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Modification of fine-grained concrete with carbon nanotubes

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Abstract: The article considers the possibility of using nanotechnology in the production of concrete. Special attention is paid to the method of administration of nano-sized additives, due to their low concentration in the composition of concrete, as well as their tendency to form aggregates. Experimental data on the selection of a type of plasticizing additive to obtain optimum plastic properties of a concrete mix are presented. Thus, on the basis of the experimental data, the plasticizer SP-3 was selected, which allowed to reduce the water-cement ratio from 0.48 to 0.36 without losing the plasticity of the mixture. Data on the development of the composition of nanomodified fine-grained concrete by introducing Taunit-M carbon nanotubes in the amount of 0.01–0.001 % by weight of the binder are also presented. Two methods of introducing carbon nanotubes are considered, namely the technology of ultrasonic dispersion and the use of a vortex layer apparatus. The possibility of combining the two technologies to introduce a complex additive into concrete is considered. The greatest increase in strength (up to 26 %) was achieved when nanotubes were introduced into the mix using a linear induction rotator together with the introduction of a plasticizer into the water using an ultrasonic disperser.

Keywords: nanomodified concrete; carbon nanotubes; linear induction rotator; ultrasonic dispersion; superplasticizer; compressive strength; bending strength.

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Модифицирование мелкозернистого бетона углеродными нанотрубками

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Аннотация: Рассмотрена возможность использования нанотехнологий в производстве бетона. Особое внимание уделено способу введения наноразмерных добавок, из-за их малой концентрации в составе бетона, а также их склонности к образованию агрегатов. Представлены экспериментальные данные по подбору вида пластифицирующей добавки для получения оптимальных пластических свойств бетонной смеси. На основе экспериментальных данных подобран пластификатор СП-3, который позволил понизить водоцементное отношение от 0,48 до 0,36 без потери пластичности смеси. Показаны данные по разработке состава наномодифицированного мелкозернистого бетона путем введения углеродных нанотрубок Таунит-М в количестве 0,01…0,001 % по массе вяжущего. Рассмотрено два метода внесения углеродных нанотрубок, а именно технология ультразвукового диспергирования, применение аппарата вихревого слоя и совместное применение двух технологий для комплексной добавки в бетон. Наибольшее повышение прочности (до 26 %) достигалось при введении в смесь нанотрубок с помощью линейно индукционного вращателя, совместно с введением в воду затворения пластификатора с помощью ультразвукового диспергатора.

Ключевые слова: наномодифицированный бетон; углеродные нанотрубки; линейно индукционный вращатель; ультразвуковое диспергирование; суперпластификатор; прочность при сжатии; прочность при изгибе.

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1. Introduction

Active mineral additives and superplasticizers occupy a special place among the available additives for concrete preparation. The analysis of literature data [1, 2] shows that the most effective additives are cement stone modifiers, which have a similar crystallo-chemical structure to inorganic nanoparticles (e.g. SiO_2). However, modification with such nanoparticles is difficult to apply as it is difficult to distribute the additive uniformly throughout the material. For this purpose, such additives can be used as a suspension with water for binder hydration [3–5]. In addition, there is the problem of distributing nanoscale additives uniformly throughout the volume of the mix. This is due to the fact that such additives are introduced in tenths and hundredths of a percent by weight of other components of the mix. In this context, the search for the most efficient and at the same time cost-effective methods of nanomodified concrete is an urgent task. The development of technologies for obtaining nanomaterials with different properties dictates the need to study the effect of introduced nanoparticles on the properties of silicate systems [6–9]. Problems related to the controlled improvement of structural concrete properties such as compressive and flexural strength, waterproofing, frost resistance, etc. [10, 11], as well as the effect on the performance of other building materials including binders (cement, gypsum, lime) are of considerable interest. There are experimental data on the use of fullerenes in the production of cinder and aerated concrete blocks, according to which the strength of conventional blocks increases by 16-18% and their density decreases by 8–10 % at a concentration of fullerene-like compounds equal to 1-10 g per ton of concrete. It also leads to a reduction in the production cycle [12].

Strotsky et al. [13] showed that in concrete production, the introduction of carbon nanoparticles with sizes ranging from 10 to 50 nm in the amount of 0.004 wt. % with respect to cement improved the effect of silica microadditive (8 wt. %) and increased the compressive strength of concrete up to 104.5 MPa. It also resulted in a significant increase in Young's modulus, Poisson's ratio, density and decrease in water permeability of concrete. The increase in concrete shrinkage, which was up to 30% with a single microsilica additive, disappeared.

Nanomodified concretes are essentially composed of the same materials as conventional concrete mixes, but their composition is chosen to ensure durability through the formation of new structural properties of the material. Concretes with improved performance characteristics are called high-performance concretes. Research in this direction is being carried out in Germany, Japan, Norway and Switzerland [15–19].

In Galinovsky et al. [20] the introduction of carbon structures into concrete mixing water is considered. Carbon nanotubes (CNTs) are an example of such additives. They are layers of graphene coiled into cylinders with lengths from 1 to 100 μm and different diameters. CNTs have improved mechanical properties [21, 22]. Depending on the number of graphene layers, there are both single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs). SWCNTs are strips of graphene sheets and have a diameter of 6–13 nm. MWCNTs have a larger number of these layers and their inner diameter varies between 6–16 nm, while their outer diameter is up to 100 nm. Due to their very small size, CNTs are subject to van der Waals forces, which cause them to agglomerate. This disadvantage of nanoscale additives requires methods to introduce them into the material composition. The most common methods are ultrasonic dispersion of such particles (ultrasonic dispersers, cavitators and ultrasonic baths), mechanical grinding and the combination of these technologies with the use of superplasticisers. There are many examples of the use of these methods in the scientific literature [23, 24]. We also previously considered the possibility of using nanomodifying additives, namely carbon nanotubes, where an increase in strength of over 15 % was observed [25, 26].

The aim of the present work is to study the effect of the introduced complex admixture in concrete containing carbon nanotubes and superplasticiser on the physical and mechanical properties of concrete and concrete mix.

2. Materials and Methods

2.1. Initial materials

For all conducted experiments the test beams of 40×40×160 mm size were made. The following materials were used for laboratory tests: cement Eurocement of M500D0 grade (JSC Cemros, Moscow); quartz sand with a grain size modulus of 1.9; plasticising additives: superplasticisers under trade mark “Linamix SP-180”, “Polyplast Premium”, “Aeroplast”, “Relamix T2”, “C-3”, “Polyplast SP-3”, “PFM NLK”, “POLYPLAST SP-4”, “Polyplast SP Sub”. All additives were introduced into the concrete mix in the form of suspension together with mixing water, in the amount of 0.5 wt. % of cement weight.

CNTs "Taunit-M" (NanoTechCenter LLC, Tambov, Russia) were used as a nanomodifier. CNTs are hollow carbon tubes with an outer diameter of 10–30 nm, inner diameter of 5–15 nm and length up to 2 μm.

2.2. Equipment for making and examining concrete samples

To investigate the mechanism of additives influence on the performance characteristics of fine-grained concrete, laboratory studies were carried out using the following equipment:

– ultrasonic device with visualization "Pulsar-1.2" (Interpribor LLC, Russia). It was used for the determination of strength characteristics by non-destructive method, including the early hardening of concrete;

– scanning electron microscope (SEM) "Versa 3d" (FEICompany, USA) was used to study the structure of nanomodified concrete;

– ultrasonic dispersant "UZG13-0,1/22" (Techcentre, Russia) was used for the introduction of additives into the mixing water;

– linear induction rotator "LIV-2" (Manufacturer – Russia) was used for introduction of carbon nanoadditives into dry mix, as well as for the activation of cement binder.

2.3. Preparation of the samples

One of the most important properties of a concrete mix is its mobility or plasticity, as this factor affects the technical and economic characteristics of the concrete works. This parameter is directly influenced by the amount of water added to the mix. Concrete mixes have their own water-holding capacity, which is determined by experimentation. It is well known that the introduction of more water leads to an increase in the porosity of the material and therefore a decrease in strength. In order to maintain strength with the same amount of water, different plasticizer additives are used. A group of plasticizers was selected for this study. The effect of each plasticizer was determined by Suttard viscometer tests. In order to study the strength properties, test beams of 40×40×160 mm were prepared with the following composition C – 500 g; sand – 1500 g; W/C – 0.36; plasticizer – 0.5 % by weight of the binder; CNT – 0.01–0.001 % by weight of the binder. The strength properties of the test specimens after 7, 14 and 28 days were investigated using the "Pulsar 1.2" device. The flexural strength of the 28-day samples was determined in accordance with Russian Standard 10180.

3. Results and Discussion

3.1. Selection of a plasticizer

The first part of the study aimed to determine the optimum performance characteristics of the concrete mix. Plasticizers were selected for the development of complex modifying additives in concrete. The results show that plasticizers have selective effects on different types of cement, due to the mineral and chemical composition of the binder on water-soluble additives. In this work, the water-cement ratio was determined experimentally in order to obtain a mix with optimum mobility. Samples were taken from each mix under normal curing conditions.

After 28 days, each sample of fine-grained concrete was tested for compressive and bending strength using a non-destructive control method with the ultrasonic device "Pulsar 1.2". The results of the research are presented in Table 1.

Table 1. Characteristics of the studied compositions using various plasticisers

Plasticiser	W/C	The blurring of the cone, mm	Density, kg·m^{-3}	Compressive strength, MPa
–	0.48	106	2220.7	53.7
SP-4	0.36	108	2277.3	68.1
PolyplastSP-3	0.36	113	2235.6	67.8
C-3	0.36	106	2281.3	64.5
PFM NLK	0.36	107	2250	62.9
Linamix SP-180	0.36	110	2286.3	65.4
Polyplast Premium	0.36	111	2264.5	66.4
Aeroplast	0.36	109	2234.4	60.7
Relamix 2T	0.36	107	2217.6	68.7
Polyplast SP Sub	0.36	109	2235.6	65.9

The standard cone blur method was used to determine the plasticizing effect of the additives on the mix. The best effect was shown by the plasticizer "Polyplast SP-3". At the same time, the fine-grained concrete samples with the superplasticizers "Relamix 2T", "SP-4" and "Polyplast SP-3" had higher strength. The increase in strength in comparison with the reference composition was 22, 21.3 and 20.6 %, respectively.

The obtained results of experimental studies confirm that the most effective plasticizers are those based on polycarboxylates at the same quantity of the added additive. Such plasticizers are less sensitive to the composition of the concrete and the type of cement binder.

The study of the obtained compositions shows a positive plasticizing effect. It should be noted that the additive "Polyplast SP-3" showed the best performance. Thus, the introduction of 0.5 % of the additive by weight of cement made it possible to reduce the water-cement ratio by 25 %, which ultimately led to an increase in strength.

3.2. Determining the efficiency of CNT introduction by ultrasonic dispersion

After selecting the superplasticizer, it was decided to use additional modifying additives, namely CNTs "Taunit-M" in small quantities (0.001–0.0001 wt. % by weight of the binder).

A staged series of tests was prepared for the research. The composition of fine-grained concrete including cement, sand and water was chosen as a control composition. The proportions of the components were chosen on the basis of optimum mobility of the mix. For additional analysis, a mix containing the plasticizer "Polyplast SP-3" was also prepared. The plasticizer was introduced in two ways: by mechanical stirring in the mixing water and by ultrasonic dispersion using the USG13-0.1/22 device. In this case, the use of ultrasonic treatment allowed the amount of plasticizer to be reduced from 0.6 to 0.5 wt. % by weight of the binder in the same mixes. On the basis of the literature data, it was decided to prepare three other mixtures with the use of plasticizer together with carbon nanotubes "Taunit-M" in the amount of 0.003, 0.004, 0.005 wt. % by weight of cement. The plasticizer and carbon nanotubes were introduced together into the mixing water under ultrasonic action for 5 minutes. Table 2 below shows the composition of the samples tested.

Table 2. Compositions of the studied mixes

Composition	Polyplast SP-3, g	CNTs Taunit-M, g	R_{com}, MPa	R_{bend}, MPa
1 (control)	–	–	27.4	5.53
2	3.0	–	35.6	5.86
3	2.5	–	35.2	6.43
4	2.5	0.003	37.4	6.53
5	2.5	0.004	39.7	6.63
6	2.5	0.005	40.2	6.80

Based on the laboratory test done, results were also obtained for the strength characteristics of the samples for each composition at 28 days of age (Figs. 1 and 2). It was found that the use of plasticizer increased the strength by 16 % both in compression (R_{com}) and in bending (R_{bend}). At the same time, however, the ultrasonic influence during the introduction of the plasticizer made it possible to reduce the amount of plasticizer additive without losing the strength properties (Compositions 2, 3).

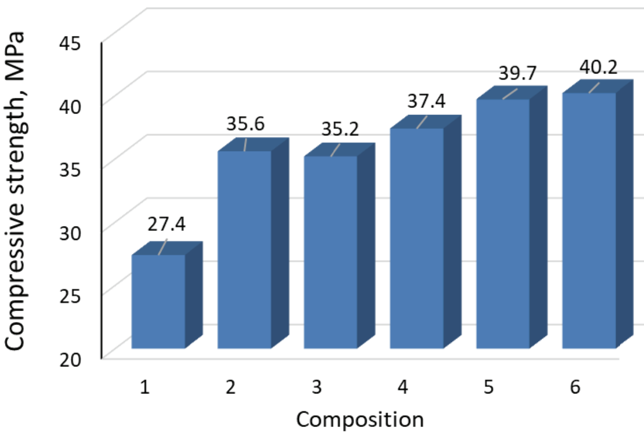


Fig. 1. Compressive strength test

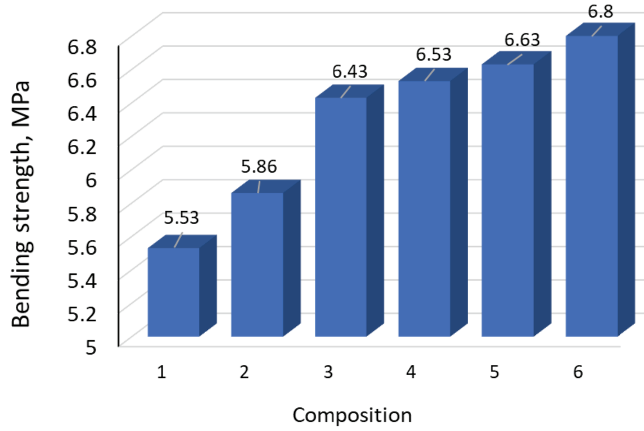


Fig. 2. Bending strength test

Table 3. The studied compositions and strength characteristics

Composition	SP-3, g	CNTs, wt. % by weight of cement	R_{com}, MPa		
			7 days	14 days	28 days
1 (control)	2.5	–	36.5	39.0	40.8
2	2.5	0.001	39.3	40.8	45.5
3	2.5	0.002	40.0	40.8	43.6
4	2.5	0.003	39.7	42.5	47.2
5	2.5	0.004	41.1	42.7	45.8
6	2.5	0.005	41.8	43.6	47.6
7	2.5	0.006	41.4	44.5	46.7
8	2.5	0.007	41.7	44.0	47.6
9	2.5	0.008	41.2	43.4	47.2
10	2.5	0.009	40.8	43.4	47.1
11	2.5	0.01	43.2	44.3	48.4

The use of a complex additive of plasticizer and CNTs increased the strength as the concentration of nanotubes increased. The introduction of carbon nanotubes increased the strength in the range of 26 to 31 %. It can be concluded that further tests are needed to determine the rational amount of additive with increasing concentration of the introduced additive.

Further, to study the effect of CNTs nanoadditive on the strength characteristics of fine-grained concrete, another series of tests was prepared from 11 compositions of C:S – 1:3, W/C – 0.36, SP-3 – 0.5 wt. % by weight of binder and different concentration of CNTs. Carbon nanotubes were introduced in the amount from 0.001 to 0.01 wt. % by weight of cement. The control samples (composition 1) did not include the nanoadditive. The investigated compositions are presented in the table below. For each composition the compressive strength was determined using non-destructive method of control with the device “Pulsar 1,2” at the age of 7, 14, 28 days. The results of the obtained data are given in Table 3.

As can be seen from the data obtained, the introduction of CNTs leads to an increase in the strength of fine-grained concrete. For example, the minimum amount of nanoadditive increased the compressive strength by 10 %. The maximum increase in strength is characteristic of Composition 11 and was 16 %. However, due to the insignificant difference in the strength values, it can be concluded that the introduction of nanotubes “Taunit-M” in the amount of 0.005–0.01 wt. % by weight of the binder has almost the same effect.

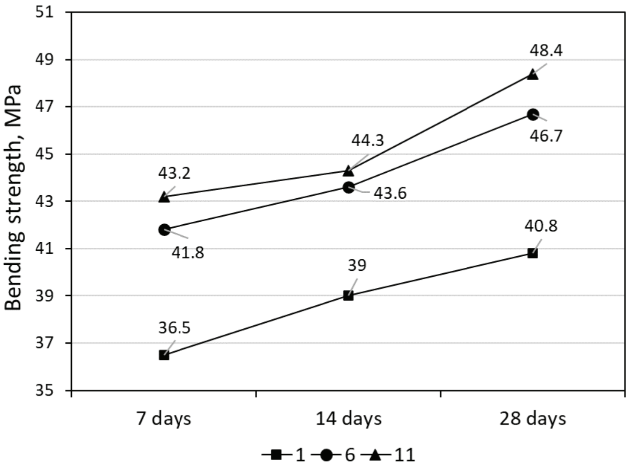


Fig. 3. The character of the strength gain of the fine-grained concrete

It should be noted that the increase in strength is already observed on the 7th day. Thus, for the composition with the maximum concentration of CNTs, the increase was 15 %. It is known that the increase in strength in the early curing period has a positive effect on the production processes associated with the manufacture of concrete products.

As can be seen in Fig. 3, the samples modified with nanoadditives showed an accelerated intensity of strength increase compared to the control composition.

3.3. Study of the method of CNTs introduction using a linear-induction rotator

In this work, the method of introduction of carbon nanostructures using a vortex layer apparatus was also investigated. A series of samples were prepared in which carbon nanotubes were introduced into the cement binder using a linear induction rotator (LIR). For comparison, one series was prepared by ultrasonic dispersion technique. The preparation technology was as follows: cement binder and sand (C:S – 1:3) were jointly loaded into the LIR chamber, together with CNTs and ferromagnetic grinding bodies. The components were processed in the apparatus for 2 min. Plasticizer was introduced into the mixing water. All components were mixed to obtain a homogeneous mixture and samples were molded. Thus, four compositions were prepared including a control one without CNTs and three compositions with inclusion of nanoadditive in different concentrations. The compositions with the obtained characteristics are shown below in Table 4.

According to the data in the table, it is clear that the introduction of CNTs by LIR technology allows obtaining an insignificant increase in strength. The compressive strength increased in the case of ultrasound by 14 %, and when using LIR – by 15 %. This is explained by an additional effect of vortex influence, namely, activation of concrete mix components by grinding with grinding bodies.

3.4. Method of introduction of the complex additive

The last stage of the research was the joint application of the two technologies. The introduction of the complex additive was carried out sequentially in two stages: 1) introduction of a plasticiser by dispersion in mixing water; 2) introduction of CNTs into cement binder in LIR chamber. After treatment, all components were mixed for further moulding of the samples. After 28 days, the compressive and flexural strength limits were determined for each sample (Table 5).

3.5. Study of the structure of nanomodified concrete

In order to study the morphology of the nanomodified concrete, the samples destroyed during the tests were crushed to a powdery state, and a small amount of them were placed in a microscope chamber.

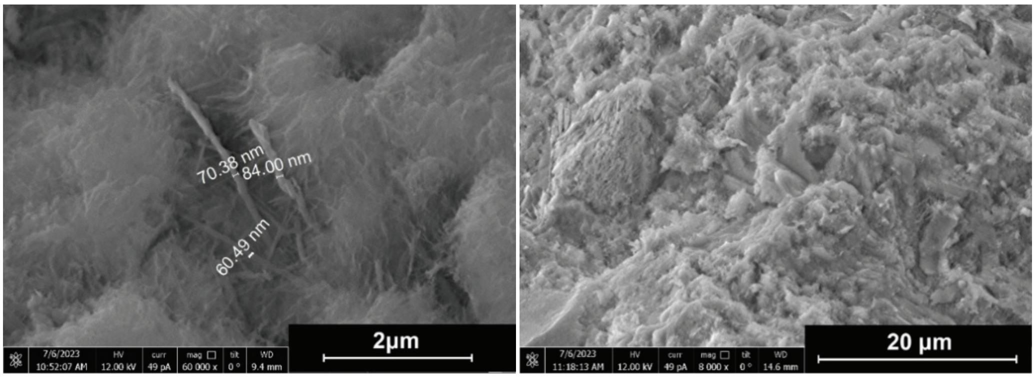
As shown in the images (Fig. 4*a*, *b*), the carbon nanotubes have a stable diameter value along the length of the fiber. This provides good conditions for the growth of cement stone around the carbon inclusions. Concrete modification at the nanoscale is evidenced by the discrete reinforcement of the cement matrix, where ettringite minerals together with nanotubes (Fig. 4*a*) allow the new formations to be combined into a single structure. The nanotube modified concrete has a dense arrangement of particles, resulting in an increase in strength properties.

Table 4. Compositions of the studied mixtures of fine-grained concrete

Composition	CNTs, wt. % per g	SP-3, g	LIR R_{com}, MPa	Ultrasound R_{com}, MPa
1	–	3	40.8	40.8
2	0.004/0.020		45.9	47.9
3	0.005/0.025		47.7	48.1
4	0.006/0.030		46.8	47.2

Table 5. Strength characteristics of the studied samples

Composition	CNTs, wt. %	R_{com}, MPa	R_{bend}, MPa
1	–	53.6	6.8
2	0.0001	65.0	8.1
3	0.0002	65.8	8.2
4	0.0003	67.0	8.2
5	0.0004	66.1	8.6
6	0.0005	68.2	9.0
7	0.0006	67.0	8.7
8	0.0007	66.2	8.4
9	0.0008	67.1	8.6
10	0.0009	67.4	8.7
11	0.0010	68.6	9.0

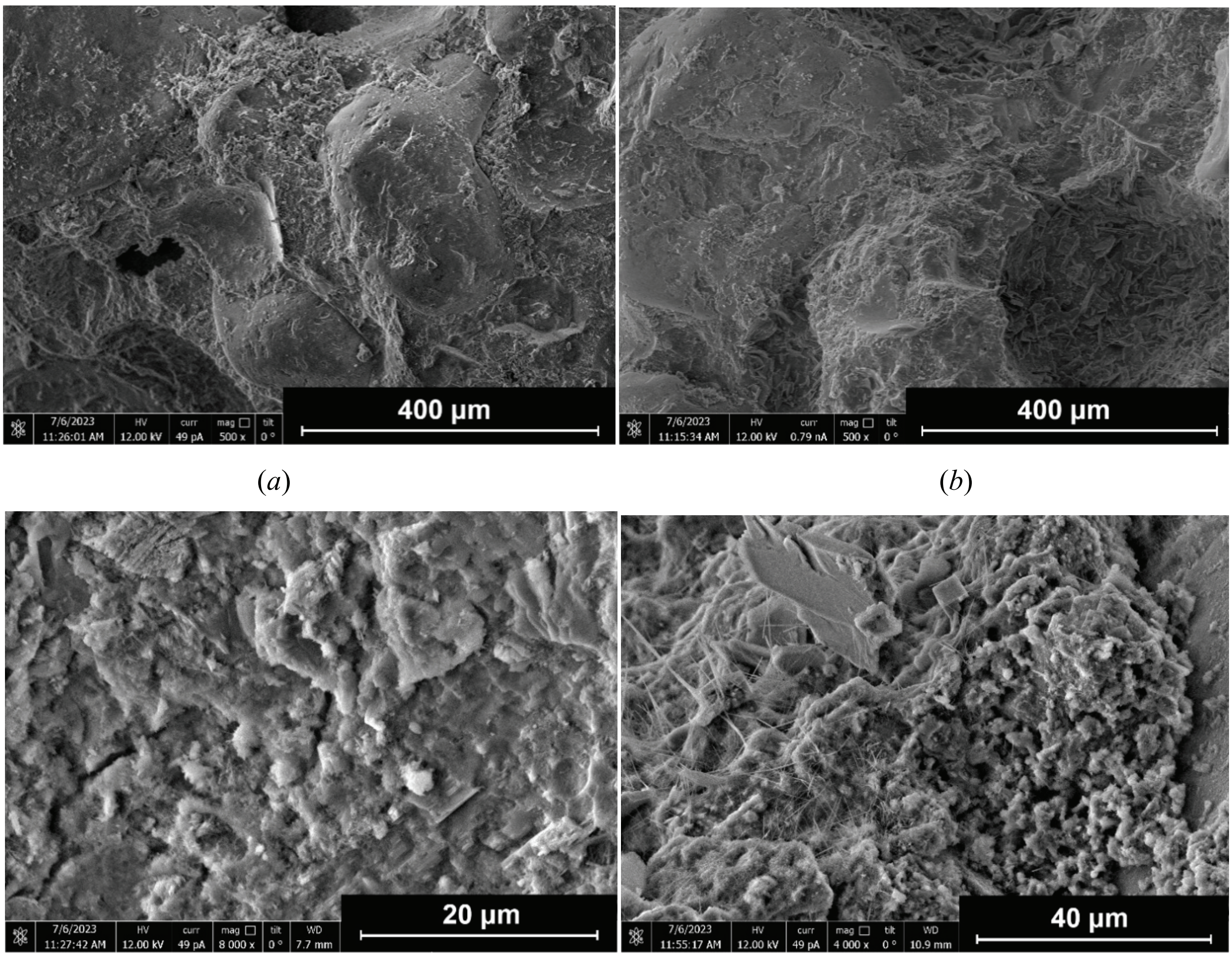


(*a*) (*b*)

Fig. 4. SEM-images of nanomodified concrete: *a* – magnification 60.000×; *b* – magnification 8.000×

The experimental data show that the maximum increase in strength is achieved at the maximum concentration of CNTs (Composition 11). This was 27 %. It should be noted that a slight difference in strength increase was achieved in composition 6 (26 %). From this it can be concluded that it would be more rational to use a lower amount of additive when selecting the concrete composition.

The microscope was also used to study the structure of samples obtained from the destruction of beams on presses. These are particles of fine-grained concrete (up to 5 mm in size).



(*a*) (*b*)

(*c*) (*d*)

Fig. 5. SEM-images of the nanomodified samples:
a, *b* – control sample; *c*, *d* – nanomodified sample

The use of the nanostructured CNTs "Taunit-M" additive results in a denser and stronger concrete structure (Fig. 5), which is the reason for the increase in strength properties.

4. Conclusion

According to experimental studies, the introduction of carbon nanotubes "Taunit-M" leads to an increase in compressive and bending strength. An insignificant amount of the introduced additive (0.0001 wt. % by weight of the binder and higher) together with the plasticizer allows to noticeably increase the strength properties. It should be noted that there is an insignificant difference in the increase of strength characteristics when using from 0.0005 to 0.001 wt. % of CNTs by weight of cement. On this basis, it can be concluded that there is an optimal amount of the additive, regardless of the method of its introduction into the concrete composition. At the same time, it should be noted that the use of LIR allows slightly higher strength values to be obtained due to the additional grinding of the dry components. The study of the morphology of the interpore partitions of concrete using electron microscopy shows that the samples modified with nanotubes have a denser structure, which increases the strength properties. The micrographs show the inclusions of nanotubes, whose reinforcement can reduce the formation of nanoscale cracks and increase the durability of modified fine-grained concrete.

5. Funding

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6. Conflict of interests

The authors declare no conflict of interests.

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