

Revisiting the Mechanism of Carbonation Shrinkage of Autoclaved Aerated Concrete

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Abstract – In the article the analysis with the generalization of experimental and theoretical studies of foreign and Russian scientists is given in order to compare, substantiate and refine existing scientific theories and statements on the nature and mechanism of carbonation factor effect on shrinkage properties and creep of cement stone and aerated concrete. It is pointed out that according to the existing opinions of foreign scientists (such as Powers, Ramachandran, Feldman, Swenson and Sereda), the mechanism of carbonation shrinkage of cement stone is considered as a topochemical process and consists in dilution of calcium hydroxide at the points of contact of crystals and formation of additional bonds during the precipitation of reaction products in new zones. The mechanism of the shrinkage of autoclaved aerated concrete during carbonation processes is explained in the framework of the scientific theories of Russian scientists E.S. Silayenkov and E.N. Chernyshov. This mechanism is classified as heterogeneous reaction, referring to its kinetic and diffusion component, where the main causes of shrinkage deformations are the intrinsic stresses of the crystalline intergrowth and the polymerization of silica gel, resulting from the interaction of hydrated calcium silicate with carbonic acid.

Keywords – carbonatization; shrinkage and creep; cement stone; aerated concrete; carbonic acid; carbon dioxide; hydrated calcium silicate and calcium carbonate; silica gel; theory and mechanism.

I. INTRODUCTION

According to E.S. Silayenkov [1, 2], E.M. Chernyshov and G.S. Slavcheva [3, 4], the main and decisive criterion for the durability of aerated concrete building envelopes is the structural crack resistance of aerated concrete in large-sized products, which is determined by its operational deformability during the processes of moisture exchange and carbonation.

The carbonation of aerated concrete under the influence of atmospheric carbon dioxide affects its strength, coefficient of elasticity, shrinkage and creep, and also causes, with the increase in the degree of carbonation, the development of tensile stresses in the surface layers of external building envelopes resulting in the decrease in their durability due to the formation of shrinkage cracks [5].

In this regard, it becomes relevant to analyze and summarize experimental and theoretical studies on this issue of foreign and

Russian scientists in order to compare, substantiate and clarify existing scientific theories and statements on the nature and mechanism of carbonation factor influence on shrinkage properties and creep of autoclaved aerated concrete.

The shrinkage deformation of cement pastes, cement-sand mortars and concretes of standard hardening during their carbonation were studied mainly by foreign scientists such as: I. Leber and F. Blake, G. Verbeck, Mayer [1], S. Lentz, T. Powers, Ramachandran and Feldman, Swenson and Sereda [6].

On the basis of research data, several scientific theories on the mechanism of carbonation shrinkage were formed, which, in general, do not comply with each other. However, three of the existing theories, namely, the theories of Powers, Ramachandran and Feldman, and Swenson and Sereda, are in closest agreement with experimental data than others, and therefore are considered the most reasonable and preferable.

The theory of T.S. Powers explains carbonation shrinkage of a cement stone by decomposition (dissolution) of calcium hydroxide crystals under the influence of carbon dioxide. It associates carbonation shrinkage with the stressed state of crystalline skeleton, weakened by decomposition of $\text{Ca}(\text{OH})_2$, and the stressed state of skeleton with capillary phenomena caused by the effect of meniscus at 50% relative humidity. At the same time, Powers believes that the interaction of hydrated calcium silicates with carbonic acid does not lead to shrinkage. This is explained by the fact that calcium ions from hydrated silicates react with CO_2 topochemically (locally in certain areas of solid material) and therefore the dissolution of this phase does not occur during the carbonation reaction. This position of the Powers theory of insolubility during the process of carbonation in the cement stone of hydrated calcium silicate crystals contradicts the experimental data obtained in the studies of Yu.M. Butt, K.K. Kuatbaev [7] and Sh.M. Rakhimbaev [8].

According to the theory of Ramachandran and Feldman [6], at the points of contact with CO_2 , the crystals of $\text{Ca}(\text{OH})_2$ are dissolved and transferred by ion diffusion in sorbing water. At the same time, the voids arise in the structure, in which the crystals enlarge due to van der Waals forces. According to Ramachandran and Feldman, these molecular forces can lead to shrinkage during carbonation, and for this purpose the compressive forces related to the appearance of meniscus are not necessary.

Ramachandran [6] based on large values of carbonation shrinkage of samples from C-C-H compared to shrinkage deformations of $\text{Ca}(\text{OH})_2$ depending on the time of exposure to carbon dioxide suggests that the effect of CO_2 on C-C-H also causes polymerization silica, as noted by Lentz in his earlier studies [6].

The scientific theory of Swenson and Sereda on the mechanism of carbonation shrinkage [6] is based on the fact that shrinkage during carbonation is the result of successive flow of a number of cycles, each of which, in turn, consists of two half-cycles: moisturizing and drying. Local moisture formation, caused by the carbonation reaction itself between $\text{Ca}(\text{OH})_2$ and CO_2 , is the first (moisture) half of the cycle; its second half

(drying) is provided by relatively impermeable films of calcium carbonate, which shield $\text{Ca}(\text{OH})_2$ and therefore inhibit its carbonation process. As a result, it becomes possible to release accumulated moisture into the atmosphere. The destruction of the shielding films during drying leads to the further course of the carbonation reaction of $\text{Ca}(\text{OH})_2$.

II. METHODS AND MATERIALS

According to the analysis of the above mentioned scientific theories and opinions, it is possible to state that the mechanism of carbonation shrinkage of a cement stone of standard hardening consists in dissolution of calcium hydroxide $\text{Ca}(\text{OH})_2$ under the influence of carbon dioxide at the points of contact of crystals, and the formation of new bonds during the precipitation of the reaction product in the form of carbonate calcium CaCO_3 in new zones in the cement stone matrix. At the same time, capillary forces, which lead to compression of the structure due to the effect of meniscus and Van der Waals forces, are important, since they provide a dense particle packing.

The problem of the influence of carbonation on the physical, mechanical and structural changes in aerated concrete was studied by many scientists: Silayenkov E.S., [1] Rozenfeld L.M., [9] Butt I.M., Kumatbaev K.K., [7] Rashkovich L.N., [10] Gumuliauskas A.D., [11] Fedin A.A., [12] Chernyshov E.N., [3, 4] Rakhimbaev Sh.M., [8] Galibina E.A., [13], Kamerlokh N.A. [14]

The chemical and mineralogical composition of the cement changes in carbonation under the influence of atmospheric carbon dioxide in autoclaved aerated concrete. There are changes in the volumes of the solid phase and its crystalline part with the formation of calcium carbonate and silica gel, as well as the appearance of micro-defects during the restructuring of the crystalline part of the solid phase. This leads to the change in strength and the increase in deformative properties of aerated concrete [1, 3, 4, 8, 11].

It was found that the carbonation shrinkage of autoclaved concretes, in particular, cellular, significantly exceeds the moisture shrinkage of these concretes. The first systematic studies of the shrinkage of autoclaved aerated concrete during carbonation, with an explanation of the causes and mechanism of carbonation shrinkage, were conducted by E. Silayenkov [1].

In order to determine the effect of atmospheric concentration of carbon dioxide on the deformation of autoclaved aerated concrete, the authors determined the shrinkage of the samples during long-term storage in a hermetic chamber containing calcium chloride and carboxide, and twin samples located on the racks in the working room. If the samples are stored in the atmosphere without CO_2 (hermetic chamber), then they are less subject to shrinkage than the samples stored on the racks, although the level of concrete drying is the same (relative humidity is 75-80%). The difference in deformation is reasoned by carbonation. This difference, calculated as the arithmetic mean for the three series of samples stored for 2.5-3 years, is 1.1 mm / m for aerated concrete, and 1.3 mm / m for a gas-ash concrete (table 1).

TABLE I. SHRINKAGE OF AERATED CONCRETE, CAUSED BY ATMOSPHERIC CARBONATION CO₂

Concrete	Shrinkage, mm/m, during storage		
	In a chamber without CO ₂	On the racks	Under the conditions of atmospheric CO ₂
Aerated concrete	0,3	1,4	1,1
Gas-ash concrete	0,4	1,7	1,3

The obtained data correspond to the results obtained by E.S. Silayenkov and G.V. Tikhomirov [1] and A.P. Merkin [15], according to which the values of carbonation shrinkage under natural conditions of storage of samples of aerated concrete with a density of 600-700 kg / m³ on a mixed binder are in the range from 1.0 to 1.62 mm / m.

The causes and mechanism of the development of shrinkage of autoclaved aerated concrete with moisture exchange and carbonation processes are most fully studied and highlighted in the scientific works of E.S. Silayenkov and E.N. Chernyshov [1, 3, 4].

They consider the mechanism of moisture deformations through the change in the stress state of a material as a result of the forces of coupling of its solid phase and pore space with water. The mechanism of carbonation deformations is revealed as a part of the macro-kinetics of physico-chemical heterogeneous processes of structure interaction with carbon dioxide. It is shown that the main causes of carbonation shrinkage of autoclaved aerated concrete are the intrinsic stresses of the crystalline intergrowth and the conversion of silica gel released during the carbonation of hydrated calcium silicates [1, 3, 4].

The studies of E.M. Chernyshov [3, 4] are mainly devoted to the structural factors controlling the moisture shrinkage of silicate autoclaved materials. According to his scientific theory, the mechanism of moisture deformations of swelling-shrinkage is considered within the framework of the statement that their manifestation is predetermined by a change in the stress state of a material and a change in its volume due to the "activation" or "release" of forces involved in the bond of its solid phase and pore space with a liquid phase. In this regard, it is indicated that when moistening-dehydrating of any elementary volume of material, the manifestation of the following forces can be observed: capillary forces and surface tension forces, if the content of free, capillary and adsorption-combined water changes in the material; forces of cohesion and adhesive interaction in the contacts of dispersed particles; internal forces in crystal hydrates, if the structure changes the content of interlayer, hydrated and inter-crystalline water; the forces of elastic resistance of the solid phase to its deformation by the forces of surface tension with complete dehydration against the background of simultaneously occurring relaxation processes in it.

As a result, the value of moisture deformations for all characteristic periods of moistening-dehydrating of the material is a function of the resultant of these forces. The measure of these forces in the material is accepted and considered in

relation to its composition and structure, since, actually, it determines the possible content of water types in the material and the balance of forces associated with the structure of the solid phase and pore space.

According to the formed structure of concrete, E.M. Chernyshov [3, 4] justified the application of the model of probable and actual deformation of the structure of aerated concrete during dehydration. In accordance with this, *type I* of shrinkage curves are inherent in materials with a structure differing in increased values of dispersion of particles of the solid phase, total surface area and stock of excess surface energy, and at the same time minimum values of pore size. This is what determines the maximum shrinkage values at all stages of dehydration. *Type III* is characteristic of a material with a structure, the characteristics of the solid phase components of which are distinguished by a relatively greater degree of crystallinity, and the stock of excess surface energy of the structure is minimal. *Type II* is characteristic of materials with intermediate values of structure characteristics.

The principle of indicators management of moisture shrinkage of a material consists in changing the nature and strength of the bond of its solid phase and pore space with water.

The structural factors of managing the operational deformability of macro-porous (aerated) concrete are formulated, the system of formulation-technological factors for their regulation is presented, and the algorithms for constructing optimal parameters of the composition and structure of the solid phase and pore space for them are developed according to a set of specified properties [4].

When assessing the mechanism of carbonation changes of the cementing material, E.S. Silayenkov and E.N. Chernyshov [1, 3, 4], unlike T.S. Powers [6], who believes that this reaction proceeds topochemically, primarily assume that the reactions of interaction between the solid phase and carbon dioxide, developing with the participation of the liquid phase, are qualified as heterogeneous reactions, in the meaning of its kinetic and diffusion components. In this regard it is necessary to take into account the peculiarity of the surface of the solid phase - its reaction area and the reactive activity of a unit of area with respect to CO₂. Such peculiarity is mainly determined by the mineralogical composition and morphology of the new formations of cementing substance.

The action of the CO₂ medium is accompanied by a deep chemical and mineralogical restructuring of the cementing substance of the material, that is, its metamorphism, which is the cause of complex volumetric changes in the material of the substance, associated with dehydration with additional shrinkage.

On the scale of the macro-volume of the material, the carbonation reaction develops by layers, along the normal to the contact surface of the volume of the material with the medium. Hence, the dynamics of the formation of the gradient and the rate of carbonation are naturally associated with the porosity and the structure of the porosity of concrete. The measure of material deformation and destruction due to metamorphic carbonation primary and secondary transformations of its

structure is assumed and appears to depend on the degree of phase transformations (degree of carbonation), the value of the gradient and the rate of manifestation of the gradient of transformations on the characteristic size of the building structure.

On the basis of extensive experiments conducted by E.S. Sylaenkov two stages in the process of development of carbonation shrinkage of autoclaved aerated concrete were distinguished: shrinkage during the carbonation reaction and shrinkage after the termination of the carbonation reaction [1]. Figure 1 shows the generalized experimental curves, which reflect the development of carbonation deformation of twin samples of autoclaved aerated concrete and concrete, hardened under standard conditions.

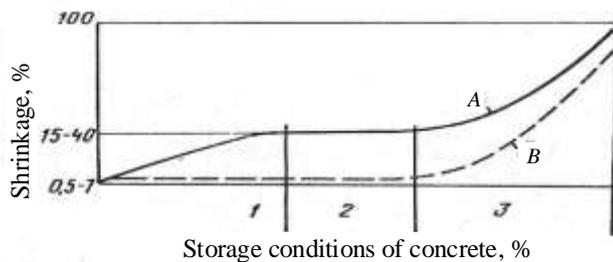


Fig. 1. Development character of carbonized shrinkage A – autoclaved aerated concrete; B – aerated concrete of standard hardening; 1 – carbon dioxide effect under conditions that release moisture from concrete to the environment; 2 – moisture storage; 3 – storage under the conditions that do not prevent the loss of concrete moisture

It can be seen that the carbonation shrinkage of autoclaved concretes is manifested during the interaction of calcium hydroxide and hydrated silicates of cement stone with carbon dioxide, even if all the initial moisture and moisture released from the hydrated silicates during their carbonation is retained. Shrinkage of autoclaved concrete at this stage is 15–40%, and non-autoclaved is 0.3–7% of total carbonation shrinkage.

Under the conditions of moisture storage after the end of the reaction, the carbonation of deformation is not observed both in autoclaved samples and in concrete of standard hardening. It begins to appear only when concrete loses moisture. At this stage, the autoclaved concrete shrinkage is 85–60%, and non-autoclaved is 93% of the total carbonation shrinkage. It is characteristic that with different shrinkage at individual stages, the final value of shrinkage of twin concrete is close, regardless of the conditions of heat and humidity hardening. Obviously, the causes of shrinkage in the first and second stages should be different.

In the first stage, shrinkage is not associated with the decrease in moisture in concrete. Moreover, shrinkage occurs despite the increase in humidity and the increase in the volume of the solid phase of concrete. The peculiarity of the development of shrinkage at this stage is that in non-autoclaved concretes it almost does not manifest itself, and in autoclaved ones it has a significant value. Consequently, shrinkage should be related to the state of the crystalline intergrowth at the time of the beginning of its restructuring due to the effect of carbon dioxide on it. At the second stage, the connection between shrinkage and loss of moisture by concrete is fixed. At the same

time, most shrinkage is irreversible and therefore not associated with capillary phenomena.

Analyzing the results of the study of shrinkage deformations during the course of carbonation, E.S. Silayenkov [1] expressed the scientific theory that the interaction of atmospheric carbon dioxide with hydrated calcium silicates of autoclaved concrete leads to its shrinkage. According to his theory the main reasons for shrinkage were the inherent stresses of the crystalline intergrowth and the conversion of silica gel released during the carbonation of hydrated silicates.

The emergence of inherent stresses in concretes of mineral binders, many researchers associate with the development of crystallization processes and the emergence of crystallization pressure during the accretion of a crystalline intergrowth, as well as with phase transformations leading to a change in the volume of the solid phase.

As E.S. Silayenkov noted, temporary concrete strength drops, the cause of which was the development of inherent stresses, were mentioned by V.I. Kind in 1932. This connection was indicated in the scientific works of P.P. Budnikov, A. V. Volzhenskii, A. F. Polak, T. I. Liubimova, P. A. Rebinder and E. E. Segalova et al. [1]. However, they did not carry out special studies on the nature of the occurrence and manifestation of these inherent stresses.

The process of occurrence of inherent stresses in cement stone due to the crystallization pressure is presented in Figure 2. In a supersaturated solution, nucleation centers appear spontaneously, on which individual elements of the primary crystalline intergrowth grow (Fig. 2a).

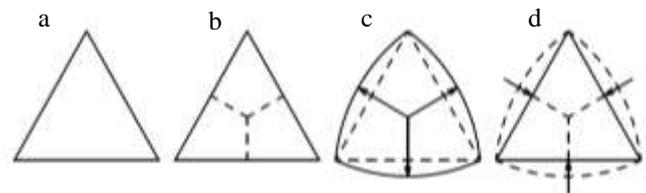


Fig. 2. Scheme of inherent stresses occurrence in a crystalline intergrowth a - primary intergrowth; b - occurrence of a secondary intergrowth; c - occurrence of crystalline pressure during the accretion of primary and secondary intergrowths; d - destruction of contacts of accretion during carbonation; 1 - elements of the primary intergrowth; 2 - elements of the secondary intergrowth

If at the same time, the supersaturation remains significant, then along with the primary intergrowth, new nucleation centers are formed, and a new secondary crystalline intergrowth is formed on them (Fig. 2, b). The secondary intergrowth tends to move the elements of the primary intergrowth and brings them into a stretched state (Fig. 2c). The elements of the secondary intergrowth will be compressed.

Thus, the crystalline skeleton of concrete is in a tense state, some of its elements are compressed, others are stretched, but on the whole the system is in balance. If such a system is affected by an atmospheric factor that is aggressive with respect to new growths of cement stone, for example, air carbon dioxide, then, first of all, the intergrowths of the secondary and primary intergrowth decompose as defective and

thermodynamically unstable. At the same time, the elements of the primary intergrowth are compressed, trying to return to the initial state (Fig. 2d) and shrinking the entire system.

This fact explains why the inherent stresses of a crystalline intergrowth, as it decomposes, lead to shrinkage, and not to swelling.

The overall qualitative picture of the effect of inherent stresses on the shrinkage of concrete in the process of their interaction with carbon dioxide was revealed by testing twin samples that were hardened under standard conditions and under high-pressure steam processing.

The quantitative effect of inherent stresses on shrinkage of aerated concrete during carbonation in the first approximation was estimated by the study of the shrinkage of twin samples with the same specific content of combined SiO₂, obtained at different intensities of crystalline intergrowth formation.

In order to determine the quantitative influence of the inherent stress level on the deformation of aerated concrete in the process of decomposition of new formations of cement stone with carbon dioxide the bars of 4x4x16 cm in size made from cement-sand aerated concrete of one grouting, were divided into 9 batches and steamed in various modes. The modes were set by extrapolating data from S.A. Mironova and L.A. Malinina on the rate of binding of SiO₂ sand at different pressures in the autoclave in order to obtain in the same specific

value of combined SiO₂ at both 0.8 and 1.6 MPa in concrete stone [1].

In the experiments, Portland cement with a calculated content of C₃S - 46.34, C₂S - 28.93, C₃A - 8.64 and C₄AF - 14.09% and quartz sand with SiO₂ content of 96.8%, ground to a specific surface area of 3000 cm²/g was used. The density of concrete is 700 kg / m³. The degree of carbonation of the samples ranged from 69.2 to 73.6%.

As results of the experiment, 3 groups of concrete were obtained, differing in the ratios of CaO to SiO₂ (Table 2.). In the 1st group the average value of this ratio is 1.2, in the 2nd - 1.07 and in the 3rd - 1.02. In each group, the compressive strength of concrete decreases and increases the intensity of a crystalline intergrowth formation. This is the effect of inherent stresses.

These stresses are as high, as fast a certain level of binding of silicon dioxide is achieved, which is reflected in the value of complete carbonation shrinkage. For example, for the 1st group of concrete, shrinkage of concrete with a maximum level of inherent stresses in the carbonation process by 1.3 mm / m exceeds the shrinkage of concrete with a minimum level of inherent stresses. The difference in the total carbonation shrinkage of these concretes is also 1.3 mm / m.

TABLE II. CHANGE IN STRENGTH AND SHRINKAGE IN THE CARBONATION OF AERATED CONCRETES WITH THE SAME SPECIFIC CONTENT OF COMBINED SILICIC ACID

Mode of steam	Autoclave pressure, MPa	Concrete compression strength		After carbonation, MPa	Concrete shrinkage during carbonation		Full carbonation shrinkage, mm / m	CaO to SiO ₂ ratio
		Before carbonation						
		MPa						
2+4+10	0.8	9	100	6.7	2.5	100	6.1	1,18
2+2+10	1.2	7.8	87	6.8	3.2	128	6.8	1,28
2.5+1+6	1.6	6.6	74	5.1	3.8	152	7.4	1,15
Average values and indicators in the group		7.8		6.2				1.2
	0.8	8.8	100	6.6	1.9	100	6.1	1,13
	1.2	7.3	83	6.2	3	158	6.6	1,08
	1.6	7.3	83	6.4	3.2	169	7.6	1
Average values and indicators in the group		7.8		6.4				1.07
	0.8	10	100	7.3	2.7	100	7.1	1,04
	1.2	7.2	74	6.7	3.6	134	7.3	1,03
	1.6	6.3	64	5.8	3.5	130	7	1,02
Average values and indicators in the group		7.8		6.6				1.02

The effect of moisture loss in concrete after the process of carbonation.

Concrete shrinkage after the carbonation process occurs only when the moisture content of the concrete decreases. The question arises whether this shrinkage is not the usual deformation observed when concrete loses moisture. However, shrinkage, related to 1% of moisture lost after carbonation, is

an order of value greater than the same value of shrinkage of the same concrete before carbonation.

For example, for autoclaved lime-sand aerated concrete, specific deformation with loss of moisture at 20°C before carbonation was 0.045 mm / m, and after carbonation - 0.25 mm / m for 1% decrease in humidity, and for cement-sand concrete, respectively 0.02 and 0.38 mm / m for a 1% decrease in humidity.

Consequently, these are different types of moisture loss. If most of the moisture lost by autoclaved concretes before carbonation is related to capillary moisture, then the same cannot be said about the moisture released after carbonation. This is evidenced by the fact that the shrinkage from the loss of autoclaved concrete moisture before carbonation is almost completely reversible, and after carbonation, most of it is irreversible. For example, for the above mentioned lime-sand concrete, the irreversible part of the shrinkage during drying before carbonation was 1.5%, and after carbonation it was 68%.

On this basis, E.S. Silayenkov [1] comes to the conclusion that the shrinkage of aerated concrete, which occurs when moisture is removed from concrete after the carbonation process, is associated with the shrinkage of the SiO₂ gel formed during the decomposition of hydrated silicates. It is known that water differences of CaCO₃ are extremely rare and special conditions are necessary for their formation. Therefore, during the carbonation of concretes, all the combined water of the new

formations of cement stone is released together with the gel — SiO₂. The water content in it can reach 30-35%. Over time, the water content in the SiO₂ gel decreases. This leads to the shrinkage of concrete. The data in Table 3 show that the shrinkage of aerated concrete with loss of moisture after carbonation varies widely. The amount of silicon dioxide gel formed during the carbonation of concretes was determined by calculation based on the SiO₂ content in cement introduced into concrete, the basic capacity of the most important hydrated silicates obtained under these hardening conditions and the degree of carbonation of concrete.

The comparison of the shrinkage of aerated concrete, which occurs when they lose moisture after carbonation process, and the value of silica dioxide, formed during the decomposition of new formations of cement stone, shows a certain dependence of these values. The ratio of the shrinkage to the amount of the silica dioxide gel is almost the same for all concretes, regardless of hydrothermal hardening conditions.

TABLE III. INFLUENCE OF HARDENING CONDITIONS OF AERATED CONCRETE ON SHRINKAGE AFTER THE CARBONATION PROCESS

Concrete	Conditions of steam processing	Concrete shrinkage with moisture loss after carbonation, mm / m	Silica gel content in cement, formed during carbonation,% by weight	Ratio of shrinkage to the amount of silica gel
Cement based	Standard hardening	3.13	6.55	0.48
	Steam hardening	3.1	6.03	0.51
	Autoclaved hardening	6.55	13.55	0.48
Lime based	Standard hardening	0.8	—	—
	Autoclaved hardening	4.31	9.1	0.49

III. CONCLUSION

The value of shrinkage of aerated concrete with loss of moisture after the carbonation process depends on the condition of heat and moisture processing, as well as on the amount of SiO₂ gel formed during the decomposition of the components of cement stone by carbon dioxide. This suggests that the value of shrinkage during carbonation is also closely related to the type of new formations.

Thus, the working theory of E.S. Silayenkov is experimentally confirmed. According to it shrinkable deformations of autoclaved aerated concrete during their carbonation are related to the level of inherent stresses of the skeleton of material and to the transformations of silica gel released during the decomposition of crystalline hydrates of cement stone.

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