

ШПИНЕЛЬОБРАЗУЮЩАЯ ФОСФАТНАЯ СВЯЗКА ДЛЯ КОРУНДОВЫХ КОМПОЗИЦИОННЫХ МАТЕРИАЛОВ**Н.В. Филатова, Н.Ф. Косенко, М.С. Малоиван, И.И. Зонина**

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Благодаря высоким температурам плавления фосфатов, они представляют особый интерес для огнеупоров и других высокотемпературных материалов. В качестве связующих используют собственно фосфорные кислоты и растворы кислых фосфатов. Фосфатные связки могут быть получены на основе химических реагентов, природных материалов, техногенных продуктов, а также применены для связывания материалов различного состава, в том числе и отходов промышленности. Большой интерес представляют алюмофосфатные связки и их производные, различные смешанные связующие. Последние перспективны тем, что при их высокотемпературной обработке могут образоваться шпинели, существенно улучшающие свойства композитов. В работе использованы брусит-алюмофосфатная (БАФС) – разновидность магнийалюмофосфатной связки – и алюмоборфосфатная (АБФС) связки для получения и исследования свойств корундового огнеупорного композита. По данным рентгенофазового анализа, при комнатной температуре в корундо-фосфатных смесях наблюдаются многочисленные рефлексы гидрофосфатов магния $Mg(H_2PO_4)_2$ и алюминия $AlH_3(PO_4)_2 \cdot 3H_2O$. При нагревании последние переходят в гидрофосфат магния $MgHPO_4 \cdot 3H_2O$, гидротирифосфат алюминия $AlH_2P_2O_7$, метаполифосфат магния $MgH(PO_3)_3$; кроме того, сразу появляется ортофосфат алюминия $AlPO_4$. При высокой температуре фосфатная фаза представлена только ортофосфатами $Mg_3(PO_4)_2$ и $AlPO_4$. Малое количество пиков фосфатов можно, по-видимому, связать с их преимущественно рентгеноаморфным состоянием. Шпинель $MgAl_2O_4$ появляется при 900 °С. Образование магнезиальной шпинели $MgAl_2O_4$ улучшает механические и термические свойства спеченного материала. В присутствии БАФС удается получить более плотные (с открытой пористостью 26,3 вместо 27,5 об.%) и прочные образцы (58 вместо 49 МПа) по сравнению с АБФС. После механоактивации корунда все характеристики улучшаются, а различия в свойствах образцов расходятся еще в большей степени, что можно связать с положительной ролью образующейся шпинели. Термические характеристики (термостойкость, линейный коэффициент термического расширения) полученных композитов мало зависят от вида выбранного связующего.

Ключевые слова: фосфаты, шпинели, магнийалюмофосфатная связка, бруситалюмофосфатная связка, корунд, огнеупорные композиты

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SPINEL-FORMING PHOSPHATE BINDER FOR CORUNDUM COMPOSITE MATERIALS

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Due to the high melting points of phosphates, they are of particular interest to refractories and other high-temperature materials. Phosphoric acids and solutions of acid phosphates are used as binders. Phosphate binders can be obtained from chemical reagents, natural materials, man-made products, and used to bind materials of various compositions, including industrial waste. Of great interest are alumina-phosphate binders and their derivatives, various mixed binders. The latter are promising in that during their high-temperature processing, spinels can form, significantly improving the properties of composites. The work uses brucite-alumina-phosphate (BAPB) – a type of magnesia-alumina-phosphate binder – and alumina-boron-phosphate (ABPB) binders to obtain and study the properties of a corundum refractory composite. According to X-ray phase analysis, at room temperature in corundum-phosphate mixtures, numerous reflexes of magnesium hydrogen phosphates $Mg(H_2PO_4)_2$ and aluminum $AlH_3(PO_4)_2 \cdot 3H_2O$ are observed. When heated, the latter pass into magnesium hydrogen phosphate $MgHPO_4 \cdot 3H_2O$, aluminum hydrogen pyrophosphate $AlHP_2O_7$, magnesium meta-polyphosphate $MgH(PO_3)_3$. In addition, aluminum orthophosphate $AlPO_4$ immediately appears. At high temperature, the phosphate phase is represented only by orthophosphates $Mg_3(PO_4)_2$ and $AlPO_4$. A small number of phosphate peaks can apparently be associated with their predominantly X-ray amorphous state. Spinel $MgAl_2O_4$ appears at 900 °C. The formation of magnesia spinel $MgAl_2O_4$ improves the mechanical and thermal properties of the sintered material. In the presence of BAPB, it is possible to obtain denser (with an open porosity of 26.3 instead of 27.5 vol. %) and durable samples (58 instead of 49 MPa) compared to ABPB. After the mechanical activation of corundum, all characteristics improve, and the differences in the properties of the samples diverge to an even greater extent, which can be associated with the positive role of the resulting spinel. The thermal characteristics (heat resistance, linear coefficient of thermal expansion) of the composites depend little on the type of binder chosen.

Key words: phosphates, spinels, magnesium alumina-phosphate binder, brucite-alumina-phosphate binder, corundum, refractory composites

INTRODUCTION

Today's high-temperature materials have exacting requirements for strength, chemical and thermal stability, corrosion resistance, including at high temperatures. These requirements only increase over time.

In recent decades, an important part of ceramic composites are chemically bound materials that acquire primary strength under normal conditions or with low-temperature processing, and during subsequent firing are significantly strengthened due to sintering. For this purpose, a variety of binders are used: calcium

aluminates (alumina cement), sodium silicates (liquid glass), magnesia compositions, phosphoric acid and phosphates, etc.

Since the 50s of the last century, astringents of phosphate hardening have been rapidly developing. The first significant work on phosphate-bound refractories was the Kingery Review [1], which described the interaction of silicates, metal oxides with phosphoric acid, and reactions of metal phosphates with refractory aggregates. Since then, astringents of phosphate hardening have constantly attracted the attention of many researchers both in the Soviet Union and then in Russia

[2] and abroad [3-6]. Due to the high melting points of phosphates, they are of particular interest to refractories and other high-temperature materials [1, 2-7]. Phosphoric acids and solutions of acidic phosphates are used as binders.

The term of *Chemically bonded phosphate ceramics* (CBPC) is widely used in recent years [4, 5, 7], but it seems to us to be inaccurate, since the formed ceramics are not essentially phosphates; in it, ceramic grains are only bound with phosphates. Any phosphate binding is chemical, as it involves the course of certain reactions leading to the material hardening. It seems to us more accurate and concise the name "phosphate-bounded ceramics".

The simplest phosphate binder, long used to produce high-temperature composite materials, is orthophosphoric acid (OPA) H_3PO_4 [2, 8]. To expand the possibilities of phosphate binding, a variety of phosphate binders (PB) have been developed, which are, as a rule, homogeneous systems obtained by neutralizing OPA with hydroxides or metal oxides or dissolution phosphates in water [2-7]. Partially neutralized OPA solutions provide more opportunities to regulate the rate of interaction with the solid phase.

The work [9] gives a classification of PB by aggregate state, form of phosphate-anion, phase state. Unlike [2], the classification includes PB not only in the form of true or colloidal solutions, but also suspensions, for example, a clay-phosphate binder. PB can be obtained based on chemical reagents, natural materials, technogenic products, as well as be used for binding materials of various compositions, including industrial waste [10-13]. Based on PB, it is possible to obtain both dense and porous products.

Selection of the binder type, the degree of neutralization, aggregate allows to vary the properties of the final composite within wide limits and achieve the desired result. Of great interest are alumina-phosphate binders and their derivatives (for example, alumina-chrome phosphate, alumina-boron-phosphate), magnesia-phosphate, various mixed binders (magnesia-alumina-phosphate, etc.). [13-14]. They are characterized by good binding capacity, high adhesive properties, low curing temperature, which distinguish them from OPA. For the synthesis of such PB, available components are required, and the process of obtaining itself does not cause difficulties. Mixed binders are also promising, in addition, because during their high-temperature processing, spinels can be formed that significantly improve the properties of composites [15-18]. For example, spinel $MgAl_2O_4$, distributed along the boundaries of corundum crystals, prevents their growth [15], which contributes to the formation of a better-

quality sintered corundum product. Besides, $MgAl_2O_4$ has a high melting point, high mechanical strength at elevated temperatures, good chemical resistance; products have increased resistance to temperature changes [16].

We have previously synthesized a brucite-phosphate binder (BPB) based on the natural mineral brucite, mainly containing magnesium hydroxide $Mg(OH)_2$, as a type of magnesia-phosphate bond [19], as well as a brucite-alumina-phosphate binder [13].

The purpose of this work is to obtain and study properties of corundum refractory composite using BAPB.

Materials and methods of research

Corundum micro powder (CP, finely ground electro corundum) with a specific surface of $150 \text{ m}^2/\text{kg}$, TU 2-036-00224450-022-90, and synthesized brucite-alumina-phosphate binder (BAPB) with a ratio of $MgO/Al_2O_3 = 1:1$ were used in the work. For comparison, we took alumina-boron-phosphate binder (ABPB), TU 113-08-606-87.

X-ray phase analysis of a mixture of corundum and BAPB at different temperatures was performed on a DRON-6 diffractometer. For this purpose, the mixtures were prepared with an excess of binders to better detect the reflexes of the formed phosphates and spinels. To decipher, the PDF database was used, as well as the data given in [17, 20].

Mechanical activation (MA) of the CP was carried out in the planetary mill AGO-2 for 30 min.

The molding of dense corundum materials was performed by the method of semi-dry two-stage pressing on a hydraulic press P-10. At the first stage, the pressure was 15 MPa with a holding of 20 s, at the second stage - 50 MPa with a holding of 30 s. Samples were prepared in the form of cylinders with dimensions of $29 \times 29 \text{ mm}$. The samples were fired in an electric furnace MP-2UM № 6497 and with inertial cooling. The resulting products were determined by bulk density, porosity, strength according to standard methods (GOST 2409-95, 4071.1-94).

The heat resistance of the obtained materials was tested in accordance with GOST 7875.2-94 according to a technique consisting in determining the number of heat shifts which the tested sample pass before destruction at a temperature difference from 950°C to the temperature of running water.

RESULTS AND DISCUSSION

To establish the phase composition of corundum-phosphate mixtures at different temperatures, X-ray phase analysis was performed (fig. 1). Strong corundum peaks, which do not change during heat treatment, are clearly visible. At room temperature (r.t.),

numerous reflexes of magnesium hydrogen phosphates $Mg(H_2PO_4)_2$ and aluminum $Al_3(PO_4)_2 \cdot 3H_2O$ are observed. When heated, the latter turn into magnesium hydrogen phosphate $MgHPO_4 \cdot 3H_2O$, aluminum hydrogen pyrophosphate $AlHP_2O_7$, magnesium metaphosphate $MgH(PO_3)_3$; in addition, aluminum orthophosphate $AlPO_4$ immediately appears. At high temperature, the phosphate phase is represented only by $Mg_3(PO_4)_2$ and $AlPO_4$ orthophosphates. A small number of phosphate peaks can, apparently, be associated with their predominantly X-ray amorphous state. Spinel $MgAl_2O_4$ appears at 900 °C.

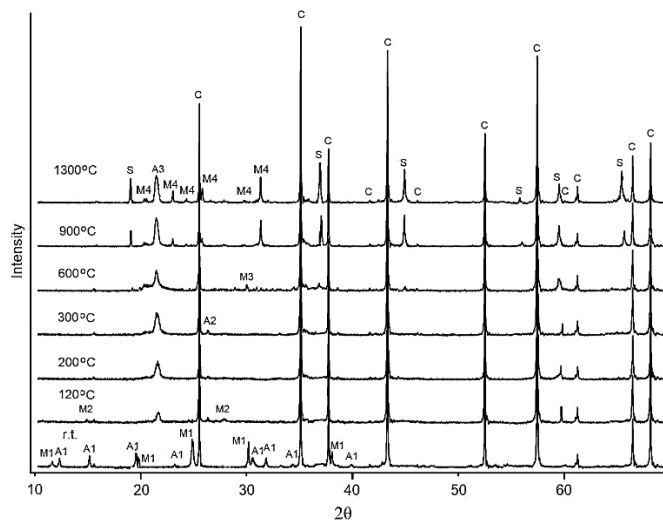


Fig. 1. Diffractograms of corundum + BAPB mixtures. Notation: C – corundum, S – spinel, A1 – $AlH_3(PO_4)_2 \cdot 3H_2O$, A2 – $AlHP_2O_7$, A3 – $AlPO_4$, M1 – $Mg(H_2PO_4)_2$, M2 – $MgHPO_4 \cdot 3H_2O$, M3 – $MgH(PO_3)_3$, M4 – $Mg_3(PO_4)_2$
 Рис. 1. Дифрактограммы смесей корунда и БАФС. Обозначения: С – корунд, S – шпинель, A1 – $AlH_3(PO_4)_2 \cdot 3H_2O$, A2 – $AlHP_2O_7$, A3 – $AlPO_4$, M1 – $Mg(H_2PO_4)_2$, M2 – $MgHPO_4 \cdot 3H_2O$, M3 – $MgH(PO_3)_3$, M4 – $Mg_3(PO_4)_2$

In our opinion, a matrix saturated with spinel new formation in a corundum composite can be represented in the form of fig. 2.

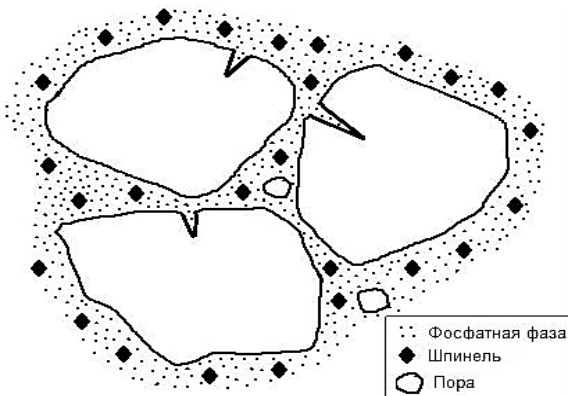


Fig. 2. Diagram of matrix formation between aggregate grains
 Рис. 2. Схема формирования матрицы между зёрнами заполнителя

The optimal number of binders for the most dense and durable composite formation was determined (Table 1).

Table 1
The effect of the amount of introduced BAPB and ABPB* on the properties of corundum materials. Firing temperature is 1300 °C

Таблица 1. Влияние количества вводимой БАФС и АБФС* на свойства корундовых материалов. Температура обжига 1300 °C

Preparation method	The content of the binder, wt. %	Open porosity, vol. %	Compressive strength, MPa
Simple mixing with a bundle	7	31.4 ± 0.6 30.6 ± 0.5	33 ± 2 30 ± 1
	8	28.0 ± 0.6 28.6 ± 0.3	47 ± 4 41 ± 5
	10	26.3 ± 0.3 27.5 ± 0.2	58 ± 3 49 ± 2
	12	28.1 ± 0.4 28.0 ± 0.4	54 ± 5 47 ± 6
Mechanical activation of corundum with a binder	7	29.1 ± 0.5 30.7 ± 0.5	67 ± 5 60 ± 4
	8	27.4 ± 0.4 27.6 ± 0.3	80 ± 6 74 ± 3
	10	24.3 ± 0.1 26.7 ± 0.6	98 ± 8 84 ± 5
	12	26.6 ± 0.6 28.0 ± 0.5	96 ± 7 93 ± 7
Mechanical activation of corundum with a binder pretreated at 180 °C	7	26.6 ± 0.3 29.5 ± 0.3	80 ± 10 79 ± 3
	8	24.0 ± 0.4 27.7 ± 0.5	96 ± 8 88 ± 6
	10	23.1 ± 0.5 23.1 ± 0.3	122 ± 10 115 ± 5
	12	23.7 ± 0.7 24.2 ± 0.3	116 ± 7 110 ± 3

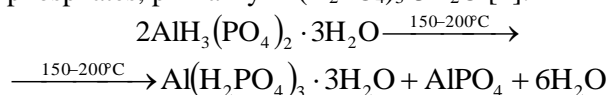
Note: The numerator shows the data obtained for samples based on BAPB, and the denominator - for ABPB

Примечание: * В числителе приведены данные, полученные для образцов на основе БАФС, а в знаменателе – для АБФС

The dependences of open porosity and strength of materials on the content of both binders contain coinciding extrema: compressive strength has the highest values at a binder content of ~10%, while open porosity reaches a minimum of 28-23%. An increase in strength with an increase in the amount of binder is because during firing a phosphate phase is formed, which combines corundum particles into a single conglomerate with sufficient strength. In the presence of BAPB, it is possible to obtain denser (with an open porosity of 26.3 instead of 27.5 vol. %) and stronger samples (58

instead of 49 MPa) compared to ABPB. After mechanical activation of corundum, all characteristics improve, and the differences in the properties of the samples diverge even more, which can be associated with the positive role of the resulting spinel. The greatest increase in the strength corresponded to a 30-min mechanical treatment.

Of interest is the fact that the preliminary heat treatment of binders at 180 °C plays a positive role, probably due to the appearance of active hydrogen phosphates, primarily $\text{Al}(\text{H}_2\text{PO}_4)_3 \cdot 3\text{H}_2\text{O}$ [2]:



The formation of this compound can be confirmed by the detection of accompanying Al orthophosphate in the diffraction pattern.

Acid phosphate interacts with the surface of corundum and improves its adhesive properties, thereby increasing the sample strength.

In addition, the MA of the totality of solid phases is more efficient compared to the processing of a mixture of solid and liquid components. However, it is difficult to evenly distribute the water introduced into the mixture after MA to ensure its formability, and hence to ensure a stable quality of the material. Obtaining and subsequent application of a heat-treated binder is a laborious process, because with an increase in temperature, it sticks to the walls of the vessel and grinds poorly. In this regard, the use of a phosphate binder in this form is hardly advisable.

Important characteristics of refractory products are the linear coefficient of thermal expansion (LCTE) and the ability to withstand standard sudden temperature changes (thermal resistance) without destruction, even though GOST 24704–2015 "Refractory corundum and high alumina products. Technical conditions" is not normalizing them. LCTE values calculated from dilatometric curves and thermal stability (difference 950 °C – water) for mechanically activated samples are given in table 2.

As the data show, there is no significant change in the thermal characteristics of corundum composites when replacing one phosphate binder with another. Both preheat-treated phosphate components contribute to better properties: less thermal expansion and greater thermal stability. Since, ultimately, the refractory composites synthesized under optimal conditions have a sufficiently high porosity, but acceptable by the standards, with a sharp temperature drop, the re-

sulting harmful stresses dissipate in the pores, thereby having a small destructive effect. In practice, refractories are prepared from a mixture of corundum grains of different grades and a powder that fills the pores, so the selection of the appropriate granulometric composition of the aggregate will further reduce the open porosity.

Table 2

Thermal stability and LCTE of corundum materials with ABPB and BAPB

Таблица 2. Термическая стойкость и ЛКТР корундовых материалов на АБФС и БАФС

Sample	Thermal stability, cycles		LCTE $\alpha \cdot 10^6$, K ⁻¹	
	for ABPB	for BAPB	for ABPB	for BAPB
CP + binder (10%)	18	17	7.95	7.86
CP + heat-treated binder (12%)	24	21	7.65	7.57

CONCLUSION

The expediency of using a magnesium alumina-phosphate binder in the form of a brucite-alumina-phosphate binder to produce sintered corundum composites has been established. It is shown that the formation of magnesia spinel MgAl_2O_4 during heat treatment contributes to an increase in the strength of samples by 17-18% and a decrease in open porosity by 4.4-9% compared to the alumina-boron-phosphate binder. The values of the linear coefficient of thermal expansion and thermal stability are practically independent of the type of binder.

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The authors declare the absence a conflict of interest warranting disclosure in this article.

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