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СИНТЕЗ И СВОЙСТВА ПОЛИМЕРНЫХ КОМПОЗИЦИОННЫХ МАТЕРИАЛОВ НА ОСНОВЕ ЭПОКСИДНОЙ СМОЛЫ

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В статье приведены результаты разработки рецептур полимерных композиционных материалов на основе эпоксидной смолы, тетраэтилентетрамина и пластификатора с заданными значениями твердости. Синтезированные материалы будут использоваться при реализации технологического процесса посадки ребер деталей в качестве губок посадочной машины. Все используемые химические соединения и стекловолокно являются продуктами отечественного производства. Для нахождения оптимальных соотношений полимерного связующего, отвердителя и пластификатора полимерных композиционных материалов использовался метод математического планирования эксперимента. Составление матрицы плана эксперимента производилось с использованием программного пакета Statistica, составлен 3-х уровневый план с числом опытов равным 9-ти и числом повторных измерений равным 3-м. Порядок проведения опытов был рандомизирован для того, чтобы условия проведения экспериментов не искажали результат за счет систематических смещений. Оценка влияний факторов была произведена с помощью дисперсионного анализа (ANOVA) на уровне статистической значимости 0,95. Значимыми регрессорами являются доля отвердителя «H», доля пластификатора «Pl», произведение «H·Pl», квадрат доли отвердителя «H^2» и произведение «Pl^2·H». Увеличение количества отвердителя приводит к росту твердости, рост функции обеспечивает как линейная, так и квадратичная составляющая. Увеличение количества пластификатора приводит к снижению твердости материала. Регрессор «H·Pl» уменьшает значение твердости, а регрессор «Pl^2·H» увеличивает ее значение. Поверхность отклика твердости композиционного материала имеет максимум при 20% отвердителя и при отсутствии пластификатора, минимум твердости соответствует сочетанию 10% отвердителя и 20% пластификатора. Достоверность построенной модели дополнительно подтверждена экспериментально на контрольных образцах.

Ключевые слова: эпоксидная смола, тетраэтилентетрамин, пластификатор, планирование эксперимента, композит, твердость

SYNTHESIS AND PROPERTIES OF EPOXY RESIN-BASED POLYMERIC COMPOSITE MATERIALS

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In the article presents the results on the creation of polymer composite materials based on epoxy resin, tetraethylenetetramine and plasticizer with the specified hardness values of the material, which are used in the implementation of the technological process of fitting the ribs of parts as jaws of the mounting machine. All chemicals and fiberglass employed in this work are domestically produced. The optimal ratios of the components of polymer composite material have been found by the mathematical planning of the experiment. The experiment plan matrix was compiled using the Statistica software package, composed three-level included 9 experiments and 3 repeated measurements. The order of the experiments was randomized to exclude systematic biases that can affect correctness of the results. The effect of factors was evaluated using analysis of variance (ANOVA) at a statistical significance level of 0.95. Significant predictors were the amount of hardener «H», the content of plasticizer «Pl», the product «H·Pl», the square of hardener «H^2» amount and the product «Pl^2·H». An increase in the amount of hardener leads to an increase in hardness, an increase in the function is provided by both a linear and a quadratic component. An increase in the amount of plasticizer leads to a decrease in the hardness of the material. The «H·Pl» regressor decreases the hardness value, and the «Pl^2·H» regressor increases its value. The hardness response surface of the composite material has a maximum at 20% hardener and in the absence of a plasticizer, the minimum hardness corresponds to a combination of 10% hardener and 20% plasticizer. The reliability of the proposed model was additionally confirmed experimentally using the control samples.

Key words: epoxy resin, tetraethylenetetramine, plasticizer, experiment planning, composite, hardness

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INTRODUCTION

Polymer composite materials (PCM) find application in the aviation, automotive, defense, chemical, pharmaceutical industries, ship building, construction, electrical and radio engineering, medicine and in everyday life [1-6]. The main advantages of PCMs over metals are high values of specific strength and elastic modulus as well as significantly lower density. In addition, PCM are employed to fabricate a wide variety of products with tailor-made combination of operational and processing characteristics by the selection of composition and properties of the material components (matrix and filler and their ratio, filler orientation) [7-13]. Therefore, the replacement of various metal elements by PCM sets the trend for materials science. For example, in the aviation industry, PCMs are used as jaws of the planting machine for fitting the ribs of parts, [14, 15], the main requirements being imposed on the hardness of the material.

The Eckold composites satisfy this requirement, but at present their supply to Russia faces certain difficulties and the issue of their replacement becomes an urgent challenge.

In the present work, PCM with a tailor-made hardness has been developed on the basis of epoxy resin and fiberglass.

EXPERIMENTAL PART

Epoxy resin of the ED-22 brand (Khimindustriya-Invest company) was chosen as a polymer binder. Mass fraction of the epoxy groups, determined according to Russian State Standard R 56752-2015 «Resins and epoxy compounds. Methods for determining the mass fraction of epoxy groups and epoxy equivalent» was 22.7. Triethylenetetraamine (analytical grade) was employed as a hardener. Dibutyl phthalate of analytical grade was used.

For the composite material was reinforced by structural fiberglass T-13 on a wax emulsion sizing agent, manufactured in accordance with Russian State

Standard 19170-2001 «Fiberglass. Fabric for structural purposes». This fiberglass has a plain weave, the thickness of the monolayer is 0.27 mm. The surface density is 285 g/m^2. The number of threads per 1 cm on the warp is 16 threads, 10 threads on the weft. This fiberglass keeps its shape well, is suitable for simple parts, and has increased mechanical characteristics. Test samples were laid in alternating layers of 0 and 90°.

Elemental analysis of PCM was performed on a THERMO FINNIGAN gas analyzer (THERMO FINNIGAN, Italy). IR spectra of PCM were recorded on an IFS-25 spectrometer (BRUKER, Germany) in KBr pellets and in vaseline oil. The structure of PCM samples was studied using a QUANTA 200 scanning electron microscope (FEI Company) and a JXA8200 X-ray spectral electron probe microanalyzer (JTOL). The curves of thermogravimetric analysis of the samples were recorded on a Q-1500 derivatograph (MOM, Hungary), the maximum temperature was 850 °C, the heating rate in air was 10 °C min^{-1}.

The hardness of PCM was determined by ball indentation test according ro the Russian State Standard 4670-2015 «Plastics. Determination of hardness. Ball indentation test». A hardened steel ball with a diameter of 2.5, 5.0 or 10 mm was indented into the tested sample under the action of a pressure applied perpendicular to the sample surface for a certain time. After removal of the pressure, the indentation diameter was measured.

The hardness was determined by the ratio of the applied pressure P (kgs) to the indentation surface F (mm^2):

$$HB = \frac{P}{F}. \quad (1)$$

The surface in the form of a spherical segment was determined by the expression

$$F = \frac{\pi D}{2}\left(D - \sqrt{D^2 - d^2}\right), \quad (2)$$

where D is a diameter of the ball, mm; d is a diameter of the indentation, mm.

The hardness was expressed in MPa or kgs/mm^2.

Preparation of the composite. To prepare a PCM, the reinforcing material (fiberglass) was cut by hand and weighed. Next, the material was impregnated with a calculated amount of binder, covered with a polyethylene film and the binder was distributed evenly over the surface of the reinforcing material using a roller. Simultaneously, excess resin and air bubbles were removed. The layered fiberglass impregnated with a binder was placed in a fluoroplastic mold, and the latter was tightly closed with a fluoroplastic piston to reach compression of the layers using threaded clamps. The mold was placed in a vacuum box with automatic heating. After loading the material, the air was discharged to the required value using a vacuum pump. The mold was kept in the box until the resin was completely cured. Then the mold was removed from the box, and the desired PCM was isolated.

RESULTS AND DISCUSSION

To synthesize a PCM with properties similar to the foreign analogue (Eckold), the latter was carried using X-ray single crystal diffraction, IR spectroscopy and elemental analysis.

According to the elemental analysis data, the analog-composite contains carbon (22.94%), nitrogen (0.50%), hydrogen (2.34%), and a significant amount of ash (67.78%). The composition of ash by the X-ray spectral analysis gave the following results: %, Si-10.09; Ca-5.68; Al-5.46; Mg -0.94; Na-0.32. Electron microscopy examination of the sample revealed that it included fiberglass of 0.1-0.15 mm thickness, located in the longitudinal direction. Consequently, it can be concluded that fiberglass was used as filler in this composite.

The foreign analogue was finely ground in a ball mill and the powder obtained was successively extracted in a Soxhlet apparatus with dimethyl sulfoxide, dimethyl formamide, and acetone. After removal of solvents, the extract was studied by IR spectroscopy. In the IR spectra, characteristic peaks of bending vibrations of the epoxy (oxirane) cycle bonds C–O–C at 835 cm^{-1}, C–O at 913 cm^{-1}, and –C–H at 3054 cm^{-1} were detected (Fig. 1) [16].

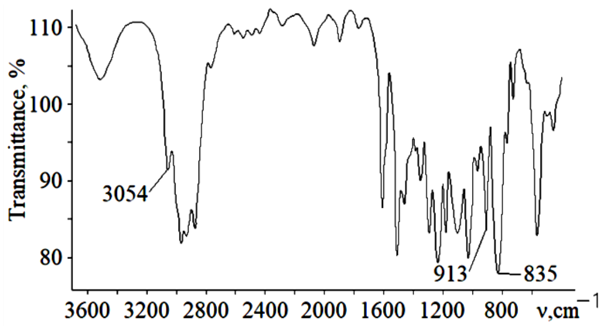

Fig. 1. IR spectrum of isolated epoxy resin
Рис. 1. ИК спектр выделенной эпоксидной смолы

This means that an epoxy resin is used as a polymer binder. It is known that cured epoxy resin has excellent strength characteristics, good adhesion to many materials, high dielectric properties, low shrinkage capacity, and high chemical resistance. Therefore, the cured epoxy resins have found numerous application in many important fields (construction; radio engineering; aviation, automobile and shipbuilding) as a polymer matrix in the creation of PCM [17-20].

To find the required properties of PCM, the hardness and decomposition temperature of the analog-composite were determined. The Brinell hardness of the sample, established by a method involving 10 parallel experiments, was 206 HB. The decomposition temperature, determined by changing the mass of the sample and rate of this changing from temperature, was ~ 300 °C.

The PCM with the tailor-made properties was prepared using the method of experiment planning. This method was aimed at obtaining a maximum of data with a minimum number of experiments [21].

As follows from literature [22, 23], the mechanical characteristics of polymer compositions, including hardness, depend on structure of the material and the mass ratios of the main components such as resin, hardener, plasticizer and filler. The content of filler in the composite is defined by on the placing and weaving of the fabric, and the formation pressure. In this experimental study, the ratio of filler to binder was constant that was ensured by the same manufacturing technology and materials. The percentage of the hardener and plasticizer to the weight of the resin in the experiment was varied and was considered as the main factors.

At the first stage of experiment planning, the levels of the main factors variation were chosen according to the existing understanding of the process and depending on the tasks. The number of levels contributed to the most complete description of the system, the complexity of the task being minimized.

A three-level design was chosen for the experiment, since it allows both linear and non-linear patterns to be simulated. Thus, three levels of variation were taken for the mass fractions of the hardener and plasticizer relative to the resin. The content of the hardener varied from 10% to 20% with an interval of 5%. The amount of the plasticizer varied from 0% to 20% with an interval of 10%. Table 1 shows the natural and normalized levels of the experimental plan factors.

Table 1

Natural and normalized levels of plan factors

***Таблица 1.* Натуральные и нормированные уровни факторов плана**

Level of factors	Plasticizer, wt. %	Hardener, wt. %
-1	0	10
0	10	15
1	20	20

The matrix of the experimental plan was compiled using the Statistica software package. A 3-level plan including 9 experiments and 3 repeated measurements was proposed. The order of the experiments was randomized so that the experimental conditions did not distort the result due to system biases.

The following designations were taken: the amount of the plasticizer in the normalized values was denoted as X_1, and the amount of the hardener was designated as X_2. Table 2 shows the matrix of the experimental plan and the obtained values of the hardness for the composite material samples.

Table 2

The matrix of the experimental plan

***Таблица 2.* Матрица плана эксперимента**

No	Plasticizer, X_1	Hardener, X_2	Hardness, *HB*
1	-1	-1	283; 269; 270
2	0	-1	262; 257; 256
3	1	-1	201; 188; 215
4	-1	0	335; 331; 348
5	0	0	271; 278; 265
6	1	0	225; 219; 234
7	-1	1	451; 465; 432
8	0	1	346; 358; 361
9	1	1	291; 284; 311

The effect of factors was assessed using analysis of variance (ANOVA) at a statistical significance level of 0.95. Significant predictors were the amounts of plasticizer "X_1" and hardener "X_2", product "$X_1 \cdot X_2$", the square of the hardener amount "X_2^2" and the product "$X_1^2 \cdot X_2$".

A regression model of the Brinell hardness of the composite material in normalized values was built by the method of regression analysis using the Statistica software package (3).

$$HB = 278{,}4 - 56{,}4X_1 + 48{,}3X_2 + 27{,}1X_2^2 - 20{,}3X_1X_2 + 19X_1^2X_2, \quad (3)$$

The determination coefficient of this model was 0.98. Analysis of the studentized residuals revealed no outliers beyond ±3σ. Thus, the constructed model can be accepted.

The normalized variables are related to the physical quantities under consideration by the following relationships:

$$X_1 = \frac{Pl-10}{10};\ X_2 = \frac{H-15}{5}. \quad (4)$$

Substituting these expressions into the regression equation and then combining similar terms, we get the regression equation in physical variables:

$$HB = 315{,}9 + 11{,}85 \cdot Pl - 15{,}0 \cdot H - 0{,}57 \cdot Pl^2 + +1{,}084 \cdot H^2 - 1{,}166 \cdot Pl \cdot H + 0{,}038 \cdot Pl^2 \cdot H. \quad (5)$$

The regression equation permits to determine various effects on the hardness of the composite material in terms of the sign and value of the coefficient. The graphical representation of the obtained regression dependence is presented as a surface. Fig. 2 shows the effect of the amount of hardener and plasticizer on the Brinell hardness (*HB*).

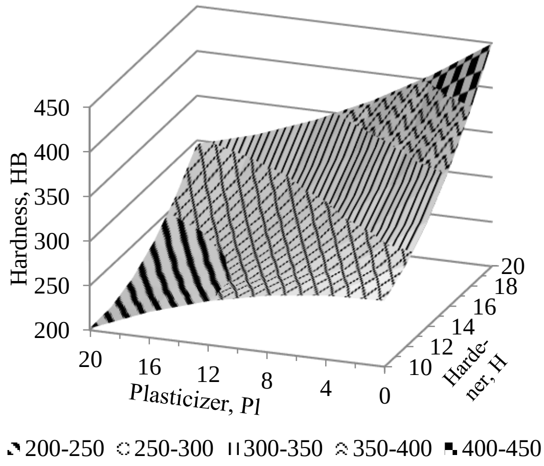

Fig. 2. The hardness of the composite material depending on the amount of plasticizer and hardener
200-250 250-300 300-350 350-400 400-450

Рис. 2. Твердость композиционного материала в зависимости от доли пластификатора и отвердителя
200-250 250-300 300-350 350-400 400-450

An increase in the amount of hardener enhances the hardness, growth of the function being ensured by both a linear and a quadratic component. In contrast, an increase in the content of plasticizer decreases the hardness of the material. The "H·Pl" predictor reduces the hardness value, while the "$Pl^2 \cdot H$" predictor augments its value.

The surface of hardness response of the composite material is maximal with 20% of hardener and in the absence of plasticizer. The minimal hardness is observed in the presence of 10% of hardener and 20% of plasticizer, which is quite consistent with the existing ideas about the characteristics of modified epoxy resin ED-22.

To check the reliability of the constructed model, three additional experiments PCM were carried out (Table 3). The ratios of the components were selected from the investigated range of variation. In these experiments, HB hardness was also measured and compared with the calculated hardness values.

Table 3

PCM composition and values of its hardness

***Таблица 3*. Состав ПКМ и значения его твердости**

No	Resin: plasticizer: hardener, %	HB hardness		Deviation, %
		Experiment	Calculation	
1	100 : 5 : 20	381	396,9	4%
2	100 : 15 : 20	337	320,2	5%
3	100 : 15 : 12,5	226	235,5	4%

The experimental values of the hardness for the control samples differ slightly from the calculated ones; the error does not exceed 5%. Thus, the constructed empirical model can be employed to calculate the predicted hardness value of fiberglass based on ED-22 epoxy resin.

CONCLUSIONS

The analysis of the foreign composite of the company "Eckold" is carried out. The obtained polymer composite materials based on domestic chemical products with a given value of material hardness. The experiments were carried out using the method of mathematical planning of the experiment. This method made it possible to reduce experimental costs and increase the amount of information received due to the optimal organization of the experiment.

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CONFLICT OF INTERESTS

The authors declare that they have no conflicts of interest requiring disclosure in this article.

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