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COMPOSITE CERAMICS FOR SPECIAL PURPOSE

As a result of the research carried out, the search for the optimal compositions of composite ceramics was carried out by the method of planning the experiment using the FFD2^{5-1} as an experiment plan. The influence of modified additives of eutectic compositions on the physical and technical properties of composite ceramics for special purposes, with a low sintering temperature, used for the manufacture of grinding bodies, has been determined.

Keywords: composite ceramics, additives, modification, grinding media, microstructure.

Background. In many industries, it is necessary to obtain substances with a small particle size. For grinding various substances, grinding equipment is used, which includes grinding bodies [1; 2].

One of the ways to increase the efficiency of grinding equipment is the use of alumina grinding bodies [3; 4], as the most wear-resistant under conditions of increased static and dynamic loads. But the synthesis of such composite ceramics is associated with the use of high temperatures, which is associated with significant energy and material consumption of production.

It is known [5–7] that for reducing the temperature of sintering of the ceramic material, there are three basic ways: increasing fineness of powder, introduction of defects in the crystal lattice and the introduction of modifying additives.

The use of modifiers of eutectic compositions due to the formation of liquid during firing makes it possible to reduce the sintering temperature [3; 5]. This ensures the crystallization of the melt during the cooling process, which contributes to the production of dense fine-crystalline materials by reducing the firing temperature. The greatest positive effect in this direction is achieved with the use of complex additives, which ultimately affect the properties of the product [6; 7].

Insufficient knowledge about the mechanism of their action in the composition of ceramics, which is far from additivity, has led to the need to use the method of planning an experiment when obtaining composite ceramics for grinding bodies.

Analysis of recent research and publications. The issue of directed regulation of the microstructure and properties of composite ceramics for special purposes by chemical modification with additives has been the subject of the work of many scientists.

In [7–9], it is noted that ceramics based on Al_2O_3, which do not contain modifying additives (modifiers), are characterized by a low level of physical and technical properties, difficult to control structure, low density and high sintering temperature. The main task of using such ceramics is to reduce its sintering temperature to the level of 1250–1300 °C.

To reduce the sintering temperature, improve the properties of alumina ceramics, various additives are used, which can be classified according to several criteria: according to the number of added additives (micro and macro additives), according to the number of additive components (single and multicomponent), according to the sintering mechanism (liquid and solid-phase sintering), on the effect on the base substance, etc. [10].

Summarizing the work on sintering ceramics based on pure oxide and oxygen-free compounds with additives, all modifiers by the nature of their interaction with Al_2O_3 are divided into 4 groups [8]:

1) completely soluble in the crystal lattice of the oxide (HfO_2 in Y_2O_3, Y_2O_3 in ZrO_2);

2) insoluble in the crystal lattice of the basic oxide, forming a liquid phase due to melting or interaction with the basic oxide with the formation of a eutectic melt ($RO–Al_2O_3–SiO_2$, $MnO–Cr_2O_3–SiO_2$, etc.);

3) insoluble in the crystal lattice of the basic oxide and not interacting with it (eutectics in the systems $Al_2O_3–ZrO_2$, $BeO–ZrO_2$, etc.);

4) entering into chemical interaction with the basic oxide with the formation of a compound (Al_2O_3 in Y_2O_3, etc.).

In the technology of modern ceramic materials, modifiers of the first and second groups are more often used.

Noteworthy is the study of issues related to forecasting fluxing ability of both traditional (system $K_2O–Al_2O_3–SiO_2$, $Na_2O–Al_2O_3–SiO_2$, $MgO–Al_2O_3–SiO_2$) and new, including the combined intensifiers sintering containing Li_2O, CaO, B_2O_3, MnO_2, etc., which will make it possible to reasonably choose the composition of ceramic masses and the firing temperature of products [11].

When choosing sintering additives, it is necessary to take into account the structure, nature and temperature of the appearance of the liquid phase; the size of the ionic radius of the modifier; the binding energy of the modifier cation – oxygen in the multicomponent melt; the geometry of the powder grains, the electrostatic state of the solid-liquid interface; surface tension at the solid-liquid interface, etc. [12; 13].

Investigations into the regularities of sintering ceramics based on Al_2O_3 containing eutectic modifiers are still ongoing.

The aim of the work is to study the effect of additives on the physical and technical properties of composite ceramics for the manufacture of grinding bodies and the search for its optimal compositions.

Materials and methods. The main component for the manufacture of ceramic composite material is alumina grade *Alumina SC-0*, containing up to 95 % α-Al_2O_3.

Aluminum oxide in the form of α-Al_2O_3 is stable when heated up to the melting point and does not have modified transformations. As accessory impurities in the bound state, there are oxides of iron, silicon, calcium and alkaline earth metals in an amount of 1–2 %.

In accordance with the technology of obtaining grinding bodies, the composition of the masses included clay from the Veselovsky deposit of the VGO, kaolin from the Nemilnyansky deposit, and bentonite from the Dashukovsky deposit.

According to the general concepts of the physicochemical nature of the modifying additives used in the preparation of composite ceramics, in order to reduce the sintering temperature and increase the operational properties, additives of a complex composition were used, including eutectic mixtures and monoxides [12]. The choice of fluxing materials was carried out based on the presence in nature and the practical positive results of their use.

To intensify the sintering processes and modify the structure of the ceramics, the following additives were used: *dolomite, colemanite, lepidolite, pyrolusite.*

Dolomite – $CaMg(CO_3)_2$ – after decomposition at a temperature of 800–1000 °C, is able to interact with other components of the ceramic mass, in the formation of fusible compounds that perform the functions of flux, which leads to a decrease in the sintering temperature and refractoriness [6; 14].

Colemanite – $Ca_2B_6O_{11} \cdot 5H_2O$ – creates favorable conditions for the intensification of physicochemical processes, the earlier appearance of low-melting eutectics and, accordingly, contributes to the formation of the required amount of the primary low-melting glassy phase and the subsequent dissolution of quartz grains in it at relatively low temperatures. The synthesized ceramics of colemanite are characterized by high strength characteristics and a wide range of values of the coefficient of thermal expansion, which is a consequence of the influence of boron and calcium oxides, which are part of colemanite.

Lepidolite – $K(Li, Al)_3(Si, Al)_4O_{10}(F, OH)_2$ – makes it possible to obtain high-quality products at lower firing temperatures. The decrease in the firing temperature is explained by the presence of Li^+ in the lepidolite concentrate, which has a smaller radius than Na^+ and

K^+ and is more active, contributing to better sintering. Lepidolite is one of the rare micas, characterized by a low coefficient of thermal expansion, which gives products a low shrinkage with high physical and mechanical properties.

Pyrolusite – $MnO_2 \cdot H_2O_2$ – additive provides a significant decrease in the sintering temperature of the charge and leads to an improvement in such parameters of ceramics as strength and hardness.

To prepare the additives of the eutectic composition for use in the composition of the masses, the loss on ignition of each of the components was preliminarily determined. The amount of starting materials was calculated in accordance with the loss on ignition and the chemical composition of the eutectics selected for the study.

To prepare the additives, the initial mixtures of the calculated composition were loaded into a ball mill, dissolved with distilled water, and ground for 8 h with corundum balls. The grinding media/water/material ratio was 3 : 1 : 1. The resulting suspensions were dried at a temperature of 80–100 °C, then the materials were rubbed through a N 01 sieve.

The powders were loaded into corundum crucibles and calcined with short holding times. Temperature regimes were selected according to petrographic studies. After calcination, the material was ground under the same conditions as before synthesis. The finished powders were used to formulate the compositions.

The compositions were prepared by joint wet grinding of the components to a final residue of 0.08–0.11 % on a sieve N 0063. The resulting suspension was dehydrated in plaster molds. After curing and processing, the masses were molded into cylindrical grinding bodies on a manual laboratory press, after drying at ambient temperature and drying in an oven at a temperature of 100–150 °C, the products were fired in an electric furnace at temperatures of 1250 and 1280 °C.

The evaluation of the sintering process of the studied compositions was carried out according to the presence of the values of shrinkage, water absorption, bulk density, abrasion, ultimate strength in compression, ultimate strength in bending according to DSTU B V.2.7-283:2011 [15].

The mathematical processing of the results was carried out using the software package Apache Open Office.

Results. To obtain reliable and objective data on the effect of modifying additives of colemanite, dolomite, lepidolite, pyrolusite on the structure and properties of a ceramic material of the following composition: alumina grade *Alumina SC-0* – 40 %, clay from the Veselovsky deposit *VGO* – 20 %, kaolin from the Nemil'nyansky deposit – 20 %, bentonite of the Dashukovskoe deposit – 5 %, the method of mathematical planning of the two-factor experiment $FFD2^{5-1}$ was applied [16; 17].

The conducted criterion-factor experiment made it possible to determine the optimal compositions of the masses containing the above-mentioned modifying additives for grinding bodies.

The efficiency of the process of obtaining the composition of the mass for grinding bodies was determined by the following optimization parameters: shrinkage (Y_1), water absorption (Y_2), bulk density (Y_3), abrasion (Y_4), ultimate strength in compression (Y_5), ultimate strength in bending (Y_6).

The choice of these optimization parameters is based on their significant effect on the performance properties that ensure the durability of products. Each of the selected parameters varied at two levels.

A priori, dolomite (X_1), colemanite (X_2), lepidolite (X_3), pyrolusite (X_4), and firing temperature (X_5) were identified as factors. These factors have a greater impact on the presented optimization parameters.

The implemented experiment plan is shown in *table 1*. The values of the factors are given in coded and natural form.

Table 1

Planning matrix in natural and coded values and optimization parameters

The coded values of the factors					Natural values of factors					Optimization parameters											
X_1	X_2	X_3	X_4	X_5	X_1	X_2	X_3	X_4	X_5	y_1	$\hat{Y}_1$	y_2	$\hat{Y}_2$	y_3	$\hat{Y}_3$	y_4	$\hat{Y}_4$	y_5	$\hat{Y}_5$	y_6	$\hat{Y}_6$
–1	–1	–1	–1	+1	0	+2	+1	0	+1280	4.50	4.40	9.40	9.49	2.50	2.49	0.369	0.369	972.67	970.11	729.67	745.27
+1	–1	–1	–1	–1	+5	+2	+1	0	+1250	7.13	7.129	4.26	4.29	2.82	2.82	0.198	0.198	862.50	859.94	748.67	733.06
–1	+1	–1	–1	–1	0	+5	+1	0	+1250	5.36	5.359	8.30	8.095	2.63	2.63	0.413	0.413	1143.00	1140.00	917.33	901.72
+1	+1	–1	–1	+1	+5	+5	+1	0	+1280	10.09	10.089	0.098	0.106	3.02	3.00	0.063	0.063	1761.50	1758.94	1048.00	1063.60
–1	–1	+1	–1	–1	0	+2	+5	0	+1250	3.66	3.66	13.69	13.659	2.31	2.32	0.287	0.287	463.00	465.57	323.00	307.40
+1	–1	+1	–1	+1	+5	+2	+5	0	+1280	9.93	9.93	0.050	0.0192	2.95	2.96	0.117	0.117	2177.00	2179.56	932.00	947.60
–1	+1	+1	–1	+1	0	+5	+5	0	+1280	9.95	9.95	0.412	0.616	3.03	3.04	0.108	0.108	3892.50	3895.05	988.00	1003.60
+1	+1	+1	–1	–1	+5	+5	+5	0	+1250	11.67	11.67	0.050	0.254	2.94	2.95	0.075	0.075	1848.00	1850.56	1048.67	1033.06
–1	–1	–1	+1	–1	0	+2	+1	+5	+1250	6.56	6.56	6.41	6.61	2.50	2.51	0.149	0.149	944.00	946.56	489.33	504.93
+1	–1	–1	+1	+1	+5	+2	+1	+5	+1280	9.31	9.31	0.04	0.24	3.03	3.04	0.069	0.069	2404.50	2407.06	1170.67	1155.06
–1	+1	–1	+1	+1	0	+5	+1	+5	+1280	9.94	9.94	0.26	0.23	3.13	3.14	0.057	0.057	1766.00	1768.56	1222.00	1206.39
+1	+1	–1	+1	–1	+5	+5	+1	+5	+1250	10.03	10.03	0.01	0.02	2.94	2.95	0.083	0.083	2126.00	2128.56	950.00	965.60
–1	–1	+1	+1	0	+2	+5	+5	+5	+1280	9.70	9.699	0.10	0.10	3.05	3.05	0.063	0.063	3299.67	3297.10	1269.33	1253.72
+1	–1	+1	+1	–1	+5	+2	+5	+5	+1250	11.19	11.189	0.05	0.15	2.98	2.96	0.082	0.082	3307.67	3307.10	1124.00	1139.60
–1	+1	+1	+1	–1	0	+5	+5	+5	+1250	12.05	12.049	0.05	0.08	3.02	3.00	0.086	0.086	2651.00	2648.44	1170.00	1185.60
+1	+1	+1	+1	+1	+5	+5	+5	+5	+1280	11.04	11.039	0.055	0.085	2.95	2.95	0.065	0.065	1664.00	1661.44	981.00	965.39

To optimize the parameters obtained regression equation:

shrinkage $\hat{Y}_1$:

$$\hat{Y}_1 = 8.882 + 1.167X_1 + 1.134X_2 + 1.017X_3 + 1.096X_4 - 0.752X_1X_4,$$

water absorption $\hat{Y}_2$:

$$\hat{Y}_2 = 2.702 - 2.126X_1 - 1.548X_2 - 0.895X_3 - 1.83X_4 - 1.4X_5 + 1.024X_1X_2 + 0.37X_1X_3 + + 1.29X_1X_4 + 0.884X_1X_5 + 0.77X_2X_4 + 0.452X_2X_5 - 0.252X_3X_5 + 0.642X_4X_5,$$

bulk density $\hat{Y}_3$:

$$\hat{Y}_3 = 2.862 + 0.091X_1 + 0.095X_2 + 0.088X_4 + 0.095X_5 - 0.086X_1X_2$$

abrasion: $\hat{Y}_4$:

$$\hat{Y}_4 = 0.143 - 0.049X_1 - 0.061X_4,$$

ultimate strength in compression $\hat{Y}_5$:

$$\hat{Y}_5 = 1955.187 + 457.666X_3,$$

ultimate strength in bending $\hat{Y}_6$:

$$\hat{Y}_6 = 944.48 + 96.146X_2 + 102.562X_4 + 98.104X_5 - 89.604X_1X_2 - 65.563X_1X_3 - 62.437X_2X_4 - 78.979X_2X_5 + 54.021X_3X_4.$$

The adequacy of the models was checked according to the Fisher criterion [16]. The hypothesis of an adequate model for all optimization parameters is not rejected at a significance level $\alpha = 0.05$.

Checking the uniformity of dispersions according to the Cochran criterion [16] showed that the values of the optimization parameters for shrinkage (Y_1), bulk density (Y_3), abrasion (Y_4), ultimate strength in compression (Y_5) are uniform for $\alpha = 0.05$, and water absorption (Y_2) and ultimate strength in bending (Y_6) – at $\alpha = 0.01$.

It was also tested the hypothesis about the importance of estimates of regression coefficients [16; 17].

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Comparative analysis of changes in shrinkage, water absorption, bulk density, abrasion, ultimate strength in compression, ultimate strength in bending of the studied compositions on the firing temperature showed that almost all studied mineralizers affect the sintering process of the studied ceramics of the compositions at given firing temperatures.

Taking into account the analysis of the regression equations, the following mass compositions were selected, with the optimal additive content *(table 2)*.

Table 2

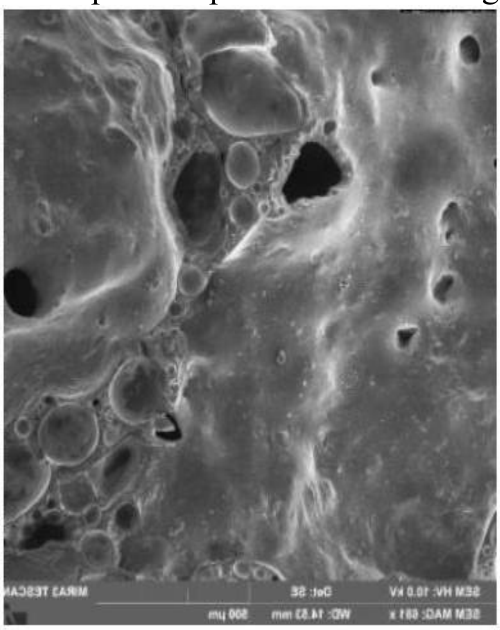
Optimal compositions of the masses for composite ceramics

Code of mass	Modifying additives, %				Firing tempe-rature, °C	Physical and mechanical properties					
	dolomite	colemanite	lepidolite	pyrolusite		shrin-kage, %	water absorption, %	bulk density, g/cm³	abrasion, % per hour	ultimate strength in compression, MPa	ultimate strength in bending, MPa
A_1	0	3.2	2.9	7	1280	10.06	0.03	3.10	0.055	233.09	120.15
A_2	5	2.0	1.75	5	1250	9.58	0.03	3.07	0.048	270.12	118.16
A_3	0	3.3	3.1	4	1280	10.18	0.02	3.11	0.050	250.64	121.16
A_4	3	2.8	3.2	6	1250	9.97	0.04	3.06	0.060	280.14	120.70

To obtain data on the phase composition and structural parameters material, the fired samples of composite ceramics were studied using a scanning electron microscope with an X-ray microscope attachment, which allows semi-quantitative determination of the elemental composition of the material by a local method.

For the study, a mass of composition A_4 was used containing, %: alumina grade Alumina *SC-0* – 40, clay from the Veselovsky deposit VGO –20, kaolin from the Nemilnyansky deposit 20, bentonite from the Dashukovsky deposit – 5, *dolomite* – 3, *colemanite* – 2.8, *lepedolite* – 3.2, *pyrolusite* – 6. The physical and mechanical properties of A_4 mass at a firing temperature of 1250 °C were obtained and presented above (see *table 2*).

Figure shows a snapshot of the A_4 composition matrix of the developed composite ceramics for grinding bodies.



Microstructure matrix of optimal composition A_4 of composite ceramics

Analyzing the image, we can conclude that the material under study is composed of corundum grains, the shape of which varies from isometric to short-prismatic, the predominant grain size is 3 ÷ 5 μm. The introduction of modifying additives leads to the formation of a network structure along the periphery of the corundum crystals. The volumetric content of the binder cementing corundum grains is 7–8 %. The phase composition of the cementing bond is represented by crystalline and amorphous glassy phases. The content of the glassy phase is 1÷1.7 %. The binder phases crystallize with a grain size of 1–2 microns, in a cubic system. The amorphous glassy phase is homogeneous in composition, the refractive index $n = 1.540 – 1.545$. The material has a closed inter crystalline porosity, distributed in the volume of the material in the form of rounded clusters with a size of 8 ÷ 10 microns. The number of pores does not exceed 2.3 % by volume.

The use as modifiers of additives of eutectic compositions *dolomite*, *colemanite*, *lepidolite*, and *pyrolusite* due to the formation liquid during firing made it possible to reduce the sintering temperature to the level of 1250–1280 °C.

In the process of cooling, crystallization of the melt occurred due to the "genetic" memory of the eutectic about crystalline phases, which contributed to the production of fine-crystalline materials of high density.

Conclusion. The studies carried out allowed us to draw conclusions about the effect of each component of the mass on the physical and mechanical properties of composite ceramics, as well as to reveal the mechanism of formation of the composition and microstructure of composite ceramics based on the main components of the mass and modifying additives, which makes it possible to predict the production of products with the required properties.

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Палієнко О. Композитна кераміка спеціального призначення.

Постановка проблеми. Композитна кераміка використовується як інгредієнт мелючих тіл, що застосовуються помольним обладнанням під час подрібнення різних речовин. Але синтезування такої композитної кераміки пов'язане з використанням високих температур. Відомо, що для зниження температури спікання керамічного матеріалу існує декілька способів, один із них – введення модифікуючих добавок.

Застосування модифікаторів евтектичних складів внаслідок утворення рідини в ході випалу дає змогу знизити температуру спікання.

Недостатнє знання про механізм їхньої дії у складі кераміки, далекий від адитивності, призвело до необхідності використання методу планування експерименту при отриманні композитної кераміки.

Мета статті – дослідження впливу добавок на фізико-технічні властивості композитної кераміки для виготовлення мелючих тіл та пошук її оптимальних складів.

Матеріали та методи. Основним компонентом для виготовлення керамічного композитного матеріалу є глинозем марки *Alumina SC-0*, що містить до 95 % α-Al_2O_3. Відповідно до технології отримання кераміки до складу маси включено глину Веселовського родовища ВГО, каолін Немильнянського, бентоніт Дашуківського родовища. Для інтенсифікації процесів спікання і модифікування

структури кераміки використано добавки: *доломіт* – $CaMg(CO_3)_2$ – 1–5 %, *колеманіт* – $Ca_2B_6O_{11} \cdot 5H_2O$ – 5–10 %, *лепідоліт* – $K(Li, Al)_3(Si, Al)_4O_{10}(F, OH)_2$ – 0–10 %, *піролюзит* – $MnO_2 \cdot H_2O_2$ – 1–3 %.

Методом регресійного аналізу експериментальних даних за ДФЕ2^{5-1} отримані математичні описи залежності властивостей композитної кераміки від вмісту модифікуючих домішок. Математична обробка результатів експерименту здійснена з використанням пакету програм *Apache Open Office*.

Результати дослідження. Обрано оптимальний склад маси A_4, що містить, %: глинозем марки *Alumina SC-0* – 40, глину Веселовського родовища ВГО – 20, каолін Немильнянського родовища – 20, бентоніт Дашуківського родовища – 5, доломіт – 3, колеманіт – 2.8, лепідоліт – 3.2, піролюзит – 6.

Фізико-механічні властивості маси A_4 за температури випалу 1250 °C становлять: усадка – 9.97 %, водопоглинання – 0.04 %, об'ємна маса – 3.065 г/см^3, стиранiсть – 0.060 % за год, межа міцності при стиску – 280.14 МПа, при згині – 120.7 Мпа.

Висновки. Доведено вплив кожного компонента, що входить до складу маси композитної кераміки, на її фізико-механічні властивості.

Встановлено механізм формування складу і мікроструктури композитної кераміки на основі компонентів маси і модифікуючих добавок, що дає змогу прогнозувати отримання виробів із необхідними властивостями.

Ключові слова: композитна кераміка, модифікуючі добавки, мікроструктура.