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QUALITY CONTROL OF MIXING COMPONENTS OF ENERGY-SATURATED HETEROGENEOUS COMPOSITE MATERIAL

Abstract. Methods of direct and indirect assessment of the quality of mixing dispersed solid-phase components in a liquid polymer binder are considered. It is shown that a number of methods for the quantitative estimation of mixing (associated with the extraction of solid-phase components), which have been developed in other areas of the technology of materials, are not suitable for controlling an energy-saturated heterogeneous composite material (EHCM). An experimental method revealed the criteria values (a range) of the EHCM density and determined the main groups of the EHCM characteristics, ensuring their acceptable operational properties. By means of modern research equipment using proven methods, a series of studies was carried out to establish the uniformity of the distribution of particles of solid-phase components in the EHCM, the presence, the shape, sizes and distribution in the volume of defects in the structure of the material, the physicomechanical properties of the EHCM after polymerization. According to a comparison of the results of instrumental determination of the main characteristics of the EHCM (structural, physical) with the operational properties of the material, depending on the duration of the mixing process on a specific equipment under otherwise equal conditions, it was found that for the successful solution of technological tasks it is acceptable to assess indirectly the quality of mixing according to the results of density determination of the EHCM after its polymerization. This can provide operational output quality control of the final product that does not require significant material and time costs during the development of technological processes and during the production of the EHCM.

Keywords: energy-saturated heterogeneous composite materials, components, mixing quality, control, research methods, assessment criteria

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КОНТРОЛЬ КАЧЕСТВА СМЕШИВАНИЯ КОМПОНЕНТОВ ЭНЕРГОНАСЫЩЕННОГО ГЕТЕРОГЕННОГО КОМПОЗИЦИОННОГО МАТЕРИАЛА

Аннотация. Рассмотрены способы непосредственной и косвенной оценки качества смешивания дисперсных твердофазных компонентов в жидком полимерном связующем. Показано, что ряд получивших развитие в других областях технологии материалов способов количественной оценки смешивания (связанных с экстракцией твердофазных компонентов), не пригоден для контроля энергонасыщенного гетерогенного композиционного материала (ЭГКМ). Экспериментальным способом выявлены критериальные значения (диапазон) плотности ЭГКМ и определены основные группы характеристик ЭГКМ, обеспечивающих приемлемые эксплуатационные свойства данного материала. С использованием современного оборудования по апробированным методикам проведен комплекс исследований по установлению степени равномерности распределения в ЭГКМ частиц твердофазных компонентов,

наличия, формы, размеров и распределения в объеме дефектов структуры материала, физико-механических свойств ЭГКМ после полимеризации. На основе сопоставления результатов инструментального определения основных характеристик ЭГКМ (структурных, физических) с эксплуатационными свойствами материала в зависимости от длительности процесса смешивания на конкретном оборудовании при прочих равных условиях установлено, что для успешного решения технологических задач приемлемым является способ косвенной оценки качества смешивания по итогам определения плотности ЭГКМ после его полимеризации. Этим может быть обеспечен оперативный выходной контроль качества конечного продукта, не требующий значительных материальных и временных затрат, при разработке технологических процессов и в ходе производства ЭГКМ.

Ключевые слова: энергонасыщенные гетерогенные композиционные материалы, компоненты, качество смешивания, контроль, методы исследования, критерии оценки

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Introduction. Energy-saturated heterogeneous composite materials (EHCMs), used as an energy source in technical systems operating on jet thrust, are produced by mixing polydisperse particles of solid-phase components in one or more liquid-phase binders. After mixing all the components of EHCM, the resulting composition is formed and its liquid phase is polymerized. Changing the performance characteristics of the product produced after polymerization becomes impossible (except for adjusting the size and shape).

Therefore, mixing of the components is one of the most critical technological operations in the general diagram for producing EHCM, which determines the quality of the final product. This circumstance makes it necessary to control the quality of mixing of EHCM components.

Formulation of the research task. For the production of EHCM, solid-phase powder components chosen in accordance with the approaches described in [1] are introduced into the composition of the polymer binder in a certain sequence. In order to obtain the densest packing of particles of these components, as a rule, fractions of various sizes are selected, forming a structure with minimal cavities during mixing [2], which are filled with a liquid-phase binder.

Much attention is paid to the quality of mixing powder solid-phase components in powder metallurgy, pharmaceuticals, agriculture, construction and other industries, where the preparation of structurally similar composite systems is carried out. In these industries, appropriate methods for controlling the uniformity of mixing of components have been introduced into the production process [3, 4]. The main part of these methods involves the use of various instrumental methods for separating fractions or components of a mixture according to their characteristic features and assessing the number of each of them in the selected sample.

Due to the presence of a polymer in the composition of EHCM, which is a liquid phase at the mixing stage, the research results carried out in Ref. [3, 4] cannot be fully used to assess the uniformity of the distribution of solid-phase components. Mixing of a polymer with polydisperse particles of a solid phase, which are introduced to strengthen it, is carried out in the construction, machine, tractor, aircraft and other industries to produce polymer-based construction materials. The mixing process of such materials was investigated in Ref. [5, 6, etc.]. Most of the traditionally used methods for assessing the quality of mixing the components of a highly filled polymer involve the burning of the binder component or the extraction of solid phase components, followed by a quantitative assessment of the extracted powder materials. The use of such methods in the technology for producing EHCM is impossible due to the reactive nature of some of its components, which are prone to chemical decomposition at relatively low temperatures ($t > 150$ °C). This can cause uncontrolled combustion of the mixed mass.

The use of methods for non-destructive control of polymer-based composite materials, considered in Ref. [7], requires their adaptation due to the increased reactivity of EHCM and the peculiarities of the technological stages of its production. Also, for the application of non-destructive control methods, a statistical database is needed to compare the results obtained with the one already available in it. Taking into account the foregoing, the task of the study is to select methods for assessing the quality of mixing of EHCM components, determine the algorithms for their application, verify these methods using the example of the existing technology for producing EHCM, formalize the input data and the resulting final values, as well as develop rules for interpreting the results obtained.

Research materials and discussion. To conduct a study and determine approaches to control the quality of mixing in accordance with the thermodynamic calculations carried out in Ref. [1], the composition of EHCM was chosen, in which the liquid phase is 14 wt.%. The solid phase is particles of micro- and nano-size. For a visual representation of the objective function of the process of mixing the components of EHCM in Ref. [8], a geometric model of a unit cell with a coordination number of 12 was developed, which is formed with a hexagonal dense packing of solid phase particles. When developing this model, in order to achieve the densest packing of the oxidant particle (the main component of the solid phase), two fractions were picked up, the median sizes of which were 240 and 50 μm.

Taking into account the characteristics of the unit cell in Ref. [1], the calculated values of the mixing quality of the EHCM components were determined, which are subsequently used for instrumental control at all stages of its production. Based on the results obtained in the course of calculations and modeling, the input requirements for the EHCM components are formulated, in accordance with which they are selected and the following characteristics are controlled:

the chemical composition of the components, which determines the required stoichiometry;

particle size distribution and morphological properties of powder components;

physicochemical properties of the polymer binder (molecular weight, number of functional groups, viscosity, moisture, and etc.).

After analyzing the measurements of the characteristics of the EHCM components, some operating modes of the technological equipment or the sequence of technological operations of the mixing process can be refined.

In the course of mixing the components of EHCM, a complex of tasks is solved. According to the results its operational properties are formed. First of all, these tasks are following [1]:

achieving a uniform distribution over the volume of EHCM of all solid-phase components and preparation of a homogeneous composition;

obtaining the densest packing of particles of the solid phase due to their rational distribution in the volume of EHCM, depending on the linear dimensions and properties;

filling cavities formed in the packing of particles of solid-phase components with a liquid polymer binder;

wetting the entire surface of the solid-phase particles with liquid-phase components.

Due to the uniform distribution of the components, the stability of the performance characteristics of the product made of EHCM is achieved, and, accordingly, the stability of technical system that includes this product. The absence of cavities in the packing of particles of solid-phase components reduces the probability of an uncontrolled increase in the area of combustion of EHCM, and the densest packing gives the best value of the energy properties per unit volume of the resulting composite material. By wetting the entire surface of the particles, the formation of a structure-forming matrix with the required value of adhesion strength is achieved, which provides the specified physical and mechanical properties for the EHCM product.

Taking into account the importance of each of the above tasks in the formation of the operational properties of EHCM in Ref. [1], the characteristics were investigated and evaluation criteria were proposed that determine the degree of their fulfillment. These characteristics include:

the degree of uniformity of particle distribution of solid-phase components in EHCM;

absence/presence of cavities in the EHCM product, their sizes, shapes and volume distribution;

the density of the resulting EHCM;

physical and mechanical properties of EHCM after polymerization.

Their measurement is carried out taking into account the physicochemical properties of the components and EHCM as a whole, typical for each stage of the technological process of its production.

Experimental set up and analysis of results. To determine the methods of studying the properties that characterize the degree of fulfillment of mixing tasks, an experimental composition of EHCM was produced. In the process of its production, the results of producing EHCM at each of the technological stages were investigated and the methods of their control were considered. Production technology of the experimental composition, due to the presence of nanoscale antioxidant powders in the composition of solid-phase components, assumed the production of EHCM in two stages.

Dynamics of surface area

Component	Specific surface area, m^2/g	Weight, kg	Total surface area, m^2
Before adhesive deposition			
Oxidizing agent (large fraction)	0.425	2.985	1417.9
Anti-oxidizing agent	430	0.015	6450
After 2 h of adhesive deposition			
Oxidizing agent (large fraction) with deposited anti-oxidizing agent	2.17	3	6491
After 4 h of adhesive deposition			
Oxidizing agent (large fraction) with deposited anti-oxidizing agent	1.965	3	5868

At the first stage, the preparation of powder components was carried out, during which nanoscale components (anti-oxidizing agent) from an alcohol suspension were deposited onto the surface of particles of the large fraction of the oxidizing agent (240 μm). The deposition of nanoscale particles was carried out in accordance with the technology described in Ref. [9]. In the course of deposition, the uniformity of the distribution of nanoscale components on the surface of the large fraction particles of the ammonium salt of perchloric acid and the change in the surface area of the particles of the mixed EHCM components were studied.

To assess the uniformity of distribution, collection of samples was carried out from time to time and changes in the surface area of the interacting particles were analyzed. The assessment of changes was carried out by measuring the specific surface area and calculating the surface area of the particles intended for subsequent wetting with a liquid-phase binder. The specific surface area was measured by the BET method on SA 3100 surface area and pore size analyzer (Beckman Coulter, Inc., USA). The results of measuring the specific surface area and total area of the particles of the selected samples of the oxidizing agent (large fraction) and anti-oxidizing agent before deposition, after 2 h and 4 h of deposition are presented in Table.

The results were evaluated taking into account changes in the surface morphology of large fraction particles of the oxidizing agent. The surface morphology was surveyed using high-resolution scanning electron microscope Mira (Tescan, Czech Republic). Thus, the results of the morphological analysis of the surface of the large fraction particles of the oxidizing agent before deposition (Fig. 1, *a*), after 2 h and 4 h of deposition of the anti-oxidant particles are shown in Fig. 1.

The analysis results showed that after two hours of adhesive deposition, the total surface area of the particles decreased by 17.5 %. At the same time, during the study of the surface morphology of the oxidant particles, the presence of agglomerates (Fig. 1, *b*) of the anti-oxidizing agent on their surface was revealed. Taking into account the obtained values, the process of mechanical impact on the deposited surface and nanoscale anti-oxidant particles continued until their relatively uniform distribution was obtained. Thus, over the next 2 h, the total surface area of the particles decreased by another 6.7 %, and a more uniform distribution of anti-oxidant particles over the deposited surface was obtained (Fig. 1, *c*).

At the second stage, solid-phase powder components were mixed in the medium of a polymer binder, including powders of the large fraction of the ammonium salt of perchloric acid, with nanoscale anti-oxidant particles deposited on the surface of their particles. The components were mixed in SP-15 plane-

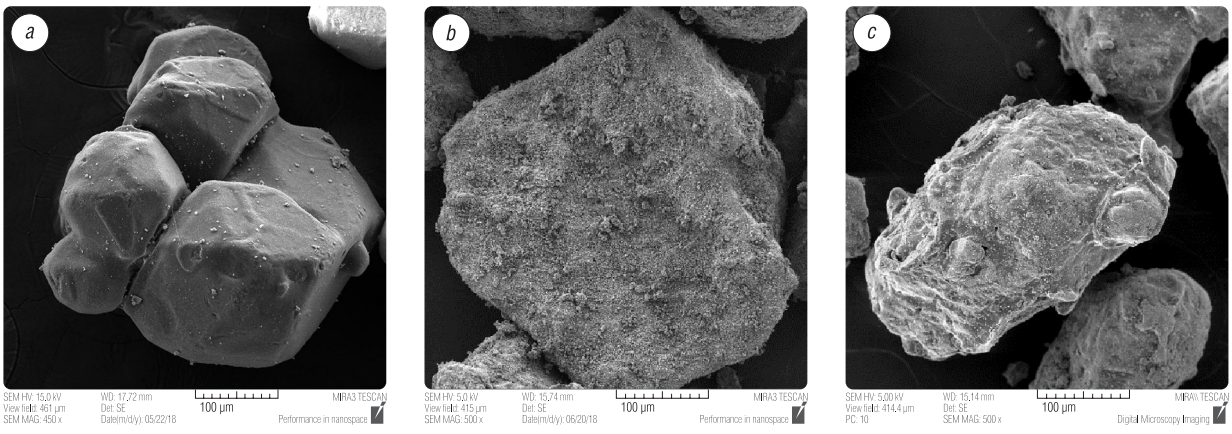

Fig. 1. The results of morphological analysis of the surface of the large fraction particles of the oxidizing agent: *a* – before the deposition of nanoscale additives, *b* – after 2 h of operation of the technological equipment, *c* – after 4 h (completion of adhesive deposition) of operation of the technological equipment

tary mixer (Russian Federation) with two mixers. The mixing bowl diameter is 335 mm and the working volume is 2.375 l. During mixing, the uniformity of the distribution of components in the volume of the EHCM was assessed by sampling, polymerization of the liquid-phase binder, and evaluation of:

the formed structure and homogeneity of EHCM afterwards;

density of the produced EHCM and its compliance with the calculated value;

physical and mechanical properties of the material.

To study the structure formed during mixing, samples were taken at intervals of 5 min. Further mixing was resumed after setting up the required pressure and temperature in the mixer bowl. After polymerization of the binder, the selected samples were subjected to fracture along the cross section and application of a conductive coating. The conductive layer on the fracture surface was applied by cathode spraying of chromium in a vacuum, and the coating thickness required for contrast was formed for 10 s of deposition.

The morphology of the samples was studied using high-resolution scanning electron microscope Mira (Tescan, Czech Republic). The accelerating voltage during the survey was 15 kV. The results of the morphological analysis of the composition obtained at various stages of mixing the components are shown in Fig. 2.

According to the results of the morphological analysis of the samples taken in the 5th minute, the presence of large (significantly larger than the particle size of powder components) cavities in their structure was determined (Fig. 2, *a*). With further mixing, an ordered structure of EHCM is formed due to a denser packing of particles of powder components and filling the cavities with the polymer binder

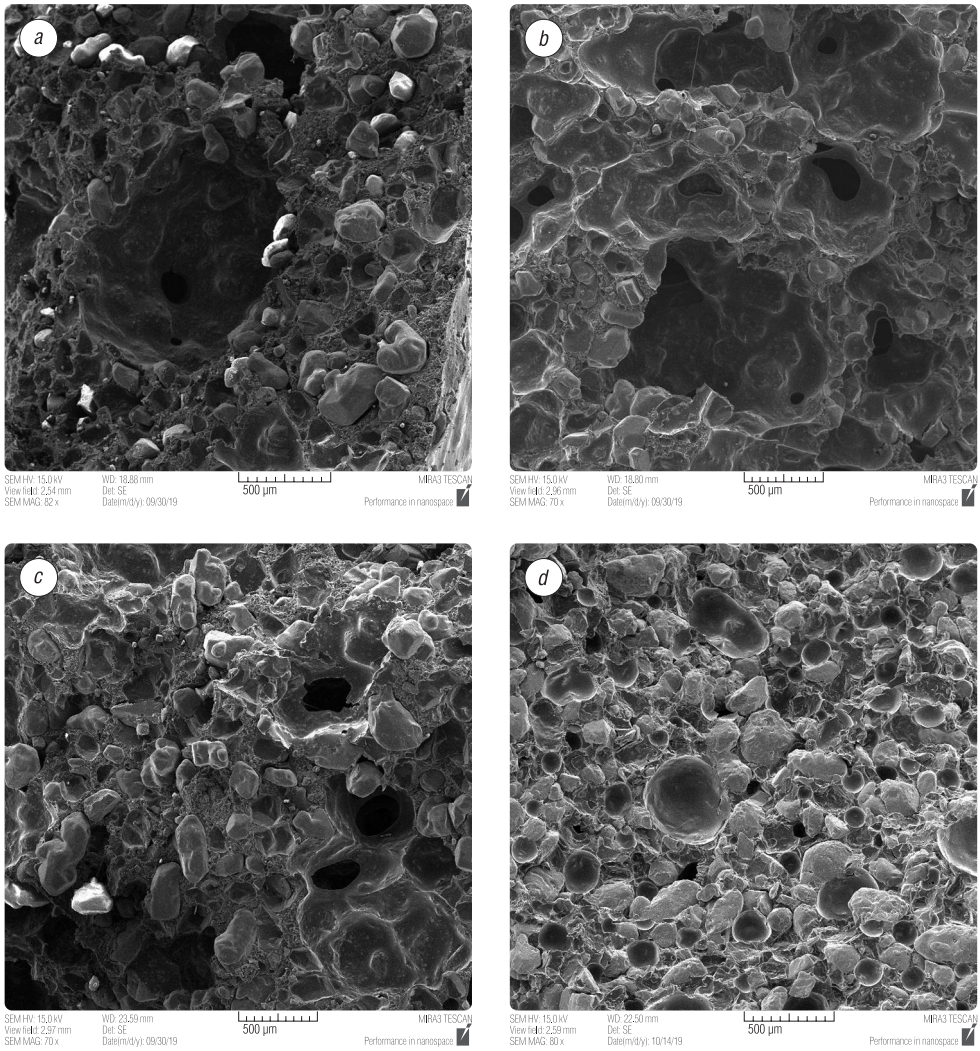

Fig. 2. Morphology of the fracture surface of polymerized EHCM samples: *a* – after 5 min of mixing, *b* – after 10 min of mixing, *c* – after 15 min of mixing, *d* – after 20 min of mixing

(Figs. 2, *a*–*c*). The best value is typical for a sample taken in the 20th minute of mixing (Fig. 2, *d*). Further mixing of the components after the ordered structuring of the EHCM led to an increase in the load on the rotating blades of the mixing equipment (it was characterized by an increase in the power output at the drive) and the destruction of particles. The morphological analysis of a sample taken in 25th minute of mixing the components revealed the presence of destruction of large fraction particles of powder components in the composition of the material. The processes (wetting, cladding, rearrangement of particles, adhesive interaction, etc.) occurring with continued mixing after the formation of a relatively ordered structure of EHCM (after 20–22 min) require additional research.

According to the results of morphological analysis, it was found that for the selected mixing equipment and the composition under consideration, mixed at a temperature in the mixing chamber $T = 303–308$ K, a pressure in the bowl $P = 0.075$ MPa, and a blade rotation speed 20 rpm, the most preferred mixing time is in the interval of 20–22 min.

Along with the morphological analysis of structure formation during the second stage of producing EHCM, the dynamics of changes in the density of the material, resulted at various stages of mixing the components, was investigated. The EHCM components were mixed for 32 min. Sampling was performed every 2 min of mixing. The other conditions and operating modes of the mixing equipment were identical to those described above when carrying out morphological analysis. The density of the samples was measured with a PZh-2 pycnometer. The dynamics of the change in the density of the material with the mixing of the EHCM components with the approximation reliability $R^2 = 0.9976$ is shown in Fig. 3.

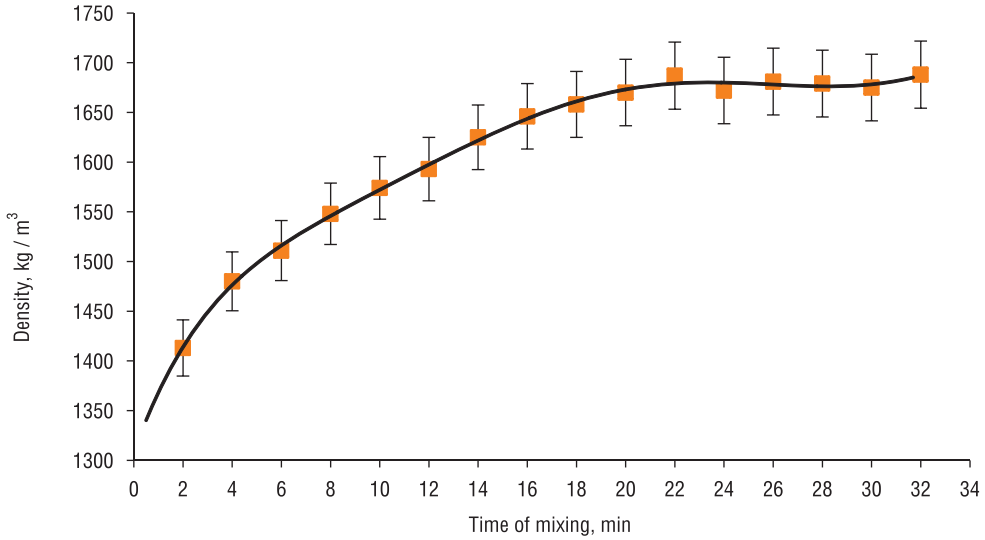

Fig. 3. Dynamics of the EHCM density during mixing of the components

It was found that the best value of the density (1687 kg/m^3, which is 0.97 of the calculated one) of the experimental EHCM was achieved at the 22d minute of mixing its components. As can be seen from the diagram, further mixing of the components does not give a significant increase in density. In this regard, the most preferred mixing time of the EHCM components was determined, which was 22 min.

Relatively identical results of the most preferred mixing time under the specified operating modes of the technological equipment confirmed the possibility of using both control methods (study of the morphology of the fracture surface of the material sample and the material density) for assessing the mixing quality. At the same time, a method that evaluates the quality of mixing by measuring the density of a sample is more accurate, since it can quantify the measured property. Also, the assessment of the quality of mixing by measuring the achieved density of EHCM is a less costly method of control, since it does not imply the obligatory production of a witness sample, which must subsequently be disposed of in the prescribed manner. However, within the framework of adapting this method for the production of specific products, it is necessary to determine the evaluation criteria. The comparison of the measured density values will make it possible for the manufacturer to decide on the quality of the produced EHCM.

To study homogeneity and establish criteria for assessing the quality of mixing of EHCM components, X-ray control of produced samples with different relative densities was carried out. X-ray control was carried out using ERESCO MF4 X-ray generator (Germany) at a voltage and current on an X-ray tube of 65 kV and 1.0 mA, respectively; a focal distance is 1000 mm and an exposure time is 0.5 s.

In the course of the radiographic analysis of samples taken from the 18th to 22d minute with a relative density of $\vartheta = 0.95–0.97$, the homogeneity of the structure of the resulting EHCM was confirmed. The results of radiographic analysis for the sample with $\vartheta = 0.95$ are shown in Fig. 4.

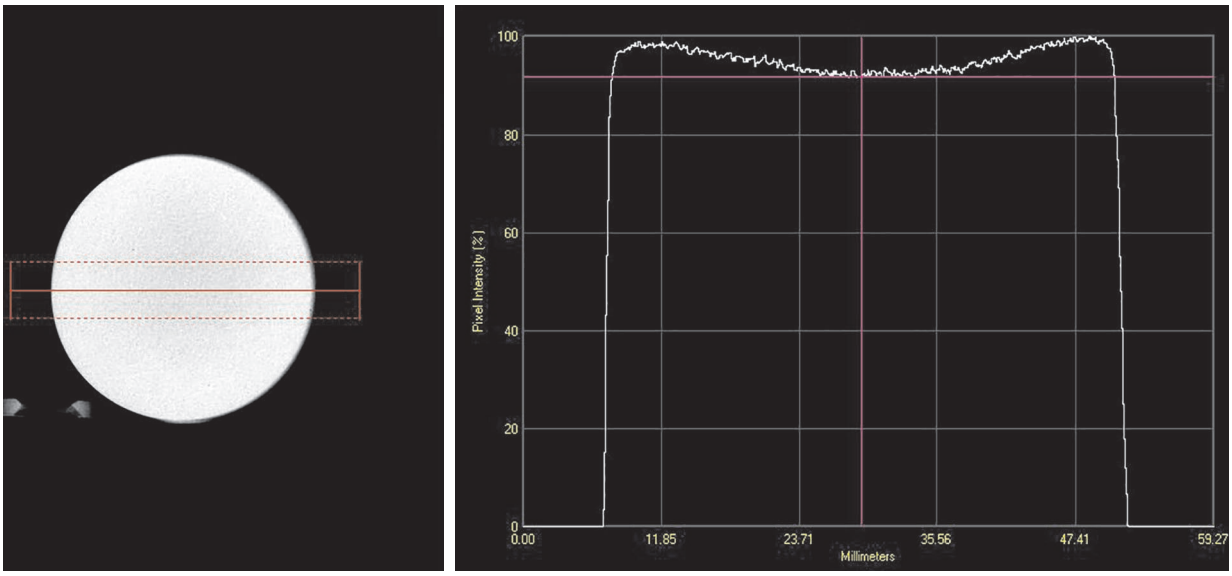

Fig. 4. Radiographic image of the EHCM sample with a relative density of $\vartheta = 0.95$

The deviations of the intensity of the measured product transmitted radiation throughout the entire cross section due to the different density were within the normal range. This confirmed the conclusion about the suitability of the resulting EHCM for operation as part of a technical system.

The study of the homogeneity of the samples taken in the 14th and 16th, as well as 24th and 26th minutes of mixing, revealed the presence of defects in the EHCM structure. The most typical examples for defects of this group of EHCM samples are shown in Fig. 5.

The reasons for the appearance of such defects can be insufficient wetting of the surface of the particles of solid-phase components with a liquid polymer binder, whereby the required contact area and, accordingly, the adhesive interaction at the phase boundary are not provided. Further mixing of the components allows such defects to be minimized. The second group of defects, which is most typical for samples taken in 24th and 26th minutes of mixing, may be a consequence of an increase in the shear stress typical for a dilatant fluid [10]. It is assumed that for this reason, the particles of the powder components are destroyed and rearranged.

To confirm the effect of the homogeneity of the EHCM structure on its operational properties, the strength and energy characteristics of the selected samples were measured. The strength of the samples was investigated in accordance with GOST 11 262-2017 (ISO 527-2:2012) "Plastics. Tensile test method". During the tests, it was found that the samples with a relative density of $\vartheta = 0.95–0.97$ showed a result that meets the requirements (tensile strength $\sigma_p = 4–10$ MPa; relative elongation $\varepsilon = 5–10$ %). Specimens made of EHCM taken in 14th and 16th, as well as 24th and 26th minutes and having hidden structural defects, corresponded in 50 % of cases to the lower limit of the specified range of admissible strength values, and the rest was destroyed at loads significantly lower than the lower threshold value.

The second group of operational properties, involving the use of EHCM products as part of a technical system, was studied on a stand that simulates the real conditions of its operation. In order to eliminate the probability of destruction of the technical system, samples made of EHCM after 14th and 16th, as well as 24th and 26th minutes as part of the technical system were not tested. The reasons for the occurrence and the nature of various defects in the operation of a technical system, which includes an

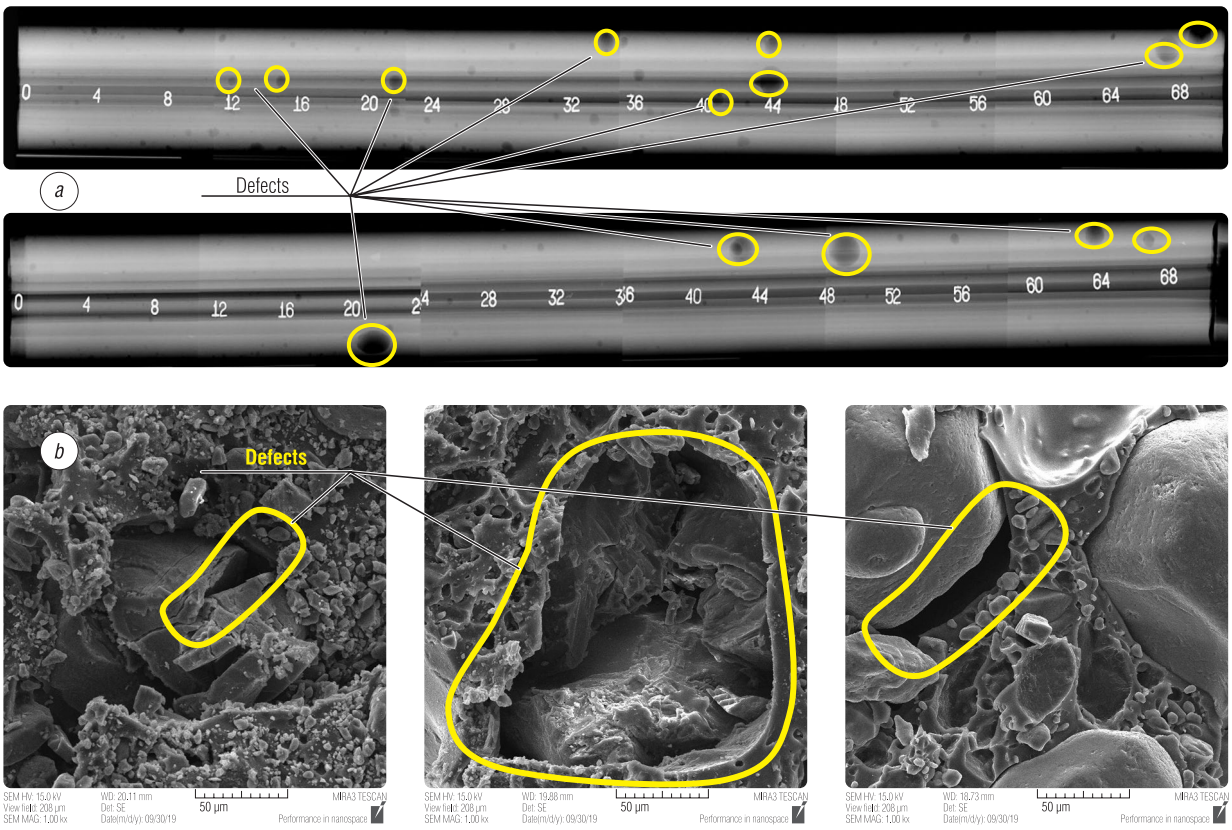

Fig. 5. Examples of defects in EHCM samples taken in 14th and 16th minute, as well as in 24th and 26th minute of mixing: *a* – the signs of lack of homogeneity and the presence of hidden defects in the product made of EHCM, *b* – the most characteristic hidden defects in the structure of EHCM (cavities formed as a result of the destruction of large fractions of the oxidizing agent, the absence of adhesion between the surface of the particles of solid-phase components and the polymer material)

EHCM product, requires additional research. Testing of products made of EHCM with a relative density of $\vartheta = 0.95–0.97$, as part of a technical system, confirmed their compliance with the requirements.

Taking into account the results obtained in the course of the study, it was shown that the density of EHCM is the most representative property characterizing the quality of mixing of its components and the formation of an ordered structure. In this case, the criterion value characterizing the degree of achievement of the required mixing quality is the relative density of the material. Its value should be in the range of $0.95 \leqslant \vartheta \leqslant 1$.

Conclusion. The paper defines the main properties of EHCM, which characterize the mixing quality of its components. Taking into account the approaches in other industries, the possibility of applying the methods used to control these properties is assessed. According to the results of the assessment, it was determined that the density of EHCM can be considered as the most representative property characterizing the quality of mixing of EHCM components. The efficiency of this choice is confirmed by the example of the manufacture of an experimental EHCM composition by other instrumental control methods. The methods of interpretation of the obtained research results have been experimentally run out. The use of instrumental control methods contributed to the optimization of technological operation modes of mixing equipment for EHCM manufacture.

An evaluation criterion has been run out to control the quality of mixing of EHCM components by analyzing the relative density of the material. The feasibility of the defined value of the mixing quality evaluation criterion is confirmed by strength and full-scale tests. It was found that going beyond the limits of the material density evaluation criterion indicates the presence of defects in the structure of the EHCM product. The possibility of using EHCM with a density lower than the defined value requires additional research.

Thus, the proposed approach to assessing the relative density of the produced material and the proposed interpretation of the results of its use makes it possible to exclude the use of a product with internal defects and thereby ensure the required quality of the sample as a whole.

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