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REPAIR MORTARS OBTAINED BY PLASMA MODIFICATION AND VORTEX ACTIVATION

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Abstract

Introduction: The service life of reinforced-concrete structures can be increased with the use of effective repair compositions obtained by activating the original components. **Purpose of the study:** We aimed to develop effective repair compounds obtained by activating the original components. **Methods:** To process the original components, low-temperature non-equilibrium plasma (LTNP) and electromagnetic activation in a vortex layer device were used. In the course of the study, we used polypropylene, steel, glass, and basalt fiber and fiber made of structured ferromagnetic microwire. Electron microscopy and X-ray diffraction analysis were applied. **Results:** It was established that the combined use of the above methods for the modification of raw components makes it possible to improve the strength of these materials by more than 50%, which is due to the characteristics of structure formation in the developed compositions. For instance, LTNP increases the amount of portlandite and reduces the main phases of cement stone — C_3S and β-C_2S, and vortex activation contributes to an increase in the total number of crystalline phases. Quartz powder particles processed in an electromagnetic mill are characterized by layered structure, high surface roughness, large developed cracking, as well as inclusions as a result of impact action. All that improves the physical and mechanical properties of the resulting repair compositions by an average of 20%. Repair compositions additionally treated with plasma modification feature new hydrated formations on quartz grains.

Keywords
activation, fiber, plasma, vortex layer.

Introduction

Improving performance of fine-grained cement concretes and repair mortars (hereinafter referred to as repair compositions) is one of the actively developing areas in construction material engineering (Chun et al., 2022; Teixeira et al., 2019). The analysis of scientific and technical literature showed that the handling ability as well as the physical and mechanical properties of building composites based on Portland cement can be improved by the following methods:

- activation of raw components:
  - mechanical, chemical, mechanochemical, and plasma activation of Portland cement (Fediuk, 2016; Ibragimov et al., 2019; Sun et al., 2021);
  - mechanical, magnetochemical, electrochemical, and plasma activation of mixing water (Fedosov et al., 2017);
  - mechanochemical and plasma activation of mineral filler and/or fine aggregate (Abbas et al., 2023; Kalyadin et al., 2019);
- dispersed reinforcement of concretes and repair compositions (metal, natural (glass and basalt), polymer (polypropylene or glass composite), vegetable (cellulose) fiber or organic (carbon) fiber) (Fediuk et al., 2017; Ruslan et al., 2022);
- the use of nano-additives of various chemical nature (Khuzin and Ibragimov, 2021);
- combination of the methods listed above to improve the performance of building composites (Ma et al., 2017).

Mechanochemical (or mechanical) activation of the binder and fine aggregates is widely used to improve the performance of building composites. Mechanochemical activation of Portland cement not only increases its specific surface area but also changes the structure of the surface layer of Portland cement particles, promotes its amorphization (Chun et al., 2022). To ensure such Portland cement activation, vortex layer devices (Ibragimov and Korolev, 2022) and vibration mills of various designs or high-energy ball milling (Khamatova et al., 2017) are most often used. During activation, the shape of Portland cement particles changes from angular to a more rounded one. At the same time, the milling fineness of Portland cement increases, which leads to a significant increase in compressive strength (up to 49%) and tensile strength in bending (up to 26%) of sand-cement mortars without increasing their water-cement ratio (in equally easily workable mixes) (Khozin et al., 2021).

Silica-containing components (fillers, fine aggregate) activated in centrifugal planetary mills and vortex layer devices play a significant role in the structure formation of cement composites. Plasma modification of raw components (Portland cement, quartz sand, and mixing water), used to improve the performance of cement composites, in particular, repair compositions, is of particular interest (Kalyadin et al., 2019). The shape of quartz sand particles after processing is quite important since it affects the hydration parameters and the structure formation of cement stone. This issue shall be studied more thoroughly (Li et al., 2022).

To obtain repair compositions with low shrinkage, high physical and mechanical properties and crack resistance, fibers of various chemical nature and size are used: metal (steel), polymer (polypropylene, polyethylene terephthalate, or glass composite), mineral (glass or basalt), and vegetable (cellulose). The effectiveness of the fiber depends on its chemical nature, and, consequently, its adhesion to cement stone, fiber diameter and length, volume content, and its distribution in the mineral matrix (Feng et al., 2020; Yang et al., 2020).

One of the effective methods to obtain repair compositions is a combination of low-temperature non-equilibrium plasma or vortex activation and effective fiber reinforcement. In this regard, the purpose of our study is to obtain highly effective repair compositions with application of low-temperature non-equilibrium plasma or vortex activation and fiber modification.

Subject, tasks, and methods

To prepare the compositions, we used Portland cement CEM I 42.5 N, corresponding to GOST 31108, and fractionated quartz sand of class I, corresponding to GOST 8736 with a content of dust-like and clay particles of less than 3% by weight.

Polypropylene, steel, glass, and basalt fibers as well as fiber made of structured ferromagnetic microwire were used as the fiber. Its physical and mechanical properties are shown in Table 1. To obtain the fiber, we used alkali-resistant glass fiber manufactured by Owens Corning (Spain), basalt fiber manufactured by Alyans-Strointelnie Tekhnologii LLC, and polypropylene fiber manufactured by Fibra Lyuks LLC.

Portland cement, quartz sand, and water were treated in low-temperature non-equilibrium plasma (LTNP) in a barrier discharge and flow mode, with the use of a laboratory installation and according to the method proposed by Bruyako et al. (2014). Portland cement, gypsum plaster, and limestone were treated in an electromagnetic mill (EM), model 297, manufactured by Regionmettrans LLC. In their paper, Ibragimov et al. (2019) presented a standard design of a vortex layer device.

The samples of repair compositions hardened under normal physical conditions: relative humidity — 100%, temperature — 20 ± 2°C. The strength of the samples in compression, bending and tension was determined according to GOST 5802 in 1, 3, 7, 14, and 28 days of hardening, with the use of an Instron 3382 hydraulic press and a WDW-100E tensile testing machine, and the setting time was determined according to GOST 56587.

The microstructure of cement stone was studied using a Merlin high-resolution field-emission scanning electron microscope by CARL ZEISS. The splits of the cement stone samples were coated with Au/Pd alloy in the ratio 80/20 using a Quorum T150 ES high-vacuum unit.

The X-ray diffraction analysis was carried out using a D2 Phaser diffractometer manufactured by Bruker (Germany) to study raw materials and new formations in the structure of hardened cement stone in the Bragg–Brentano geometry with application of monochromatic CuK αradiation (λ = 1.54178 Ǻ), in step scanning mode.

To evaluate the effectiveness of the developed repair composition, industrial construction mixes of such brands as Mapegrout Thixotropic, Structurite 100, and CarbonWrap® Repair ST, manufactured by Mapei, Thoro, and NTsK LLC, were used.

The repair compositions were prepared in accordance with GOST 58277. The consistency of

Table 1. **Physical and mechanical properties of fiber used in the course of study**

Fiber	Dimensions		Density, kg/m^3	Tensile strength, MPa	Relative elongation at break, %	Modulus of elasticity in tension, GPa
	Length, mm	Diameter, μm				
Steel	15	300	7800	1870	3.6	200.6
Glass	6	14	2680	2500	2.5	72.4
Basalt	6	16	2670	2200	2.5	76.9
Polypropylene, grade VSM 6 (0.6)	6	20	910	240	212	3.8
Fiber made of structured ferromagnetic microwire	10	15.2	7300	3500	3.2	154.7

mortar mixes was evaluated in accordance with GOST 5802. The bond strength of the repair compositions with concrete was determined according to GOST 58277, frost resistance — according to GOST 10060, water resistance — according to GOST 12730.5.

Results and discussion

Mechanical and thermal action in an EM destroy the crystalline structure of cement clinker minerals, which is manifested in reduced coherent scattering regions. The most significant changes are observed for C_3S (the average size of crystallites decreases by 21%), C_2S (by 18%), and periclase (by 29%); the average size of C_3A cubic and C_4AF crystallites remains virtually unchanged. Table 2 shows the analysis of the X-ray diffraction patterns of cement stone obtained by treating Portland cement with LTNP.

Under the action of low-temperature plasma, the crystal hydrate layer on the surface of Portland cement particles is destroyed and water evaporates from their surface. This is confirmed by a large difference in the hardening rates of aged and partially hydrated Portland cement and Portland cement treated with LTNP.

The action of LTNP in Portland cement treatment (or vortex activation in the EM) changes the mineralogical composition of cement stone studied after 28 days of hardening. LTNP increases the amount of portlandite and reduces the main phases of cement stone — C_3S and β-C_2S, which indicates a more complete hydration of Portland cement. Vortex activation contributes to an increase in the total number of crystalline phases, portlandite, which naturally causes a decrease in the content of the initial phases of Portland cement, increasing the degree of its hydration.

In the course of the study, we analyzed the microstructure of cement stone obtained as a result of Portland cement treatment with LTNP (Fig. 1). The influence of Portland cement activation on the structure of cement stone is characterized by such a distinctive feature as new crystalline formations with far less dispersion. The new formations crystallize in a more finely dispersed form, smaller pores and capillaries of cement stone are formed. Together with the higher degree of hydration, this leads to the formation of a dense structure of cement stone, which naturally has a higher strength.

Table 2. **Mineralogical composition of cement stone based on the initial Portland cement and Portland cement treated with LTNP**

Phase composition of cement	Phase content in Portland cement, % wt.		
	Initial cement	Cement treated with LTNP	Cement treated in the EM
Ca_3SiO_5 (C_3S)	22.7	21.5	19.9
Ca_2SiO_4(β-C_2S)	14.4	14.0	13.8
Amorphous phase	40.0	40.0	38.6
C_4AF	8.5	8.3	7.9
$Ca(OH)_2$	8.5	11.2	12.8
Ettringite	4.0	3.5	5.4
$CaCO_3$	1.9	1.5	1.6

The compressive strength of cement stone during Portland cement treatment with LTNP increases by 16.2–20.4%, and the bending strength of sand-cement mortar increases by 18.1–22.3%. Portland cement activation in the EM makes it possible to increase the compressive strength of cement stone on the first day 2.2 times, on the 28th day — by 35% (Fig. 2).

The treatment of quartz sand grains with LTNP results in a decrease in the total surface area of the grains by 10.6–20.3%. It was established that with an increase in the geometric dimensions of quartz

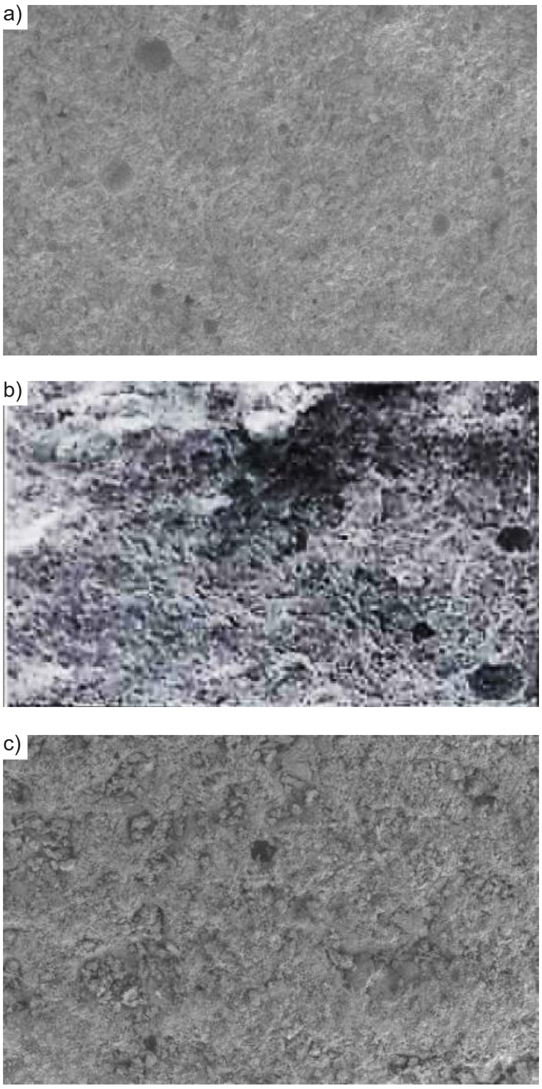

Fig. 1. Microstructure of cement stone (X100 magnification): a — reference composition; b — obtained by Portland cement treatment with LTNP; c — obtained by activation in the EM

sand grains, a more significant decrease in the pore surface area is observed. The latter is associated with sand particle surface melting, which naturally leads to amorphization (Fig. 3b).

Quartz powder particles processed in an electromagnetic mill are characterized by layered structure, high surface roughness, large developed cracking, as well as inclusions as a result of impact action. The treatment in an EM is characterized by the following distinctive feature: when quartz sand particles are milled, the grains are separated along individual conglomerates (Fig. 3c) and not along cleavage planes as with the use of a ball or centrifugal mill, which indicates high EM energy density.

The treatment of mixing water and quartz sand with LTNP, followed by repair composition obtaining, ensures a synergistic effect in strength increasing (Fig. 4).

Fig. 5 shows the microstructure of the obtained repair compositions No. 1 (based on quartz sand two times treated with LTNP and mixing water consisting of a mixture of untreated and plasma-modified water at a ratio of 1:1) and No. 5 (reference composition) with X500 magnification. The data presented in Fig. 5 show that the hydration of the compositions obtained by Portland cement treatment with LTNP differs from that of the reference ones. The compositions additionally treated with plasma modification feature new hydrated formations on quartz grains.

An important parameter of repair compositions is their crack resistance. Rong et al. (2021) as well as Wang et al. (2021) proposed to indirectly evaluate crack resistance by the following ratio: R_{bt}/R_b (R_{bt} — tensile strength, R_b — compressive strength). Table 3 shows how the type of fiber affects the ratio of the samples of the repair compositions obtained by Portland cement activation in the EM.

The data in Table 3 show that the highest crack resistance (value of the ratio) of the repair compositions is observed with the introduction of metal fiber (the indicator increases by 40%). With the introduction of polypropylene fiber, this indicator increases by 27%.

Shen et al. (2020) indicated that the introduction of fiber makes it possible to increase the impact strength of building materials. Also of interest is the study of the effect of polypropylene and metal fibers on the impact strength of the repair compositions obtained by Portland cement activation in the EM (Fig. 6). The data in Fig. 6 show the obvious result of dispersed reinforcement, which is an increase in impact strength. Of practical interest are the abscissas of functions , at which the impact strength as well as its relative increase have the maximum value. For instance, the introduction of

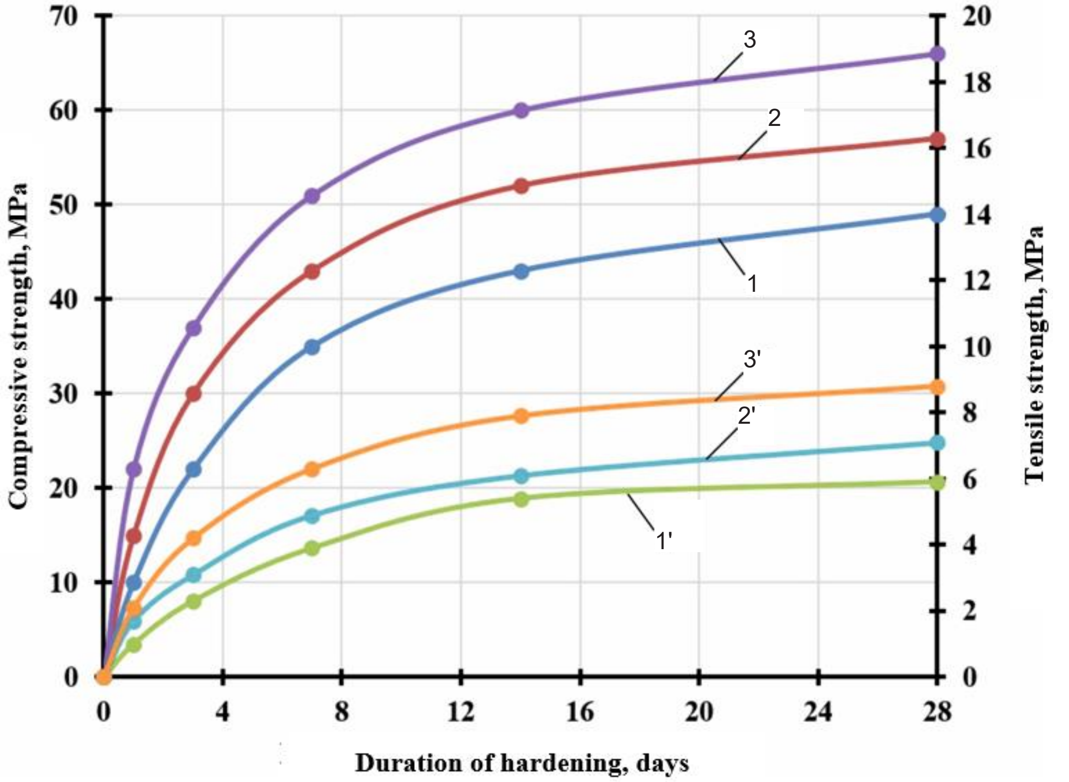

Fig. 2. Compressive strength (1, 2, 3) and bending strength (1', 2', 3') of cement stone vs. duration Portland cement hardening:
1 — reference composition; 2 — treated with LTNP; 3 — obtained by vortex activation

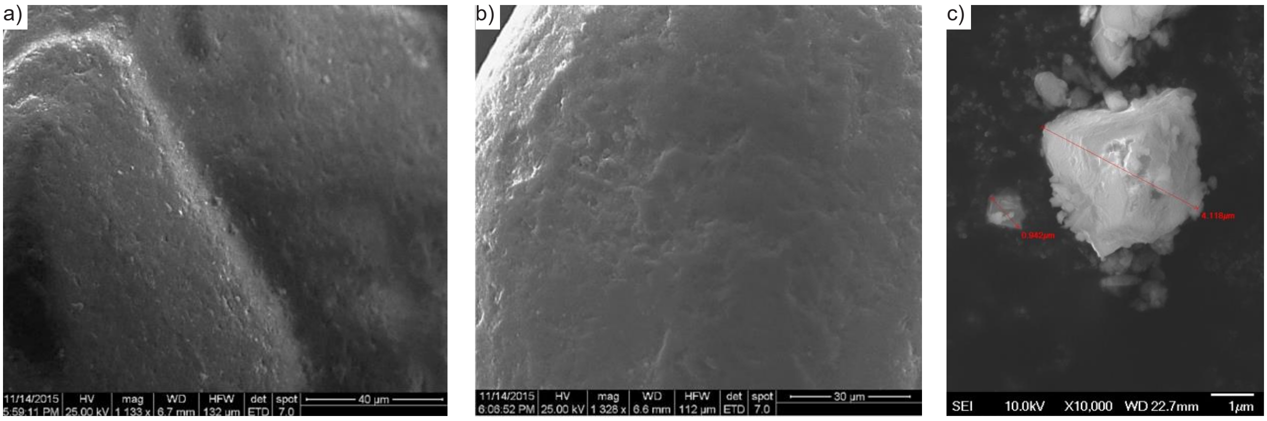

Fig. 3. Photos of quartz sand grains: a — before treatment (X1000) b — after treatment with LTNP (X1300); c — in a vortex mill (X10000)

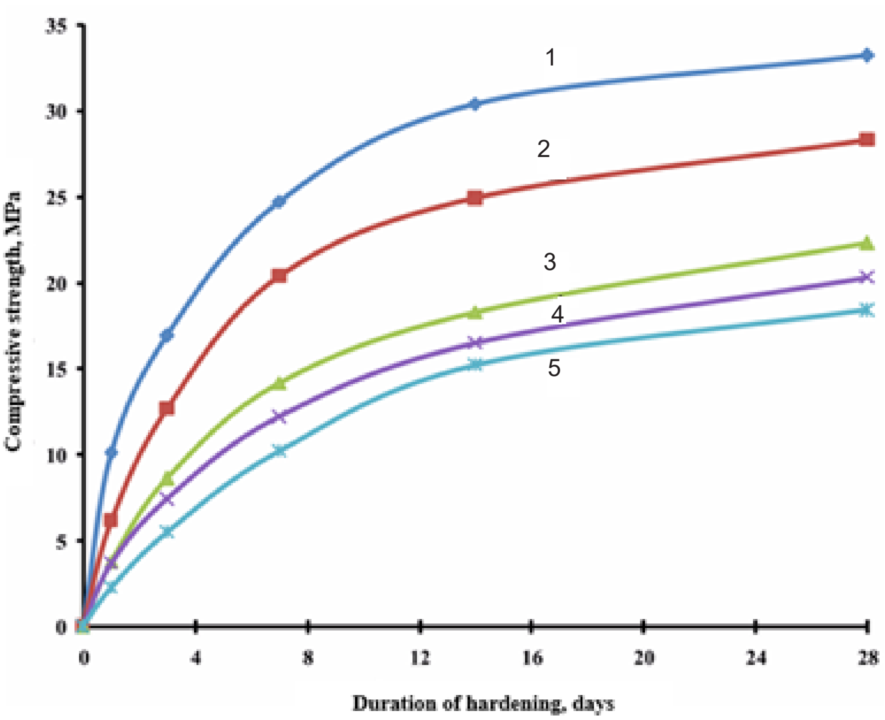

Fig. 4. Dynamics of strength gain in the studied compositions

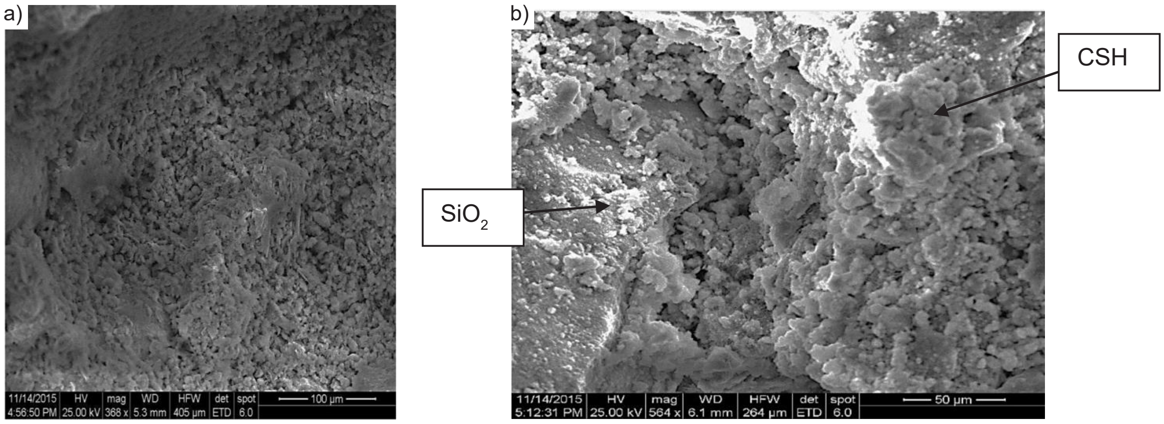

Fig. 5. Microstructure of the repair compositions: a — No. 5; b — No. 1

Table 3. **Ratio for the repair compositions under study**

Fiber type	Geometric characteristics of the fiber		R_{bt}/R_b
	d, mm	L, mm	
Reference composition			0.131
Metal	0.3	15	0.183
Polypropylene	$20\cdot10^{-3}$	6	0.167

polypropylene fiber increases the impact strength 1.72 times at its volume content of 1%, and the introduction of metal fiber — 2.6 times at its volume content of 1.5%. It should be noted that the volume content of fiber , at which is more than . Moreover, for polypropylene fiber, more than for metal fiber and equals 2. Although they noted an increase in impact strength with fiber introduction, Shen et al. (2019) did not show how the ratio depends on the type of fiber.

One of the most important structural indicators of repair compositions is durability. In this regard, the influence of aggressive media on the corrosion resistance of cement stone obtained by Portland cement activation in the EM was studied. As aggressive media, 0.1 N nitric, sulfuric and hydrochloric acids were used. The test results show that Portland cement treatment in the EM allows for the formation of cement stone that is more resistant to the effects of the considered aggressive media, which complements the findings of our colleagues (Shen et al., 2019). The resistance of the repair compositions in aggressive media increases by 11–15%, depending on the medium, which increases the durability of such compositions and their service life.

Table 4 shows the handling ability of the developed repair composition as well as its physical and mechanical properties in comparison with the existing repair compositions. The developed repair composition has steel fiber reinforcement in the amount of 0.4% by volume.

The data in Table 4 show that the developed dispersion-reinforced repair composition obtained by Portland cement treatment with LTNP or activation in the EM outmatches the known industrial repair compositions used to eliminate defects and damages in reinforced-concrete structures.

Conclusions

1. LTNP increases the amount of portlandite and reduces the main phases of cement stone — C_3S and β-C_2S, which indicates a more complete hydration of Portland cement. Vortex activation contributes to an increase in the total number of crystalline phases, portlandite, which naturally causes a decrease in the content of the initial phases of Portland cement, increasing the degree of its hydration. The influence of Portland cement activation on the structure of cement stone is characterized by such a distinctive feature as new crystalline formations with far less dispersion. The new formations crystallize in a more finely dispersed form, smaller pores and capillaries of cement stone are formed.

2. The treatment of quartz sand grains with LTNP results in the amorphization of the surface. Quartz powder particles processed in an electromagnetic mill are characterized by layered structure, high surface roughness, large developed cracking, as well as inclusions as a result of impact action. All that improves the physical and mechanical properties of the resulting repair compositions by an average of 20%. Repair compositions additionally treated with plasma modification feature new hydrated formations on quartz grains.

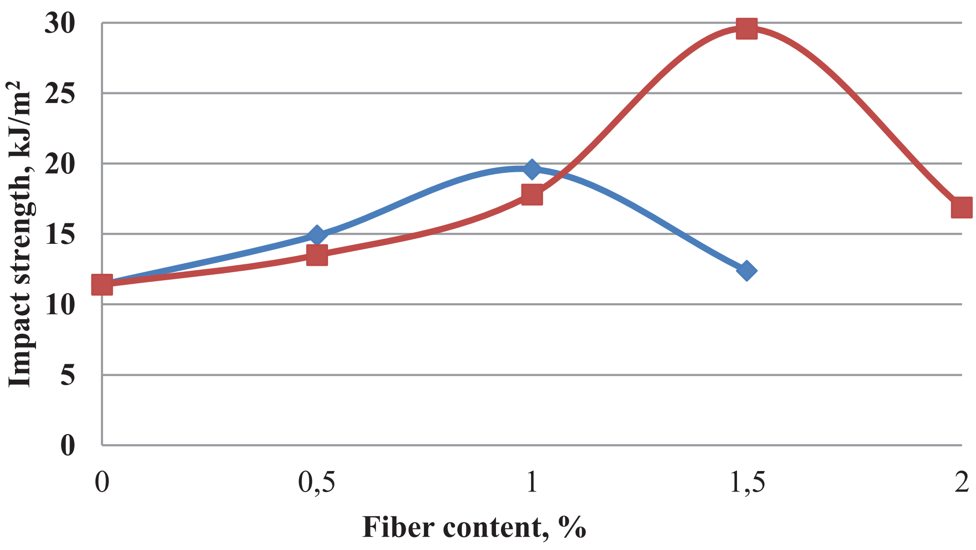

Fig. 6. Impact strength vs. fiber content

Table 4. **Handling ability and physical and mechanical properties of the repair compositions**

Indicator	Industrial repair compositions			Developed repair composition
	Structurite 100	**Mapegrout Thixotropic**	**CarbonWrap® Repair ST**	
Mortar mix consistency	PK 2	PK 2	PK 2	PK 2
Mortar mix properties preservation, min	60	About 60	60	45–50
Maximum filler size, mm	1.5	2.5	2.5	1.5
Mortar mix strength after 28 days of normal hardening, MPa, under: compression bending	65 >7	≥60 ≥8.5	50 ≥8.5	>72 >9.5
Mortar mix adhesion strength after 28 days of hardening, with a concrete base, MPa	>1.5	≥2.0	1.5	>2.8
Frost resistance, number of cycles	200	300	200	300
Watertightness, grade	W10	–	W8	W10

3. Dispersion reinforcement improves the physical and mechanical properties of the developed repair compositions. The value of crack resistance increases with the introduction of metal fiber 40%. With the introduction of polypropylene fiber, this indicator increases by 27%.

4. It was established that the developed dispersion-reinforced repair composition obtained by Portland cement treatment with LTNP or activation in the EM outmatches the known industrial repair compositions used to eliminate defects and damages in reinforced-concrete structures.

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РЕМОНТНЫЕ СТРОИТЕЛЬНЫЕ РАСТВОРЫ, ПОЛУЧЕННЫЕ ПЛАЗМЕННОЙ МОДИФИКАЦИЕЙ И ВИХРЕВОЙ АКТИВАЦИЕЙ

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Аннотация

Введение: Повышение срока службы железобетонных конструкций возможно использованием эффективных ремонтных составов, полученных активацией исходных компонентов. **Цель исследования:** разработка эффективных ремонтных составов, полученных активацией их исходных компонентов. **Методы:** для обработки исходных компонентов применяется низкотемпературная неравновесная плазма (НТНП) и электромагнитная активация в аппарате вихревого слоя. В работе используется полипропиленовая, стальная, стеклянная, базальтовая фибра и из структурированного ферромагнитного микропровода. Применялась электронная микроскопия, рентгенофазовый анализ. **Результаты:** Показано, что совместное применение рассмотренных выше способов модификации сырьевых компонентов позволяет повысить прочность указанных материалов более чем на 50 %, что связано с особенностью структурообразования разработанных составов. Так, воздействие НТНП вызывает увеличение количества портландита и снижение основных фаз цементного камня – $C3S$ и β-$C2S$, а вихревая активация способствует увеличению общего количества кристаллических фаз. Частицы кварцевого порошка, обработанного в электромагнитной мельнице, имеют слоистую структуру, высокую степень шероховатости поверхности, большую, развитую сетку трещин, а также вкрапления в результате ударных воздействий. Указанное повышает физико-механические свойства получаемых ремонтных составов в среднем 20 %. В ремонтных составах, дополнительно обработанных плазменной модификацией, наблюдается скопление гидратных новообразований на зёрнах кварца.

Ключевые слова: активация, фибра, плазма, вихревой слой.