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Mechanical and electrical properties of concrete modified by carbon nanoparticles

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Abstract. The article presents the study of obtaining electrically conductive concrete using carbon nanoparticles obtained by processing coal in an electric-discharge plasma reactor. An electric-discharge method for producing a sedimentation-resistant and highly dispersed suspension consists in treating coal powder with an electric current in an aqueous medium. Using laser diffraction, it was found that carbon nanoparticles have a particle size in the range from 50 to 500 nm. The microstructure of hardened cement paste was studied using SEM. The compressive strength was tested on cubes with an edge of 100 mm according to EN 12390-6. Determination of the electrical resistance of concrete was carried out on specimens with an edge of 100 mm, placed between two brass plates, through which direct current was passed. The optimum content of carbon nanoparticles (0.01–0.1 % wt.) in the binder is evaluated, which allows to obtain high mechanical properties (30–35 % higher compressive strength compared to a control specimen. It is proved that the mechanism of action of nanoscale modifiers is most manifested in small doses. The dependences of the physicomechanical properties of Portland cement upon the addition of various amounts of carbon nanoparticles are determined. Physicomechanical and exploitation properties of heavyweight electrically conductive concrete are determined. The kinetics of changes in the electrical resistance of concrete at different curing periods is established.

1. Introduction

Cement composites with improved electrical properties in recent years have the potential to be used as deformation sensors for structures, as well as heating elements or anodes in various electrochemical methods [1–5].

Traditionally, the production of such concretes was associated with the addition of conductive particles into the concrete mix, such as multilayer carbon nanotubes [6], soot [7-8], iron oxide nanoparticles [9], nickel powders [10], graphite [11]. The use of electrically conductive aggregates, for example, piezoceramics, also reduces the electrical resistance of the composite [12–13].

The use of these materials allowed expanding the range of cheap electric heaters, especially for extended spaces (floors, walls of garages, parking lots and special structures). In addition, knowledge of electrical parameters is useful for developing accelerated curing processes for concrete mixtures, for example, with electromagnetic heating. The deformation and length of cracks in cement composites are closely related to their electrical resistance, so electrically conductive concrete can be used to monitor structures. Multifunctional smart concrete can be used as a deformation, fire and moisture sensor, acting as a structural material [14–16]. The areas of application of electrically conductive concrete and their required characteristics of electrical resistance are listed in Table 1.

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Table 1. The areas of application of electrically conductive concrete.

Areas of application	Specific electrical resistance, Ohm·cm	References
Anodes	$2\cdot10^2$ - $8\cdot10^3$	[2, 17, 25, 26, 34, 36]
Deformation sensors for structures	$3\cdot10^2$ - $5\cdot10^4$	[5, 6, 9–13, 20, 27]
Anti-icing road surfaces	$7\cdot10^4$ - $4\cdot10^5$	[8, 17, 22, 32, 33]
Heating elements	$2\cdot10^6$ - $5\cdot10^7$	[1, 4, 8, 21, 31, 35, 37]

However, a certain instability of properties associated with increased moisture content, blocking of electrically conductive particles by hydration products, as well as the problems of ensuring a high initial strength and density of such concretes significantly limit their scope [17–21]. It should be noted that the use of electrolyte additives in this case is extremely undesirable.

Shi [22] examined the electrical conductivity of concrete from the perspective of the conductivity of the liquid phase in the pore space. Higher electrical conductivity (lower electrical resistivity) suggests higher porosity. He considered hardened concrete to be a semiconductor or dielectric. The same theory is confirmed by other researchers [23–25].

Velay-Lizankos et al. [26] searched for a connection between the elastic modulus and electrical conductivity, but they did not get a clear dependence. Demircilioglu et al. [27] investigated smart concrete containing brass fibers. Despite the good results, this composition is quite expensive. In addition, the authors were convinced that the temperature and humidity at which it is used are of great importance for the electrical characteristics of concrete.

According to the results of many studies, [28–30] the electrical properties of cement pastes were quite sensitive to minor changes in the microstructure of cement materials.

As a number of authors indicate in their studies, the mechanism of electrical conductivity in composite electrically conductive materials is rather complicated [31–32]. The mechanism of conduction through, for example, dielectrics can be both ionic and electronic in nature. A promising issue is the search for a concrete component with a relatively high electrical conductivity, which will have an effect on the overall electrical conductivity of the composite [33]. Researchers emphasize that electrically conductive components are divided into two groups: metal and carbon [34-36].

Carbon electrically conductive components include soot, graphite and carbon black. They are relatively cheap, available in any region, and have a very low electrical resistivity. Carbon, like metal, has electronic conductivity. However, these materials lower the strength of the composites. So, with a volume content of carbon black above 30 %, a complete loss of the mechanical strength of concrete occurs [37]. One of the solutions to this problem is to reduce the size of low-strength carbon materials. The use of carbon nanoparticles with a high specific surface in this regard to reduce the electrical resistance of concrete is interesting.

In general, it should be noted that the properties of electrically conductive concrete are relatively poorly understood. In world construction practice, there are no standards for the study of the electrical characteristics of concrete. Especially few studies relate to studies of carbon nanoparticles obtained by low-cost methods. The problem of ensuring the sedimentation stability of these particles in suspension has not yet been solved.

The scientific novelty lies in the use of carbon nanoparticles obtained in the processing of coal in a plasma-arc reactor as electrically conductive particles in concrete.

The aim of the article is the development of fine-grained concrete and the study of their physico-mechanical, exploitation, as well as electrical properties. The scope of these concretes is quite extensive – from paving slabs [38] to structures of especially important structures [39].

2. Materials and Methods

To achieve the aim of paper, an electrically conductive concrete composition was developed. It consists of (per 1 m^3): Portland cement CEM I 32.5 N (Spasskcement, Russia) – 375 kg; quartz-feldspar sand with the fineness modulus 3.84 – 670 kg; granite crushed stone fractions of 5–20 mm – 1160 kg; water – 170 L and carbon nanoparticles (CNP) obtained from coal in a plasma-arc reactor – 0.01–1% by wt. of cement). Moreover, the same proportions were used for the control composition, but without carbon nanoparticles.

Both chemical and mineralogical composition of used Portland cement are listed in Table 2.

Table 2. Chemical and mineralogical composition of Portland cement CEM I 32.5N.

Chemical composition (%)							Mineralogical composition (%)			
CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O	C_3S	C_2S	C_3A	C_4AF
65.94	21.70	5.02	4.20	1.25	0.40	0.78	61.0	16.3	6.2	12.8

An electric-discharge method for producing a sedimentation-resistant and highly dispersed suspension from coal at low energy costs and without the use of plasticizers consists in treating a coal powder with a fraction of 200 μm with water by electric current. Under the influence of an electric arc plasma from the material of the electrodes and coal supplied for gasification, synthesis gas ($CO + H_2$), activated carbon (sorbent) and carbon nanoparticles — fullerene-containing soot - are formed in one setup. Fullerene-containing soot in this installation is formed along the way, which favorably distinguishes this method of producing carbon nanoparticles in comparison with others. The results of determining the phase composition indicate that the content of fullerene C_{60} in the additive is approximately 1.5–2 %. As a result of the treatment, a sedimentation-stable water-coal suspension (WCS) is obtained.

Figure 1 shows the results of a study of the particle size of CNPs determined by laser diffraction on an SALD-7101 Shimadzu instrument (Japan). The results obtained by laser diffraction show that the carbon nanoparticles lie in the range from 50 to 500 nm.

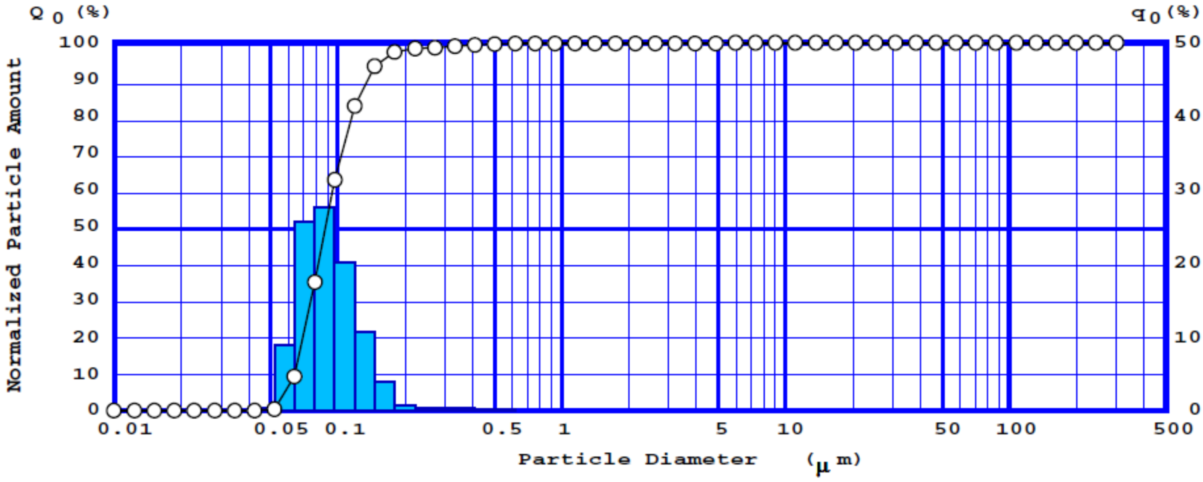

Figure 1. The distribution of CNP particles in the volume of mixing water.

The microstructure of fiber cement compositions was determined using a Jeol JSM 6510 LV scanning electron microscope (Japan) with a magnification of ×1000. The porosity of the hardened cement paste was determined by mercury porosimetry by a Quantachrome PoreMaster 33 instrument.

Concrete cubes of dimensions 100×100×100 mm were prepared for compressive strength test at the age of 2, 7 and 28 days for all mixes. The concrete specimens were unmolded after 24 hours of casting and then immersed in curing tank at room temperature and relative humidity at 65 ± 5 % until the age of testing. This test was carried out using a Shimadzu (Kyoto, Japan) tester machine with a capacity of 200 kN according to EN 12390-3.

Water permeability was determined on cylinder specimens with a diameter of 150 mm, h = 50 mm on an Agama-2RM instrument (Russia). Water absorption was determined by weighing the specimens every 24 hours to obtain a constant weight. The study of freeze-thaw resistance was carried out on specimens of 100×100×100 mm. Specimens were immersed in water at first at 1/3 of the height for a day, then at 2/3 of the height also for a day, and then immersed in water completely for two days. Then the specimens were placed in a Polair CV-105S freezer (Russia) at a temperature of -18°C. Each freezing cycle lasted 2.5 hours, the thawing cycle at a temperature of 20°C is 2 hours.

Determination of the electrical resistance of concrete was carried out on specimens with an edge of 10 cm by the following method. A specimen was placed between two 3 mm thick metal brass plates through which direct current was passed. A multimeter was connected to the plates. Ensuring reliable electrical contact between the plates and the specimen was carried out using a press, which was isolated from the plates with rubber gaskets.

3. Results and Discussion

Figure 2 shows the results of a study of the compressive strength of hardened cement paste with different contents of carbon nanoparticles. It was revealed that higher results were obtained with a CNP content in the range of 0.01–0.1 wt. %, at which there was an improvement in strength by 30–35 % compared with the control specimen. With an increase in the content of additives to 1 %, a decrease in the strength of hardened cement paste is observed, while the results are comparable with the control specimen. In our opinion, this is due to the mechanism of action of nanoscale modifiers, which are most manifested in small doses.

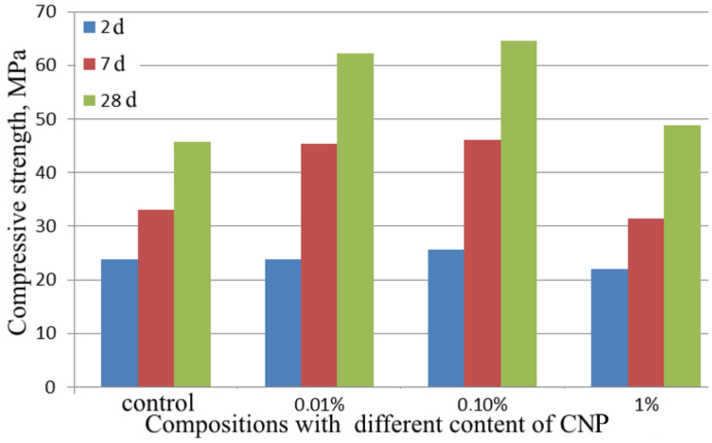

Figure 2. The dependence of the strength of the modified cement paste on the content of CNP.

The microstructure of the specimens using Portland cement (OPC) and CNP is denser compared to the control composition (Figure 3). Carbon nanoparticles act as centers of crystallization of new growths, thereby compacting the microstructure of hardened cement paste. In the control composition, there is a larger amount of pores that are filled with lime crystals of $Ca(OH)_2$ during the curing process. The addition of CNPs leads to a decrease in capillary porosity, to an increase in the amount of tiny gel pores that are part of the calcium silicate hydrogel. When observing the contact zone of the formed portlandite, there is a dense microreinforcement and its binding to additional calcium hydrosilicates, which leads to an increase in the density and strength of the composite. In the modified composition, the germination and thickening of needle-like spokes-like crystals of calcium hydrosilicates is visible.

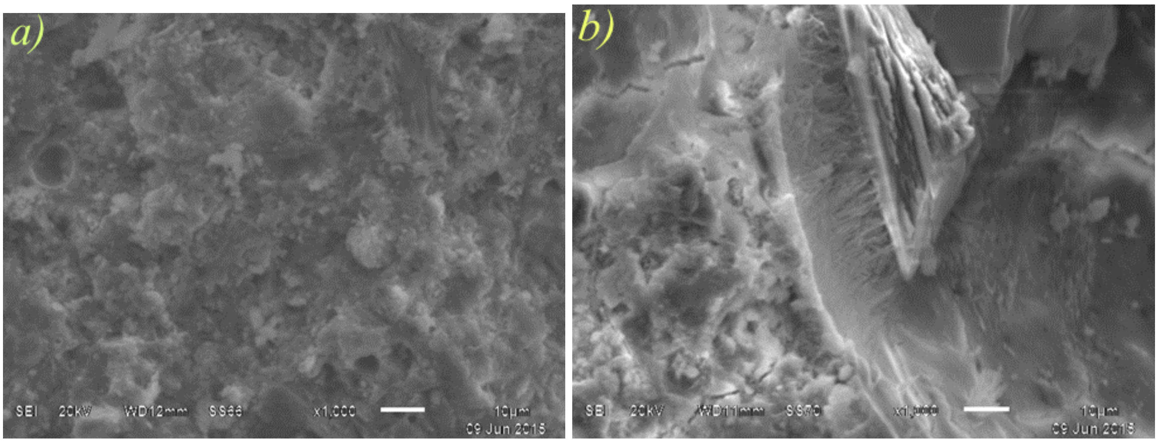

Figure 3. SEM-images of hardened cement paste cleavage (x1000):
***a* – control composition, *b* – OPC + CNP 0.01%**

All this favorably affects the change in the compressive strength characteristics of the modified cement paste, as indicated above, in Figure 3.

The increased strength of the modified cement paste occurs not only due to the acceleration of its hydration processes, but also due to a change in the structure and porosity of the hardened cement paste. Using mercury porosimetry, it was found that the addition of CNPs leads to a decrease in the total porosity by 12 % compared to the control composition (Table 3).

Table 3. The porosity of hardened cement paste at the age of 28 days.

Composition	Total porosity, cm^3/g	Pore diameter, µm					
		1–0.1		0.1–0.01		0.01–0.001	
		cm^3/g	%	cm^3/g	%	cm^3/g	%
OPC	0.094	0.02	21.2	0.072	76.4	0.002	2.4
OPC+0.01%	0.083	0.008	9.6	0.073	87.7	0.002	2.7

Table 3 shows the improvement of the capillary-porous structure of hardened cement paste with the addition of CNP in comparison with the structure of hardened cement paste without additives. In concrete technology, there is a generalized idea that micro- and macropores with a radius not exceeding 10^{-4} cm, which should be mostly closed or dead-ends, should prevail in the structure in cement concrete. The pores size distribution in a hardened cement paste with CNP shifts toward an increase in the amount of small pores, the pore content with a diameter of 1–0.1 μm decreased by 11.6 %, while the pore content in the range 0.1–0.01 μm increased by 11.3 %. A change in the nature of porosity in the direction of increasing the amount of small micropores with the addition of CNPs contributes to the creation of a dense hardened cement paste and the improvement of its physicomechanical properties.

The results of differential thermal analysis (Figure 4) of the cement matrix indicate an increase in the intensity of the endoeffect in the temperature range 515–520 °C during the modification of cement with the addition of a coal-water suspension. This indicates an increase in the content of calcium hydroxide, which is associated with an acceleration of cement hydration with the addition of a water-coal suspension. It is worth noting that along with the acceleration of cement hydration with the introduction of the additive, a change in the basicity of the formed calcium hydrosilicates is observed: the endothermic effect in the temperature range 800–840 °C shifts to the right, in the direction of lowering the temperature from 838 to 806 °C.

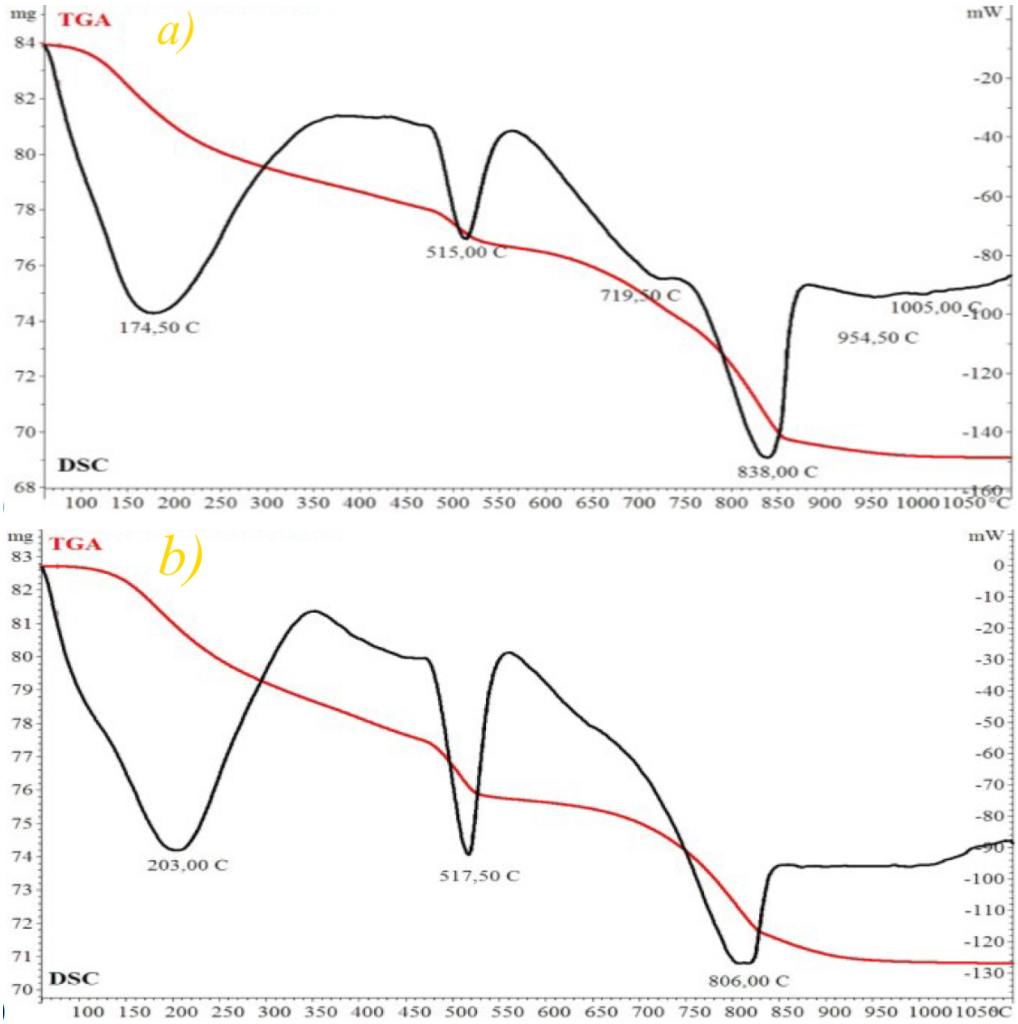

Figure 4. Differential thermal analysis of hardened cement paste: *a* - OPC, *b* - OPC + 0.01% CNP

The revealed positive effect of CNP on the properties of hardened cement paste leads to an improvement in the properties of modified concrete. When selecting the composition of modified concrete of class C20, a CNP was used with an optimum concentration of 0.1% by weight of a binder, the consumption of which amounted to 375 kg/m^3 (Table 4).

Table 4. Technological and physicomechanical properties of modified concrete.

Properties	Value	
	0% CNP	0.1% CNP
Compressive strength, MPa:		
2 d.	26.5	30.9
7 d.	42.3	43.8
28 d.	51.1	55.0
Water permeability, grade	W12	W18
Water absorption by weight,%	1.4	1.1
Freeze-thaw, cycles	300	300

The addition of CNP increases the compressive strength of concrete by 15-20% compared with the control non-additive composition. The combined effect of CNPs at different stages of concrete curing contributes to the creation of a high-density microstructure, a change in the nature of porosity, and an improvement in the hydrophysical and exploitation characteristics of modified concrete.

Having achieved a certain increase in the physicomechanical characteristics of the modified concrete, they simultaneously sought to increase its conductive properties. An important technical result in this direction is the creation of a high-density microstructure of hardened cement paste. This will prevent the saturation of concrete with water during exploitation. Thus, the hardened cement paste itself will be a dielectric, while allowing the conductive function to perform carbon nanoparticles. At the same time, it should be noted the use of cement concrete as the electrically conductive composition. As it was proved earlier [3, 7, 12], cement concrete (in comparison with, for example, polymer or polymer cement binders), in addition to high structural and technical and economic indicators, has a fairly good corona resistance and arc resistance.

The second step is the use of carbon nanoparticles as a conductive additive. As shown by experimental results, the electrical resistance of concrete decreased by a factor of two with the addition of CNP in a small amount in comparison with the known electrically conductive additives (Table 4). It is known that carbon conductive components (for example, carbon black or graphite) create continuous conductive chains in electrically conductive concrete, while their concentration is quite high (it reduces strength of concrete). When using carbon nanoparticles, in our opinion, due to the high specific resistance of CNP particles and subject to their uniform distribution in concrete, continuous conductive chains are also created, leading to a decrease in the electrical resistance of the material.

Table 5. Electrical resistance of modified concrete.

Additive type	The amount of additive,% by weight of cement	Specific electrical resistance, Ohm·cm		
		2 d	7 d	28 d
CNP	0.5	$2.0 \cdot 10^6$	$3.0 \cdot 10^6$	$3.4 \cdot 10^6$
	0.1	$1.1 \cdot 10^6$	$3.2 \cdot 10^6$	$4.5 \cdot 10^6$
Carbon black	20	$1.8 \cdot 10^6$	$3.2 \cdot 10^6$	$5.1 \cdot 10^6$
Graphite	20	$1.7 \cdot 10^6$	$3.0 \cdot 10^6$	$8.9 \cdot 10^6$

The decrease in the electrical resistivity of concrete as a result of the use of carbon nanoparticles is explained both by the influence of CNP on the cement hydration process and by the high dispersion of conductive particles. A change in the nature of porosity in the direction of increasing the amount of small micropores with the addition of CNPs contributes to the creation of a dense hardened cement paste and a more uniform distribution of nanosized conductive particles. This creates a homogeneous microstructure of electrically conductive concrete, allowing it to be used for the various applications listed above. In addition, low porosity will prevent the saturation of concrete with water during exploitation, thereby providing more stable electrical characteristics.

4. Conclusion

Based on the results obtained, the following conclusions can be drawn:

- carbon nanoparticles act as centers of crystallization of new growths, thereby compacting the microstructure of hardened cement paste.;

- a change in the nature of porosity in the direction of increasing the amount of small micropores with the addition of CNPs contributes to the creation of a dense structure of hardened cement paste and improve its exploitation properties.

- the electrical resistance of concrete was halved with the addition of CNP in a small amount compared to known electrically conductive additives.

- a small amount of conductive additives compared, for example, with soot and graphite, gives a more durable structure of the concrete matrix.

The relationship between the electrical conductivity of concrete and macroscopic strength correlates with the results of early studies [4, 8, 21].

Prospects for further research can be conducted in two directions:

1. The creation of electrically conductive concrete with low electrical resistivity and the stability of electrical parameters over time under changing exploitation conditions.

2. The study of the electrical properties of existing concrete and the creation of concrete with improved electrical insulation properties: high electrical resistivity, low dielectric loss and permittivity, high electrical strength.

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Механические и электрические свойства бетона, модифицированного углеродными наночастицами

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Ключевые слова: цементы, цементные композиты, вяжущие, бетоны, углеродные наночастицы

Аннотация. В статье представлены исследования по получению электропроводного бетона с использованием углеродных наночастиц, полученных при переработке угля в электроразрядном плазменном реакторе. Электроразрядный способ получения седиментационно устойчивой и высокодисперсной суспензии заключается в обработке угольного порошка электрическим током в водной среде. С помощью лазерной дифракции было обнаружено, что углеродные наночастицы имеют размер частиц в диапазоне от 50 до 500 нм. Прочность на сжатие была испытана на кубах с ребром 100 мм в соответствии с EN 12390-6. Определение электрического сопротивления бетона проводилось на образцах с ребром 100 мм, размещенных между двумя латунными пластинами, через которые пропускался постоянный ток. Оценено оптимальное содержание углеродных наночастиц (0,01–0,1 % мас.) в вяжущем, что позволяет получить высокие механические свойства бетона (на 30–35% более высокую прочность на сжатие по сравнению с контрольным образцом). Доказано, что механизм действия наноразмерных модификаторов наиболее выражен в малых дозах. Определены зависимости физико-механических свойств цементного камня при введении различного количества углеродных наночастиц. Исследована микроструктура цементного камня при введении углеродных наночастиц методом растровой микроскопии. Определены физико-механические и эксплуатационные свойства тяжелого электропроводящего бетона. Определена кинетика изменения электрического сопротивления бетона в различные сроки твердения.

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