

## **Preparation of composite materials based on oxide ceramics by the free SHS-compression**

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**Abstract:** This article deals with a problem relevant for modern industry – the development of new composite materials based on oxide ceramics used in application of protective coatings to parts and cutting tools. The authors proposed the free SHS compression method as a technology for producing such materials; it combines the process of self-propagating high-temperature synthesis of materials and their subsequent high-temperature shear deformation of the resulting products. The influence of free SHS compression process parameters on the process of manufacturing products from the selected research objects has been studied. It has been found that for the tested materials there is an optimal temperature-time treatment interval that allows manufacturing high-quality products. A study of the phase composition and microstructure of the obtained products has been conducted. The paper also studies the physical and mechanical properties of the obtained products, and conducts heat resistance tests in the temperature range of 1000–1300 °C for 10 hours.

**Keywords:** free SHS compression; self-propagating high-temperature synthesis; aluminum oxide; high-temperature materials.

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## **Получение композиционных материалов на основе оксидной керамики методом свободного СВС-сжатия**

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**Аннотация:** Настоящая статья направлена на решение актуальной для современной промышленности задачи, а именно разработку новых композиционных материалов на основе оксидной керамики, предназначенных для нанесения защитных покрытий на детали и режущий инструмент. В качестве технологии получения таких материалов в данной работе предложен метод свободного СВС-сжатия, который сочетает в себе процесс самораспространяющегося высокотемпературного синтеза (СВС) и последующее высокотемпературное сдвиговое деформирование полученных продуктов. Изучено влияние технологических параметров свободного СВС-сжатия на процесс получения изделий из выбранных объектов исследования. Установлено, что для исследованных материалов существует оптимальный температурно-временной интервал переработки, позволяющий получить изделия высокого качества. Проведено исследование фазового состава и микроструктуры полученных изделий. Изучены физико-механические свойства полученных изделий, проведены испытания на жаростойкость в интервале температур 1000...1300 °C в течение 10 ч.

**Ключевые слова:** свободное СВС-сжатие; самораспространяющийся высокотемпературный синтез; оксид алюминия; высокотемпературные материалы.

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## **1. Introduction**

In modern industry, there is a major problem of wear of processing tools and technological equipment. This problem is especially acute in industries associated with high-temperature production [1–3]. The problem of increasing the service life of processing tools and technological equipment can be solved by applying wear-resistant protective coatings to these parts. Among promising materials used as coatings we can note composites based on oxide ceramics [4–6]. Such materials have high hardness, wear resistance, crack resistance, corrosion resistance and chemical inertness [7–11]. Today, there are many technologies for applying coatings. At the same time, most of them require first obtaining the material that will be applied to the part, and then obtaining a product from this material – an electrode or a target for further application of coatings. To obtain such composites based on oxide ceramics, various methods are used, such as hydrothermal synthesis [12–14], sol-gel method [15, 16], carbothermal synthesis [17, 18], mechanochemical synthesis [19, 20], etc. Then, electrodes and targets used for applying protective coatings are made from the powder materials obtained by the above methods using various consolidation methods. Methods, which allow obtaining bulk products with low porosity, include hot pressing, sintering, spark plasma sintering, etc. [21–23].

A common feature of the listed consolidation methods is the need to use complex technological equipment, external heating, and the duration of the process of obtaining products [24, 25]. In addition, with such a technological chain of obtaining products, the processes of synthesis of the material and its compaction are separated in time, which can create additional difficulties. From this point of view, a promising approach is one that consists of synthesizing the material and obtaining products based on it in one technological stage. This approach is implemented in the free SHS compression method, which combines the processes of obtaining a material in the self-propagating high-temperature synthesis (SHS) mode and subsequent high-temperature shear deformation [26, 27].

SHS is characterized by high temperatures of the process, especially for synthesis with a reduction stage. Due to this, the synthesis products, including refractory and difficult-to-deform ones, are in a heated state and have the ability to deform, which was shown earlier. In the free SHS compression method, the heated synthesis products are subjected to shear plastic deformation immediately after the

combustion wave passes through the sample. In this case, special equipment is used, the lateral boundaries of which are capable of moving in the direction perpendicular to the axis of pressure application. This deformation scheme allows minimizing the wall external friction on the mold walls, which allows reducing the pressing pressure, and also provides the most favorable scheme of the stress state and plastic deformation of the material, which leads to a decrease in the number of pores and cracks in the final product.

Previously, the work was carried out on obtaining various ceramic and metal-ceramic composite materials by the free SHS compression method. For example, composites based on TiC–NiCr, MAX-phases of the Ti–Al–C system, TiB–Ti,  $\text{Al}_2\text{O}_3$ –TiB<sub>2</sub>,  $\text{Al}_2\text{O}_3$ –CrB<sub>2</sub> and other materials were obtained [28–30].

This work is aimed at studying the influence of process parameters on the process of free SHS compression of materials based on oxide ceramics, studying the properties of the obtained products, as well as expanding the range of materials obtained by the free SHS compression method.

## **2. Materials and Methods**

### **2.1. Initial materials**

In this paper, the following powders were used as initial powders to obtain materials based on oxide ceramics: titanium oxide (99.3 % < 15  $\mu\text{m}$ ), boron oxide (> 99 %, < 2  $\mu\text{m}$ ), tungsten oxide (99.97 %, < 3  $\mu\text{m}$ ), chromium oxide ( $\geq$  99.9 %, 1  $\mu\text{m}$ ), aluminum, zirconium (> 99 %, < 50  $\mu\text{m}$ ), silicon (99 %, < 250  $\mu\text{m}$ ), magnesium ( $\geq$  99 %, 250–450  $\mu\text{m}$ ), titanium (< 45  $\mu\text{m}$ , 99.1 %), chromium (99.2 %, < 150  $\mu\text{m}$ ), and boron (99.5 %, 10  $\mu\text{m}$ ). The ratio of the initial components is given in Table 1.

### **2.2. Implementation of the free SHS compression method**

The method used to obtain products from the selected objects of study in this work is free SHS compression. A distinctive feature of this method is the use of special equipment, the lateral boundaries of which are capable of moving in the direction perpendicular to the axis of pressure application. Unlike SHS extrusion, the free SHS compression method allows obtaining finished products from materials with a lower capacity for high-temperature deformation, since there is no extrusion of the material through a narrow forming channel.

**Table 1.** Characteristics of research objects

Composition	Ratio of source components, mol	Composite material
1	4TiO <sub>2</sub> –8B–4Al–Zr	Al <sub>2</sub> O <sub>3</sub> –ZrO <sub>2</sub> –TiB <sub>2</sub>
2	4B <sub>2</sub> O <sub>3</sub> –4Al–3Si–4Ti	Al <sub>2</sub> O <sub>3</sub> –SiO <sub>2</sub> –TiB <sub>2</sub>
3	2B <sub>2</sub> O <sub>3</sub> –Cr <sub>2</sub> O <sub>3</sub> –4Al–2Cr	Al <sub>2</sub> O <sub>3</sub> –Cr <sub>2</sub> O <sub>3</sub> –CrB <sub>2</sub>
4	3WO <sub>3</sub> –2Al–4Cr–3B	Al <sub>2</sub> O <sub>3</sub> –Cr <sub>2</sub> O <sub>3</sub> –WB
5	B <sub>2</sub> O <sub>3</sub> –2Al–Mg–Zr	Al <sub>2</sub> O <sub>3</sub> –MgO–ZrB <sub>2</sub>

To carry out free SHS compression, square samples with a side of 50 mm and a relative density of 0.7 were prepared. These samples were covered on all sides with asbestos cardboard to prevent heat loss through the substrate and the environment. The SHS process was initiated by a tungsten spiral 1 mm thick.

### 2.3. Analytical methods

The phase composition of the obtained materials was studied using X-ray diffraction analysis (XRD). For this purpose, a DRON-3M powder diffractometer and ARL XTRA on copper radiation were used. The microstructure of the obtained materials was studied using a LEO-1450 scanning electron microscope in combination with an INCA Energy energy-dispersive microanalyzer (EDS system). Density was measured by hydrostatic weighing. Hardness was measured using the Vickers method. To measure the ultimate bending strength, three-point bending tests were carried out on a REM-20-A-1-4 universal tensile testing machine. Crack resistance was determined by microindentation of a diamond pyramid and subsequent analysis of cracks obtained at the vertices of the indentation. Heat resistance was determined according to Russian Standard 6130-71 by measuring the specific weight gain of the tested samples at certain time intervals. To measure electrical resistance, a four-contact method was used based on measuring the voltage drop when an electric current flows in a section of the circuit.

### 3. Results and Discussion

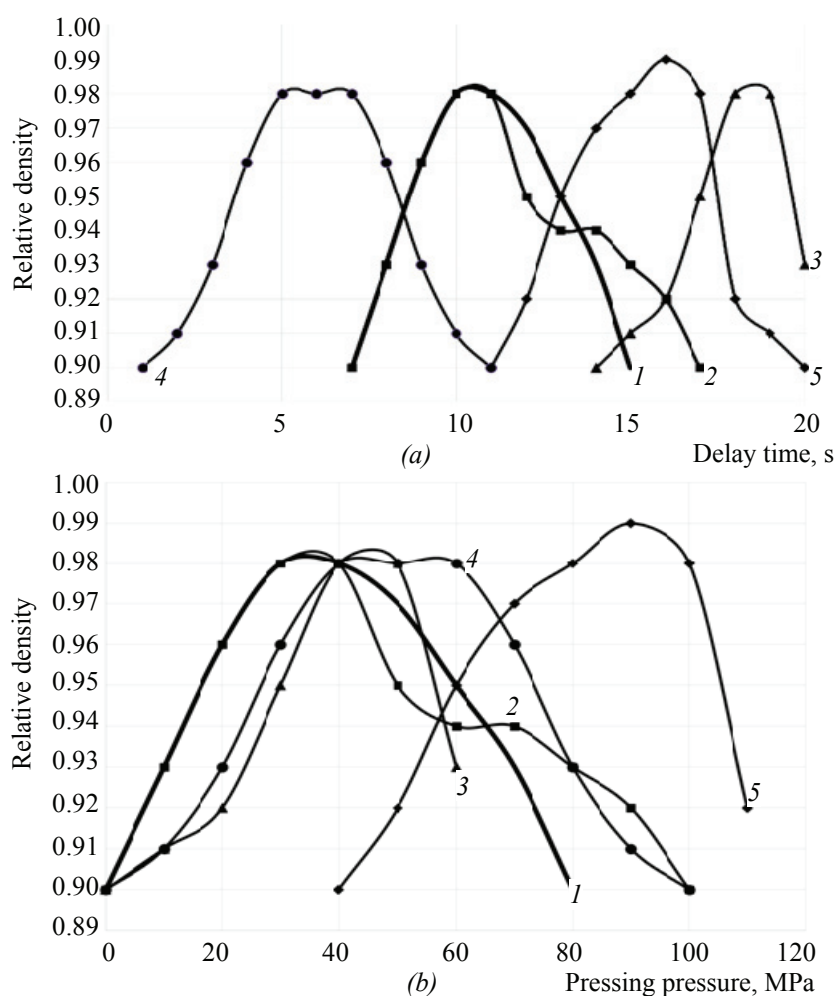
The object of the study in this paper is composite materials representing an oxide matrix reinforced with solid particles of borides and carbides. The matrix material is a composite based on aluminum oxide in combination with zirconium, silicon, chromium and magnesium oxides. The use of such a combination improves the properties of aluminum oxide. Thus, chromium oxide has unlimited solubility with aluminum oxide, which contributes to the formation of a solid solution of these compounds. In addition, chromium oxide is

a common component of refractories. Silicon oxide also has refractory properties and promotes the formation of mullite. The introduction of magnesium oxide into a system containing aluminum oxide promotes the formation of aluminum-magnesium spinel, which has a fairly high hardness and chemical inertness. The listed materials have high values of hardness, wear resistance, chemical inertness, heat resistance, and high availability. However, oxide ceramic materials have some limitations in application due to their low ability to resist thermal shock, low crack resistance of aluminum oxide, and the inability to conduct electric current. It is possible to eliminate these disadvantages by creating composite materials based on the listed oxides, reinforced with solid particles of carbides and borides, for example, TiC, TiB, TiB<sub>2</sub>, SiC, CrB, CrB<sub>2</sub>, etc. The introduction of the specified particles of borides and carbides allows both to increase heat resistance and crack resistance, and to impart electrically conductive properties to the developed materials, since carbides and borides are good conductors of electric current. In addition, the use of such compositions allows the implementation of the SHS process with a reduction stage, which provides a high thermal effect and has a positive effect on the ability of materials to high-temperature deformation.

In the course of this work, the influence of such process parameters as the delay time before applying pressure and the pressing pressure on the process of free SHS compression of the selected objects of study was studied. The delay time is defined as the time from the initiation of the SHS process to the beginning of applying external pressure. Since the SHS process includes not only the combustion stage, but also post-processes during which phase and structure formation of the material occurs, it is important to carry out the deformation of the synthesis products in the optimal temperature-time interval of processing. Since different materials have different temperatures and combustion rates, and upon completion of SHS, the cooling and crystallization processes also occur differently, the

delay time for different initial compositions can differ significantly. Since in free SHS compression the shear deformation scheme is implemented in a way that favorably affects the ability of the synthesized material to plastic flow, the optimal temperature-time interval of material processing in this case is wider than in SHS extrusion. Nevertheless, the delay time still has a significant effect on the quality of products obtained by free SHS compression. At low values of this parameter, the synthesized materials contain a large amount of liquid phase, which leads to its extrusion from the reaction zone through process gaps, which reduces the ability to compact the remaining synthesis products. When the optimal values of this parameter are exceeded, the ability of materials to high-temperature deformation gradually decreases. This ability decreases gradually, since the synthesized material cools unevenly. The inner part of the material is still deformed, while its outer part loses the ability to plastic flow. As a result, pores and cracks are formed on the surface of products. The dependence of the relative density of products on the delay time before applying pressure is shown in

Fig. 1a. When studying the effect of pressing pressure on the process of free SHS compression of the developed research objects, it was found that this dependence, shown in Fig. 1b, has a maximum. The density of the products obtained monotonically increases with increasing pressing pressure, and a maximum is observed upon reaching optimal values. Then, the density begins to decrease monotonously due to the fact that exceeding the optimal value of pressing pressure leads to mechanical damage, cracking and destruction of the resulting products. The difference in the values of pressing pressure for different compositions is due to the fact that the studied materials have different temperatures and combustion rates, as well as different amounts of liquid phase in the synthesis products, and thus have different abilities for high-temperature shear deformation. Using the obtained optimal values of the technological parameters of free SHS compression, given in Table 2, a pilot batch of electrodes was produced, the appearance of which is shown in Fig. 2.

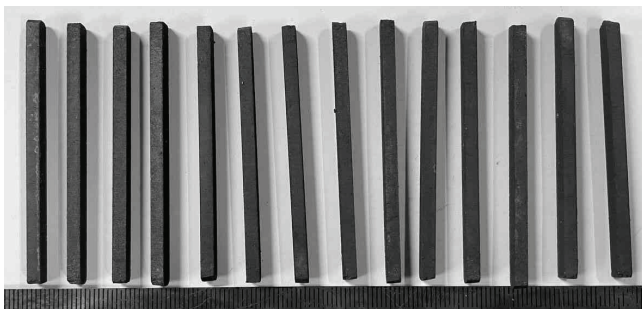


**Fig. 1.** Dependence of the relative density of products obtained by the free SHS compression method on delay time (a) and pressing pressure (b): 1 – composition 1; 2 – composition 2; 3 – composition 3; 4 – composition 4; 5 – composition 5



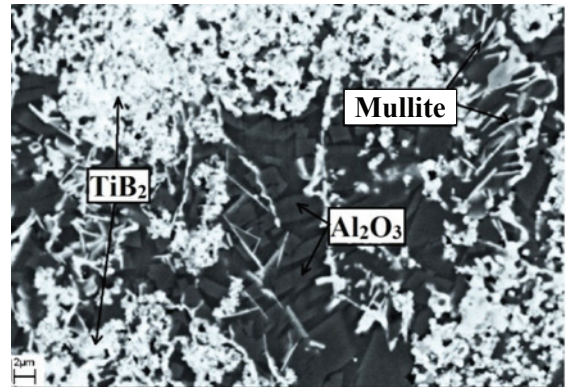
**Table 2.** Optimal values of technological parameters of free SHS compression of the developed research objects

Composition	Delay time, s	Pressing pressure, MPa
1	10–11	50
2	10–12	40
3	18–19	40
4	5–7	60
5	15–17	90



**Fig. 2.** Appearance of electrodes obtained by the free SHS compression method

The results of X-ray diffraction analysis and scanning electron microscopy showed that the obtained products had a composite structure. Thus, composition 1 was a composite material in which a mixture of aluminum and zirconium oxides acted as a matrix, where strengthening particles of titanium diboride were uniformly distributed. Composition 2 contained aluminum oxide as the main phase, which was the matrix. Strengthening particles of titanium diboride, as well as mullite, were uniformly distributed in the aluminum oxide matrix. Mullite was formed as a result of the interaction of aluminum and silicon oxides. The matrix of composition 3 was a solid solution of aluminum and chromium oxide. These oxides have unlimited solubility in each other [4, 31], which makes it possible to obtain composite materials with a matrix based on their solid solution.



**Fig. 3.** Microstructure of the obtained products based on composition 2

Chromium diboride acted as a strengthening phase in composition 3. Composition 4 had a similar structure, in which the matrix also consisted of a solid solution of aluminum and chromium oxides. However, in the case of composition 4, tungsten boride acted as a strengthening phase. Composition 5 had a matrix based on a mixture of aluminum and magnesium oxides in its structure, which partially formed spinel  $\text{MgAl}_2\text{O}_4$ . Zirconium diboride particles were present as a strengthening phase in composition 5. Figure 3 illustrates a typical microstructure of the obtained products using composition 2 as an example.

In the course of this work, physical and mechanical properties of the products obtained were studied. The results of the study are presented in Table 3.

The heat resistance of the obtained products was also studied in the temperature range of 1000–1300 °C for 10 hours. The results are shown in Fig. 4.

It was found that the obtained products have low specific weight gain values in the studied temperature range. At the same time, in [4] it was shown that for pure titanium diboride the specific weight gain for 10 hours at a temperature of 1000 and 1200 °C was 0.004 and 0.008  $\text{g}\cdot\text{cm}^{-2}$ , respectively, which is 10 and 2 times greater than for the obtained samples, respectively.

**Table 3.** Physical and mechanical properties of the obtained products

Composition	Density, $\text{g}\cdot\text{cm}^{-3}$	Hardness, GPa	Bending strength, MPa	Crack resistance, $\text{MPa}\cdot\text{m}^{1/2}$	Specific resistance, $\text{Ohm}\cdot\text{m}$
1	4.38	18	635	3.9	$2.7 \cdot 10^{-5}$
2	3.62	17	685	4.3	$2.9 \cdot 10^{-5}$
3	4.50	18	651	5.2	$1.3 \cdot 10^{-5}$
4	8.03	19	690	5.4	$3.1 \cdot 10^{-5}$
5	4.29	17	657	4.1	$2.7 \cdot 10^{-5}$

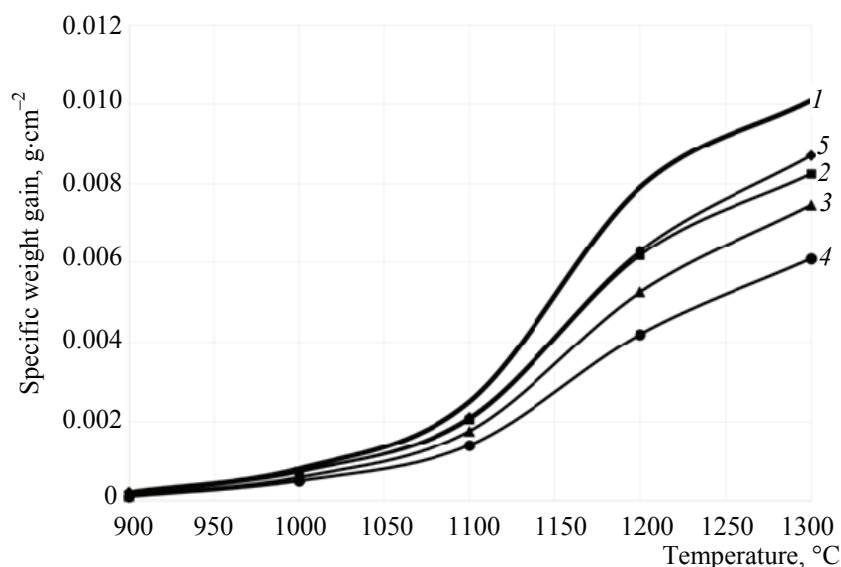


Fig. 4. Finding on heat resistance of the obtained products:

1 – composition 1; 2 – composition 2; 3 – composition 3; 4 – composition 4; 5 – composition 5

For example, for a single-phase material made of SiC obtained by hot pressing, at a temperature of 1350 °C the specific weight gain for 10 hours of annealing was 0.0004 g·cm<sup>-2</sup> [5].

Let us consider several works in which similar studies were carried out in order to determine the feasibility of the goals and objectives set in this paper. Thus, in [6] a composite material with a matrix based on aluminum oxide was obtained by impregnation followed by sintering. The authors report that the bending strength of the obtained composite material was (414.7 ± 41.6) MPa. The process of obtaining the specified material included preliminary heat treatment of the workpiece at 700 °C, sintering in a vacuum at a temperature of 1100 °C for 4 hours, and sintering in air for 1 hour also at 1100 °C. The work [7] describes composite materials based on Al<sub>2</sub>O<sub>3</sub>–Cr<sub>2</sub>O<sub>3</sub> with additives of various amounts of titanium dioxide. The hardness of the composite materials obtained in the work reached 17.2 GPa. In this case, to obtain the described composite materials, the sintering process at a temperature of 1600 °C for 5 hours was used.

The authors of the paper [8] obtained a ceramic composite material Al<sub>2</sub>O<sub>3</sub> – 10 % wt. Co. Powders of aluminum and cobalt oxide were used as the source materials. The authors report that the density and hardness of the obtained materials were 91.60 % and 14.37 GPa, respectively. The obtained samples had the following overall dimensions: 40 mm in diameter and 6 mm in height. In [9], ceramic composite materials based on Al<sub>2</sub>O<sub>3</sub>–ZrO<sub>2</sub>–SiC with a silicon carbide content of 0 to 25 vol. % were obtained by spark plasma sintering. The hardness values of the obtained materials were in the range from 14.4 to 16.1 GPa, depending on the silicon carbide content.

The study [10] describes the production of Al<sub>2</sub>O<sub>3</sub>–WC composite material by spark plasma sintering. The hardness of the obtained material reached 19 GPa. A composite material based on Al<sub>2</sub>O<sub>3</sub>, reinforced with silicon carbide particles, was obtained in [11]. The composite bending strength and hardness were 652 MPa and 19 GPa, respectively. Compact blanks from this material were produced by spark plasma sintering.

Thus, materials and products based on them obtained in the course of the work are at the level of world analogues in their properties. However, the method of obtaining products used in the work, namely free SHS compression, allows this to be done in one technological stage in tens of seconds directly from the source powders. In this case, there is no need to use complex technological equipment, long-term heat treatment at high temperatures and a large number of technological operations.

#### 4. Conclusion

The free SHS compression method was used to obtain composite materials based on oxide ceramics of the following compositions: Al<sub>2</sub>O<sub>3</sub>–ZrO<sub>2</sub>–TiB<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub>–TiB<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>–Cr<sub>2</sub>O<sub>3</sub>–CrB<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>–Cr<sub>2</sub>O<sub>3</sub>–WB, Al<sub>2</sub>O<sub>3</sub>–MgO–ZrB<sub>2</sub>. The influence of technological parameters of the production process on the quality of final products was established. It was shown that there is an optimal temperature-time interval for processing the above materials by the free SHS compression method, which ensures achieving maximum values of relative density. When studying the effect of pressing pressure on the process of obtaining products from the selected

research objects, it was shown that the dependence of the relative density of the products on pressure has a maximum. The results of studying the phase composition and microstructure of the obtained products showed that they have a composite structure. Namely, an oxide matrix with various strengthening particles distributed in it. In addition, the work studied physical and mechanical properties of the obtained products, conducted tests for heat resistance in the temperature range of 1000–1300 °C for 10 hours. It is shown that the obtained products have low indicators of specific weight gain in the studied temperature range.

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This study received no external funding.

### 6. Conflict of interest

The authors declare no conflicts of interest.

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