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On the Existence of Ternary Compounds in the CaO-BaO-Al_2O_3 System

SHABANOVA Galina[1,a], TARANENKOVA Victoria[1,b],
MYRGOROD Oksana[2,c*], PIROHOV Oleksandr[2,d]

[1]National Technical University "Kharkiv Polytechnic Institute", 2, Kyrpychova str., Kharkiv, Ukraine, 61000
[2]National University of Civil Defence of Ukraine, 94, Chernishevska str., Kharkiv, Ukraine, 61023

[a]gala-shabanova@ukr.net, [b]taranenkova@ukr.net, [c*]mir-oksa@ukr.net,
[d]pir.s@ukr.net

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Abstract. In this article in order to verify the probability of the formation of the compound $CaBaAl_4O_8$, a thermodynamic analysis of the following possible solid-phase reactions of its formation was carried out: formation of $CaBaAl_4O_8$ from the initial components - calcium carbon dioxide, barium carbon dioxide and aluminum oxide; the probability of formation of dual compounds $CaAl_2O_4$ and $BaAl_2O_4$ from the same raw materials (since the compound $CaBaAl_4O_8$ is located on the $BaAl_2O_4$-$CaAl_2O_4$ conjugate) and the possibility of formation of the compound $CaBaAl_4O_8$ from binary compounds $CaAl_2O_4$ and $BaAl_2O_4$. As a result of our experimental studies, the existence of ternary compounds $Ba_3CaAl_2O_7$ and $BaCa_2Al_8O_{15}$ was confirmed, and it was found that the $Ba_3CaAl_2O_7$ compound exists in the system at least up to a temperature of 1400 °C. Thus, our studies have determined an increase in the temperature limits of its existence, in contrast to the data of previous researchers, who indicated 1250 °C as the upper temperature of existence of $Ba_3CaAl_2O_7$.

1 Introduction

A comprehensive solution to the problems of increasing the durability of various materials for the construction of nuclear installations [1] and research reactors, as well as reducing labor costs for construction and repair [2], is provided by refractory and heat-resistant cements and concretes based on them, which have high thermomechanical properties [3]. The problem of creating new multi-purpose cements with a set of specified operational properties and concretes based on them is very urgent [4]. Therefore, materials based on oxide systems in which part of CaO is replaced by BaO are of interest. It is this replacement that gives the materials a number of valuable properties: increased fire resistance and specific gravity, protective properties against the action of ionizing radiation, etc. [5, 6, 7, 8]. That's why, the three-component system CaO–BaO–Al_2O_3 is of interest, which includes compounds with binding properties, fire resistance and a high mass absorption coefficient, which makes it possible to create new high-strength composites based on the cements of the CaO–BaO–Al_2O_3 system, which are capable effectively resist the influence of elevated temperatures and weaken hard ionizing radiation.

The study of ternary compounds of the CaO-BaO-Al_2O_3 system is of undoubted interest, since alkaline earth element aluminates [9, 10] are widely used in various fields of technology.

For example, aluminates with a high content of barium oxide are used as thermal emission materials in metal-porous cathodes [11, 12, 13]. In addition, a number of binary compounds of the system are characterized by high hydraulic activity and refractoriness and can serve as the basis for the creation of new types of refractory alumina binders [14].

However, the ternary compounds of the CaO-BaO-Al_2O_3 system have not yet been sufficiently studied, and the literature lacks data on their physical and mechanical properties. In connection with the above, the aim of our work is to study ternary compounds of the system that can be used to produce alumina cements.

The literature contains rather contradictory information about the existence of three triple compounds in the system.

Lapin [15] obtained the triple compound $BaCaAl_4O_8$ from slag formed during the smelting of aluminum-barium alloys. In a slag consisting of 91-92 wt. % BaO and Al_2O_3, and 6.6-9.8 % CaO, was found to be predominantly barium monoaluminate, as well as the presence of new calcium and barium aluminate. The chemical analysis data of the new phase are as follows (wt. %): CaO - 13.90; BaO - 36.46; Al_2O_3 - 48.25; MgO - 0.82; SiO_2 - 0.31.

The authors of [16], who first studied the structure of the CaO-BaO-Al_2O_3 system at 1250°C, did not confirm the existence of the $BaCaAl_4O_8$ compound. However, in an Al_2O_3-rich region, the compound $BaCa_2Al_8O_{15}$ (stable at least up to a temperature of 1450 °C) was found to exist in equilibrium, on the one hand, with $BaAl_2O_4$ and $BaAl_{12}O_{19}$, and on the other hand, with $CaAl_4O_7$ and a solid solution of (Ca, Ba)O-Al_2O_3. The existence of this compound was confirmed by further studies by Mazazz [17], who studied the system in the temperature range of 1200-1400 °C, and by Theorean [14].

In work [18], a new triple phase of $Ba_3CaAl_2O_7$ was obtained at a temperature of 1250 °C, characterized by a narrow homogeneity interval, the existence of which was confirmed by researchers [19].

2 Main Part

There is no information in the literature on the thermodynamic description of the reactions of formation of $Ba_3CaAl_2O_7$, $BaCa_2Al_8O_{15}$, and $BaCaAl_4O_8$ compounds, so we performed thermodynamic calculations of the probability of formation of these compounds by calculating the value of the change in the Gibbs free energy as a function of temperature, taking into account the heat capacity of the compounds. The initial thermodynamic constants were taken from our work [20].

The thermodynamic estimation of the probability of formation of the compounds $Ba_3CaAl_2O_7$ and $BaCa_2Al_8O_{15}$ from the initial raw materials: barium carbon dioxide, calcium carbon dioxide, and alumina according to the reactions was given:

$$CaCO_3 + 3\ BaCO_3 + Al_2O_3 = Ba_3CaAl_2O_7 + 4\ CO_2 \quad (1)$$

$$2\ CaCO_3 + BaCO_3 + 4\ Al_2O_3 = BaCa_2Al_8O_{15} + 3\ CO_2 \quad (2)$$

The calculation was carried out in the temperature range 800-2000 K. The obtained values of the change in the Gibbs free energy with temperature are given in Table 1. The analysis of the results shows that for the $Ba_3CaAl_2O_7$ compound, the formation reaction becomes thermodynamically possible at a temperature of 1100 K, and for the $BaCa_2Al_8O_{15}$ compound - at 1200 K.

In order to verify the probability of the formation of the compound $CaBaAl_4O_8$, a thermodynamic analysis of the following possible solid-phase reactions of its formation was carried out: (3) - formation of $CaBaAl_4O_8$ from the initial components - calcium carbon dioxide, barium carbon dioxide and aluminum oxide; (4) - the probability of formation of dual compounds $CaAl_2O_4$ and $BaAl_2O_4$ from the same raw materials (since the compound $CaBaAl_4O_8$ is located on the $BaAl_2O_4$-$CaAl_2O_4$ conjugate); (5) - the possibility of formation of the compound $CaBaAl_4O_8$ from binary compounds $CaAl_2O_4$ and $BaAl_2O_4$:

$$CaCO_3 + BaCO_3 + 2Al_2O_3 = CaBaAl_4O_8 + 2CO_2 \quad (3)$$

$$CaCO_3 + BaCO_3 + 2Al_2O_3 = CaAl_2O_4 + BaAl_2O_4 + 2CO_2 \quad (4)$$

$$CaAl_2O_4 + BaAl_2O_4 = CaBaAl_4O_8 \quad (5)$$

Table 1. Variation of the Gibbs free energy with temperature

Temperature [K]	Gibbs free energy [kJ/mol], for the reaction	
	1	2
800	144.335	157.751
900	87.378	110.071
1000	31.564	62.398
1100	-23.197	14.695
1200	-76.973	-33.064
1300	-129.819	-80.902
1400	-181.776	-128.835
1500	-232.883	-176.876
1600	-283.169	-225.037
1700	-332.661	-273.327
1800	-381.380	-321.754
1900	-429.348	-370.326
2000	-476.585	-419.051

The change in the value of the Gibbs free energy $\Delta G = f(T)$ for the studied reactions is presented in Table 2.

Table 2. Variation of the Gibbs free energy with temperature

Temperature [K]	Gibbs free energy [kJ/mol], for the reaction		
	3	4	5
800	93.528	80.332	-1107.317
900	64.334	49.416	-1113.262
1000	35.720	18.912	-1119.612
1100	7.654	-11.226	-1126.277
1200	-19.891	-41.039	-1133.185
1300	-46.938	-70.556	-1140.277
1400	-73.501	-99.803	-1147.502
1500	-99.598	-128.801	-1154.819
1600	-125.238	-157.568	-1162.190
1700	-150.434	-186.119	-1169.586
1800	-175.194	-214.469	-1176.977
1900	-199.526	-242.629	-1184.340
2000	-223.439	-270.612	-1191.650

The analysis of the results obtained indicates the thermodynamic advantage of the formation of binary compounds $CaAl_2O_4$ and $BaAl_2O_4$ in a raw material mixture consisting of calcium carbon dioxide, barium carbon dioxide, and aluminum oxide (reaction 4), compared to the formation of $CaBaAl_4O_8$ in a raw material mixture of the same composition (reaction 3).

The reaction of the formation of $CaBaAl_4O_8$ from binary compounds (reaction 5) is not possible in the entire temperature range under consideration.

Thus, thermodynamic calculations indicate the fundamental impossibility of the formation of the ternary compound $CaBaAl_4O_8$ in the system and the predominant probability of the formation of binary compounds $CaAl_2O_4$ and $BaAl_2O_4$ instead.

Materials and Methods. To experimentally verify the theoretical calculations and literature data on the existence of ternary compounds in the CaO-BaO-Al_2O_3 system, the following compositions were synthesized: $Ba_3CaAl_2O_7$, $BaCa_2Al_8O_{15}$, and $BaCaAl_4O_8$. For the synthesis of samples of a given phase composition, the raw material mixtures were sequentially crushed, mixed, and fired. The starting materials used were calcium and barium carbonates of the "KhCh" grade and aluminum oxide of the "ChDA" grade. The raw materials were taken in a strictly defined stoichiometric ratio. Thorough grinding and mixing of the components was carried out in a laboratory ball mill using the "wet method" (slurry moisture content - 50 wt%). The grinding fineness was controlled by sieve analysis (complete passage through sieve No. 006).

Before firing, samples-cylinders with a diameter of 50 mm were formed from the raw material mixtures by two-sided pressing at a specific pressure of 60-80 MPa. The samples were fired in a krypton furnace at the specified synthesis temperatures and isothermal exposures. The completeness of the synthesis of the compounds was controlled by X-ray diffraction analysis and chemical analysis for the absence of free calcium and barium oxides. The chemical composition of the ternary compounds and synthesis parameters are given in Table 3, and the results of the studies are shown in Figs. 1-3.

Table 3. Chemical composition and synthesis parameters of ternary compounds of the CaO - BaO - Al_2O_3 system

Compound	Chemical composition [wt. %]			Synthesis temperature [°C]	Exposure [h]
	CaO	BaO	Al_2O_3		
$Ba_3CaAl_2O_7$	9.07	74.43	16.50	1380-1400	3
$BaCa_2Al_8O_{15}$	16.66	22.77	60.57	1400	3
$BaCaAl_4O_8$	13.57	37.10	49.33	2400 (from the melt)	–
				1400	3

As a result of our experimental studies, the existence of ternary compounds $Ba_3CaAl_2O_7$ and $BaCa_2Al_8O_{15}$ was confirmed (Figs. 1 and 2), and it was found that the $Ba_3CaAl_2O_7$ compound exists in the system at least up to a temperature of 1400 °C. Thus, our studies have determined an increase in the temperature limits of its existence, in contrast to the data of previous researchers [18], who indicated 1250 °C as the upper temperature of existence of $Ba_3CaAl_2O_7$.

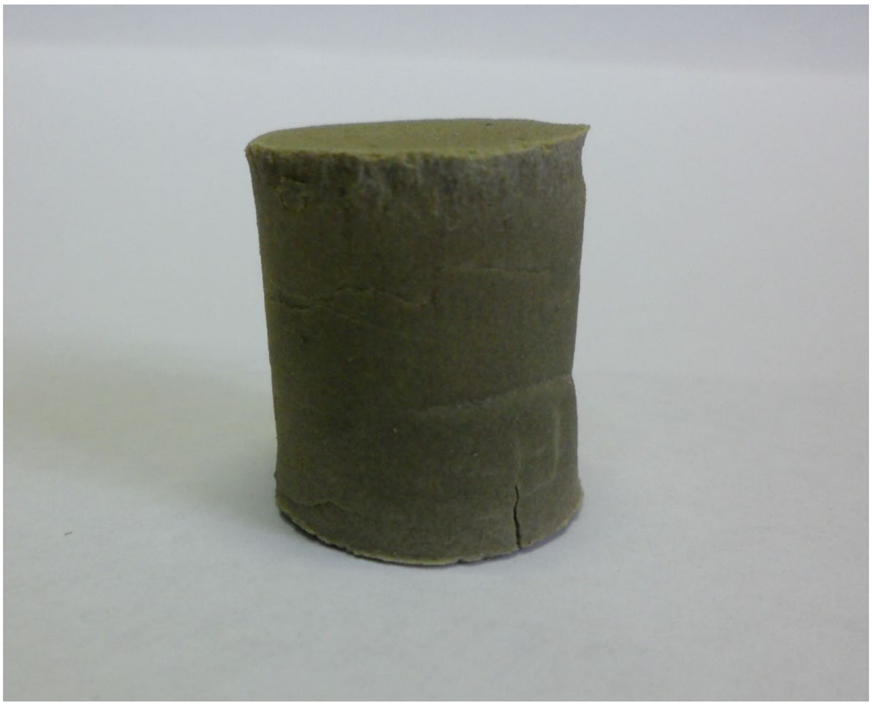
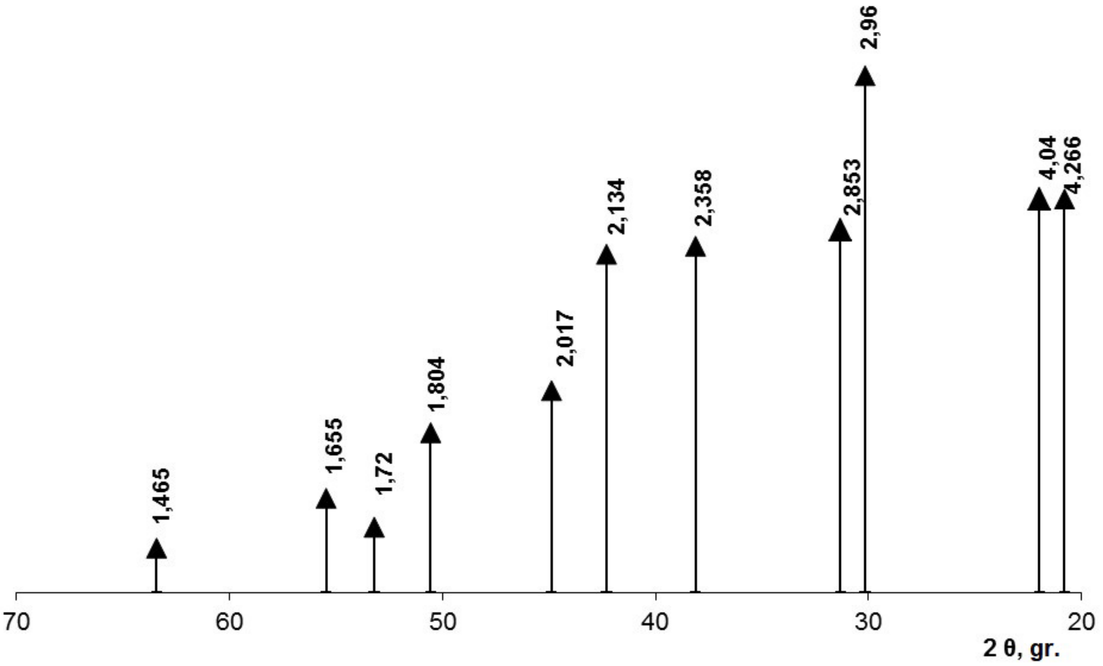
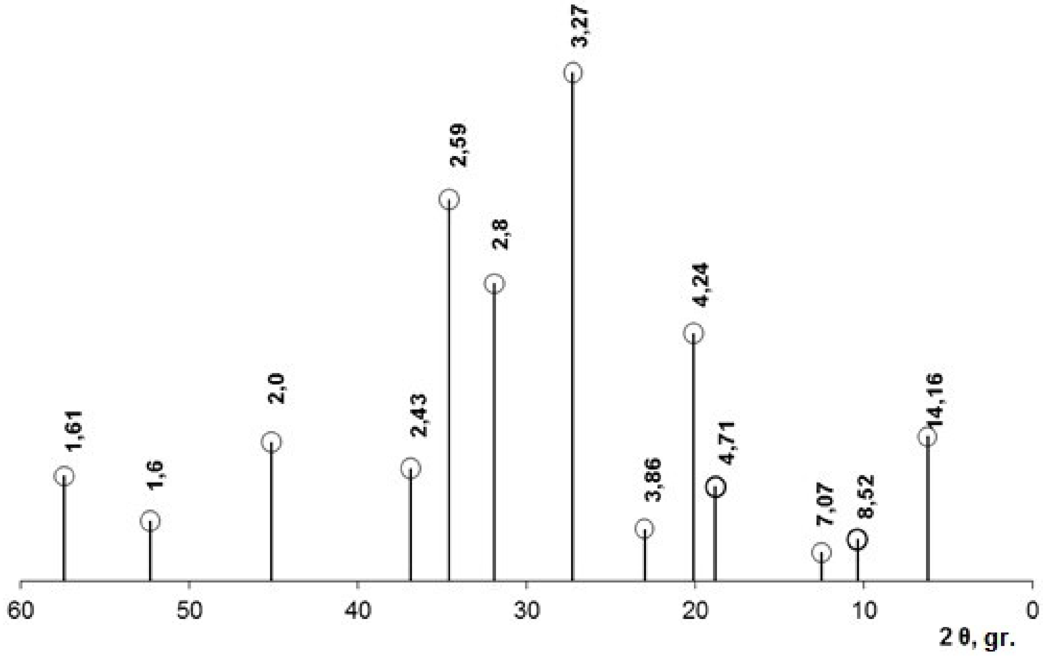


Fig.1. Sample appearance $Ba_3CaAl_2O_7$ (t_{firing}=1380-1400 °C, τ=3 h)



a)



b)

▲ – $Ba_3CaAl_2O_7$; ○ – $BaCa_2Al_8O_{15}$

Fig. 2. X-ray diffraction patterns of the synthesized ternary compounds:
a – $Ba_3CaAl_2O_7$ (t_{firing} = 1380-1400 °C, τ = 3h.); b – $BaCa_2Al_8O_{15}$ (t_{firing} = 1400 °C, τ = 3h.)

The X-ray diffraction patterns of the samples designed to obtain $BaCaAl_4O_8$ by solid-phase synthesis (see Fig. 3a), there are diffraction maxima corresponding to barium monoaluminates ($d \cdot 10^{-10}$ = 4.514; 4.01; 3.124; 2.60; 2.231; 1.999; 1.668; 1.585; 1.501 m) and calcium ($d \cdot 10^{-10}$ = 4.716; 3.001; 2.536; 2.173; 1.956 m). No other unidentified diffraction maxima were observed on the X-ray diffraction pattern. Since the author of [15] found the compound $BaCaAl_4O_8$ in slag, released from the melt at higher temperatures than those possible in solid-phase synthesis, the corresponding conditions for obtaining the compound by melting the raw material mixture at

2400 °C were reproduced. The results of the X-ray phase analysis of the crystallized melt (see Fig. 3b) show that a solid solution based on barium monoaluminate is present in the material, as evidenced by slightly shifted diffraction maxima corresponding to $BaAl_2O_4$ ($d\cdot10^{-10}$ = 4.514; 4.008; 3.124; 2.60; 2.228; 1.997; 1.701; 1.583 m) and calcium aluminate $Ca_{12}Al_{14}O_{33}$ ($d\cdot10^{-10}$ = 4.907; 2.999; 2.682 m).

For additional verification, compositions on the $CaAl_2O_4$-$BaAl_2O_4$ connode with different proportions of calcium and barium monoaluminates were studied. The X-ray diffraction patterns of the fired samples shown in Fig. 4, indicate the presence of only $CaAl_2O_4$ and $BaAl_2O_4$. Thus, the results of our experimental studies on the existence of the ternary compound $BaCaAl_4O_8$ are in good agreement with our thermodynamic calculations and those of other authors [16, 21] and prove the impossibility of its formation in the CaO-BaO-Al_2O_3 system, both from the melt and by solid-phase synthesis. In our opinion, the formation of the $BaCaAl_4O_8$ compound in the aluminum-barium slag in [15] can be explained by its special composition (the presence of a small amount of SiO_2, MgO, and F).

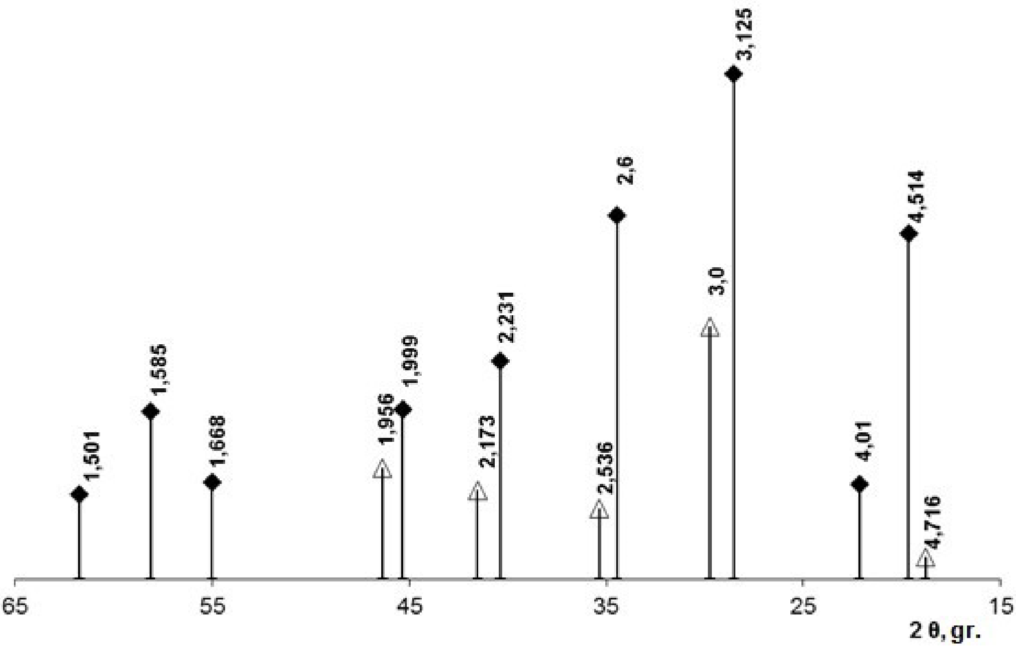
Evaluation of the manifestation of binding properties. To predict the nature of the interaction with water of ternary compounds and three-component compositions of the CaO-BaO-Al_2O_3 system, we used the methodology refined by N.F. Fedorov [22], which is based on the concept of electronegativity and allows us to assess the reactivity of inorganic oxide compounds with respect to water. Using this method, we calculated the electronegativity of compounds (ENsol.) as the geometric mean of the electronegativities of the elements that make up the compound. Comparison of the values of the relative electronegativity of compounds (ENvs.), obtained by dividing ENs. by the electronegativity of water (EN_{H2O}), with the presence of binding properties and the conditions of their manifestation allowed us to assume that binding properties are manifested only in those compounds whose ENvs. values are in a certain interval. The initial data for calculating EN were taken from [23].

According to our studies, aluminates of alkaline earth elements, for which the relative electronegativity is in the range of 0.69-0.82, are capable of exhibiting binding properties [22]. In accordance with the developed methodology, the binding properties of the ternary compounds $Ba_3CaAl_2O_7$ and $BaCa_2Al_8O_{15}$, as well as the three-component composition of $BaCaAl_4O_8$, were evaluated. Although the existence of a ternary compound $BaCaAl_4O_8$ in the CaO-BaO-Al_2O_3 system has been refuted by our theoretical and experimental studies, the composition of this composition is on the $CaAl_2O_4$-$BaAl_2O_4$ conjugate and can be the basis for the creation of a special calcium-barium alumina cement.

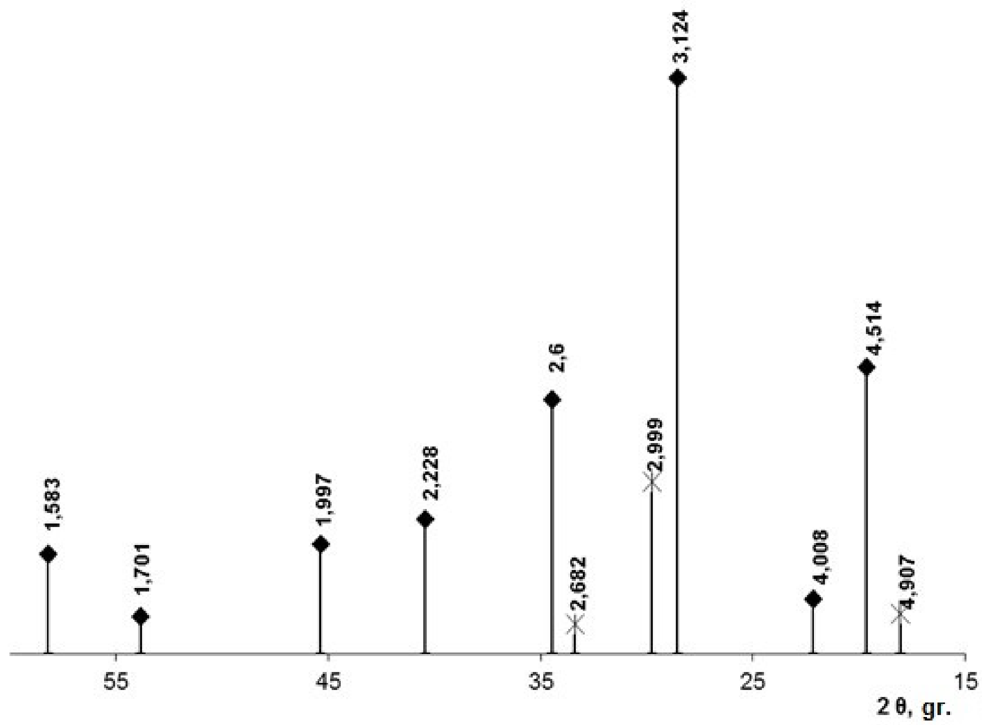
According to our calculations, $BaCaAl_4O_8$ (EH_V = 0.78) and $BaCa_2Al_8O_{15}$ (EH_V = 0.80) can exhibit binding properties, while the ternary compound $Ba_3CaAl_2O_7$ (EH_V = 0.68) will interact too actively with water, and, as a result, will not form a strong cement stone.

Physical and mechanical tests. Samples were made and their physical and mechanical properties were studied in accordance with the method of small samples by M.I. Strelkov [24]. The test results are shown in Table 4.

It was found that the compound $Ba_3CaAl_2O_7$ reacts rapidly when mixed with water, and the interaction reaction is accompanied by an increase in temperature to 55 °C and an increase in the volume of the mixture. In addition, an increase in temperature causes intense evaporation of water, and, as a result, the water demand increases sharply (W/C = 1.64). The samples are characterized by high porosity and low compressive strength (2 MPa after 28 days of curing).



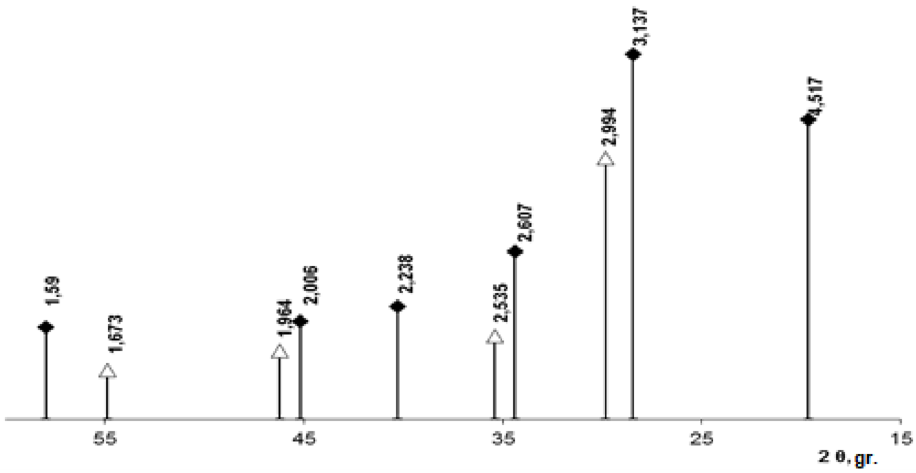
a)



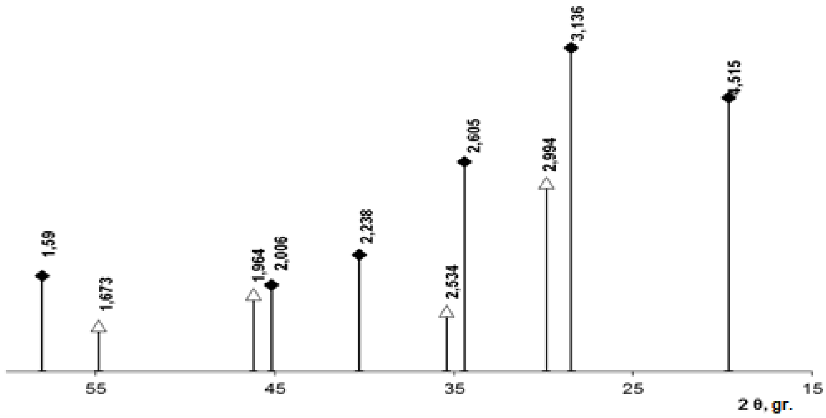
b)

♦ – BaAl2O4; △ – CaAl2O4; × – Ca12Al14O33

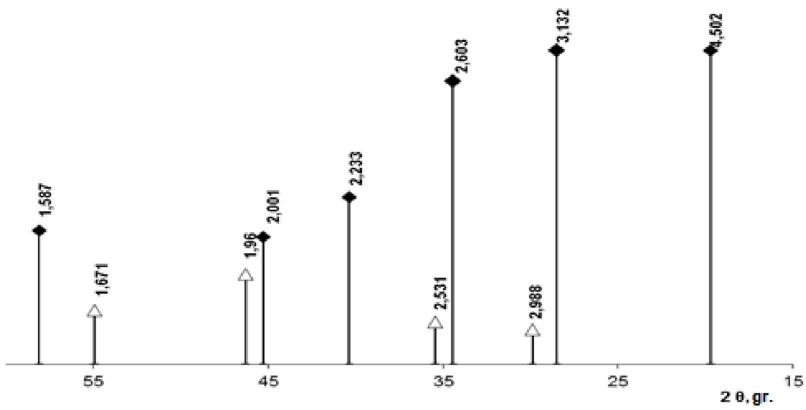
Fig. 3. X-ray radiographs of heat waves BaCaAl4O8:
a – (tfiring =1400 °C, τ = 3h.) ; b – (tfiring = 2400 °C, molten)



a)



b)



c)

♦ – BaAl2O4; △ – CaAl2O4

Fig. 4. X-ray radiographs of connode spicules BaAl2O4–CaAl2O4 (tfiring =1400 °C, τ = 3h), (m.%): a) 30 % BaAl2O4 : 70 % CaAl2O4; b) 60 % BaAl2O4 : 40 % CaAl2O4; c) 80 % BaAl2O4 : 20 % CaAl2O4

Table 4. Physico-mechanical properties of ternary compounds of the system $CaO–BaO–Al_2O_3$

Compound	W/C	The term of stiffening [hrs. - min.]		Compressive strength [MPa]			
		Start	Finish	1 day	2 days	7 days	28 days
$BaCa_2Al_8O_{15}$	0.31	0-55	1-11	No data	9.2	12.2	12.2
"$BaCaAl_4O_8$"	0.25	0-17	0-33	40,8	51.0	63.2	57.1
$Ba_3CaAl_2O_7$	1.64	0-15	1-15	No data	0	2.0	2.2

3 Conclusions

The presence of binding properties in the ternary compound $BaCa_2Al_8O_{15}$ was confirmed, the samples of which showed a compressive strength of 12 MPa in physical and mechanical tests after 28 days of curing. This compound can be used to produce calcium-barium alumina cement, but in combination with compounds characterized by higher mechanical strength, for example, in combination with calcium and barium monoaluminates.

Despite the fact that the existence of the compound $BaCaAl_4O_8$ has not been confirmed by our studies, the composition of this chemical composition containing calcium and barium monoaluminates can be used as a basis for the synthesis of refractory alumina cement, since it is characterized by a fast hardening period (beginning - 17 min, end - 33 min.), low water-cement ratio (W/C = 0.25), is a fast-setting and high-strength binder (compressive strength after one day of curing reaches 40 MPa, after 7 days - 63 MPa).

Thus, the experimental data fully confirm the results of the calculations. The obtained results made it possible to identify three-component compositions in the $CaO-BaO-Al_2O_3$ system, characterized by the presence of binding properties and can be the basis for the creation of new heat-resistant and refractory binders.

All in all, as a result of theoretical and experimental studies, the existence of triple compounds $Ba_3CaAl_2O_7$ and $BaCa_2Al_8O_{15}$ in the $CaO-BaO-Al_2O_3$ system was proved, and the existence of $BaCaAl_4O_8$ was refuted. It was found that the $Ba_3CaAl_2O_7$ compound exists in the system at least up to a temperature of 1400 °C. The binding properties of the synthesized ternary compounds and compositions of the system were evaluated. It is proved that the results of physical and mechanical tests fully correspond to the results of theoretical calculations.

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