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The phase composition and properties of aluminate cements after early loading

Фазовый состав и свойства алюминатных цементов при раннем нагружении

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Ключевые слова: глиноземистый цемент; высокоглиноземистый цемент; раннее нагружение; прочность на сжатие; прочность на растяжение при изгибе; рентгенофазовый анализ; дифференциально-термический анализ

Abstract. It is widely recognized that the effect of loading at the early stages of hardening enables to increase strength characteristics of cement systems and composites based on them. Of particular interest is a study on the effect of compression of aluminate cements on physicomechanical characteristics, hydration process and phase transformations. The research focuses on maximum compressive and flexural strength, the peak intensity of the main phases and hydrate products, characteristics of DTA curves after early loading, studied by means of physical and chemical methods. The authors note an increased flexural strength of specimens exposed to loading at an early age. The comparison of diffractograms showed that the peaks of the main phases were reduced during the compression stage, as well as the changes in the amorphous structure of the stone. The differential thermal analysis showed no change in bound water content.

Аннотация. Воздействие нагрузки на ранних этапах твердения позволяет получать прирост прочностных показателей цементных систем и композитов на их основе, что является общеизвестным фактом. Интерес представляет исследование влияние сжатия структуры алюминатных цементов на физико-механические характеристики, процесс гидратации и изменение фазового состава. В работе при помощи физико-химических методов изучались предел прочности на сжатие и растяжение при изгибе, интенсивность пиков основных фаз и гидратных продуктов, характер эффектов на кривых ДТА при раннем нагружении. Авторами отмечается увеличение прочности на изгиб образцов, подвергнутых нагрузке в раннем возрасте. Путем сравнения дифрактограмм установлено снижение пиков основных фаз при сжатии структуры, а также различия в аморфности структуры камня. Несмотря на отмеченные изменения, дифференциально-термический анализ показал отсутствие изменений связанной воды.

Introduction[1]

Calcium aluminate cements (CAC), known as Fondu cements, have become widespread in construction industry, including the production of high-performance concretes (HPC) [1], because of the following properties [2–5]:

- Rapid strength development;
- High resistance to corrosion;
- Stability at high temperatures and flame resistance.

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[1] Notation: C=CaO; A=Al_2O_3; H=H_2O; S=SiO_2.

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An intensive hardening is accompanied by an increased heat release during hydration, and within 24 hours about 70–90 % of all heat should be released, while the temperature of material can reach up to 1000°C [6]. The development of structure is mainly occurs through the hydration of calcium monoaluminate CA [7]. The most important hydroaluminates are CAH_{10}, C_2AH_8, C_4AH_x (x = 13-19), C_3AH_6 [8–9] (cubic phase), and AH_3 [10] as an amorphous gel which crystallizes to gibbsite.

The following chemical equations demonstrate the effect of temperature on the composition of hydration products [11–12]:

- ($T \leq 15^0C$) $CA + 10H \rightarrow CAH_{10}$;
- ($15^0C < T \leq 30^0C$) $2CA + 11H \rightarrow C_2AH_8 + AH_3$;
- ($30^0C < T$) $3CA + 12H \rightarrow C_3AH_6 + 2AH_3$.

Over time, crystallization of metastable CAH_{10} and C_2AH_8 leads to their conversion to a thermodynamically stable cubic C_3AH_6. As a result of conversion reactions, some of the bound water within the crystal structure is liberated resulting in an increase in porosity of CAC matrix and consequently in a decrease in strength, which limits the scope of application of the CAC [2].

An opportunity to apply pre-stress to cement and concrete composites at an early stage of hardening and to achieve design requirements (along with the obvious acceleration of construction works [13]) without any loss of performance characteristics is particularly relevant. The review [14] presents some data on changes of properties after early loading, and many studies have been devoted to the application of this method (for example, [15–24]), in the course of which Portland cement silicates (with crystalline, submicrocrystalline and amorphous structure) silicates had been exposed to compression.

The study of the effect of early loading of a structure consisting mostly of calcium aluminates (CA) will allow us to gain a better understanding of how the aluminate component affects the effectiveness in comparison with silicate component, to investigate the nature of the changes, taking into account the properties and structure of aluminate cements. Rapid strength development makes it possible to apply a significant loading at the earliest stages of hardening (24–72 hours from the moment of molding). Obtaining data on the effects of loading (changes of compressive and bending tensile strength, hydration, bound water content, composition and number of new formations) is of particular interest.

The main purpose of the study is to analyze the changes occurring in the structure of aluminate cement after early loading, which determines the following tasks:

- obtain values of compressive and bending tensile strength after preliminary short-term compression;
- conduct X-ray phase analysis and describe the changes of intensity of the peaks of crystalline phases, take an assessment of amorphousness of the deformed structure;
- analyze differences in weight loss using the DTA methods.

Materials and Methods

High aluminate cement GC-50 (according to Russian State Standard GOST RF 969-91) produced by Pashiya Metallurgical Cement Plant was used (Table 1) as a binding component. The X-ray phase analysis of initial cement stone (before mixing with water) was carried out to identify main phases. The main mineralogical phase is calcium monoaluminate CA. There are also $C_{12}A_7$, C_2AS, C_4AF and CA_2 to be found. The size of the prism is 40 × 40 × 160 mm. The samples have been molded from cement-sand grout with the following shares of components: cement : sand : water = 2.5 : 2.5 : 1, using fractional sand 0–0.63 mm, purified from any foreign and clay particles as a fine aggregate. Bending under tension tests were conducted on prisms, while compressive strength was determined by testing prism halves. Thus, each point of the generated strength curve indicates average value obtained from 3 measurements of bending tensile strength and 6 measurements of compressive strength values.

Table 1 Chemical composition of cements applied (%)

Al_2O_3	CaO	SiO_2	Fe_2O_3	MgO	TiO_2
38–42	27–29	10–12	5–8	<5	<10

Correct load distribution was provided through the use of hinged bearings. Samples that were not exposed to loading (hereinafter referred to as "control samples"), as well as samples before and after loading, were stored under the same normal conditions of humidity.

Curing duration of samples subjected to loads is 24-hours from the moment of their manufacture. For the experiment, the value of the compressive load was taken as a constant and was equal to 10% of the daily strength of the sample. It was expected that effect of earlier loading would be the most significant at the stage of formation of composite structures. Cracks were not allowed, as well as the eccentricities corresponding to the points of load application. Taking into account rapid strength development of aluminous cement (grade strength R = 50.3 MPa is achieved after 72 hours), a period of short-term loading should be 24 hours. Bending under tension test for prisms was performed in accordance with Russian State Standard GOST RF 310.4, for cubes – according to Russian State Standard GOST RF 10180. Calculation and statistical methods were applied for analysis of the obtained data. The accuracy rate (the ratio of the mean error to the arithmetic mean) did not exceed 2.6 % for bending under tension test, and it reached the value 3.9 % for compression test.The results were assessed for 5 % significance level. The total test duration was 15 days. After 10 days, the increase of strength did not exceed 5 %, and the slope of a line tangent to strength curves tended to a constant, therefore, in the present work, changes in compressive and bending strength are presented for 10 days.

X-ray phase analysis was performed using an XRD-7000 Shimadzu diffractometer (Japan). The peaks identification in the diffractograms was carried out using the PDWin 4.0 and Crystallographica Search-Match software, integrated into the hardware software complex of the device. The shooting conditions were the following: copper anode, the wavelength of radiation Kα 1.54051Å, 40kV, 30mA, the angle range 5 to 70 degrees, the shooting speed 1 deg/min.

The differential thermal analysis was performed by Netzsch STA-409 PC Luxx, temperature range of 25–1000 °C. The test has been carried out under the air atmosphere conditions in platinum crucibles at a heating rate of 10 °C/min.

Observational data are presented as strength curves (Fig. 1), X-ray diffraction patterns of alumina cement before mixing with water (Fig. 2a), comparision of overlaid diffractograms of loaded and control samples (Fig. 2b, 2c), and comparision of their derivatograms (Fig. 3).

Results and Discussion

The effect of a 24-hour static compression on the strength development of aluminate cement for 10 days is shown in Figure 1. The prisms subjected to the loading showed an increase of ultimate tensile strength (up to 29 % on the second day). Observations indicate that in the course of aging an increase in strength development is reducing.

At the early loading, an increase in the compressive strength was not detected (Fig. 1). Comparison of diagrams demonstrates that the increase rate reduces, and it becomes close to a constant after 3 days. The fact that the compression strength of aluminate cement decreases when loaded for 1 day is very much in line with the data of [25].

A comparative analysis of X-ray diffraction patterns of the sample subjected to early short-term compression (drawn in blue color in the Fig. 2b) and control sample (drawn in red color in the Fig. 2b) has been carried out. Nine days later, another X-ray phase analysis of the same samples was carried out (Fig. 2c).

Figure 2b shows the peaks of control and pre-loaded samples related to hydroaluminates of CAH_{10} type (2Θ = 6.1°; 12.3°), whose intensity decreases by the 10th day due to recrystallization (Fig. 2c), as well as because of the formation of C_2AH_8 and aluminum hydroxide (amorphous gel). The arc of amorphous cement without any load in the angle range 2Θ = 5-17 ° is higher, which indicates an increasing content of loosely bound water in its structure. The authors of [26–31] drew attention to the almost instantaneous change in moisture content during the compression of cement systems, relating it to the intense shrinkage and redistribution of water in capillaries and interlayer space under compressive loading.

As it is seen in X-ray diffraction patterns of cement stone which was subjected to early short-term compression (Fig. 2b), the peak intensity of the main phase – calcium monoaluminate CA is decreasing (2Θ=16.1°; 18.8°; 22.8°; 24.07°; 28.85°; 31.14°; 40.14°; 41.01°; 59.14°), as well as peak intensity of $C_{12}A_7$ (2Θ = 18.02°; 36.56°) и CA_2 (2Θ = 28.85°; 34.27°). However, the number of peaks related to CAH_{10}, as well as the peak intensity, is higher in the X-ray patterns of the control sample (Fig. 2b). This is true for C_2AH_8 (hexagonal phase) and for AH_3 (microcrystalline phase). Recorded decrease of the number of peaks of crystalline hydration products in the cement stone after early short-term compression whilst decreasing peak intensity of main mineralogical phases in the X-ray pattern is probably due to increase of the amorphous content in the pre-loaded samples, which is difficult to identify with X-ray phase analysis. In this case the structure of the cement stone subjected to load at an early stage can be more amorphous, which is consistent with the conclusions [32, 33], and it may have more specific surface area [34]. Data on crystallinity decreasing of water containing structures subjected to compression are given in the sources [35, 36].

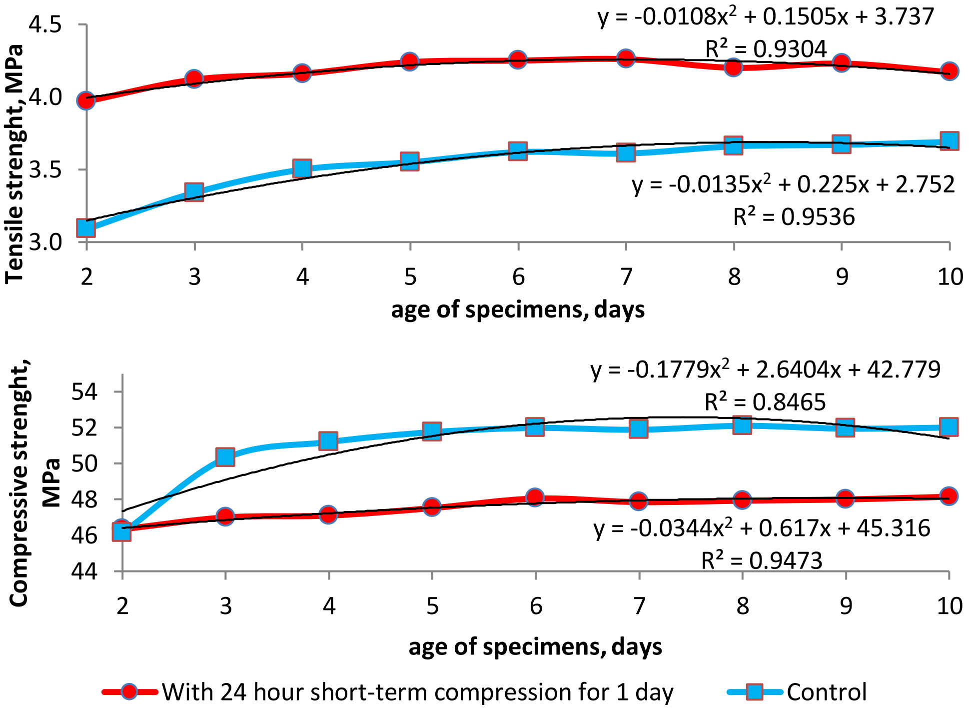
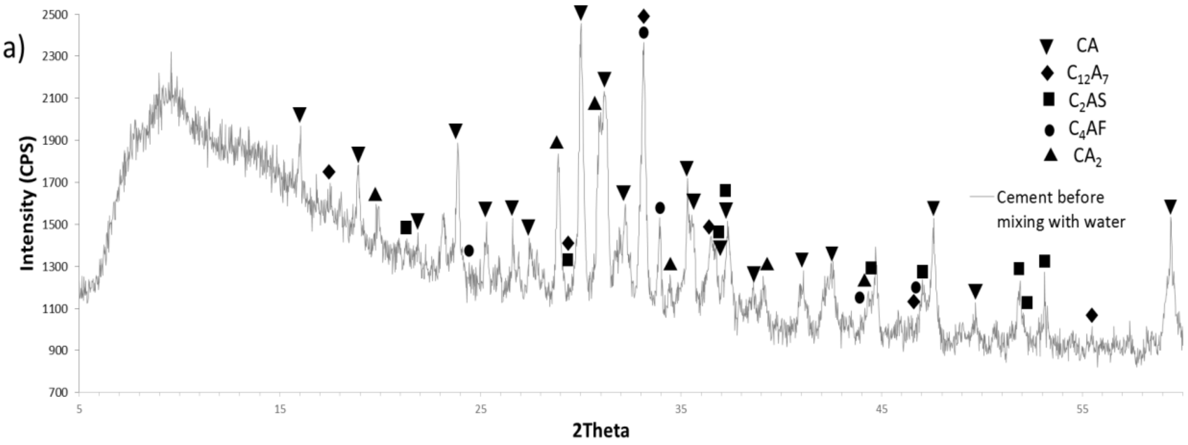


Figure 1. Effect of short-term compression on increase of compressive and bending strength

X-ray diffraction patterns of sample which was not exposed to loading (see red X-ray diffraction pattern in the Fig. 2b) indicates the presence of hydroaluminate C_3AH_6 (cubic phase) (2Θ = 19.89°; 22.6°; 26.8°; 39.1°; 44.47°), while the peak intensity related to C_3AH_6 in pre-loaded samples is lower. The aluminum hydroxide gel affects the stability of hexagonal hydroaluminates and reduces the tendency of recrystallization into cubic crystals [37]. Perhaps the increasing amorphousness (which was noted above) because of early compression leads to slowing down of recrystallization process and to the formation of cubic hydroaluminates, which caused these differences of X-ray diffraction patterns. AH_3 gel plays an important role in the strength increasing [36]. Higher tensile strength in bending (Figure 1) may result from changes in the structure occurred under load (the increasing amorphousness noted above due to the gel component). Interconnection between layers [34], compaction and change in porosity [39–43], which accompany deformation of cement systems, probably affect the bending strength characteristics.



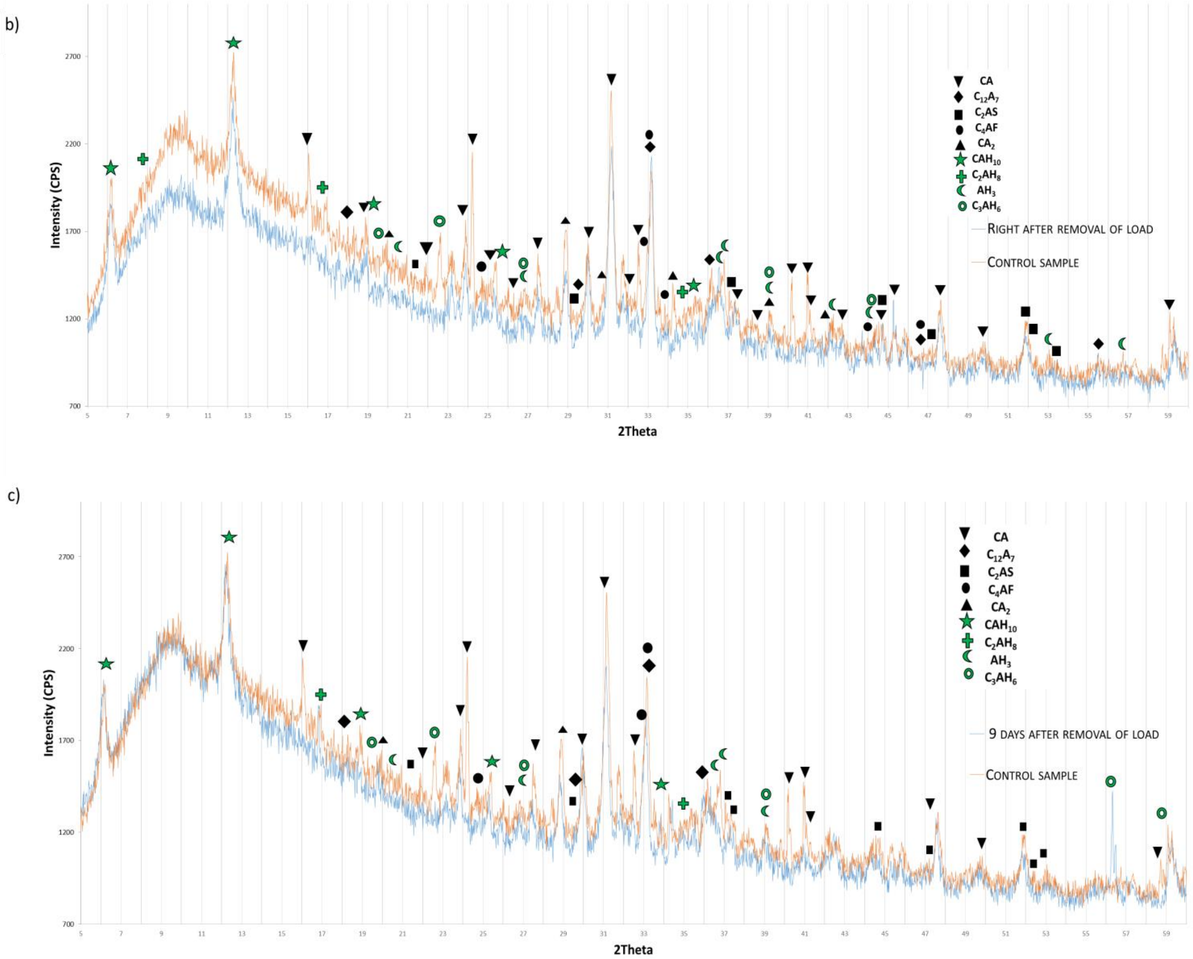


Figure 2. X-ray diffraction patterns of the cement GC-50 before mixing with water (a), immediately after removal of the load (b) and 9 days later (c)

A second analysis of the phase composition 9 days later showed that recrystallization of hexagonal hydroaluminates occurred in the cement stone under compression. Load removal and hardening of samples subjected to preliminary compression under absence of compressive stresses contributed to formation of cubic structure. It is confirmed by the appearance of a peak of cubic C_3AH_6 (peak at 2Θ = 56.3° in Figure 2c) and increasing intensity of hydrate peaks (for example, microcrystalline gibbsite from 2Θ = 20.58°; 26.8°). It is also appropriate to assume that the recrystallization rate under compressive stresses should be lower than in uncompressed structures. The delay in this process led, apparently, to a strength decrease of the specimens after 10 days of compression [2] (Fig. 1). An almost instantaneous partial transformation of the amorphous structure into a crystalline structure after removal of the load was indicated in researches [35–37, 42]. Taking the above into account, it should be noted that the short-term load at an early age changes the nature of structure formation processes that occur in aluminate systems.

According to the data of DTA (Fig. 3), it can be seen that the thermogravimetric curves of the samples practically coincide with each other. Despite the changes mentioned above (noted in the analysis of X-ray diffraction patterns), water content values in the structures of the pre-loaded and control samples are quite close. Coincidence of endo-effects at 275 °C (which corresponds to the temperature of boehmite formation [38]) may indicate that the weight percentage of the amorphous component of the control sample and pre-compressed sample is equalized within 10 days.

It is confirmed by overlaying of amorphous phase arcs in the angular range 2Θ = 5-17° 9 days later (Fig. 2c). As S.V. Aleksandrovsky [44] indicated earlier, the water molecules in crystalline structures are loosely bound. Under certain conditions, they can be removed again, and then to be re-absorbed without changing the crystal structure.

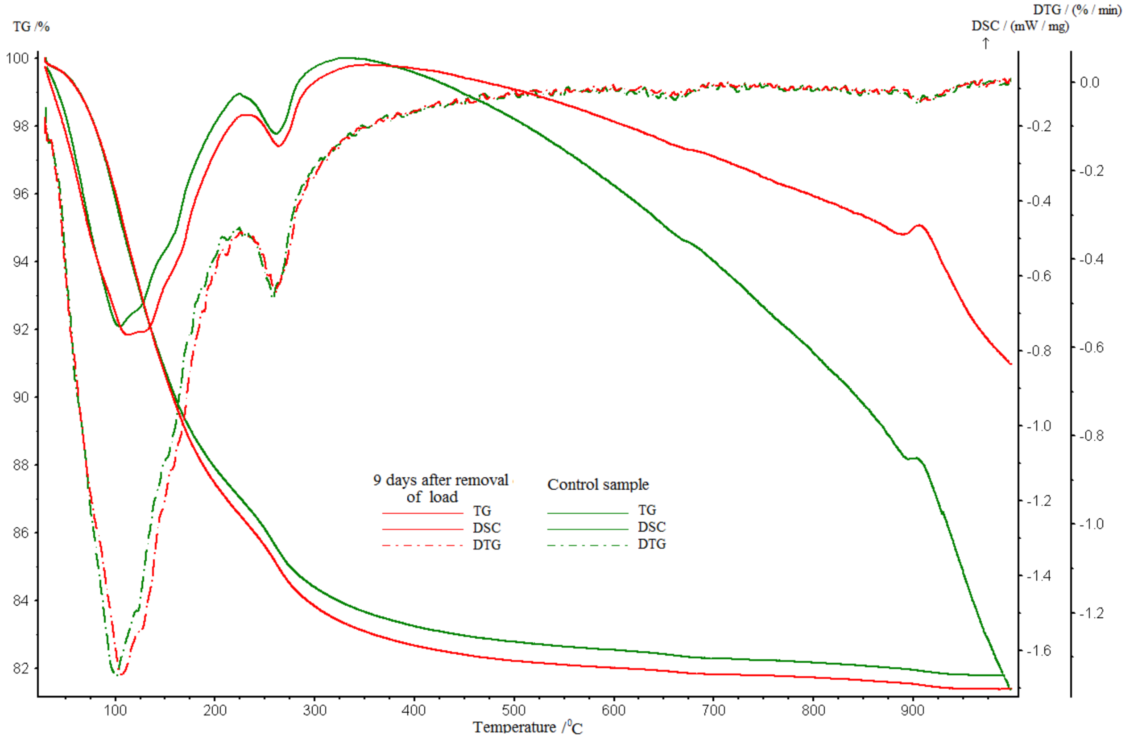


Figure 3. Comparison of the compressed and control specimens through differential thermal analysis 9 days after removal of the load

Conclusions

1. Preliminary short-term compression at an early age contributes to increase of bending tensile strength.

2. At the same time the compressive strength of the samples loaded at early age decreases, which may be due to the later recrystallization of hexagonal hydroaluminates into cubic phase and dumping of strength accompanying this process.

3. The peak intensity of the main phases of aluminate cement reduces in the case of load application. On the basis of the literature review, an assumption has been made that the amorphousness of the hydrated aluminate structure tends to increase under compressive stress.

4. Comparison of the derivatograms of the samples did not reveal any changes in the bound water content. The absence of such changes and simultaneous formation of a different crystal structure indicated by the X-ray analysis is of particular interest for the further study of aluminate cements after early loading.

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