Study of Quality Parameters of Long-Term Strength of Cement Composites

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Abstract. The currently widespread assessment of concrete quality, including reliability, by traditional mechanical characteristics is ineffective, since these state parameters do not take into account changes in the strength and deformation properties of concrete under the influence of temporary processes of microcracking during loading when diagnosing structural materials. Modern development of fracture mechanics has made it possible to establish scientific principles for evaluating materials and testing methods and to propose together force, strain, energy and acoustic criteria for crack resistance that determine the actual limit States of their structure in terms of strength and deformability and thus allow predicting the performance of the material under load. This paper presents the results of experimental evaluation of mechanical failure criteria and regularities of changes in acoustic emission parameters from the loading intensity during non-equilibrium mechanical tests for three-point bending of experimental samples with an initial incision depth of 13 mm. Analysis of the influence of the concentration and the introduction of SP-3 on regularities of changes in the coefficients of increase of controlled criteria of crack resistance in time relative to 28-day age quite clearly suggests, first, that the change of parameters of criteria of crack resistance in time has a wavelike nature, and the change in strength under axial compression, and, secondly, about, that the modified structures of cement stone samples relative to the control composition generally give significantly better indicators not only of the growth coefficients of the compared quality criteria, but also of the absolute values of the criteria for crack resistance up to 18 years of age.

1. Introduction

Analyzing the trends in the development of modern construction in foreign countries, we can see that they are increasingly linked to the development of concrete based on new technologies (non-traditional concrete) [1, 2, 3, 4, 5, 6, 7, 8, 9]. Researchers identify a factor of long-term economic effect from the use of non-traditional concrete in structures, which is caused not only by their increased strength, but also by high performance properties: frost resistance, corrosion resistance, high water resistance, etc. In addition, such concretes have an increased workability of the concrete mix. In the West, a special term "High performance Concrete" has been introduced for such concretes – high-quality concrete. Some new technologies for the production of high-quality concrete are found in Russia. Among them we can distinguish the production of concrete with the use of multifunctional modifiers [10, 11, 12, 13, 14, 15, 16].

Based on their own experimental data, the authors considered the problem of increasing the long-term strength of the hydration structure of cement stone modified with superplasticizer C-3 [17].
Modification of the cement–water paste with A / C in the range from 0.18 to 0.27 was carried out both by the procedure of introducing SP and by changing its concentration, which corresponds to the modern concept of obtaining a new generation of high-strength concretes, which is based on the idea of using primarily super-and hyperplasticizers and dispersed mineral additives, which together allow increasing the volume of the rheological matrix phase and obtaining highly mobile concrete mixtures with low water content, high strength, density and structure uniformity [18, 19].

However, the currently widespread assessment of concrete quality, including reliability, by traditional mechanical characteristics is ineffective, since these state parameters in the diagnosis of structural materials do not take into account changes in the strength and deformation properties of concrete under the influence of temporary processes of microcracking under loading [20].

The specificity of this problem lies in the fact that the usual ideas about the influence of structure parameters on the usual strength characteristics in relation to the characteristics of crack resistance are in some cases incorrect. Thus, it was shown in [21] that the value of the critical stress intensity coefficient for a stone made of cements of different activity increases much less than the compressive strength. This problem of crack resistance is particularly acute in connection with the intensive development and practical implementation in construction practice of so-called high-quality concretes of high and extra-high strength [22], for which the assessment of the actual limit States of the structure acquires special scientific and practical significance, since according to the concept of the kinetic nature of strength, destruction is a process that develops over time, and can occur at different stress levels.

Modern development of fracture mechanics has made it possible to establish scientific principles for evaluating materials and testing methods and to propose together force, strain, energy and acoustic criteria for crack resistance that determine the actual limit States of their structure in terms of strength and deformability and thus allow predicting the performance of the material under load.

As is known, during the process of deformation and destruction, various materials emit elastic vibrations in a wide range of frequencies and amplitudes. A joint study of the nature of changes in radiation and the parameters of this phenomenon, called acoustic emission in the literature, when evaluating mechanical failure criteria on experimental samples using the GOST 29167-91 method can not only provide valuable information about the patterns of deformation and destruction of structural material in real time, but also predict its future performance [23].

Objective: to Study the quality parameters of long-term strength of cement composites

Research objectives:
1. Study of mechanical and acoustic parameters of cement stone destruction depending on the duration of hardening.
2. Identify the nature of the increase in mechanical criteria for crack resistance and long-term strength of cement stone.
3. Determine the effect of plasticizer additives on the processes of structure formation of cement stone both at the initial and long-term stages of hardening.

2. Materials and methods

The article presents the results of experimental evaluation of mechanical failure criteria and regularities of changes in the parameters of acoustic emission from the loading intensity during non-equilibrium mechanical tests for three-point bending of type I prototypes with an initial incision depth of 13 mm, obtained when forming samples by laying a steel plate according to GOST 29167-91. After testing the 4x4x16 cm prism samples for bending, the resulting two halves of the sample were tested for axial compression strength, respectively. Thus, both mechanical and acoustic criteria for crack resistance of the compared series of samples were evaluated on three twin samples during each control period of the test, and the axial compressive strength was evaluated on six twin samples, respectively. Simultaneous evaluation of mechanical and acoustic criteria for crack resistance and axial compressive strength on the same samples in the age range from 28 days to 18 years, naturally, increases the reliability of the results. Here it is appropriate to note that the coefficient of variability of the strength
of cement stone for axial compression at the age of 28 days and 18 years for the compared six series of samples made at A / C of 0.24, respectively, was for the series: 1-0.05 and 0.105; 2-0.037 and 0.04; 3 – 0.061 and 0.039; 4 – 0.055 and 0.08; 5 – 0.031 and 0.065; 6 – 0.04 and 0.094. From the experiments the values of this coefficient testify, first, about a high level of homogeneity of the considered properties of the values of this coefficient testify, first, about a high level of homogeneity of the considered properties of the structure, and secondly, on the positive influence of SP-3 the uniformity of strength of the modified cement stone structure of the considered series of samples, which is confirmed as numerical values of coefficients of variability, and the kinetics of its change in the considered range of time, despite the fact that the compaction of cement paste samples series 3-6 was slightly (0.5-1.7 percent) lower than the control composition, what follows from the data [24, 25].

3. Results and discussion

Table 1 shows the results of evaluating the mechanical and acoustic criteria for the destruction of experimental samples of cement stone aged from 28 days to 18 years, indicating the numerical values of the coefficient of change of the controlled parameters in the named time interval.

Table 1. Mechanical and acoustic parameters of cement stone destruction.

| Quality parameters of cement stone | Quality indicators of the compared series of samples |
|-----------------------------------|-----------------------------------------------------|
|                                    | 28 days     | 420 days    | 4.5 years old | 9.5 years old | 18 years old |
| Series 1                           |             |             |              |              |             |
| $R_{cn}$, MPa                      | 76.7 / 1    | 87.5 / 1.14 | 96.9 / 1.26  | 100.3 / 1.31 | 90 / 1.17   |
| $R_{f}$, MPa                       | 5.19 / 1    | 6.38 / 1.23 | 6.97 / 1.34  | 6.77 / 1.3   | 5.35 / 1.03 |
| $K_{cr}$                           | 0.068 / 1   | 0.073 / 1.07| 0.072 / 1.06 | 0.067 / 0.98 | 0.059 / 0.85|
| $K_{c}$, MPa · m$^{-0.5}$          | 0.51 / 1    | 0.626 / 1.23| 0.7 / 1.37   | 0.661 / 1.3  | 0.525 / 1.03|
| $G_{s}$, J/m$^2$                   | –           | 85.6        | –            | –            | 115.6       |
| $E_{acr}$, W$^2$/cm$^{-2}$         | 0.17 / 1    | 0.21 / 1.23 | 0.427 / 2.5  | 0.607 / 3.57 | 0.79 / 4.65 |
| Series 2                           |             |             |              |              |             |
| $R_{cn}$, MPa                      | 68.2 / 1    | 74.5 / 1.1  | 70.6 / 1.04  | 84.2 / 1.23  | 81.8 / 1.2  |
| $R_{f}$, MPa                       | 3.88 / 1    | 6.69 / 1.72 | 6.37 / 1.63  | 7.35 / 1.89  | 6.53 / 1.68 |
| $K_{cr}$                           | 0.057 / 1   | 0.09 / 1.58 | 0.09 / 1.58  | 0.087 / 1.53 | 0.08 / 1.4  |
| $K_{c}$, MPa · m$^{-0.5}$          | 0.381 / 1   | 0.656 / 1.72| 0.626 / 1.64 | 0.697 / 1.83 | 0.641 / 1.68|
| $G_{s}$, J/m$^2$                   | –           | 75.3        | –            | –            | 122.7       |
| $E_{acr}$, W$^2$/cm$^{-2}$         | 0.108 / 1   | 0.27 / 2.5  | 0.308 / 2.85 | 0.333 / 3.08 | 0.39 / 3.61 |
| Series 3                           |             |             |              |              |             |
| $R_{cn}$, MPa                      | 94.1 / 1    | 103.2 / 1.1 | 96.1 / 1.02  | 94.3 / 1     | 104.7 / 1.1 |
| $R_{f}$, MPa                       | 3.98 / 1    | 6.9 / 1.73  | 6.71 / 1.68  | 6.55 / 1.64  | 6.18 / 1.55 |
| $K_{cr}$                           | 0.042 / 1   | 0.067 / 1.6 | 0.07 / 1.67  | 0.069 / 1.64 | 0.059 / 1.4 |
| $K_{c}$, MPa · m$^{-0.5}$          | 0.392 / 1   | 0.677 / 1.73| 0.658 / 1.68 | 0.643 / 1.64 | 0.606 / 1.55|
| $G_{s}$, J/m$^2$                   | –           | 94.2        | –            | –            | 159.6       |
| $E_{acr}$, W$^2$/cm$^{-2}$         | 0.102 / 1   | 0.136 / 1.33| 0.267 / 2.62 | 0.355 / 3.48 | 0.47 / 4.6  |
| Series 4                           |             |             |              |              |             |
| $R_{cn}$, MPa                      | 100.4 / 1   | 108 / 1.08  | 101.8 / 1.01 | 95 / 0.95    | 119.5 / 1.19|
| $R_{f}$, MPa                       | 4 / 1       | 7.46 / 1.86 | 7.2 / 1.8    | 6.98 / 1.74  | 7 / 1.75    |
| $K_{cr}$                           | 0.04 / 1    | 0.069 / 1.72| 0.071 / 1.77 | 0.073 / 1.82 | 0.059 / 1.46|
In the growth coefficients of the controlled crack resistance criteria, but also of the absolute values of the criteria, compared to the control composition of series 1, give generally noticeably better indicators not only of the crack resistance criteria over time is undulating, as is the change in the strength property relative to the 28-day age.

The energy characteristic of crack resistance was determined by the formula

$$E_{ac} = \frac{3P_c^*L}{2tb^2(1-\lambda)^2},$$

where $P_c^*$ - the maximum (destructive) load, N; $L$ - the distance between the supports, m; $t$ and $b$ - respectively, the width and height of the section, m; $\lambda = a/b$ - the relative length of the incision.

Crack resistance coefficient $K_{cr} = R_{fs}/R_{cr}$, where $R_{cr}$ is the compressive strength. The conditional critical stress intensity coefficient $K^C$ was calculated according to the dependence (9) of GOST 29167-91. The energy characteristic of crack resistance was determined by the formula $G_c = A_c/F$, where $A_c$ is the total work of failure, J; $F$ - the area of failure, m². In turn, $A_c = Pf$, where $f$ is the deflection of the sample, m. The energy of acoustic emission of $E_{ac}$ as a criterion of crack resistance of samples was determined by [23] by the ratio of the Eae recorded by the acoustic emission device (W2) when loading the sample to failure to the surface of its failure (cm²).

The analysis of the influence of the concentration and procedure for the introduction of SP C-3 [24, 25] on the regularities of changes in the growth coefficients of the controlled crack resistance criteria over time relative to the 28-day age clearly shows, firstly, that the change in the parameters of the crack resistance criteria over time is undulating, as is the change in the strength under axial compression [25], and, secondly, that, that the modified structures of cement stone samples of series 2-6 relative to the control composition of series 1 give generally noticeably better indicators not only of the growth coefficients of the compared quality criteria, but also of the absolute values of the criteria.

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### Table 1: Crack Resistance Coefficients

|       | Series 5        | Series 6        |
|-------|-----------------|-----------------|
| $R_{fs}$, MPa | 93.2 / 1       | 93.9 / 1       |
| $R_{cr}$, MPa  | 4.88 / 1       | 4.88 / 1       |
| $K_{cr}$      | 0.056 / 1      | 0.052 / 1      |
| $E_{ac}$, W²/cm² | 0.117 / 1   | 0.121 / 1     |
| $K^C$, MPa · m⁰⁵ |               |                |
| $G_c$, J/m²   |               |                |
| $E_{ac}$, W²/cm² |             |                |

Note: before the line – numerical values of properties; after the line - the coefficient of growth of the property relative to the 28-day age.
for crack resistance up to 18 years of age. According to the optimal quality indicators, it is necessary to note the cement stone of series 4 samples made at the expense of SP C-3 in the amount of 0.5% of the cement mass, which was introduced into the cement paste with the second half of the mixing water after preliminary five-minute grinding with the first half of the mixing water [25]. However, at the age of 28 days, the numerical values of the criteria $R_{fs}$, $K_{cr}$ and $K'$ of cement stone with C-3, i.e. samples of series 2-6, showed lower values relative to the control composition, especially on samples of series 2-4 with a consumption of C-3 in the amount of 1 and 0.5% by weight of cement. However, the axial compressive strength of the samples of these series, with the exception of series 2 samples, was 20-30% higher than that of the control samples at this age. The obtained data on the formation of initial and long-term strength and crack resistance criteria suggest that the synthesis of these mechanical properties, which reflect the different stress state of the material when evaluating these quality criteria, is based on different mechanisms, the flow of which over time can be controlled to a certain extent by chemical and technological effects on the basic elementary processes of adsorption, dissolution, hydration and structure formation at the very beginning of the preparation of a cement disperse system, that is, in other words, at the initial stage of mixing, nothing should interfere with the interaction of cement minerals with water, and this should be the fundamental technological principle for modifying the hydration structure of the cement disperse system with modern superplasticizers [17]. From the analysis of the numerical values of the coefficient of increase in the energy of acoustic emission $E_{ac}$, it follows that for all compositions with increasing age of cement stone, it increases, which is in full accordance with the position [26] that the strengthening of the structure over time results as a transition of part of the adhesive contacts of electromagnetic and electrostatic nature into crystallization bonds of valence nature, the destruction of which, naturally, is characterized by the release of more elastic energy generated in acoustic signals.

Figure 1 shows the experimental results of evaluating the quality coefficients-criteria for long-term thermal stability of modified hydration structures of cement stone series 2-6 relative to the control unmodified composition of series 1 from the logarithm of its age. The numbers for the graphic dependencies correspond to the series of samples under consideration.

Analysis of the relative numerical values of mechanical crack resistance criteria $R_{fs}$, $K_{cr}$ and $K'$ of modified and unmodified cement stone structures shown in the graphic dependences of figure 1 allows us to formulate a number of conclusions about the influence of procedural and concentration factors in the preparation of cement paste on the formation of criteria $R_{fs}$, $K_{cr}$ and $K'$ in the time interval from 28 days to 18 years.

First, it should be noted that at the age of 28 days, the named criteria for crack resistance of modified structures were significantly lower than the control composition of series 1, which is in full agreement with the basic technological principle of modifying cement paste formulated above.

At the age of 28 days to 420 days, there was a fairly intensive increase in the $R_{fs}$ and $K'$ criteria with a noticeable excess relative to the control composition, with the exception of samples from series 5. However, the $K_{cr}$ criterion showed, with the exception of series 2 samples, lower values relative to the control composition, which is in full accordance with the nature of the increase in the Rcs criterion.

In the time interval from 420 days to 4.5 years, a symbiotic decrease in the numerical values of the criteria $K'$, $R_{fs}$ and compressive strength $R_{cs}$ was recorded, and the $K_{cr}$ criterion showed a slight increase in this period. At the age of 4.5 years, almost all samples of cement stone with a modified structure gave numerical values of the quality coefficients $R_{fs}$ and $K'$, which are less than those of the control composition, that is, less than one. However, in the future, with an increase in the age of cement stone with the addition of superplasticizer to 9.5-18 years, there is a clear tendency to improve the crack resistance criteria $R_{fs}$ and $K'$ by 15-32% relative to the control composition of series 1. Moreover, as follows from the graphical dependencies of Fig., that is, based on the angle of inclination of logarithmic segments, more intensive growth of these criteria was observed in the age range from 9.5 years to 18 years. It should also be noted that the nature of the change in the coefficients of the
criterion for compressive strength of $R_c$, relative to the control composition of series 1 has a more complex relationship, as can be seen from the graphs in figure 1.

Figure 1. Dependences of quality coefficients of long-term crack resistance criteria of modified cement stone structures of series 2-6 relative to the control composition of series 1 on the logarithm of its age.
Thus, the conducted studies have shown that the increase in both long-term axial compressive strength and mechanical criteria for crack resistance of modified and unmodified cement stone structures in the considered time range has a wave-like (oscillatory) character. It can be regulated to a certain extent by the introduction procedure and the concentration of the plasticizing additive, bearing in mind that the conditions under which elementary hydration processes begin at the initial stage have a greater impact on the structure than its further change, which is of great practical importance when designing the structural strength of new-generation cement dispersion systems.

4. Conclusion
1. The results of experimental studies of quality coefficients – criteria for long-term crack resistance of modified hydration structures of cement stone depending on its age are presented.
   2. Conclusions about the influence of procedural and concentration factors in the preparation of cement paste on the formation of strength criteria, stress intensity coefficients and crack resistance are formulated.
   3. It was found that the increase in long-term compressive strength, mechanical criteria for crack resistance of modified and unmodified cement stone structures in the time range up to 18 years has a wave-like (oscillatory) character.

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