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STRUCTURE FORMATION OF DISPERSED-REINFORCED BUILDING COMPOSITES

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Abstract. The results of the study of the mechanism of structure formation of cement compositions reinforced with finely dispersed monofilament are presented in the article. The mechanism of microstructure organization of construction composites was studied on models of dispersed systems, with different qualitative and quantitative composition of linear and dispersed particles. At the same time, restrictions had been placed on particle size – fiber diameter and diameter of dispersed particles are proportional to each other. Study of cracking formation kinetics was carried out on disk-shaped samples made of water-clay and water-cement compositions. Physical and mechanical characteristics of dispersed-reinforced cement stone, including non-reinforced stone, have been defined on prisms-shaped samples of square section with size $40 \times 40 \times 160$ mm.

The analysis of physical models showed that cluster structures filling with particles of various nature and shape increases structural diversity of entire dispersed system. An inserting of linear particles changes nature of system structure formation. Depending on the characteristics, structural components of the system, substructures are formed, which differ in the periods of their formation and geometric parameters. It has been established that dispersed particles of different nature are structured in different ways into clusters with discrete fibers of different length. Linear particles were more active in the creation of structural aggregates (clusters) comparing to dispersed grains.

The impact of highly dispersed fibers on the structure organization of the binder compositions was quantified by the damage coefficient determined on samples of different types. The presence of discrete fibers in the composition of the material leads to modify the qualitative characteristic of compositions cracking formation. Improvement of physical and mechanical properties of the dispersed-reinforced composite confirms the ability of the fiber to change a mechanism of material destruction due to a probable deposition of hydration products on monofilaments, to densify and strengthen the interfacial transitional zone.

Keywords: structure of concrete, dispersed system, cluster, cracks, dispersed particle, fiber.

Introduction. The durability of composite materials is one of the important operational properties of products (structures), characterized by various structural parameters – geometric dimensions, shape, material composition, etc. For many decades, scientists have been trying to obtain a building material which is capable of maintaining established functional properties in products for a specified period of time under certain operational loads (power, climatic, etc.). It is known that the main mechanical characteristics (strength, crack resistance, etc.) for different types of products, in particular decorative quality indicators for finishing products, are directly related to the material structure on the basis of which they are made. Therefore, the relentless improvement of the mechanism of structure formation of cement composites, against a background of increasing demands for structural elements and architectural details of modern buildings and constant climatic changes of the natural environment, provides an opportunity to obtain high-quality products with the initially given operational reliability of its material.

Analysis of recent research. A review of the scientific literature showed that it is necessary to

apply an integrated approach to a design of composite structures at each level of its structural heterogeneity in order to obtain an optimal structure of a composite material. At the same time, it is worth considering the mutual influence of structural characteristics in a structure formation and operation of the material of products. According to V.I. Solomatov, V.N. Vyrovoy et al. [1-3], it is advisable to identify two levels of structural heterogeneity: micro- and macrostructures in polystructural materials, which is concrete. This is due to the fact that although these levels are fundamentally different among themselves in the structure organization of the material over time, they sufficiently reflect objective regularities of a structure formation and establishment of the concrete properties.

The main aspect in obtaining an optimal concrete structure is the prevention of a formation and development of technological defects (pores, cracks, internal interfaces, etc.) at all levels of its structural heterogeneity during the manufacturing and operation of the material of products [3]. It is known that these defects cause a concentration of high stresses, even with low internal and external impacts. In this regard, it is necessary to keep under control all imperfections of the concrete structures in order to ensure an operational reliability of the material of products. At the same time, it is important that the cracks from lower levels do not sprout into higher levels, as this will negatively affect the durability and bearing capacity of products (structures), and especially the appearance of finishing products, such as architectural elements.

The microstructure of cement stone is significant for concrete, because its properties and durability are based on it. However, it is known that the microstructure of concrete is quite heterogeneous in its composition even at the initial W/C ratio of 0.30, especially in a porous and weak interfacial transition zone, which exists at the interface of cement stone with filler, dispersed reinforcing fiber, etc. [4-7]. It should be noted that the interface is one of the important microstructural components of concrete through which stresses are transmitted. Micromorphological studies presented in [4, 8] show that the microstructure of the interfacial zone, which mainly consists of large crystals of portlandite CH and extends from the filler surface by about 10-50 µm, is sufficiently porous, than the bulk of hydrated cement. In particular, E.I. Kolokolnikova in [9] draws attention to the fact that cracks mainly develop in areas of cement stone with a high CH content and less uniform structure. Taking this into account, the creation of a volumetric and homogeneous highly dense and, as a consequence, stronger interfacial zone of the microstructure of concrete, will reduce the risk of «easy» formation and spread of defects of various kinds under the influence of internal stresses and climatic factors. In this context, internal stresses refer to voltages not related to the application of operational loads to the material.

Numerous papers have shown that it is possible to control the process of concrete structurization at a microlevel by using organo-mineral additives [1, 2, 8, 10, etc.]. The use of these additives makes it possible to obtain a more uniform dense and high-strength concrete microstructure with a markedly lower number of calcium hydroxide crystals of smaller size and volume in the transition zone [4-6, 8]. However, there are many disputes regarding the qualitative and quantitative composition of active mineral additives in cement composites. This is due to the fact that the microstructure of the transition zone is closely related to the dispersed nature of the cement-matrix. For cement binder, it is necessary to select fillers of optimal size and surface activity, which are able to take part in the processes of organizing the microstructure of concrete, while changing the conditions for genesis and spread of cracks [1-3]. However, the inclusion of the above-mentioned additives in the concrete composition leads to an increase in its fragility. In order to increase the viscosity of destruction and control of technological defects of various kinds in the general structure of high-strength or high-efficiency concrete, disperse fibers of different nature and geometry are mainly used in the manufacture [11]. In turn, the use of fibers to strengthen the microstructure of concrete has its own features, in particular in its interfacial zone. In [7] A. Bentur and S. Mindess note that in order to obtain a denser structure of cement stone, it is worth using discrete monofilaments that are well dispersed in the mixture. This is due to the fact that the entire surface of the monofilament is in direct contact with the matrix, the same cannot be said about bound fibers bundle (e.g. asbestos, polypropylene, etc.) in which only the external surface can have direct access to the cement-matrix. However, too strong matrix-fiber relationship is not desirable [12] because the fiber flexibility is reduced and thereby the breaking mode changes - from extraction to rupture. Equally important is the particle size of all components of the concrete microstructure. In the article [13], the authors state it is necessary for the filler particle size and cross-section of the fibers to be smaller or proportional to the grain of the cement binder in order to achieve an optimal matrix-fiber relationship. In their view, a fiber can be efficiently laid between cement grains to obtain a more uniform dense microstructure of the composite without any transition zone only with such sorting of the mixture. There is also a significant length of fiber under this condition. As noted in [7, 14-16], the ease of fiber dispersion in the cement-matrix composites markedly improves with decreasing its length ($l_f < 10 \text{ mm}$). Therefore, it is not enough to determine only the relationship between the components of cement stone (e.g., binder, mineral additive and discrete fibers) and know their geometric parameters. It is worth understanding, how they will be oriented in the volumetric space of the cement-matrix in order to identify its optimal reinforcement and obtain at the same time a more stable microstructure of the composite to the effects of various internal and external factors.

The purpose of research is to open the mechanism of structure formation of cement compositions reinforced with highly dispersed fiber.

Research methodology and starting materials. The studies were carried out in two stages to better understand the mechanism of microstructure formation of construction composites.

At the first stage, the mechanism of organizing the microstructure of the composite was studied during the physical interaction of discrete fibers with dispersed particles of different nature. Due to the fact that the microstructure of concrete represents a highly concentrated coarsely dispersed system [1-2], consisting of various structural aggregates (clusters), a modeling of a formation process of cluster structures (at $0 < \tau \le 9$ min) was carried out by means of model systems. The following types of physical models of dispersed systems were adopted in the study: models of dispersed particles of various nature, models of linear particles (or discrete monofilaments), models of dispersed and linear particles located in any order on a surface of the model of aquatic environment. During a simulating interparticle interactions in the Petri dish in the microstructure of the material, the following restrictions were imposed: a diameter of dispersed particles and diameter of fibers are equal to each other; discrete fibers correspond to a length of 6 and 12 mm; a concentration of particles in the system is such that an attractive force F_a occurs between them; a bonding force between particles is greater than force of gravity F_g . Foam polystyrene granules and millet grains were used as dispersed particles, and a line above-mentioned length - as lineal particles. Therefore, several clustering mechanisms should be implemented in the system depending on particles nature, inter-particle distances and ratios of the forms of dispersed and linear particles.

At the second stage, physical and mechanical characteristics of dispersed reinforced cement stone at the age of 7 days were determined. The samples were tested on square prisms $40 \times 40 \times 160$ mm in size. White Portland cement produced by CIMSA (Turkey) and Owens Corning (USA) alkaliresistant fibers with a length of 6 mm were used in research. An unreinforced material composition was taken as a base (comparison) in order to confirm the advisability of highly dispersed fiber reinforcement of binder compositions. Also at this stage, subjects of the research of binder stone were cracks and internal interfaces, the total number of which determines technological damage of material samples on the surface [2]. In turn, the technological damage was assessed through the damage coefficient (K_D , cm/cm²). Thus, experiments on kinetics of cracking formation on models of dispersed systems made of water-clay and water-cement compositions (W/C = 0.6 and W/C = 0.5, respectively) were additionally implemented. A choice of clay as a dispersed phase is due to the fact that the mechanism of occurrence, distribution and redistribution of deformations in the microstructure of these dispersed systems, is independent of their type and features of transition from one rheological state to another [3].

Analysis of research results. The studies conducted in the first stage on modeling of separate spatial orientation of particles of same nature showed that a different number of structural aggregates of a different quantitative composition of particles are formed in primary disordered dispersed systems over the same time period (as an example, two physical models are shown in



Fig. 1. Modeling of a spatial orientation of identical particles in a step-by-step arrangement of clusters within 9 minutes:dispersed grains (a) and linear particles with a length of 6 mm (b)

Fig. 1, a and b). It is worth noting that an organization of clusters is carried out spontaneously without applying external forces in the aquatic environment. First of all, aggregates are formed, the particles of which are located at smaller distances from each other. Linear particles are more active in creating cluster structures in comparison to dispersed grains of different nature. This is most likely due to their larger surface area, as well as different values of surface tension with aqueous medium. At the same time, a formation of linear clusters ($\tau < 9$ min) generally starts with the contact of the end parts of the fibers, and further they can reciprocally rotate relatively to each other, in seeking to come into contact with parts of a surface of the same state. In this case, the value of orientation angles between individual monofilaments changes at intervals during their regrouping. In particular, it is important to note that the interparticle interaction of the particles did not change in similar experiments, but on the surface of the model of the aqueous medium plasticized with a polycarboxylate additive (in the amount of 0.3%).

Analysis of model systems of a mixture of particles of a different nature and shape made it possible to establish that dispersed particles are variously structured into clusters with linear particles of different length (Fig. 2 shows two models as an example). When foam polystyrene granules interact with monofilaments, linear clusters with a greater length and less volume are formed, and a coupling between the components of the substructures of the system occurs exclusively through the active centers of the end parts of the fiber (Fig. 2, a). Mostly, fibers are not symmetrically oriented, but are fixed in the condition in these discrete linear aggregates. Besides linear clusters in the system, there are also aggregates, consisting only of the equal particles. The aggregates are not uniform on the composition of discrete particles in such a system.

An organization of cluster structures formed from mineral grains and linear particles occurs a little differently. Dispersed particles with fibers of different length are joined along the entire length (Fig. 2, b), thereby forming more volumetric clusters, because linear particles serve as the interface in such aggregates. Furthermore, the grains are regrouped in structural aggregates. Dispersed particles, changing their position in substructures, increase a density of cluster packing. In these model systems, no cluster structures were found that would consist only of the same particles.



Fig. 2. Self-organization of cluster structures ($\tau = 9 \text{ min}$) in interaction: a) foam polystyrene granules with linear particles, 12 mm long; b) mineral grains with discrete fiber, 6 mm long

Therefore, the general structure of dispersed system is organized a more volumetric and uniform on the composition of aggregates. These discrete structures are more diverse due to the fact that the monofilaments are oriented from a different angle. There is also a symmetrical arrangement of the fiber relative to itself through dispersed particles. It is worth noting that the structure of the model system, which is formed from a mixture of mineral grains and short fiber over a period of 9 minutes, is organized slightly faster compared to a long fiber.

Thus, the analysis of physical models (Fig. 1 and 2) showed that a filling of cluster structures with particles varied in nature and shape increases a structural diversity of the entire dispersed system. An introduction of linear particles changes the structure formation mechanism of the system. Depending on the characteristics of structural components of the system, substructures are formed which differ in the periods of their formation and geometric parameters. In our opinion, the variety of such substructures at the microlevel of concrete will improve the physical and mechanical properties of cement stone.

The influence of discrete fibers on the structure formation of cement composites has been studied in different models. Clay was used jointly with the cement composition to visually observe an organizing process of the structure of dispersed systems. At the same time, the research was carried out on samples in the form of disks. Experiments have shown that dispersed fibers are actively involved in the organization of the microstructure of these mineral systems (Fig. 3). Therefore, preliminary conclusions are confirmed regarding the impact of a qualitative composition of particles of the dispersed phase on the the structure formation of systems at the microlevel. In Figure 3, it can be seen that a qualitative characteristic of cracking formation of clay and cement compositions changes completely depending on a structure of discrete particles of the dispersed phase. The use of fibers allows to change the distribution of volumetric deformations that arise during hardening of binder systems (Fig. 3c and d). A net of thin curved cracks and internal interfaces with a sufficiently small opening width is observed on a surface of samples of compositions reinforced with highly dispersed fiber. In particular, the effect of the fibers on the structure organization of the compositions is also quantified by the damage coefficient $K_{\rm D}$ on some areas of a surface of samples-disks (Fig. 3). A comparison of the numerical values of the coefficient $K_{\rm D}$, using clay compositions as an example (Fig. 3a), demonstrates that despite a large technological damage of reinforced clay composition (Fig. 3c) - it remains a whole «indivisible» into fragments system. Consequently, the ability of fibers to perceive and evenly redistribute deformations is confirmed.

Analysis of the results presented above shows that a presence of highly dispersed fiber in binder systems changes their structural characteristics. Therefore, this should lead to a change in the physical and mechanical properties of cement stone. Preliminary studies of the characteristics of the binder stone confirmed that the use of discrete fibers with a length of 6 mm contributes to an increase in strength of samples, both of bend and compression test, of 40 and 10%, respectively. An increase in strength of the dispersed-reinforced composite confirms an ability of a fiber to change



Fig. 3. Technological damage of non-reinforced (a and b) and short fiber reinforced (c and d) binder compositions made on the basis of clay (a and c) and cement (b and d)

the mechanism of material destruction due to a probable deposition of hydration products on monofilaments, to densify and strengthen the interfacial transition zone. In particular, a presence of fibers in the cement stone composition does not affect its average density and does not contribute to the growth of water absorption. However, it has an impact on the technological damage of the composite, because the coefficient K_D determined on beam samples is more than 10% higher than the K_D of non-reinforced stone.

Conclusions. Previous knowledge of the concrete microstructure allows for better understanding of its mechanical behavior under an influence of operational loads. An introduction of linear particles into the material composition changes the structure formation mechanism of the system at the microlevel. Depending on the characteristics of the structural components of the dispersed system, substructures are formed which differ in the periods of their formation and geometric parameters. The use of highly dispersed thin fibers improves structural, physical and mechanical properties of cement stone.

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СТРУКТУРОУТВОРЕННЯ ДИСПЕРСНО-АРМОВАНИХ БУДІВЕЛЬНИХ КОМПОЗИТІВ

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Анотація. У статті наведено результати дослідження механізму структуроутворення цементних композицій, армованих високодисперсним моноволокном. Механізм організації мікроструктури будівельних композитів вивчався на моделях дисперсних систем, з різним якісним і кількісним складом лінійних й дисперсних часток. Водночас введено обмеження щодо розміру часток – діаметр волокон і діаметр дисперсних часток є співрозмірними між собою. Дослідження кінетики тріщиноутворення виконувалось на зразках у формі дисків, виготовлених з глиняно- і цементно-водних композицій. Фізико-механічні характеристики дисперсно-армованого цементного каменю, зокрема й неармованого, визначено на зразкахпризмах квадратного перерізу розміром 40×40×160 мм.

Проведений аналіз фізичних моделей показав, що наповнення кластерних структур різноманітними за природою і формою частками збільшує структурне різноманіття всієї дисперсної системи. Введення лінійних часток змінює характер структуроутворення системи. В залежності від характеристик структурних складових системи утворюються підструктури, що відрізняються періодами свого формування та геометричними параметрами. Встановлено, що дисперсні частки різної природи по-різному структуруються в кластери з дискретними волокнами різної довжини. Більш активними в створенні структурних агрегатів (кластерів), в порівнянні з дисперсними зернами, були лінійні частки.

Вплив високодисперсних волокон на організацію структури в'яжучих композицій кількісно підтверджено коефіцієнтом пошкодженості, визначеним на зразках різних типів. Присутність в складі матеріалу дискретних волокон призводить до зміни якісної характеристики тріщиноутворення композицій. Покращення фізико-механічних властивостей дисперсно-армованого композиту підтверджує здатність фібри змінювати механізм руйнування матеріалу за рахунок ймовірного осадження продуктів гідратації на моноволокнах, ущільнювати й зміцнювати міжфазну перехідну зону.

Ключові слова: структура бетону, дисперсна система, кластер, дисперсна частка, тріщини, волокно.

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Аннотация. В статье представлены результаты исследования механизма структурообразования цементных композиций, армированных высокодисперсным моноволокном. Механизм организации микроструктуры строительных композитов изучался на моделях дисперсных систем, с разным качественным и количественным составом линейных и дисперсных частиц. При этом, введены ограничения по размеру частиц – диаметр волокон и лиаметр лисперсных частиш соразмерны межли собой. Исследование кинетики трещинообразования выполнялось на образцах в форме дисков, изготовленных из глиняно- и Физико-механические цементно-водных композиций. характеристики дисперсноармированного цементного камня, в том числе неармированного, определены на образцахпризмах квадратного сечения размером 40×40×160 мм.

Проведенный анализ физических моделей показал, что наполнение кластерных структур разнообразными по природе и форме частицами увеличивает структурное разнообразие всей дисперсной системы. Введение линейных частиц изменяет характер структурообразования системы. В зависимости от характеристик, структурных составляющих системы, образуются отличающиеся периодами своего формирования и геометрическими подструктуры, параметрами. Установлено, что дисперсные частицы разной природы по-разному структурируются в кластеры с дискретными волокнами разной длины. Более активными в создании структурных агрегатов (кластеров), по сравнению с дисперсными зернами, были линейные частицы.

Воздействие высокодисперсных волокон на организацию структуры вяжущих композиций количественно подтверждено коэффициентом повреждения, определенным на образцах разных типов. Присутствие в составе материала дискретных волокон приводит к изменению качественной характеристики трещинообразования композиций. Улучшение физико-механических свойств дисперсно-армированного композита подтверждает способность фибры изменять механизм разрушения материала за счет вероятного осаждения продуктов гидратации на моноволокнах, уплотнять и укреплять межфазную переходную зону.

Ключевые слова: структура бетона, дисперсная система, кластер, дисперсная частица, трещины, волокно.

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