Methods of assessing the durability of high performance concrete

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Abstract. To ensure concrete durability in reinforced concrete structures is one of the main problems of modern concrete technology. In the State Standard (GOST 31384-2017) we can find the restrictions to concrete composition depending on the class and index of operating media, but there is no information on how they affect the durability, what kind of service life of concrete and reinforced concrete structures they guarantee. This articles shows that the restrictions to concrete composition are necessary but not sufficient to ensure concrete durability especially under harsh conditions of its operation. This is due to the fact that in the national standards the role of the basic concrete strength index, which is the microstructure of hydrated phases of cement stone, is not taken into consideration. The cement gel formed by calcium hydrocilicates and being the densest and strongest component of the structure, determines the concrete durability provided it is stable in specific environmental operating conditions. The proposed methods of assessing the stability of concrete microstructure make it possible to optimize technological parameters of production of reinforced concrete structures of high performance and durability.

1. Introduction
It is known, that the concrete durability depends on the features of reinforced concrete structures operating environment, concrete resistance and density, moreover frost resistance mark is more often assigned as concrete resistance indicator [1-2]. According to [3] the duration of failure-free operation of building structures is one year for 5–15 cycles of frost resistance testing, which is determined by the first standard method. State Standard (GOST 31384-2017) sets the requirements for frost resistance of concrete saturated with mineralized or fresh water depending on minimum winter temperature and the saturation degree from F2100 to F2450 or F1100 up to F1400.

In practice, quite often we obtain the results that are not consistent with the regulatory requirements. For example, the standard sets specific restrictions to the concrete composition for XF operating environment class, but does not take into account the hardening features. According to our data [4,5] concrete samples of the same composition but hardening in different conditions are characterized by frost resistance differing up to 10–12 times. The change in the cement type can change its frost resistance by 2–3 times with the same concrete capillary porosity [6]. These and similar research help to consider the need of additional consideration of structural features of cement stone (apart from water-cement ratio) affecting concrete frost resistance and durability.

The products of hydrolysis and hydration of cement minerals are introduced by crystalline, semi-crystalline and gel-like phases. In addition, cement stone porosity is formed, which is contraction,
closed and gel (with a diameter of less than 10 nm), water in which does not freeze to very low temperatures (-196°C), microcapillary (10–100 nm) with water freezing temperature of -20...-35 °C, and macrocapillary (over 100 nm) with water freezing temperature of -3...-6 °C [7].

According to modern concepts [8], cement gel is the basic structure-forming component of the cement stone, possessing the highest adhesion, cohesion and density, which is a thermodynamically unstable component unlike crystalline hydration products. In [9] it is indicated that even a single drying of a cement stone reduces its shrinkage irreversibly. This is due to the aging of the cement gel, as a result it loses its X-ray amorphism, its particles are increased in size and the amount of absorbed moisture decreases. We [4-5] have studied the process of cement gel aging and revealed that it proceeds under any hardening conditions, but heat treatment, cyclic temperature and humidity effects accelerate it. The noticeable stabilization of the cement stone microstructure is achieved by pozzolanic.

According to GOST 31384-2017, for all reinforced concrete structures of different operating environmental classes CEM I and CEM I SR (sulfate resistant) are recommended with less stable microstructure of hydrated phases of cement stone, hence we have high cost and low durability of construction sites. CEM II is less often recommended, CEM IV is used for underground and underwater structures and it can’t be used for precast concrete due to its increased water demand, which is easily eliminated by the introduction of water-reducing additives. We recommend CEM III/A for prefabricated reinforced concrete.

High performance concrete (HPC) is characterized by low water-cement ratio (0.2–0.4) and strength in the range of 40–130 MPa [10-11]. The reduced water content of mixtures for such concrete contributes to the formation of a dense texture without macrocapillary porosity [12]. Therefore, the durability of such reinforced concrete products made of such concrete will be determined only by the stability of hydrated phases of cement stone under real operating conditions. It follows that to ensure durability of concrete and reinforced concrete structures and constructions the development of comprehensive methods for assessment the stability of gel-like cement hydration products is a topical issue. The solution of this problem is especially significant for harsh operating conditions, under which cement gel aging proceeds more intensively.

2. Methods for assessing the durability of concrete

At the Department of Building Materials and Products of South Ural State University, we have made research using various materials and additives, which meet the requirements of the corresponding regulatory documents to produce samples hardening under different conditions and to assess the stability of gel-like structure of cement hydration products [5]. As the main aggressive effect on a grade concrete sample, we apply the third accelerated method of concrete frost resistance determination according to GOST 10060-2012, which is freezing up to -50 °C and thawing at +20 °C in 5 % sodium chloride solution. In this case, the following methods of assessing concrete durability are used.

1. By the Portlandite content. When using CEM I, cement stone samples have been made from the slurry of normal density, in which due to pozzolanization (silica fume is used) and complex formation of polycarboxilate superplasticizers the Portlandite content at the age of 28 days of water hardening is reduced up to 3–5 % by weight [13]. The samples have been tested through different amount up to 80 cycles, which corresponds to F 2500, while the microstructure of hydrated phases does not change. According to our thoughts, such frost resistance ensures reinforced concrete durability for at least 100 years in severe climatic conditions. For concrete on CEM II/A-D it is desirable to have silica fume (SF) content of 10 % by weight of a binder and additionally infuse a polycarboxylate plasticizer to control the Portlandite content within the required limits.

2. By the change in the amount of low-basic calcium hydroxysilicates in cement stone with C/S ratio not more than 1.5. The cement stone samples are subjected to differential thermal analysis before and after a various number of freezing and thawing cycles. Low-temperature gel-like hydroxysilicates dehydrate upon heating, at 840–870 °C they are crystallized in the form of
wollostanite $\beta$ CaOSiO$_2$ with an exo-effect. If the value of exo-effect has not decreased after a various number of cycles, the content of gel-like phase of hydration products has not changed as well and the cement stone is resistant and durable.

3. The dilatometric method, which calculates the change in the volume or linear size of the sample after a various number of freezing and thawing cycles. We use cement stone or fine grain concrete samples hardened in water. The first freezing to a temperature of -3...-6 $^\circ$C is accompanied by a decrease in sample dimensions both due to the coefficient of thermal compression according to a linear law and additionally due to cryogenic contraction, caused by an increase in the density of adsorbed moisture or gel shrinkage during dehydration under water migration effect [14-16]. If the sample dimensions are unchanged with an increase in the number of cyclic freezing, the structure of the hydrated phases of the cement stone is stable, and the number of cycles will determine the durability of concrete.

4. By residual deformations of concrete samples expansion, with different water/binder ratio (W/B), during the frost resistance testing (Figure 1).

![Figure 1](image)

Figure 1. Elongation deformations of 1:3 fine grain concrete samples at cyclic freezing: 1 – W/B=0.2, SF=10 %; 2 – W/B=0.25, without SF; 3 – W/B=0.25, SF=10 %, 4 – W/B=0.25, SF=20 %; 5 – W/B=0.3, without SF; 6 – W/B=0.3, SF=10 %; 7 – W/B=0.3, SF=20 %.

Samples 1,4 withstood 60 cycles – frost resistance mark more than F$_2$400, samples 2, 3 withstood 40 cycles – more than F$_2$300, samples 6, 7 withstood 20 cycles F$_2$200. Hence, concrete durability in harsh operating conditions is approximately not less than 80 years (1 and 4 compositions), 60 years (2 and 3 compositions), not more than 40 years (6 and 7 compositions).

5. KraShT method (Kramar, Shuldiakov, Trofimov) of comparing moisture absorption of basic and control samples of high performance concrete when testing frost resistance. The samples are characterized by very poor and long-term water absorption, since diffusion absorption prevails, which is facilitated by contractional vacuum and self-drying at hardening of HPC samples. If the value of moisture absorption of basic and control samples is similar (both samples are always in the 5 % sodium chloride solution and are weighted at the same time), this allows us to consider the microstructure of hydrated phases to be identical in control and basic samples. For concrete samples with silica fume and SP-1 plasticizer additives after a stable period of frost resistance testing, provided the mass increment of basic and control samples is the same, a destructive method follows, which is a sharp increase in moisture absorption by the basic samples with constant strength. In this case we observe the cement gel aging, the pores are enlarged to macrocapillaries, and afterwards,
there is a greater increase in weight of basic samples with the loss of strength due to ice formation in macroporipilaries. This method helps to compare different technological effects not only on resistance and density directly in concrete, but also to reveal the stability of cement gel and its aging during frost resistance testing. As a result we observe the optimization of the content and properties of HPC of the required durability.

6. Method for cyclic concrete strength testing. For concrete samples with an unstable structure of hydrosilicate gel there is an increase in strength at the very first cycles of frost resistance testing in comparison with the control samples. This is caused by the penetration of moisture to relic clinker grains, which have been covered with a dense layer of hydration products before cyclic freezing. The increase in strength is due to poor stability of hydrosilicate phases, which leads to fast concrete destruction at cyclic freezing and does not ensure the required concrete durability. Thus, the greater the increase in concrete strength at initial cycles of frost resistance testing (up to 15–18 % in comparison with the control ones), the lower frost resistance and durability of reinforced concrete structures are.

7. Method of determination of diffusion concrete permeability before and after cyclic freezing. In the GOST 31383-2008 there are methods of testing diffusion concrete permeability for carbon dioxide and chlorides. The diffusion coefficient is the result of testing, if it is increased in comparison with the control samples after a different number of cycles, the structure consequently coarsens, ages and leads to poor resistance and concrete durability. The increase in time of ultrasonic signal transition by 15 % in comparison with the initial value is taken as the destruction. The same criterion can be taken for increasing the diffusion coefficient.

In addition to the materials, the monolithic and precast concrete and reinforced concrete technology significantly affects the properties of concrete. One of the main technological process stages is the preparation of a concrete mix. Modern concrete mixing machines provide quick preparation of mixes with a low water content through the use of counter currents, water injection to prevent flocculation, automatic moisture content corrections of aggregates. Forms or molds should be rigid and tight. Physical and chemical processes occurring on the mold-lubricant-concrete mix contact should provide easy demolding of concrete without damaging the products. The values of the tearing or shear forces of concrete applied to the mold model are usually used for the quantitative assessment of various lubricants, if there is a lubricant between them. The most important indicator of the concrete mix quality is its homogeneity and absence of water or solution gain. The quality of concrete is determined by the degree of the concrete mix compaction; the compaction coefficient should be at least 0.98 for high-flow and at least 0.96 for hard concrete mixes. If concrete is intended for structures used in XF1...XF4 environments, it should contain at least 4 % of entrained air. It is not explained how the degree of the concrete mix compaction is assessed during molding.

The cement and water interaction processes begin during molding of products after laying and compacting the concrete mix. Concrete deformations at this stage are conventionally divided into the following types: 1) Internal deformations of the concrete mix (incipient shrinkage); 2) Deformations of hardening concrete: moisture shrinkage and swelling, carbonization and contraction shrinkage; 3) Temperature deformations both due to external heating and heat generation in cement under the influence of the hydration reaction; 4) Deformations due to the action of loads: during short-term (elastic and plastic deformations) and long-term (creep) loading; 5) After laying and compaction of the concrete mix, there may be sedimentation segregation (especially in high-flow and cast mixtures). Water is separated on the surface, the volume of the mix decreases, and the open porosity of the concrete is formed. It is obvious that segregated mixes cannot be used for reinforced concrete structures with increased strength and resistance requirements. The water gain of the concrete mix can be reduced (up to 7.5 times) by replacing a part (about 30 %) of the coarse 10...20 mm fraction of crushed stone with a fine 5...10 mm fraction, with the simultaneous use of screening from crushed stone breakage instead of natural sand. With a decrease in the water gain of the concrete mix, the quality of the concrete surface improves - the number and size of large pores decreases. The
uniformity of the concrete strength increases due to a more uniform compaction degree of the mix when concrete products are molded.

The volume of the cement – water system decreases due to the adsorption and chemical binding of water. Thin films of the cement paste on the surface of the aggregates will experience tensile stress, which can cause cracks - the initial defects, which are stress concentrations, and reduce the strength and durability of reinforced concrete.

Molding based on the use of vibrocompression is effective when using rigid concrete mixes, which have significant internal friction and need a forced movement of particles for their most compact placement in the mold and compaction. The obtained products have a regular shape with clear edges and can be partially or fully stripped immediately after pressing, hardening is accompanied by minimal shrinkage deformations. Compression molding enables us to obtain quick-hardening, dense, and durable concretes, however, it requires significant energy costs. The presence of entrained air in the concrete mix can cause an elastic reaction - expansion of the concrete mix after molding with the formation of cracks.

The accelerated hardening of molded products is a widespread processing technique to increase labor productivity in construction. Most effective are steam treatment methods to accelerate concrete hardening without water evaporation, but as a result of heating, the steam-air mixture expands, which increases the open porosity and reduces the durability of concrete. Thus, the properties of steamed concrete differ from the properties of the concrete hardened under normal conditions due to the following reasons:

- “coarsening” of the structure of the hardened cement paste during the stream treatment of concrete, as the content of the cement gel decreases, and hydrated newgrowths are larger.
- After stream treatment, the hardened cement paste and concrete have an increased (by 8...15 %) porosity depending on the stream treatment mode and conditions. In some cases, the porosity can increase up to 2 times mainly due to an increase in the volume of macropores with a radius of over 100 nm.
- This leads to an increase in the concrete brittleness, a decrease in the fracture toughness, crack resistance, adhesive strength, as well as bending and axial tensile strength and bond to steel. The increased macrocapillary porosity reduces frost resistance, water resistance, and protective properties of concrete in relation to steel reinforcement.
- The main reason for the increase in the volume of the macrocapillary porosity is considered to be the thermal expansion of the liquid and, especially, gas-vapor phases during the heat treatment of concrete, which causes the appearance of pressure and destructive processes with an increase in the external volume.

To stop the negative consequences of the concrete mix expansion during heating, Professor A.S. Arbenev developed installations to preheat the concrete mix; its subsequent compaction reduces the defectiveness of concrete and increases its durability.

3. Conclusions

1. The required durability of high performance concrete of reinforced concrete structures in frost resistance grades can be achieved only taking into account the stability of gel-like cement hydration products.
2. Theoretically, stability is ensured when the content of portlandite in the cement stone is 3–5 % by weight (at the age of 28 days of normal hardening) when using special cements or when CEM I pozzolanization and a polycarboxylate plasticizer capable of binding calcium ions to complexes.
3. The proposed methods for assessing the stability of hydrated gel-like calcium hydrosilicates help us to test cement stone samples, fine-grained and ordinary concrete samples. At the stage of designing the composition of concrete, these methods optimize the materials used and the technological parameters of concrete and reinforced concrete structures production.
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