

Magazine of Civil Engineering

ISSN 2712-8172

journal homepage: http://engstroy.spbstu.ru/

Research article UDC 691.32:620.191.33 DOI: 10.34910/MCE.119.9



Strength and crack-resistance of concrete with fibre fillers and modifying nano-additives

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Keywords: concrete construction, stress rates, strength of materials, fibre, nano-additives, crack resistance, methods of analysis, strength criteria

Abstract. The article dwells on a comprehensive method for determining concrete strength and crackresistance in sample testing with varying loading rate. The method allows for receiving simultaneously kinetic parameters of the microstructure defects and length and rate of the main crack growth. The authors consider the impact of effective involved volume on the formation and growth of minor structural defects in cement gel with their eventual phased transition to larger defects. It facilitates increased stress concentration and the development of main cracks at the matrix-aggregate interface. Control over the development of the main crack allows for determining the time till destruction for concrete (longevity). The suggested method and the obtained formula helped applying a wide range of loading rates for determining the critical length of the main crack in concrete. The testing results were used to identify the coefficients of dynamic strengthening for the sample sets with and without an artificially created main crack. These coefficients were applied for calculating the main crack critical length. The article presents the results of experiments aimed at development of concrete compositions comprising fibre fillings and modifying additives including nano-additives followed by determination of their strength and crack-resistance as per the presented comprehensive method. The found values of efficient involving volume, activation energy characterise the microstructure parameters and critical length of the main crack in concrete. The resulting conclusion was that the main crack length in all of the developed concrete compositions exceeded the half of the section in the sample where it developed. Comprehensive assessment of concrete strength and crack-resistance and accumulation of experimental data improve the reliability of scientific research due to the emerging integrated approach to determining quantitative parameters of crack-resistance and longevity of the developed concrete compositions with forecasted properties.

Citation: Perfilov, V.A. Strength and crack-resistance of concrete with fibre fillers and modifying nanoadditives. Magazine of Civil Engineering. 2023. 119(3). Article no. 11909. DOI: 10.34910/MCE.119.9

1. Introduction

Today strength.crack-resistance and longevity of solid bodies including high weight concrete are determined by means of fracture mechanics methods [1–5 and other]. These methods suggest that under mechanical load microfractures present in the solid body develop with further formation of an unbalanced main fracture that can break the sample with sound speed that results in the loss by the sample of its bearing capacity and its full destruction. Even most break-through technologies for preparation of different types and compositions of concrete do not produce a homogeneous dense structure having no micro-pores and micro-fractures that serve as stress points and under the impact of external loads result in the course of operation into reduction of strength and fracture-resistance of concrete. Similar studies have been performed by foreign researches for ceramics [6, 7], HW concrete [8–10] and cellular concrete [11–14]. Unfortunately, no universal method for a reliable determination of main fracture growth parameters in concrete has been found so far.

To describe the mechanism of solid bodies destruction it is necessary to take into account the microstructure defects that include point (to the extent of atoms) dislocations, micro-pores and micro-fractures that are concentrated around concrete structure irregularities. Under mechanical load the concentration of point defects increases and they joint with larger dislocations. While applied load is small, movement of differently directed dislocations results in their annihilation, i.e. their mutual intertwinement, temporary slowdowning and stress relaxation. This does not produce micro-deformations in concrete. As externally applied load increases, there grow similarly directed dislocations that result in formation and development of larger structure defects in the form of micro-fractures. At that deformations develop in a thermallyactivated mode that is addressed from the kinetic theory of strength perspective [15–18].Such approach was considered with respect to concrete by S.N. Leonovich and S.I. Karpenko [19].

It is understood that the behavior of solid bodies under increasing external load is based on the destruction of micro-pores that contribute to creation and further development of micro-fractures with their further transition to macro-level. The Russian Standard GOST 29167 recommended for determination of fracture-resistance properties of concrete based on the use of energy and force criterion of fracture mechanics has a number of shortfalls. In particular, the method for determination diagram and fracturing of standard concrete samples is restricted by stage-wise stopping of the testing implied by technology. However, in concretes with heterogeneous micro- and macro-structure that includes different agents fracture length and width. Thus, there arises the necessity to develop a new method for measuring the length of the main fracture that is continuously growing with time. Moreover, testing during receiving full diagrams of concrete fracturing is impossible in the majority of process construction laboratories because of the high cost of equipment and technical complexity including processing of the obtained results.

Another known method is tensiometric identification of the dimensions and growth rate of the main fracture that implies fixation of its path during the process of stable fracturing or during dynamic testing with varying rate of concrete samples loading [20]. Such testing with the use of modern high capacity registration equipment allows determining fracturing energy, stress intensity coefficient and main fracture dimensions and growth rate simultaneously.

Integrated use of methods for determining dependences between fracture toughness, strength and, for example, rate of samples mechanical loading (stressing) is promising at the moment. In earlier studies conducted by S. Mindess [21], I. M. Grushko, A.I. Kazachuk, A.P. Vashchenko [22–24] et al. samples stressing rate varied from 10⁻³ to 10³ MPa/s. Impact stress was checked at rates 10⁴–10⁵ MPa/s. Use of dynamic tests to determine strength, fracture resistance and longevity is an urgent task for different concrete types of compositions with expected properties.

In order to receive more information on mechanical properties of concrete it is necessary to consider the use of an integrated approach that allows for receiving fracture mechanics parameters, kinetics of microdefects development, macro-fractures length and growth rate under the conditions of stable fracturing. The objective of the research is the development of a comprehensive method for determination of strength and fracture-resistance parameters (time till destruction) of known and newly created concrete compositions with pre-set physical and mechanical properties. The following tasks were implemented:

- develop a comprehensive method for determining strength and fracture-resistance of concretes that includes the methods of fracture mechanics, kinetic strength theory and samples testing with varying loading rate;
- based on the offered comprehensive method determine the values of activation energy U_0 and efficient involving volume γ in the samples of the developed concrete compositions modified with plasticising and nanocarbon additives to identify their impact on the amount of defects and micro-structure strength;
- determine parameters of critical length of the main fracture in the samples of the developed concrete compositions by means of testing by standard fracture mechanics methods and by means of testing with varying loading rates.

2. Methods

Based on the large quantity of accumulated theoretical and experimental data [25–28] it was determined that during operation concrete under the impact of external loads in samples demonstrate the development of elastic, quasi-elastic and plastic deformations. Their further growth depends on the accumulated structure defects and on their size that further on can result in the formation and development of micro- and macro-fractures of shearing and split types. Under small loads small-size, point (to the extent of one or several atoms) defects are formed in nano-structure. Such defects are thermally activated within

approximately one atom. From the point of view of strength theory [15, 17, 18, 23] the development of micro-fractures depends on the change in activation energy U_0 and efficient involving volume γ ($\gamma = v \cdot n$, where v is involving volume, n is excessive stress coefficient) that is characterised by the development of deformations under the impact of one thermal activation.

The numerous studies performed by the author [25–27] have established the impact of loading (stress) rate of concrete samples on changing of kinetic characteristics of micro-structure and their significant impact on the obtained values of strength and fracture-resistance. The mechanism of impact of efficient involving volume on possible phase transition of point defects of one level into dislocations that results in the formation and development of the main fracture (fractures), changes in strength depending on the stress rate are shown in Fig. 1.



Figure 1. Scheme describing the influence of different defects on concrete strength at differing stress rates.

In order to determine time till destruction (longevity) of concrete, it is necessary to control the development of the main fracture in the process of its slow growth. The existing theories and few experiments have so far contradictory results and cannot be considered reliable.

Of the few papers there is one method for determination of the critical length of a macro-fracture for concrete [2] that includes static tensile in bending testing of several samples with an initiated main fracture and of the samples with the same composition and dimensions, but without an artificially initiated macro-fracture. The testing results were used to determine ultimate stresses and critical length of macro-fractures. This method [2] has certain deficiencies, the concrete samples strength in all tests was determined under standard loading with the same rate. At that the obtained values of the main fracture critical length is 10 times less than the dimensions of the tested samples that is not reliable. Accumulation and development of the theoretical and experimental databases allowed for developing the method for determining the main fracture critical size [25, 27] using different loading rates when testing samples. At that loading is performed in bending tension of samples with an artificially initiated fracture and similar samples without such fracture. The results of testing a set of samples with different rate allow for determining dynamic strengthening coefficients based on which critical length of the main fracture in concrete can be identified by the following formula:

$$l_{\kappa p} = h \cdot Y \cdot \frac{K'_{\partial.y.}}{K_{\partial.y.}},\tag{1}$$

where l_{cr} is main fracture critical length, m; *h* are linear dimensions of the sample (its thickness or height), by which the fracture is developed, m; *Y* is function depending on the size of the sample and testing scheme); $K'_{d.s}$ is dynamic strengthening factor for the samples with an artificially induced fracture; $K_{d.s}$ is dynamic strengthening factor for the samples without an artificially induced fracture.

Use of different loading rates and determination of dynamic strengthening coefficients that are characterised by the relation of the concrete strength limit registered at the maximum rate to the sample strength obtained at the minimum (below standard) loading rate allowed for receiving periods of growth of

the main fracture. The moment of the fracture development start is correlated with the minimum strength received at the lowest loading rate, and the moment of the sample full destruction is characterised by maximum strength registered at the highest loading rate.

The essence of the testing is as follows. According to the developed composition there are two sets of standard samples in the form of prisms for further in bending tensile testing. In one set the main fracture is initiated by means of applying a cut with a diamond tool or placing in advance a metal plate when preparing the sample. There was no artificially induced main fracture in the other set of the samples. The size of the induced fracture *a* shall exceed about twice and more the maximum size of the aggregates, and the ratio between the fracture length and the sample section height *h* shall make 0.2–0.4 [2]. The prepared samples after hardening were tested at a various rage of loading rates at the special unit ranging from 10^{-7} m/s to 10^{1} m/s. Such concrete loading rate range has been widely used for concrete testing by other researchers [21–24 and other]. The strength values obtained as a result of the tests were further used to determine dynamic strengthening coefficients in the sample sets with and without an artificially induced fracture for calculating the main fracture critical length by formula (1).

During the experiments we determined optimal compositions of the concrete mixtures and the method for their production with the view to increase strength and fracture-resistance parameters by means of using components that modify macro-, micro- and nano-structure of concrete.

For production of the concrete mix we used Portland cement brand PC M500 D0-N produced by ZAO Oskolcement, quartz sand from the Orlovsky sandpit with a fraction of 1.9-2.0. Coarse filler was granite crushed stone from the Bystrorechensky pit.

Micro-structure of concrete mixes were modified by plasticising agents and nanocarbon components. It is known that plasticising agents, when they completely dissolve in the mix water, improve the solution wettability, its fluidity, decrease water-cement ratio and create conditions for more even distribution of all mix components and for the formation of a dense micro-structure not participating in the processes of the cement stone microcrystal formation [29–34]. Introduction of firm nanocarbon hollow tubes facilitates directed formation of hydrosilicates crystals formation in the cement stone micro-structure. During cement gel structure hardening hydrosilicates adsorb on the internal and external surfaces of hollow nanocarbon tubes and form more strong crystallohydrates. The modified reinforced micro-structure formed after hardening has better physical and mechanical properties. The advantages of the use of nano admixtures for concrete are considered in detail in papers [35–37]. Superplasticising agent Poliplast SP-3 was used as modifier as per TS 5870-006-58042865-05 as well as carbon nano-tubes with a length of 4–45 mcm and a diameter of up to 35 nm.

Fibres were used to increase concrete strength and fracture-resistance at the micro- and macrolevel; cement hydration nano-activated products adhesion occurred on their surfaces, too. Steel fibre MIxarm produced by Severstal-metiz was used as macro-filler, its specification is given in TS 1211-205-46854090-2005. The fibre structurally has cone-shaped anchors with the help of which up to 95 % of it is retained in concrete that facilitates blocking of the formation and development of micro- and macro-fractures in concrete.

Concrete mix production with account of the differences in the properties of its components was as follows. Appropriately selected and measured fibres, portland cement and quartz are mixed electromagnetically in a linear induction spinning device for 5 minutes. Under the influence of alternative electromagnetic field steel fibres, acting as ferromagnetic elements, facilitate better mixing, dispergation and increase specific surface of the obtained powdered dry mixture. Under the influence of magnetic field cement and quartz sand particles are adsorbed on the surface of steel fibres that increases the effectiveness of further reactions in water medium with other concrete mix components. Advantages of such devices with vortex layer and the impact of mechanochemical activation and ultrasonic dispergation have been considered in detail in papers [38–40].

At the same time water insoluble nanocarbon admixtures and superplasticising agent are mixed with water during 1–2 minutes in ultrasonic disperser with a frequency not less than 20 kHz. The obtained dry mix with fibres is further treated during 4–5 minutes in the standard concrete mixers adding coarse aggregate and mix water activated in the ultrasonic unit.

In order to compare the results we prepared a reference composition as per the conventional technology without using the linear induction spinning device and ultrasonic disperser. The quantity of fibre fillers and modifying admixtures in the reference samples complied with composition No. 1 (Table 1).

The compositions of the fibre fillers and modifying admixtures that significantly influence the physical and mechanical properties of concrete mixes are given in Table 1.

Name	Content of the modifying admixtures components, % of the cement weight			
	1	2	3	
Superplasticising agent Poliplast SP-3	0.5	0.55	0.6	
Carbon nano-tubes with diameters 5–35 nm and lengths 4–45 mcm	0.005	0.0075	0.01	
Steel fibre Mixarm with a diameter of 1 mm and a length of 54 mm,	0.75	1.25	1.75	
% of the mix volume				

Table 1.Compositions of modifying admixtures and fibre filler.

With the results of determining the main fracture critical length and rate of its growth the time till destruction (longevity) can be found by formula:

$$\tau = \frac{l_{cr.}}{V} = \frac{h \cdot Y \cdot K'_{\partial.y.} / K_{\partial.y.}}{V}, \tag{2}$$

where l_{cr} is critical length of the main fracture, m; V is fracture growth rate, m/s.

Thus, the offered samples testing method implying testing within a wide range of loading rates for determination of the main fracture critical length and growth rate as well as kinetic parameters of the concrete micro-structure corresponds to the known method of concrete fracture mechanics as per GOST 29167. The developed comprehensive method for determining strength and crack resistance parameters, followed by forecasting the time until concrete is erected, demonstrates high accuracy and reliability.

3. Results and Discussion

The quantitative confirmation of the offered scheme describing the influence of structural defects on changing strength was obtained in the course of special experiments implying testing of concrete samples with aggregates of various origins (Fig. 2 and Table 2). According to the obtained data when the rate of mechanical load grows, strength parameters in all samples increase. At stress rate equalling 1 MPa/s the strength vs. stress rate diagram showed a "change" to a sharper strength growth in all concrete samples with different aggregates (Fig. 2). Apparently, nearby this stress rate there is the area of possible transition of defects of one level (point defects, vacancies) to the other (dislocations) because of the increasing involving volume that corresponds to the conclusions of the kinetic strength theory.

Increasing of concrete strength parameters with growing stress rate takes place to a certain value that corresponds to the rate of stress relaxation. It has been experimentally determined that strength growth stops at stress rate approximately equalling 10² MPa/s. With further stress rate growth in some cases there was observed a slight reduction of strength values, which can be related to instantly occurring over stresses that result in an uncontrolled drastic growth of the main fracture (macro-fractures) and destruction of the sample. Such experiments with the same results were reflected in surveys [21–24, and other].



Figure 2. Changing of strength depending on stress rate logarithm for concrete with aggregate of: 1 – granite, 2 – limestone, 3 – keramzit.

Kinetic parameters of the structure defects, i.e. activation energy U_0 and efficient involving volume γ in concrete samples with different aggregates tested at differing stress rates were determined by formula [23]:

$$\sigma_p = \frac{KT}{\gamma} \ln \left[1 + \frac{\tau_0}{KT} \exp \frac{U_0 \omega \gamma}{KT} \right], \tag{3}$$

where σ_p is applied stress, MPa, *K* is Boltzmann's constant; *T* is absolute temperature, K; τ_0 is preexponential factor, 10⁻¹² s; U_0 is activation energy, J; γ is efficient involving volume, m³; ω is stress rate, MPa/s.

Quantitative values of kinetic parameters of the tested concrete samples with different aggregates given in Table 2 showed decrease in efficient involving volume γ at slight reduction of activation energy U_0 with stress rates exceeding 1 MPa/s (after the "change").

	<u>γ</u> *10 ⁻	²⁶ , m ³	U_0 *1	0 ⁻¹⁹ , (J)
	"before the change"	"after the change"	"before the change"	"after the change"
Keramzit	4.19	2.65	1.3	1.11
Limestone	2.94	1.81	1.37	1.15
Granite	2.82	1.66	1.45	1.18

	Table 2.Kinetic	characteristics of	concrete strength
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Consequently, when the stress rate of the samples grows, point defects of structure concentrate in some volume that does not promote a large-scale development of micro-fractures, and concrete strength grows.

The offered method showed that concrete samples testing with different stress rates demonstrate the kinetic process of the structure point defects transition into dislocations, micro- and macro-fractures both in the cement gel and nearby the matrix-aggregate structural irregularities. Thus, there is the possibility to refuse from direct registration of the main fracture dimensions and growth rate that reduces the process complexity and the reliability of the obtained results is high. The proof is that the offered method is related to fracture mechanics that determines the parameters of the main fracture development and to the kinetic parameters of the development of different defects of concrete micro-structure.

Based on the offered comprehensive method for determination of strength and fracture-resistance the authors have performed experiments with the aim to develop concrete compositions with fibre fillers and modifying additives. During testing there were used standard units as well as the units developed by the author aimed at determination of the quantity parameters of fracture formation and destruction of concrete.

Concrete samples with dimensions $100 \times 100 \times 400$ mm were manufactured for testing with the view to determine strength parameters and kinetic characteristics when loaded at different rates. Testing with the scheme implying three-point bending was performed at the special unit with loading rates ranging from 10^{-7} m/s to 10 m/s.

Quantitative measurement of values of activation energy U_0 and efficient involving volume γ that allow for determining the influence of different micro-structure defects on the processes of fracturing was performed following the method [23, 25]. The results of the performed experiments are given in Table 3.

The analysis of the data presented in Table 3 showed that the offered concrete compositions with the Mixarm fibre filler, plasticising and nanocarbon additives and the methods of their preparation with the indicated component ratios bring to the increasing of compression strength and tensile strength in bending as compared to reference samples. It has been identified that maximum compression strength gain of 28.2 %, and bending tensile strength gain of almost 40 % was received in composition No. 2. At that all samples demonstrated reduction of kinetic parameters of the efficient involving volume compared to the reference samples. The developed compositions with the plasticising and nanocarbon additives have strong micro-structure that testifies to the prevailing development of point defects in small amount that does not lead to a large-scale fracturing. Use of fibres with cone-shaped anchors at the ends promoted slowing of the processes of formation and propagation of main fractures in concrete. With account of the obtained experimental data we have selected an optimal concrete composition with maximum strength

characteristics and better kinetic parameters of fracturing (Table 2, composition 2). The developed fibreconcrete composition is recommended for use in reinforced concrete beams [41, 42].

Micrographs of the structure of fibre-concrete with the Mixarm fibre filler, superplasticising agent Poliplast SP-3 and nanocarbon additives that were made using "Altami LCD" stereomicroscope showed compaction and strengthening of the cement-sand matrix due to filling of the interparticle space with stringer steel fibres (Fig. 3).



Figure 3. Micrographs of the structure of fibre-concrete with the Mixarm fibre filler and complex modifying additive that includes superplasticising agent Poliplast SP-3 and nanocarbon tubes: 1) 100 X, b) 200 X.

In order to determine the characteristics of macro-fracturing and destruction of the selected compositions (Table 2) the authors performed testing of the concrete samples by fracture mechanics methods with the receipt of full deformation diagrams and by the offered testing method with different loading rates and identification of dynamic strengthening coefficients. During the experiments with the receipt of the full destruction diagrams we used capillary (as per GOST 29167) and tensiometric [20] methods to measure the length of the developing main fracture.

For the tests we prepared a set of samples with dimensions $100 \times 100 \times 400$ mm with an artificially induced fracture having a length of 40 mm made by a cutting tool. No artificial macro-fracture was made in the other set of concrete samples. The testing unit loading rates ranged from 10^{-7} m/s to 10 m/s. As a result we received esperimental daata and determined the quantitative values of the main fracture critical length in concrete by the standard fracture mechanics method $l_{cr.st.}$ and by the offered method $l_{cr.}$ finding dynamic strengthening coefficients [25] (Table 3).

No. of mix	R_b , MPa	$R_{b\!f}$, MPa	γ.10 ⁻²⁶ , m ³	$U_{0^{\cdot}}$ 10 ⁻¹⁹	$K_{d.s.}$	$K'_{d.s.}$	<i>l_{cr.st.}</i> ,	<i>l</i> _{cr.} ,
	WI G	WI a	111	0			m	m
Reference	61.2	11 3	<u>2.81</u>	<u>2.43</u>	1 87	1.05	0.065	0.063
sample	01.2	11.5	1.94	1.72	1.07	1.00	0.005	0.005
			2.68	2.51				
1	70.38	12.75	1.82	1.86	1.94	1.12	0.066	0.065
			2.47	2.87				
2	78.43	15.81	2.41	2.07	2.0	1.21	0.069	0.068
			1.62	2.29				
2	74.05	10.00	<u>2.58</u>	<u>2.68</u>	10	1 1 2	0.067	0.067
3	74.25	13.62	1.73	1.99	1.9	1.13	0.067	0.067

Table 3.Concrete strength and tracture-resistance parameters.

Comments. R_b is strength limit of concrete at compression received with standard loading rate; R_{bf} is strength limit of concrete at bending received with standard loading rate; γ is efficient involving volume, above the line are values of γ "before the change", under the line – "after the change". U_0 is activation energy, above the line are values of U_0 "before the change", under the line – "after the change".

It has been determined that an increased sensitivity to the formation and growth of the main fracture is characteristic of the concrete having a significant difference in strength limits of the samples with an

artificially induced fracture and without such fracture, and, consequently, the smaller ratio $\left(\frac{K_{d.s.}}{K_{d.s.}}\right)$. At that

the main fracture critical length has minimum values. And vice versa, the smaller the difference in the

strength values of the samples with and without a fracture is, the higher fracture-resistance of the concrete is and the larger the macro-fracture critical length is.

It has been established that the obtained size of the main fracture in all concrete samples has values more the half of the section of the sample in which it developed that demonstrates a significant deviation from the known results of other researchers [2]. The development of the formed main fracture in the developed concrete compositions with modifying additives and steel fibres is accompanied by constant overcoming of the coarse aggregate in the contacts with the cement-sand matrix and highly firm fibre that reinforces the macro-structure of concrete. As a result the growth slows down and the value of the macro-fracture critical length determined as per GOST 29167 and the offered method grows (Table 3). However, the developed method for determination of the main fracture critical size in concrete is more accurate and reliable as compared with the fracture mechanics method because the macro-fracture growth was controlled constantly, but not stage-wise. Based on the performed research and determination of the main fracture critical length and growth rate under the impact of external mechanical load the time till full destruction (longevity) of the concrete samples was determined by formula (2). The offered comprehensive method for determination of fracture-resistance parameters can be applied in practice, including for the structures manufactured of fibre-concrete.

4. Conclusions

1. A comprehensive method of an uninterrupted testing of concrete samples at different loading rates has been developed. It allows for simultaneous determination of the micro-structure defects growth kinetics and fracture mechanics parameters related to determination of the main fracture critical length and growth rate as well as forecasting of the time till destruction (longevity) under the impact of mechanical load.

2. The developed concrete compositions with fibre fillers, plasticising and nanocarbon additives and their preparation method in the course of testing with different loading rates have demonstrated a reduction of the efficient involving volume γ that testifies to the micro-structure increased strength by means of formation of a small amount of defects that does not result in a large-scale formation of micro-fractures. The optimal composition of concrete has been selected that has the maximum compression strength gain of 28 % and tensile strength gain of 40 % compared with the reference composition.

3. Based on the offered method the characteristics of formation of macro-fractures have been determined for the developed concrete compositions with fibre fillers and modifying additives prepared using a linear induction spinning device and an ultrasonic disperser. It has been determined that in all samples the values of the main fracture maximum critical length determined by capillary method of standard fracture mechanics and by the offered method implying the use the samples dynamic strengthening coefficients in a wide range of loading rates varied from 63 to 68 mm with the height of the section where the macro-fracture was developing equalling 100 mm. The time of the main fracture growth and its length is related to the strength of the micro- and nano-structure of the developed concrete compositions with modifying additives and to the use of non-standard fibres with cone-shaped anchors that slow down the macro-fractures propagation.

Thus, the use of the offered comprehensive method for determining concrete strength and fractureresistance parameters demonstrated the required accuracy and requires less effort that expands the scope of application and accumulation of experimental data and improves their reliability. This article has a more methodical orientation for subsequent wide practical use in the development of new and well-known concrete compositions with specified physical and mechanical properties and service life.

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Received 10.11.2021. Approved after reviewing 23.03.2023. Accepted 29.03.2023.