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ФОРМУВАННЯ ВЛАСТИВОСТЕЙ БЕТОНУ ПІД ВПЛИВОМ МОДИФІКАТОРОВ НА ОСНОВІ ПОВЕРХНЕВО-АКТИВНИХ РЕЧОВИН

В. В. Пісчанська, Г. С. Войтюк, Я. М. Пітак

ФОРМИРОВАНИЕ СВОЙСТВ БЕТОНА ПОД ВЛИЯНИЕМ МОДИФИКАТОРОВ НА ОСНОВЕ ПОВЕРХНОСТНО-АКТИВНЫХ ВЕЩЕСТВ

В. В. Песчанская, А. С. Войтюк, Я. Н. Питак

FORMATION OF THE PROPERTIES OF CONCRETE UNDER THE INFLUENCE OF MODIFIERS BASED ON SURFACE ACTIVE SUBSTANCES

V. Peschanska, A. Voytyuk, Y. Pitak

Проведеними дослідженнями впливу триполіфосфату натрію та комплексного модифікатору на процеси тужавлення і твердіння, фазо- і структуроутворення цементного каменю встановлено механізми процесів... Доведено, що вони полягають у... Завдяки цьому стале можливим визначення формування структури та властивостей цементного каменю на ранніх стадіях твердіння. Експериментальними дослідженнями підтверджено, що... за рахунок переугрупування груп полімерних молекул триполіфосфату натрію і суперпластифікатору суттєво змінюється щільність упаковки молекул в адсорбційному шарі, структура і товщина поверхневого шару новоутворень на поверхні гідратованих фаз цементу. Це приводить до підвищення механічної міцності цементного каменю та формування заданих властивостей низькоцементного бетону. Зокрема встановлено, що завдяки... механічна міцінісць бетону підвищується з ... до.. Це дозволяє стверджувати про відповідність виявленого механізму формування властивостей та практичну привабливість запропронованих технологічних рішень. Останні, зокрема, стосуються визначення кількості модифікатора, ... Показано, що...

....

Таким чином, є підстави стверджувати про можливість спрямованого регулювання процесів формування міцної структури низькоцементних бетонів шляхом використання комплексного модифікатору, який містить поверхнево-активні речовини.

Ключові слова: *низкоцементный бетон, кальцийалюминатный цемент, гидратация, модификатор бетона, аморфная фаза, кристаллогидраты*

The research we conducted into effect of sodium tripolyphosphate and a complex modifier on the processes of setting and hardening, phase- and structure-

formation of the cement stone established the mechanisms of processes ... It was proven that they consist of ... This made it possible to determine the formation of structure and properties of the cement stone at the early stages of hardening. Experimental research confirmed that ... due to the regrouping of groups of polymer molecules of sodium tripolyphosphate and superplasticizer, the packaging density of molecule arrangement in the adsorption layer substantially changes, as well as the structure and thickness of surface layer of new formations at the surface of the hydrated phases of cement. This leads to an increase in mechanical strength of the cement stone and to the formation of predetermined properties of low-cement concrete. It was determined, in particular, that due to ... mechanical strength of concrete increases from ... to ... This allows us to argue about the alignment of the discovered mechanism of properties formation with practical application of the proposed technological solutions. The latter, in particular, are related to determining the amount of modifier ... It is shown that ...

....

Thus, there are reasons to argue about the possibility of targeted control over the processes of formation of solid structure of low-cement concretes by using a complex modifier that contains surface active substances.

***Keywords:** low-cement concrete, calcium aluminate cement, hydration, concrete modifier, amorphous phase, crystalline hydrates*

1. Introduction

The implementation of strategic direction in the development of refractory industry regarding the rational use of fuel and energy, as well as material resources, is aimed at increasing the volumes of production of unshaped refractories, which include low-cement refractory concretes. Effective combination of physical-chemical properties of low-cement concretes with high adaptation capability to temperature operating conditions, substantial technical and economic advantages of using the monolithic linings predetermined the application of these concretes at almost all process stages in metallurgical production [1–4].

....

Low-cement concretes represent a multi-component compositional system and contain in the physical composition coarse and finely-dispersed fireproof filler, calcium aluminate cement with an aluminium oxide content exceeding 70 %... [5]

Given the polyfunctional effect of deflocculants and plasticizers, the research that is considered to be relevant is aimed at further improvement and development of the technology of refractory concretes with high operational resource, as well as a directed control over the processes of phase- and structure-formation of the cement stone by using efficient modifying additives.

2. Literature review and problem statement

Progress in the materials science of fire-resistant composite materials based on hydraulic binders has contributed to the deepening of examining chemical aspects of the hydration processes of calcium aluminate cements whose mineralogical composition is presented by calcium monoaluminate $CaO{\cdot}Al_2O_3$ (CA), calcium dealuminate

$2CaO \cdot Al_2O_3$ (CA_2), $12CaO \cdot 7Al_2O_3$ ($C_{12}A_7$), and corundum [6]. The research into the system "calcium aluminate cement–water" established that the rate of formation of these or other hydrated phases: CAH_{10} ($CaO \cdot Al_2O_3 \cdot 10H_2O$), C_2AH_8 ($2CaO \cdot Al_2O_3 \cdot 8H_2O$) and the amorphous phase of alumogel, or structures of the C–A–H type, is determined by the mineralogical composition… [7]. It should be noted, however, that the given paper fails to outline thermodynamic conditions for the formation of hydrated phases… This means that the course of the process under conditions of temperatures in the range of…. is not defined. From a practical point of view, it can result in difficulties related to determining the optimal amount of a mixture of deflocating additives. This circumstance is connected to the fact that their introduction to concrete mixtures significantly changes the mechanism and kinetics of processes of hydration of clinker minerals. In order to overcome this problem, in article [8], authors conducted research into effect of deflocating additives on the processes of hydration of calcium aluminate cements. It is shown that due to the established analytical dependences it becomes possible to adjust the content of modifiers to ensure reotechnological characteristics of concrete masses. Despite the practical significance of such results, kinetic patterns of hydration processes were not studied sufficiently enough. This is obviously related to the complexity in determining indicators of the properties of concretes in the process of thermal treatment under conditions of ...

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The feasibility of employing hybrid modifiers containing sodium tripolyphosphate is confirmed by the improved rheological properties of concrete masses and by significant improvement in the mechanical strength of concrete [9]. According to the authors of [9], this is predetermined by the formation during calcium aluminate cement hydration in the composition of concretes with limited humidity of the large amount of nanostructures. which are compacted at drying and form a strong frame, destruction of which corresponds to ... However, no appropriate physical-chemical calculations were provided to confirm this hypothesis…

Thus, there is a reason to believe that the lack of certainty in determining the impact of hybrid modifiers on the morphological features and characteristics of structures that are formed with their participation during hydration of calcium aluminate cements necessitates research in this direction.

3. The aim and objectives of the study

The studies we conducted set the aim of determining features of the processes of hardening and structure-formation of the cement stone with an individual and a complex modifier and the formation, under conditions of thermal treatment, of structure of low-cement corundum concrete, which would enable improvement in the physical and mechanical properties of the composite.

To achieve the set aim, the following tasks have been solved:

– to determine the effect of sodium tripolyphosphate, superplasticizer, and a complex modifier on a change in the character of the processes of setting, phase- and structure-formation of the cement stone at the early stages of hardening at different values of water-cement ratio;

– to establish the features in the formation of microstructure of the thermally-treated corundum concrete containing sodium tripolyphosphate and a complex modifier, in the relationship with physical and mechanical indicators of the concrete properties.

4. Materials and methods for examining effect of modifiers on the processes of hardening of cement slurry, microstructure, and properties of low-cement corundum concrete

4. 1. The studied materials and equipment used in the experiment

The study was carried out using the calcium aluminate cement "Gorkal-70" and modifies – surface active substances (SAS): sodium tripolyphosphate (STPP), superplasticizer of organic origin S-3 (Polyplast, Russia), and integrated deflocculant containing STPP and S-3.

A study of morphological characteristics of particles, phase composition and structure of the cement stone was carried out using an X-ray phase analysis (diffractometer DRON-3), electron microscopy (electronic microscope "REM-106I"), and infrared spectroscopy (spectrophotometer Spectrum One (Perkin Elmer)).

Experimental samples of concrete were prepared from concrete mixtures containing 75 % of electrocorundum, fractions 6–0.063 mm (6–3 mm – 18 %, 3–0.063 mm – 57 %), and 25 % of matrix component (electrocorundum – 71 %, cement – 29 %). Components of the concrete mixture were wetted to a humidity of 5.0–5.2 % and then the mass was agitated for 4 min. Additives were introduced as a water solution, calculated as STPP – mass of the concrete mixture, C-3 – per mass of cement.

Experimental samples, cubes with an edge of 50 mm, were fabricated using the method of vibrational shaping, which, after hardening in the air for 3 days, were thermally treated at 110 °C (24 h) and temperature 1550 °C (aging –5 hrs.). Studying the microstructure of concrete was conducted using a polarization microscope (MIN-8).

4. 2. Technique for determining indicators of the properties of samples

An indirect estimation of effect of modifiers on the kinetics of cement hardening at a water-cement ratio (W/C) of 0.31 and 0.27 was performed by the results of determining time of setting on the Vick's device, registering in time the depth of the needle's device immersion into the cement slurry. Determining the influence of modifiers on the hardening of cement stone at the early stages was carried out on the samples of cement with a W/C ratio of 0.31, 0.27, and 0.2.

Basic indicators for the properties of concrete samples that were determined in the course of experiment were: apparent density (ρ), open porosity (Π), limit of strength at compression (σ).

Determining the apparent density was carried out according to the following procedure: ...

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Determining the open density was conducted according to the following procedure: ...

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In order to determine the limit of strength at compression, we employed…

5. Results of examining the indicators of properties of the concrete samples

Results of determining the time of setting of the cement slurry (W/C 0.31) depending on the type of modifier (STPP and S-3) and its amount are given in Table 1.

Table 1

Influence of surface active substances on spreading index and setting time of cement slurry

No.	Type and amount of modifier, %		Setting time, h-min	
	STPP	S-3	start	finish
1	–	–	2–21 / –	4–19 / –
2	0.15	–	0–35 / 0–05	6–39 / 1–33
3	–	0.05	2–17 / 0–09	7–03 / 2–10
4	0.15	0.05	0–12 / 0–07	4–40 / 1–40

Note: numerator – W/C 0.31; denominator – W/C 0.27

It should be noted that the integrated modifier, when compared with an individual additive of STPPN and S-3, accelerates by 1.4–1.5 times the period of setting finish. At a decrease in W/C to 0.27 due the deflocating action of modifiers and dilution of the cement slurry [23] by the degree of accelerating action on the time of setting, the additives can be arranged in the following series S-3 → complex modifier → STPP (Table 1).

By the experimental data on determining the setting rate of cement slurry, we selected periods, which correspond to the depth of immersion into the cement slurry of the Vick's device needle at 10 mm, 20 mm, 30 mm, and 38-39 mm (Fig. 1).

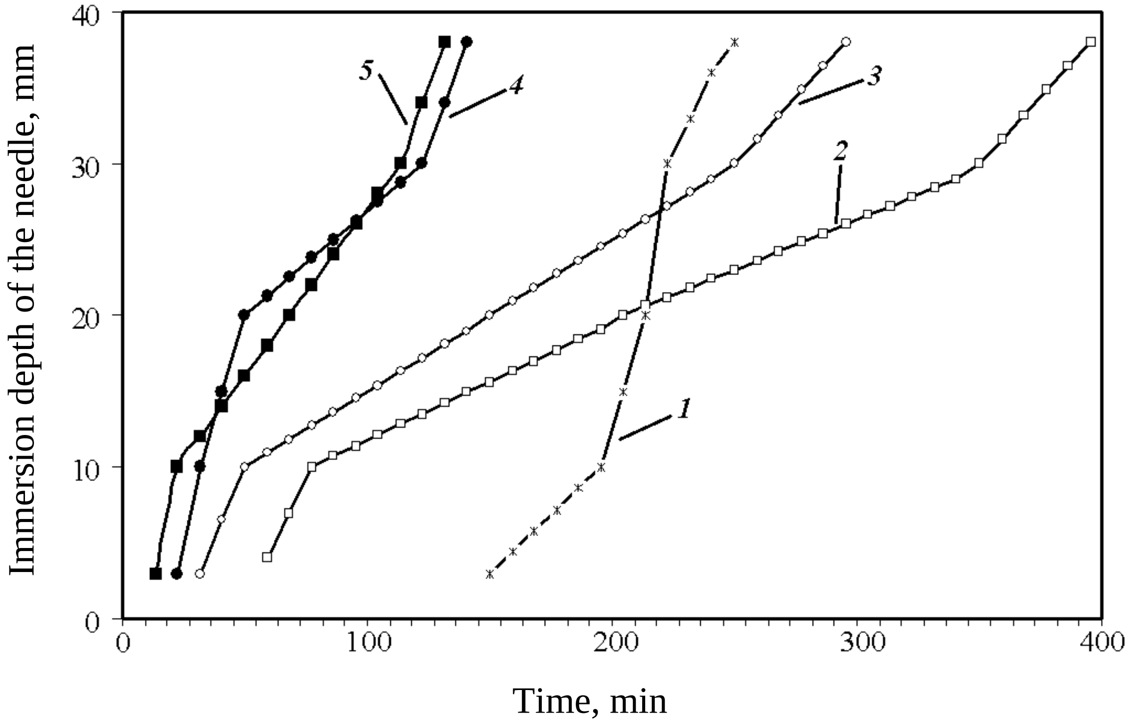

Fig. 1. Kinetics of the cement hardening: 1 – without additives; 2, 4 – with STPP; 3, 5 – with a complex modifier; 1, 2, 3 – W/C 0.31; 4, 5 – W/C 0.27

Attention should be paid to the fact that the first period of the cement slurry setting (depth of the neddle's immersion – 10 mm) is characterized by a sharp rise in the curves and at a W/C of 0.31 lasts for 196 min.; 55 min., and 20 min. for cement without additives (Fig. 1, curve 1), with an STPP additive (Fig. 1, curve 2), and a complex modifier (Fig. 1, curve 3), respectively. Slowing down the processes of hydration and setting of the modified slurry at a W/C of 0.31 distinguishes the second and third periods (Fig. 1 curves 2 and 3) that correspond to the depth of needle's immersion at 20 mm and 30 mm.

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Results of determining mechanical strength of the samples of cement stone at the early stages of hardening with a W/C of 0.31, 0.27, and the samples shaped by vibration with a W/C of 0.2, are shown in Fig. 2.

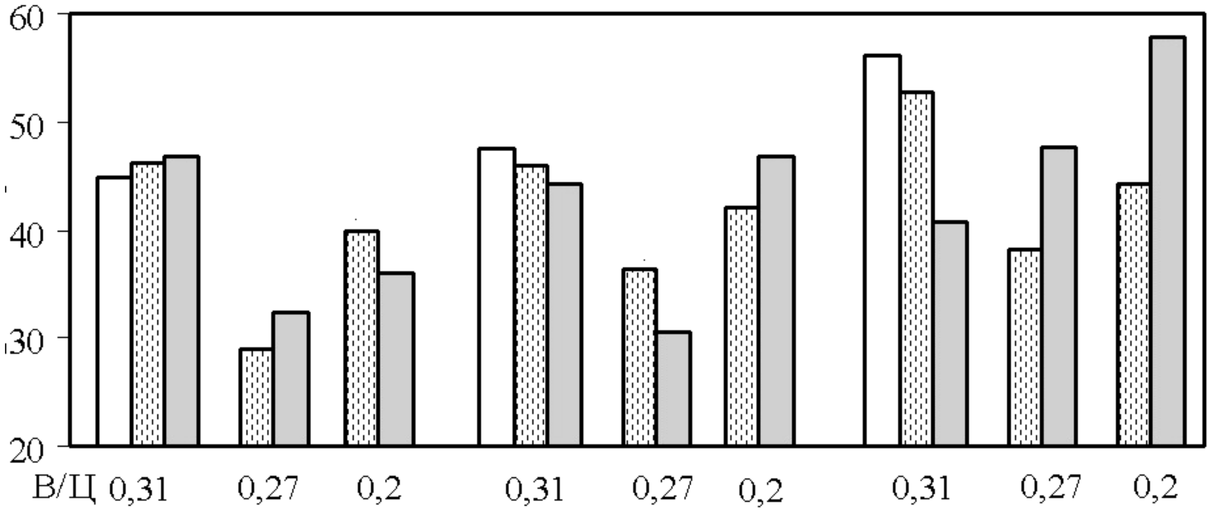

Fig. 2. Effect of W/C and modifiers on strength of the samples at the early stages of hardening of cement stone

As can be seen from Fig. 2, at a W/C of 0.31, strength of the samples with modifiers after 3 days and 7 days is practically on the same level within a range of 44.4–47.5 N/mm^2, but after 28 days the strength of cement stone with the complex modifier is 1.4 times lower than that in the samples with STPP, and without modifiers.

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Results of the X-ray phase analysis of cement stone with a W/C of 0.31 and 0.2 at the age of 3 days are shown in Fig. 3.

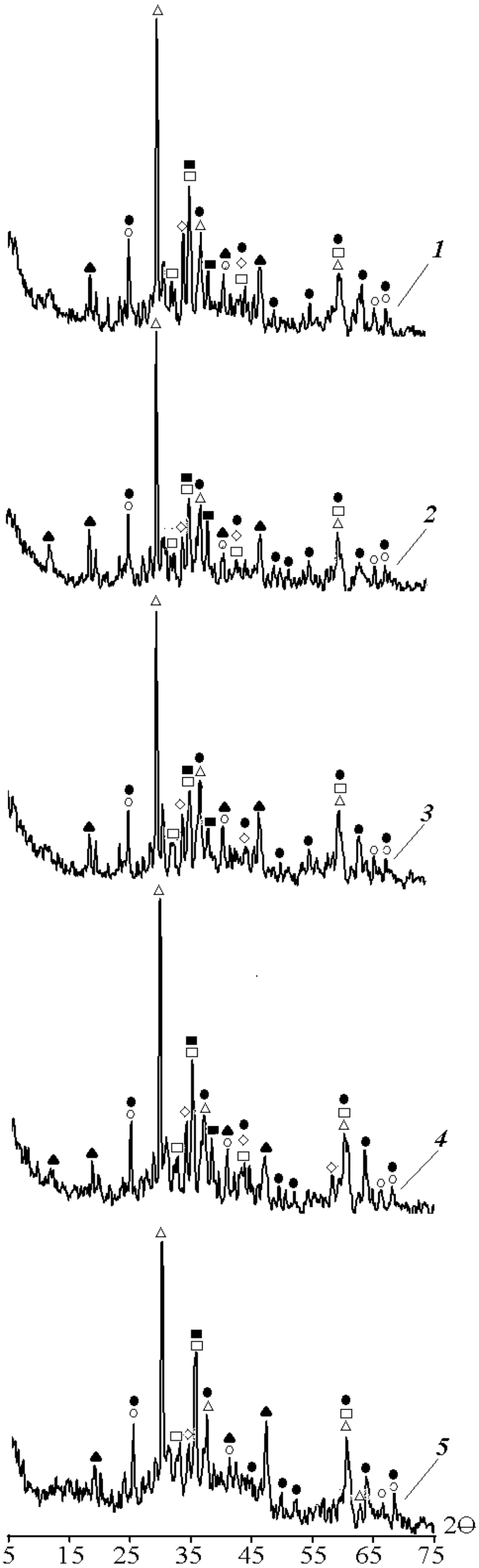

Fig. 3. Diffractograms of cement stone at the age of 3 days: 1, 2, 3 – W/C 0.31; 4, 5 – W/C 0.2; 1 – without an additive; 2, 4 – with STPP; 3, 5 – with a complex modifier; ○ – α -Al_2O_3; Δ – CA; □ – CA_2; ▲ – CAH_{10}; ■ – C_2AH_8; ● – AH_3; ◊ – C_3AH_6

Special attention should be paid to the fact that at a decrease in W/C the intensity of peaks of starting phases (CA, CA_2) and CAH_{10} of the modified cement (Fig. 3, lines 2–5) reduces due to the formation of C_2AH_8 and a significant amount of AH_3.

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Using results of the IR-spectroscopy (Fig. 4), we determined absorption bands in the region of frequencies 3680–3460 cm^{-1}, 2930–2850 cm^{-1}, 1640–1400 cm^{-1} corresponding to the valency oscillations of the OH hydroxyl groups, methylene groups, and to the deformation fluctuations of molecular water (H-O-H), as well as in the frequency range of 1370–1420 cm^{-1}, corresponding to the carbonate complexes.

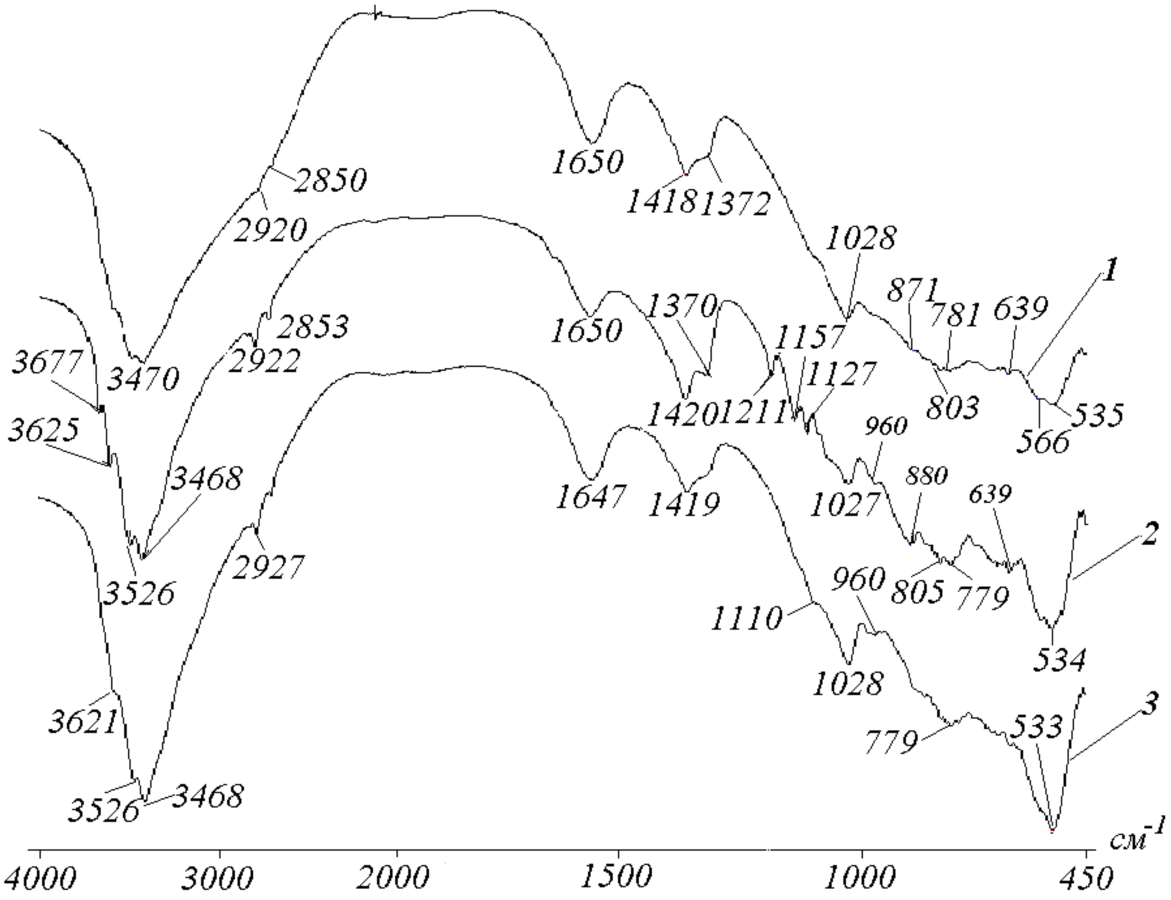

Fig. 4. Infrared spectra of cement stone at the age of 3 days: 1 – without a modifier (W/C 0.31); 2 – with STPP (W/C 0.2); 3 – with a complex modifier (W/C 0.2)

Special emphasis when studying spectrum, shown in Fig. 4, should be placed on the frequency range of 900–1500 cm^{-1}, which corresponds to the deformation fluctuations of the AlO–OH bonds in the alumogel Al $(OH)_3$.

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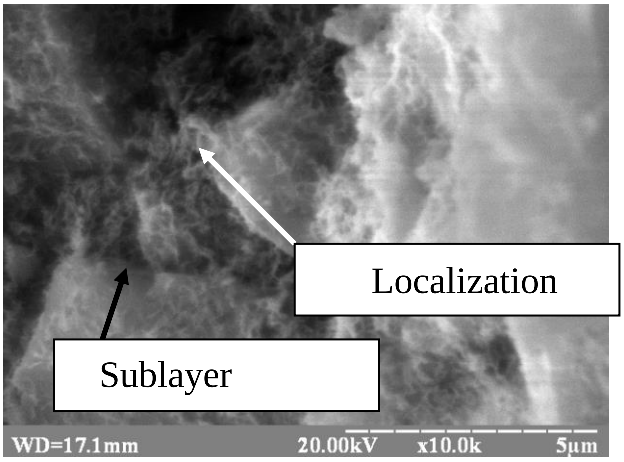
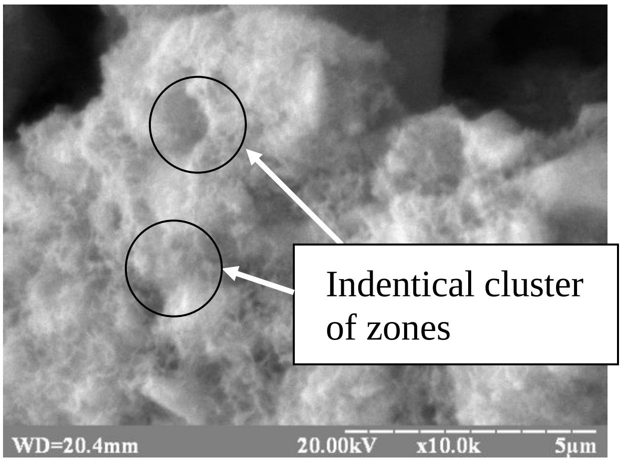
Electron-microscopic images of the fracture of samples of the modified cement stone (W/C 0.2) at the age of 3 days (Fig. 5) illustrate the formation of a zeolite structure; in the case of modifier with STPP (Fig. 5*a*), it is distributed along the plane and is characterized by new formations with a thickness and a length of 9–11 nm and 80–100 nm, respectively. A generalization of this fact can be formulated in the form of the following thesis – the complex modifier provides structures with volume and a denser character (Fig. 5*b*), which is associated with the rearrangement of polymeric structures of molecules of different structure of STPP and superplasticizer S-3.



a



b

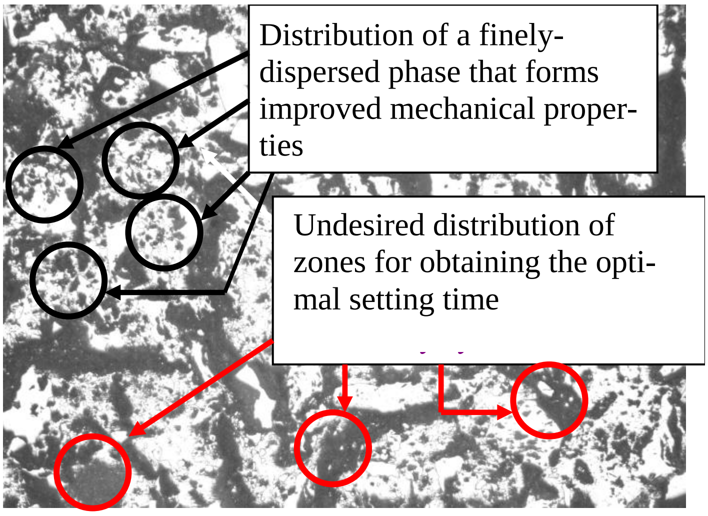
Fig. 5. Microphotographs of cement stone: *a* – with STPP; *b* – with a complex modifier

Special features in the action of modifiers on microstructure of the cement stone define their impact on a change in the indicators of properties (Table 2) and microstructure of low-cement corundum concrete after firing (Fig. 6).

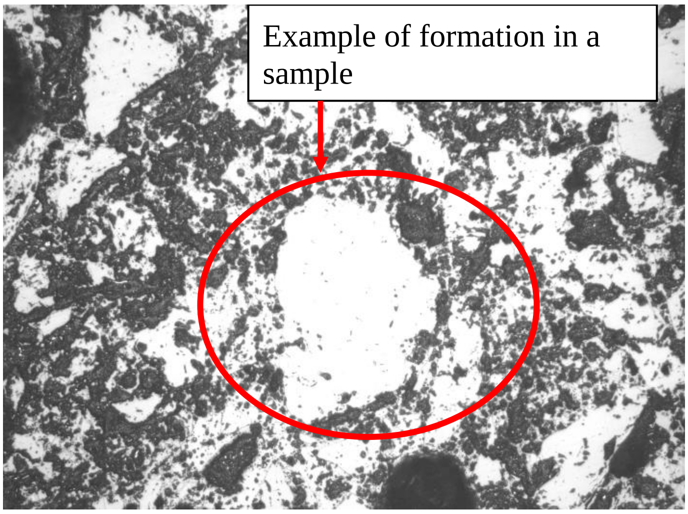
Table 2
Effect of modifiers on the properties of low-cement corundum fire-resistant concrete

Content of modifier, %		Strength limit at compaction, N/mm², at hardening for		Indicators of properties after thermal treatment		
STPP	S-3	1 day	3 days	П, %	ρ, g/cm³	σ, N/mm²
0.15	–	20.5	30.8	11.91 / 12.88	3.19 / 3.37	39.5 / 146.7
0.15	0.05	18.7	38.6	12.35 / 13.68	3.24 / 3.33	47.3 / 185.9

Note: numerator – temperature 110 °C; denominator – 1550 °C



a



b

Fig. 6. Microstructure of low-cement corundum concrete after firing at 1550 °C (magnification ×50 times): *a* – with addition of STPP; *b* – with complex additive; black color indicates pores

Based on these results, we can state the presence of an interesting pattern related to the formation of microstructure. In particular, the microstructure of concrete with the addition of STPP (Fig. 6*a*) is represented mainly by small capillary microcracks in the branch-shaped form with a width of 50–90 μm, and by an insignificant number of isolated round-shaped pores the size of 90–180 μm (in some places, up to 360 μm). In this case, concrete with the complex modifier (Fig. 6*b*) is distinguished by the fine-porous structure, homogeneous in shape with micropores of size that on average is 2.5 times less than the previous one (its absolute value, as derived from Fig. 6*b*, is in the range of 18–90 μm).

6. Discussion of results of research into effect of modifiers on the processes of hydration, phase- and structure-formation of the cement stone

When determining effectiveness of the complex modifier on the process of setting, as follows from the obtained results (Table 1 and Fig. 1), it is logical to extend the time of completion of the modified cement setting. This is predetermined by the dispergating action of STPP and the formation of adsorption layers on the surface of clinker minerals that inhibit diffusion processes in the ions of hydroxyls and the formation of crystalline hydrates. It should be noted that the presence of the SAS additives leads to ... Obviously, such a mechanism of the SAS inpact is the very factor in controlling the process due to which the complex modifier contributes to reducing the second period by … %. However, at the same time, this mechanism adversely affects duration of the third period in setting.

In this sense, of special interest is the interpretation of results of the X-ray phase analysis and infrared spectroscopy, shown in Fig. 3, which confirms the fact of establishing a reduction in W/C. This testifies to the formation of amorphous phases, in particular, CAH and CAPH, which cannot be identified using the method of an X-ray phase analysis [22].

In order to prove this argument, it will suffice to carefully investigate absorption bands in the frequency region of 1370–1420 cm^{-1}. Moreover, comparing the intensity, half-width, and the degree of diffusion of bands of the spectra of samples testifies to the depth of the course of hydration processes. This means that taking this fact into account opens up the possibility to effectively control properties of the stone under actual industrial conditions.

Comparing the time of setting and the duration of periods of the modified cement hardening with a different W/C indicates acceleration of the setting processes of cement slurry. This does not disagree with practical data, well known from papers [7, 13], whose authors also attribute a change in the process duration to the formation in the composition of concrete the less stable compounds, in particular over the third interval of setting time. However, in contrast to the research results published in [7, 13], obtained data on the effect of STPP and the complex modifier on the process of setting allow us to argue about the following:

– the main regulator of the process is not so much the formation of a significant quantity of nanostructures but rather the deflocculating action of modifiers and the dilution of cement slurry;

– considerable effect on the process is exerted mainly by the correct arrangement of additives in the series S-3 → complex modifier → STPP.

Such conclusions can be considered expedient from a practical point of view as they substantiate the approach to determining the required amount of a modifier. From a theoretical point of view, they make it possible to argue about defining the mechanism of hydration processes, which constitute certain benefits of present research. However, it is impossible not to note that the results of determining … (Fig. 2) point out an ambiguous impact of the modifiers on a change in mechanical strength. This is evident in the first place, in ... Such uncertainty imposes some constraints on the application of results obtained, which can be interpreted as the shortcomings of the given study. The inability to remove specified limitations within the framework of present study gives rise to a potentially interesting direction for the further research. It could be aimed, in particular, at detecting the moment at which a decline in the properties occurs. Such a detection will make it possible to explore the microstructural transformations that start to take place at this time, as well as to determine input variables of the process, which significantly influence the onset of "negative" transformation.

7. Conclusions

1. The research we conducted established special features of spatial orientation and density of nanostructures on the surface of hydrated minerals that ... Based on this, it can be argued that ... Sodium tripolyphosphate, superplasticizer, and a complex modifier significantly affect a change in the character of processes of setting, phase- and structure-formation of the cement stone at the early stages of hardening. This is evident in ...

2. The features of microstructure formation of the thermally-treated low-cement corundum concrete that contains sodium tripolyphosphate and the complex modifier are the following: ... Due to such mechanism, which was established based on ..., mechanical strength of the cement stone increases from… to ... Compared to ... this allows us to argue about the effectiveness of using a modifier under the following technological modes: ... This testifies to the possibility of targeted control over the processes of formation of solid structure of low-cement concretes by using a complex modifier, which contains surface active substances.

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Песчанская Виктория Викторовна
Кандидат технических наук, доцент
Кафедра химической технологии керамики и огнеупоров
Национальная металлургическая академия Украины
пр. Гагарина 4, г. Днепр, Украина, 49005

E-mail: v_peschanska@gmail.com
Контактный тел.: 095-75-257-00
Количество статей в общегосударственных базах данных – 22
Количество статей в международных базах данных – 8
Номер ORCID: http://orcid.org/0000-0001-7500-336X

Войтюк Анна Сергеевна
Аспирант
Кафедра химической технологии керамики и огнеупоров
Национальная металлургическая академия Украины
пр. Гагарина 4, г. Днепр, Украина, 49005
E-mail: makaronchik2@gmail.com
Контактный тел.: 093-93-524-00
Количество статей в общегосударственных базах данных – 4
Номер ORCID: http://orcid.org/0000-0001-7156-2025

Питак Ярослав Николаевич
Доктор технических наук, профессор, заведующий кафедрой
Кафедра химической технологии керамики, огнеупоров, стекла и эмалей
Национальный технический университет
«Харьковский политехнический институт»
ул. Кирпичева, 2, г. Харьков, Украина, 61002
E-mail: pyarn1@gmail.com
Контактный тел.: 050-81-840-00
Количество статей в общегосударственных базах данных – 53
Количество статей в международных базах данных – 34
Номер ORCID: http://orcid.org/0000-0001-5421-6702

Пісчанська Вікторія Вікторівна
Кандидат технічних наук, доцент
Кафедра хімічної технології кераміки і вогнетривів
Національна металургійна академія України
пр. Гагаріна 4, м. Дніпро, Україна, 49005
E-mail: v_peschanska@gmail.com
Контактний тел.: 095-75-257-00
Кількість статей у загальнодержавних базах даних – 22
Кількість статей у міжнародних базах даних – 8
Номер ORCID: http://orcid.org/0000-0001-7500-336X

Войтюк Ганна Сергіївна
Аспірант
Кафедра хімічної технології кераміки і вогнетривів
Національна металургійна академія України
пр. Гагаріна 4, м. Дніпро, Україна, 49005
E-mail: makaronchik2@gmail.com

Контактний тел.: 093-93-524-00
Кількість статей у загальнодержавних базах даних – 4
Номер ORCID: http://orcid.org/0000-0001-7156-2025

Пітак Ярослав Миколайович
Доктор технічних наук, професор, завідувач кафедри
Кафедра хімічної технології кераміки, вогнетривів, скла і емалей
Національний технічний університет
«Харківський політехнічний інститут»
вул. Кирпичова, 2, м. Харків, Україна, 61002
E-mail: pyarn1@gmail.com
Контактный тел.: 050-81-840-00
Кількість статей у загальнодержавних базах даних – 53
Кількість статей у міжнародних базах даних – 34
Номер ORCID: http://orcid.org/0000-0001-5421-6702

Peschanskaya Victoria
PhD, Associate Professor
Department of chemical technology of ceramics and refractories
National Metallurgical Academy of Ukraine
Gagarina ave., 4, Dnipro, Ukraine, 49005
E-mail: v_peschanska@gmail.com
Contact tel.: 050-69-396-00
The number of articles in the national database – 22
The number of articles in international databases – 8
Number ORCID: http://orcid.org/0000-0001-7500-336X

Voytyuk Anna
Postgraduate student
Department of chemical technology of ceramics and refractories
National Metallurgical Academy of Ukraine
Gagarina ave., 4, Dnipro, Ukraine, 49005
E-mail: makaronchik2@gmail.com
Contact tel.: 093-93-524-01
The number of articles in the national database – 4
Number ORCID: http://orcid.org/0000-0001-7156-2025

Pitak Yaroslav
Doctor of Technical Sciences, Professor, Head of Department
Department of chemical engineering ceramics, refractories, glass and enamel
National Technical University «Kharkiv Polytechnic Institute»
Kyrpychova str., 2, Kharkiv, Ukraine, 61002
E-mail: pyarn1@gmail.com
Contact tel.: 050-81-840-00
The number of articles in national databases – 53

The number of articles in international databases – 34
Number ORCID: http://orcid.org/0000-0001-5421-6702

Поштова адреса для відправки журналу:
Пісчанська Вікторія Вікторівна
Відділення Нової пошти №45 (м. Київ, вул. Ватутіна, 24)
Контактний тел.: +38050-33-77-888