doi: 10.18720/MCE.75.16

Expanding cements hardening within the limited deformations conditions

Гидратация и структурообразование при твердении расширяющихся цементов в условиях ограниченных деформаций

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Key words: hydration product; amorphous phase; calcium sulfoaluminate; x-ray diffraction; Portlandite; ettringite

Ключевые слова: продукты гидратации; аморфная фаза; сульфоалюминат кальция; рентгеновская дифракция; портландит; эттрингит

Abstract. The features of expanding cement strength and structure formation at different constrained deformation conditions were studied (without constraint of deformations, uniaxial constraint of deformations, biaxial constraint of deformations, and triaxial constraint of deformations). The study was conducted for three doses of 5 %, 15 % and 25 % of expander on the calcium sulfoaluminate basis in Portland cement of CEM 42.5 mark. For experimental data interpretation in the analysis of structure and phase composition, the combination of methods of x-ray diffraction, differential scanning calorimetry and scanning electron microscopy were used. The constraint of expansion deformations has a significant impact on the volume and nature of the expanding cement pore structure, and leads to the formation in the volume of cement stones of black agglomerates, which represent the mixture of portlandite and ettringite crystals, uniformly distributed in the amorphous CSH-phase. It has been established that by increasing the expander amount and the degree of deformation constraint increases the number of black agglomerates and an increase in compressive strength.

Аннотация. Изучались особенности набора прочности и структурообразования расширяющихся цементов в различных условиях ограничения деформаций (без ограничения деформаций, одноосное ограничение деформаций, двухосное ограничение деформаций, трехосное ограничение деформаций). Исследования проведены для трех дозировок 5 %, 15 % и 25 % расширяющейся добавки на основе сульфоалюмината кальция в портландцемент марки CEM 42,5. Для интерпретации экспериментальных данных при анализе структуры и фазового состава использовалась комбинация методов рентгеновской дифракции, дифференциальной сканирующей калориметрии и сканирующей электронной микроскопии. Ограничение деформаций расширения оказывает существенное влияние на объём и характер поровой структуры расширяющегося цемента, а так же приводит к формированию в объеме затвердевшего цементного камня чёрных агломератов представляющих собой смесь кристаллов портландита и эттрингита, равномерно распределённых в аморфной CSH-фазе. Установлено, что при увеличении количества расширяющей добавки и степени ограничения деформаций возрастает число черных агломератов и наблюдается рост прочности при сжатии.

Харченко И.Я., Пустовгар А.П., Пашкевич С.А., Еремин А.В., Иванова И.С., Баженов Ю.М., Харченко А.И. Гидратация и структурообразование при твердении расширяющихся цементов в условиях ограниченных деформаций // Инженерно-строительный журнал. 2017. № 7(75). С. 161–170.

Introduction

One of the areas of effective use of expanding cements and concretes based on them is butt joints sealing of concrete and reinforced concrete structures [1, 2]. Controlled expansion allows ensuring the functional characteristics uniformity of structures [3–5]. For these purposes, high workability mixtures are mainly used [6], which evenly fill the butt joints volume and the hardening at the flat or volumetric constraint deformation conditions expansions [7–8]. The energy, released during the expanding cement system hardening, is spent for prestressing of the butt joint concrete that provides the hardening concrete structure compaction and creates reinforcement stress when butt joint reinforcing. The uniformity of sectional construction functional characteristics with butt joints sealing using expanding concretes is determined not only by the concrete composition and expanding cement properties [9–12], but also by the expansion deformation constraint conditions.

There are publications of volumetric expansion magnitude studies [13–16], which describe the structure and properties formation mechanisms, depending on the expansion deformation magnitude, expanding cement composition and hardening conditions in the absence of constraint of expansion deformations, while for sulfoaluminate systems the main criterion for expansion is the crystallization pressure, caused by ettringite intense crystallization [17, 18]. However, the structure and properties formation mechanisms of expanding cements at the constrained deformation conditions have been studied to date only in part of the reinforcement post-stress by the expanding concretes [19].

Materials and method

In order to study the influence of constrained deformation conditions on the structure and properties of hardened cement stone and concrete, on its basis the comprehensive studies of expanding cement were made, prepared by homogenizing of the base portland cement of CEM 42.5 mark with low content of C3A= 3.2% and expander on the calcium sulfoaluminate and calcium sulfate dihydrate basis in stechiometric ratio, required for the ettringite formation. The expanding cement is prepared by intergrinding of Portland cement with expander components in the amount of 5 %, 15 % and 25 % by Portland cement mass [20].

The study of expanding cement properties at the different constrained deformation conditions were conducted in accordance with method [16], through the preparation of cement-sand mortar in the ratio of the expanding cement: sand = 1 : 1 with water-cement ratio of 0.30.

To determine the linear expansion the beam samples with dimensions 40x40x160 mm were made. Measurement frequency was 1, 2, 7, 14, 21 and 28 days. Linear expansion of each beam sample (%), was calculated according to the following formula:

$$\Delta l = \left[\left(n_2 - n_1 \right) / l \right] \times 100, \tag{1}$$

where n1 – a reading taken during the measurement of a beam sample that was released from the mold after 1 day from the start of cement mixing, mm;

l – length of the standard sample, l =160 mm.

As a linear expansion of tensile cement the average results of three measurements of beam samples was assumed.

The result was rounded to 0.01 %.

The constraint of deformations was carried out using the dynamometric rings for biaxial constraint of deformations and with the use of dynamometric conductors for uniaxial constraint of deformations (Fig.1). The expansion pressure of the beam sample in the ring (MPa) was calculated according to the following formula:

$$\sigma = 2\Delta l, \tag{2}$$

where Δl – deformation value of the ring in the direction of the sample axis, mm.

The cement expansion pressure was calculated as an average result of three measurements of the beam samples molded from a single batch. Calculations were carried out with accuracy of 0.01 MPa.

Figure 1. The influence of expansion deformation constraint degree on the change in volume of the expanding concrete of B 35 grade (2 ÷ 4).
1 – fine-grained concrete without expanders at normal hardening; 2 – fine-grained concrete with 15% of expander, the hardening without the deformation constraint (expansion is 185 mm/m); 3 – fine-grained concrete with 15% of expander, uniaxial expansion constraint (expansion pressure is 3.9 N/mm2); 4 – fine-grained concrete with 15% of expander, biaxial expansion constraint (expansion pressure is 15.3 N/mm2)

The study of the structure and properties of expanding cement at the triaxial expansion constraint conditions was conducted according to the method [21], the cement-water paste of normal density for the manufacture of samples was used. For studies of the influence of triaxial deformation constraint on the structure and properties of expanding cements and concrete, the measuring chamber was used. The overall view of the measuring chamber is shown in (Fig. 2). The camera consists of a massive metal vessel with the screw cap. The cap is equipped with a liquid pressure gauge and valve for air drain from the chamber. The samples of cylindrical shape of diameter 50...70 mm from the cement stones or cement-sand mortar are prepared for studies, the ends of which are closed with plastic washers of appropriate diameter, which eliminates the contact of the sample with outdoor environment.

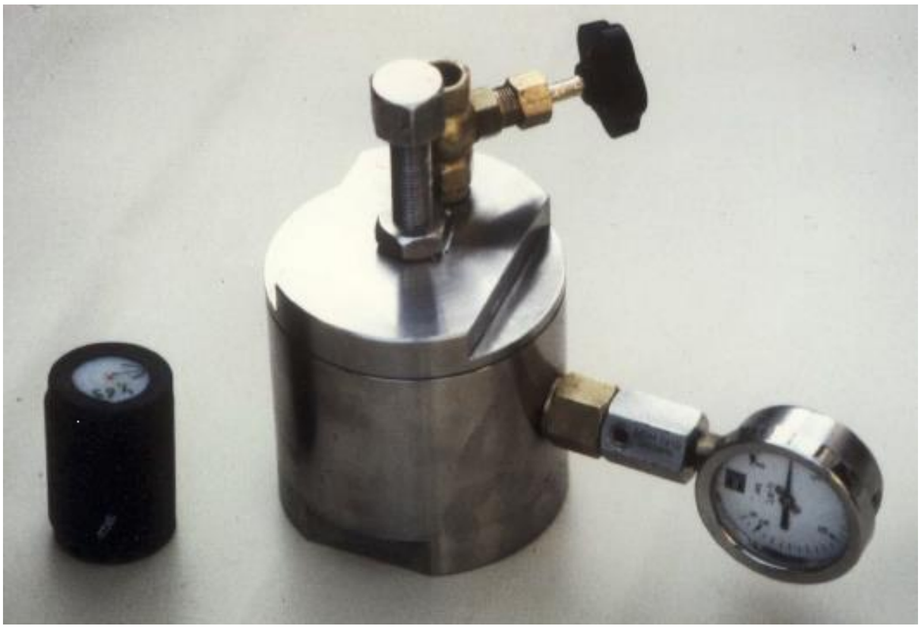

Figure 2. Measuring chamber for the structure and properties of expanding cement study that hardens at the isotropic deformation constraint conditions

After hardening at normal conditions for 24 hours, the samples are released from the formwork and placed in a heat-shrinking tube of the appropriate diameter, which, after short-time heating with hot air, is securely pre-stress the sample all around except for its contact with the outdoor environment. In this case, the expandable tube can deform without preventing the expansion process. The prepared sample is placed in a measuring vessel filled with water, went trough a preliminary treatment for air elimination. The vessel is tightly closed with screw cap. In the vessel there is a pre-stress of 0.01 MPa, which is taken as the initial reference. The measuring chamber is in the room with a constant temperature of 20 ^{0}C to complete stabilization of the expansion process. Indicators of the pressure gauge are recorded every hour in an automatic mode with the use of special recording device.

Studies of the composition of hydration products were carried out using quantitative X-ray diffraction (XRD) and differential scanning calorimetry (DSC) methods. X-ray phase analysis was carried out on a powder X-ray diffractometer ARL X'TRA (Switzerland) equipped with a narrow-focus tube 2200 W (Cu anodes) and an energy-dispersive solid-state detector with a Peltier cooler. The registration of the diffractograms was carried out in a step-by-step mode (with a step of 0.02° 2Θ), in the angle range 2Θ°=4-70°. For qualitative phase analysis, the database ICDD PDF-2 was used. The analysis was carried out on interplanar distances in manual mode using the Hanavalt method and in semi-automatic mode using the software Oxford Crystallographica Search Match. Non-standard quantitative X-ray phase analysis by the Rietveld method was carried out using the software Siroquant 3 Sietronics Pty Ltd. For all phases, the following parameters were specified: scale factor, zero offset of the instrument counter, background parameters (the Chebyshev polynomial of 5th degree), unit cell parameters. Also, the profile parameters varied during the adjustment - the profile function Pearson VII (U, V, W in the Kalotti dependence) was used.

To study the thermal behavior of the samples, the apparatus for carrying out thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) SETARAM LABSYS TGA / DSC / DTA (France) was used. The obtained samples were abraded in an agate mortar with an agate pestle and then sieved through a sieve with a 008 cell. The abrasion process was repeated until the entire sample passed through a sieve. Then we weighed a sample of each sample with a mass of 50 ± 2 mg and weighed on an analytical scale with an accuracy of 0.0001 g. The sample was then placed in a corundum crucible for analysis. Measurements of the thermal behavior of the samples were carried out in a temperature range of 50 °C to 1000 °C with a stabel heat rate of 10 °C/min in the atmosphere of air.

The microstructure of the samples was studied on a FEI Quanta 200 scanning electron microscope with an Apollo 40 elemental analysis attachment. Samples were fixed to the sample holders with carbon scotch tape. Studies of the microstructure were carried out in the regime of deep vacuum.

Results

Figure 3 presents the results of a study of the compressive strength of fine-grained concrete, depending on the conditions of limited expansion deformations.

It was found that with an increase in the degree of deformations restriction, an increase in the compressive strength is observed. In the case of triaxial and biaxial constraints, an increase in strength is observed with an increase in the amount of the expanding admixture in fine-grained concrete. With uniaxial deformation constraint, the drop in strength is observed when the content of the expanding additive in the cement is 15 %. During the hardening process of the fine-grained concrete with expanding admixture without deformation restrictions, the compressive strength decreases even when the content of the expanding admixture in cement is 5 %.

It has been established that with increase of the deformation constraint degree there is the increase in strength under compression. In the case of biaxial deformation constraint, the strength increase is observed with the expander amount increase in the fine-grained concrete composition. The strength growth under compression for uniaxial deformation constraint is terminated when the expander content in the fine-grained concrete is 2.5 % by the total mass. While fine-grained concrete hardening with the expander without deformation constraint, it significantly reduces the strength with the expander content increase more than 0.5 % by the total mass. That confirms the results of fine-grained concrete strength properties studies on the expanding cements basis [22–26], where a sharp decrease in strength with calcium sulfoaluminate content increase in the expanding cement composition was observed [16, 27, 28].

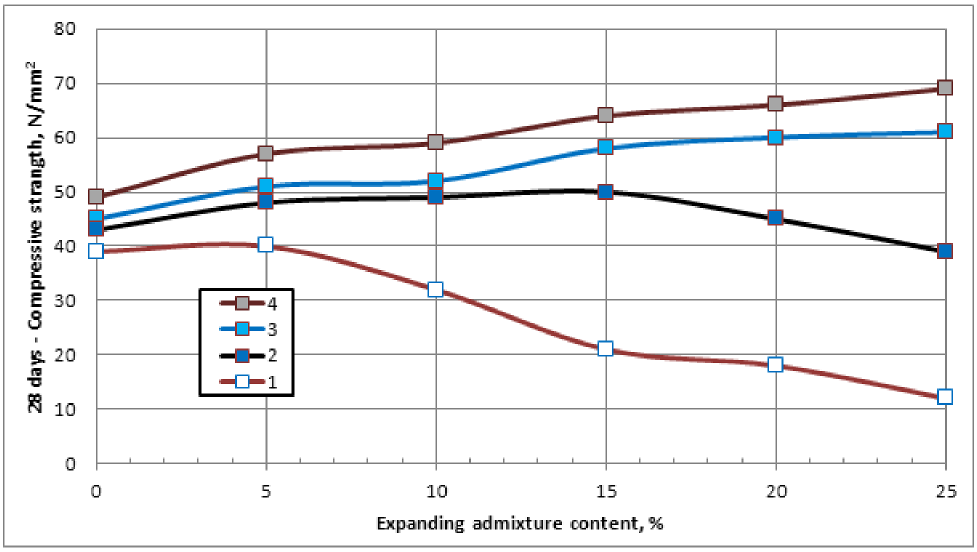

Figure 3. The influence of expansion deformation constraint conditions on the strength under the concrete compression: 1 – without deformation constraint; 2 – uniaxial deformation constraint; 3 – biaxial deformation constraint; 4 – triaxial deformation constraint

The analysis of the study results showed that the expansion deformation constraint has a significant impact on the volume and nature of the expanding cement pore structure while hardening. It also has been established that the extent of this influence increases in proportion to expansion potential capacity increase.

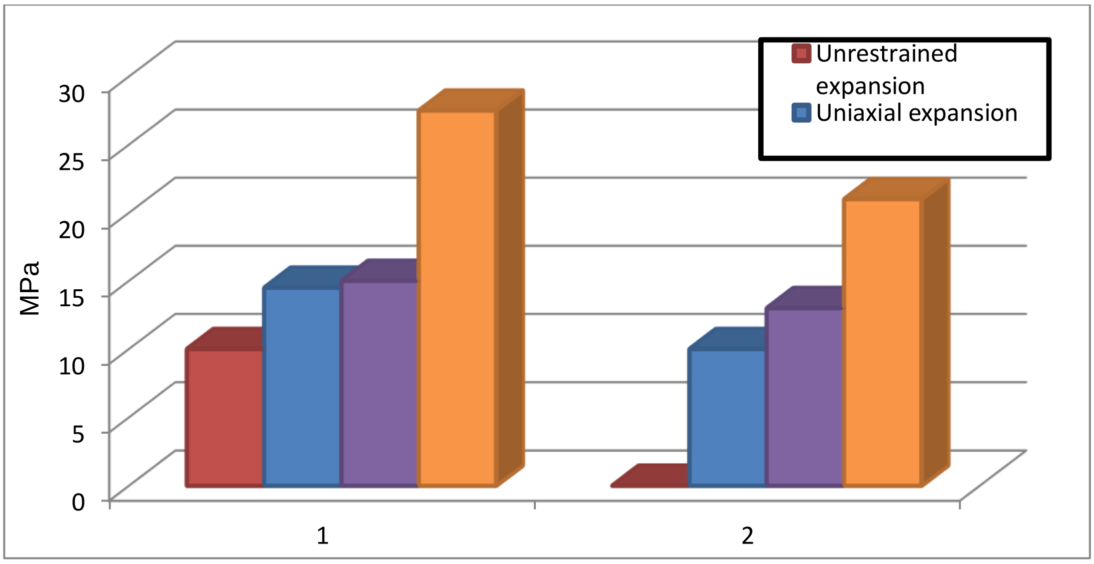

Figure 4. The influence of constraint degree of the expansion process on the strength under compression, porosity and expansion pressure: 1 – strength under compression; 2 – pressure under stable expansion

As the expansion potential capacity, the expansion value during sample hardening without deformation constraint at normal temperature-humidity conditions has been established (temperature – 20 ^{0}C, humidity – 50%) [21, 29]. During the introduction in the base cement composition of 5 % of expander, the deformation constraint degree has not substantially influence on the cement stone strength and pore structure. Compared to the base Portland cement strength under compression, the strength under compression increases by 6-8% regardless of the constrained deformation conditions.

During the introduction in the cement composition of 15% of expander, the expansion potential capacity increases by 12 mm/m and in this case, the sample strength under compression, hardening under the uniaxial and biaxial expansion constraint increases by 7 % and 10 % respectively (Fig. 4). At the same time, the significant differences in volume and nature of the pore structure were not established. While expanding cement hardening, which contains in its composition 25% of expander, the expansion potential capacity increases by 185 mm/m. In this case, while uniaxial expansion deformation constraint the self-destruction of concrete structure at the sample centerline is observed that do not have expansion constraint (Fig. 1). While biaxial deformation constraint, a fairly dense and solid structure of cement stone is formed (Fig. 3) and deformation in “free” direction does not exceed 5% of the expansion potential capacity value.

Of special scientific and practical interest are the study results of expanding cement structure formation processes under triaxial expansion deformation, when trough the entire volume of hardened cement stone the anomalous inclusions of the black color are fairly formed, which optically resembles the filler grains with a size of 0.1 to 5.0 mm. (Fig. 5)

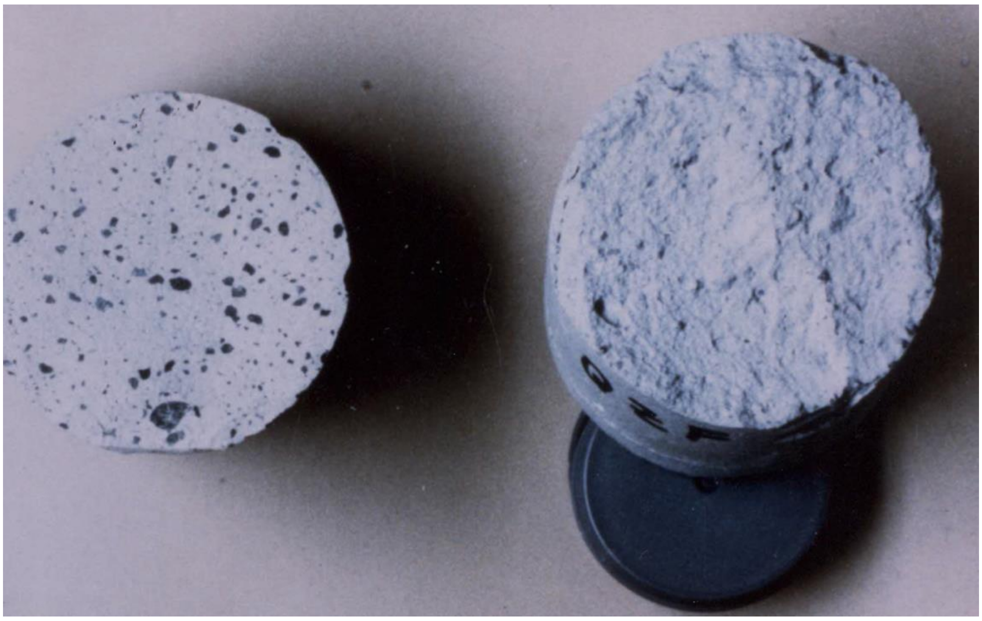

Figure 5. The formation of black aggregates in the cement stone volume, hardening at isotropic deformation constraint conditions (left sample) and the absence of aggregates during hardening without deformation constraint (right sample) at an expansion potential capacity of 185 mm/m

The studies performed using optical polarizing microscope and electronic scanning microscope, did not allow identifying a clear border zone between the cement stone matrix, which has a gray color and black agglomerates. The agglomerate microstructure is extremely dense, continuous, and substantially free from micro cracks and pores. In this case the studies of hydration products, performed using DSC and XRF analysis has allowed establishing that for the cement stone matrix the predominant content of hydrosilicate phase and hydrosulfoaluminate of monosulfate form is typical.

Table 1. The results of XRF, DSC and ISA of hydration products after the statistical processing

Hydration products	**Sample 1**	**Sample 2**	**Sample 3**	**Sample 4**
Ettringite	36.7 %	62.3 %	57.7 %	55.8 %
Monosulfate	22 %	small amount	small amount	small amount
Portlandite	4 %	5.8 %	5.4 %	4.9 %
Calcite	6.4 %	9.0 %	10.2 %	8.9 %

Note: sample 1 – grey part or matrix; samples 2 and 4 – black agglomerates; sample 3 –transition zone between the “matrix” and black agglomerates.

In the study of the black agglomerate phase composition, a preferential content of ettringite was established, as well as the high content of portlandite and calcite, the number of which is more than 1.5 times higher than their content in the cement matrix (Table 1).

Thus, the above crystallohydrates are located in a dense environment of CSH-phase. Using X-ray microstructural sensing of different zones of black agglomerates, the presence of only AFt-phase was established, while in the section zone of black agglomerates and the gray matrix, a preferential content of AFm-phase was established. This can be explained by increased water demand of crystallizing AFt - phase, at which point in adjacent regions, containing sulfates and calcium, reactive water and the sulfate component is sufficient only for the AFm-phase formation. In the main volume of the black agglomerates there is a mixture of crystals of portlandite and ettringite, uniformly distributed in the CSH-phase. In this area there are ordinary ettringite crystals of in the form of needle-like prisms, and its morphological structure resembles the form of fused crystals (Figs 6 and 7).

Figure 6. Mixed crystal of hydration products (ettringite, portlandite, hydrosilicate) in the black aggregate volume: zoom x10000

Figure 7. Transition zone between the black agglomerate and gray matrix, zoom x10 000: ettringite, portlandite, silicate hydrate, calcite

Discussion

The number and density distribution of black agglomerates in the cement matrix volume increases in proportion to the pressure magnitude, developing in the process of expanding cement hardening in the complete expansion deformation constraint. The emergence of black agglomerates was identified in the expansion pressure exceeding 0.4 MPa, The process of their formation is probably associated with the reaction system equilibrium state disturbance at the isotropic expansion constrained deformation conditions and change of component chemical potential, involved in the development of hydration reactions. It has a significant impact on changing the crystallohydrate habit with preferential formation of microcrystalline forms, as well as high content of CSH-phase in the black agglomerate volume. In accordance with the Taylor’s theoretical views [30], the morphology of the forming crystallohydrates is primarily determined not only by the ion ratio in the reaction medium, but the temperature and humidity and adiabatic conditions, under which the hydration processes are developing.

The distribution of individual black agglomerates by the volume of hardening cement stone may be a result of sulfoaluminate expander mixing with the base Portland cement while preparing a dry mixture and cement-water paste and during the introduction of water of mixing. In this case the calcium aluminates function as centers of crystallization, and the growth rate and the size of the producing crystallohydrates in the aquatic environment are determined by diffusion processes at the interface of the reaction phases.

Optical anomalies, such as established in the study of structure formation processes of expanding cements at the full expansion constrained deformation conditions, exist in cement silos and filters on the cement plants, when while their cleaning the layers of hardened cement stone with separate inclusions are removed, which are of the dark gravel form [31].

Conclusions

Experimentally determined anomalous phenomena during expanding cement hardening on the calcium sulfoaluminate basis at the expansion constrained deformation conditions are determined by the combination of two processes – structure strengthening due to hydration sulphoaluminates and silicates of calcium and the ettringite formation, thus the constraint of the deformation on the one hand increases the degree of self-stress, and on the other hand reduces the role of structure plasticity of cement stone newgrowths while hardening, which allows in wide ranges modifying the concrete properties on the expanding cement basis and ensuring the uniformity of the structure functional characteristics. Further theoretical and experimental studies of expanding cement hardening on the calcium sulfoaluminate basis at the expansion constrained deformation conditions opens up new possibilities for obtaining concrete products and constructions with the structure and properties, commensurate with ceramics, but without the use of cost demanding processes, only by chemical energy, released during the hydration of sulfoaluminate systems.

Acknowlegement

This work was financially supported by Ministry of Education and Science of the Russian Federation (7.6666.2017/8.9). All tests were carried out using research equipment of The Head Regional Shared Research Facilities of the Moscow State University of Civil Engineering.

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Kharchenko I.Ya., Pustovgar A.P., Pashkevich S.A., Eremin A.V., Ivanova I.S., Bazhenov Yu.M., Kharchenko A.I. Expanding cements hardening within the limited deformations conditions. *Magazine of Civil Engineering*. 2017. No. 7. Pp. 161–170. doi: 10.18720/MCE.75.16.