy of properties.

Temperature of the composition during treatment must not exceed *T*=333–335 K. In order to ensure such treatment conditions, we applied the PID control algorithm. Coefficients for the PID-algorithm were selected experimentally.

It was established that for the content of a ferromagnetic filler *q*=30 mass shares, the optimal value of the coefficient of proportionality is K_p=3. When increasing the content of a filler above q>30 mass shares, the value for the coefficient of proportionality should be reduced to K_p=2. When increasing the coefficient of proportionality K_p>3, we observed a short-term temperature increase in the treatment zone, above T>353 K. In this case, the resulting material was characterized by the elevated values of residual stresses σ=3.7±0.1 MPa. This can be explained by the fact that at the elevated values for K_p, the PID-controller ensures a rapid increase in voltage in the electromagnet winding and reacts too late on the temperature rise. In such a material, we observed the presence of pores. It was established that at small values K_p<2 the resulting composite material is characterized by the lower values of impact strength a_n=9.5–10 kJ/m^2. This is due to the excessive time required for the device to reach operating voltage. In the treated composition, viscosity grows as a result of hardening. The particles of a filler failed to fully orient along the force lines of a magnetic field. Therefore, we subsequently treated the samples of epoxy compositions containing ferrite in the amount of q=30 mass shares and K_p=3.

The next stage involved studying the influence of integrating coefficient K on the physical-mechanical characteristics of the treated material. We observed the maximum values of impact strength a_n=11.3 kJ/m^2 at K=0. At value K=0.2, temperature in the treatment zone was constant and amounted to T=333 K, which is the upper permissible limit for the applied selected hardener. The result is the fact that the treated material is characterized by the elevated values of residual stresses (σ=3 MPa) and the low value of impact strength (a_n=9±0.5 kJ/m^2). At value K=–0.3, temperature in the treatment zone did not exceed T=315 K, and the value of impact strength was a_n=9.5±0.5 kJ/m^2.

Next, we investigated the influence of differentiation coefficient K_d on physical-mechanical characteristics of the treated material. It was established that the optimal value for the coefficient of differentiation is K_d=0.2. At value K_d<0, we observed a short-time increase in temperature in the treatment zone. When increasing K_d>0.25, the system is unstable, we observed the system leaving the equilibrium in 5...8 min from the start of treatment.

Therefore, taking the above into consideration, it can be argued that the advantage of the specified technique for treating the epoxy compositions with an alternating magnetic field using the PID-control algorithm is the possibility to modify the composite material at the higher values of magnetic induction without overheating the material. This ensures the elevated orientation of the filler's particles along the force lines of the magnetic field and the resistance of the material to the sedimentation of a filler. However, the shortcoming is the complexity to control the technological process, as well as energy consumption of the magnetic treatment.

The research results will make it possible to improve the operating properties of epoxy-composite materials in order to obtain parts and components for electrotechnical purposes with the elevated structural strength. This work is one of the stages in our research into epoxy composite materials modified by force fields. In the future, it is planned to conduct a study that would make it possible to stabilize the temperature-time processes when fabricating products from epoxy composites.

7. Conclusions

1. It was determined that the highest indicators of physical-mechanical characteristics of epoxy composites for a given method of treatment are demonstrated by those formed at frequency of the magnetic field of ν=1...10 kHz and at a temperature in the treatment zone to t<360 K. In this case, the impact viscosity of the treated material is a_n=11.3 kJ/m^2, heat resistance T=415 K.

2. It was established that the selection of optimal values for the PID-control algorithm's coefficients in the process of magnetic treatment makes it possible to ensure stability of the environment temperature in the treatment area. That makes it possible to perform treatment at the higher values of the magnetic field induction than those during treatment without using the PID-algorithm. In this case, the highest strength characteristics are demonstrated by the material, which was formed under the influence of an alternating magnetic field with the following control parameters: coefficient of proportionality K_p=3, integration coefficient K=0, and differentiation coefficient K_d=0.2.

3. It was established that the material modified with an alternating magnetic field possesses the smallest residual stresses with a magnitude of σ_r=3...3.5 MPa. The optimum content of a ferromagnetic filler is q=30...35 mass shares per 100 mass shares of the binder.

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