Boron Carbide Composites produced by Self-Propagating High-Temperature Synthesis

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Abstract.- This work is a study of the conditions for obtaining boron carbide, magnesia, and aluminum oxide composites by self-propagating high temperatures synthesis (SHS). The substances used to synthesize the composites were boron oxide, magnesia, and aluminum oxide. The SHS with the reduction stage was conducted as the following aggregate reaction: $2\,B_2O_3 + 6\,Mg + xC \longrightarrow ByCx + 6\,MgO, 2\,B_2O_3 + 4\,Al^{+}xC = 2\,Al_2O_3 + ByCx$. The metallothermic SHS using boron oxide seems more attractive than the current carbide production methods characterized by long-time and multistage physico-chemical processes requiring increased material, energy, and financial costs. The thermodynamic calculations of the phase composition of the products and the adiabatic combustion temperature for systems $B_2O_3 - Mg - C$, $B_2O_3 - Al - C$ were made in the FastStage program. The calculations and tests allowed identifying the optimal conditions of SHS. The products of SHS were examined by X-ray phase analysis and the SEM method. The compositions of the $B_2O_3 - Mg - C$ system obtained by SHS were represented mainly by boron carbide, magnesia, magnesium borate, and $Mg_3B_2O_6$. The products of the $B_2O_3 - Al - C$ system obtained by SHS contained boron carbide, alumina, and aluminum boride.

Keywords: composite; boron carbide; self-propagating high-temperature synthesis; thermal dynamic analysis.

Materiales Compuestos de Boro Producidos por Síntesis Autopropagante a Temperaturas Elevadas

Resumen.- Este trabajo es un estudio de las condiciones para la obtención de compuestos de carburo de boro, magnesia y óxido de aluminio por medio de síntesis autopropagante a temperaturas elevadas (SAPTE). Las sustancias utilizadas para sintetizar los compuestos incluyeron óxido de boro, magnesia y óxido de aluminio. Se llevó a cabo la SAPTE con la fase de reducción como la siguiente reacción agregada: $2\,B_2O_3 + 6\,Mg + xC \longrightarrow ByCx + 6\,MgO, 2\,B_2O_3 + 4\,Al + xC = 2\,Al_2O_3 + ByCx$. La SAPTE térmica metálica que utiliza óxido de boro parece más atractiva que los métodos actuales de producción de carburo que se caracterizan por procesos fisicoquímicos de larga duración y múltiples etapas que requieren mayores costos de materiales, energía y financieros. Los cálculos termodinámicos de la composición de fase de los productos y la temperatura de combustión adiabática para los sistemas $B_2O_3 - Mg - C$, $B_2O_3 - Al - C$ se realizaron en el programa FastStage. Los cálculos y pruebas permitieron identificar las condiciones óptimas de SAPTE. Los productos de SAPTE se examinaron mediante análisis de fase de rayos X y el método SEM. Las composiciones del sistema $B_2O_3 - Mg - C$ obtenido por SAPTE estuvieron representadas principalmente por carburo de boro, magnesia, borato de magnesio y $Mg_3B_2O_6$. Los productos del sistema $B_2O_3 - Al - C$ obtenido por SAPTE contenían carburo de boro, alúmina y boruro de aluminio.

Palabras clave: material compuesto; carburo de boro; síntesis autopropagante a temperaturas elevadas; análisis termodinámico.

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1. Introduction

Boron carbide is the basis of high-refractory materials with a unique combination of a lot of properties, such as high thermal conduction, high electrical resistance, hardness, chemical inertia, high thermal shock resistance, high corrosion

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resistance, and others [1, 2]. The main method of making ceramic B_4C items is hot pressing at about 2300 K [3, 4]. Another popular method of making them is the sintering in flash plasma [5]. The synthesis of amorphous boron and carbon is conducted in vacuum or inert atmosphere at temperatures above 1500 °C. The synthesis is a slow process despite the reaction's strong exothermal effect due to the difficulties with the hard-phase diffusion of the reacting particles through the layers of synthesized B_4C. The high price of pure boron used as a reactant makes the process economically unviable but for cases when the object of synthesis is highly pure B_4C is intended for experimentation purposes. The method is indispensable in synthesizing ^{10}B-enriched B_4C for nuclear engineering purposes [6].

However, despite its high hardness and wear resistance, B_4C has low strength and cracking resistance. To improve the strength of ceramics based on B_4C, various kinds of additives are used in the course of sintering that make the material more ductile and resistant to cracking [3]. For this reason, a lot of attention is currently being paid to the technology of making composites based on borides, carbides, and nitrides together with more ductile materials used as binders. Self-propagating high-temperature synthesis provides ample opportunities for using magnesium or aluminum as reducing agents in synthesizing materials by combustion. Thus the aluminum oxide or magnesia contained in synthesized composites can function as high-temperature binders and fillers [7, 8, 9]. The SHS method is used at high temperatures in the condensed phase and fully uses the chemical energy of the system but not of external sources of heat. In the course of SHS strongly exothermal chemical interactions are observed among the reactants, which are attended by the release of heat in large amounts. High thermal effect values lead to high temperatures, whereas the maximal temperature achieved during SHS is called the combustion temperature. It plays a key role in the development of SHS and the formation of the final products of combustion. This is why, a lot of attention is being paid to both, experimental temperature measurements and theoretical temperature calculation [10, 11, 12, 13, 14, 15].

Together with the high speed of the process, SHS allows achieving increased output at minimal costs. This synthesis is energy-saving, self-controlled, and ecofriendly. In addition, SHS allows adjusting the size of the particles and structure and synthesized composites. In these processes, the wasteless chemical synthesis of high-refractory boron, carbide, and other compounds occurs. These compounds are the basis of a broad range of modern nonorganic materials usable in extreme conditions [16, 17, 18, 19, 20, 21, 22].

An important role in obtaining materials by SHS is played by preliminary mechanochemical activation (MCA) that allows attaining a high level of particle dispersion, changing the structure and energy intensity of the material, and, therefore ensures its high reactivity [17, 18, 19, 20, 21, 22].

The pulping of components reduces the kinetic obstacles to chemical reactions in heterogeneous media. The MCA of SHS is characterized by high heat release levels and speed, low initial reaction temperatures, and high chemical and structural heterogeneity of synthesized products [17]. The mechanical activation of reactants before SHS is a very important stage. It can make heterogeneous processes far more intensive and increases the chemical activity of processed particles by increasing their defect rate and/or expanding their reactive surface through their reduction in size. Thus MCA prepares the particles for response activity. Mechanical activation may affect both, the speed and conditions of combustion wave front propagation (macrokinetic effect) and the shape and size of the crystallites and structural porosity (structural effect) [10].

The reason, why the inclusion of SHS and MCA in the process flow of creating new kinds of material is of interest, is that the accumulation and release of energy is observed in each of them and exothermal effects advance the synthesis. These processes allow saving energy, which is especially important in making materials with a high melting point. Their synthesis by conventional technologies, not by SHS, requires using a lot of energy and

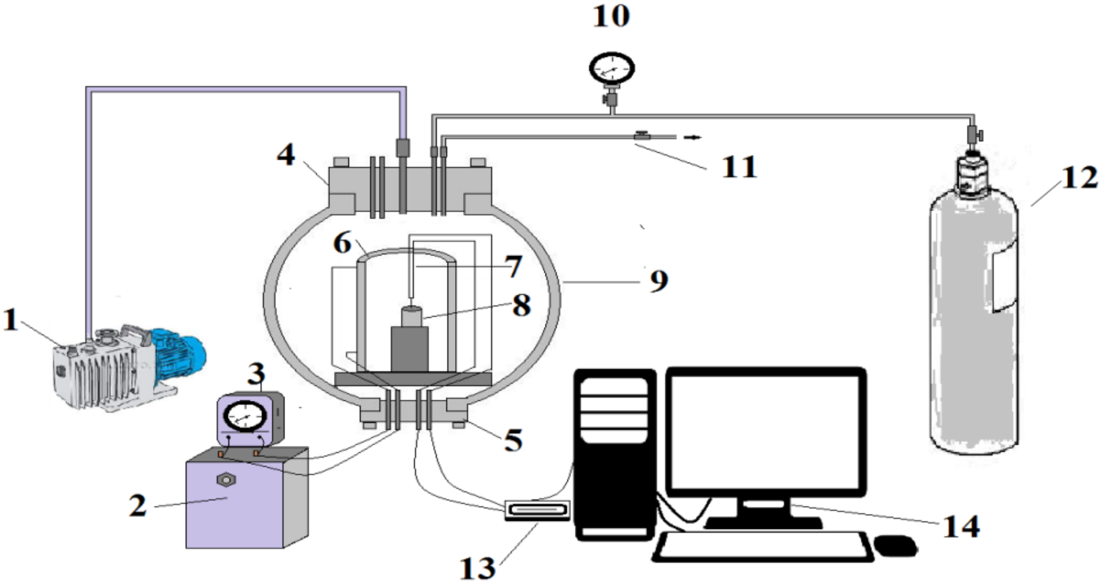


Figure 1: High-pressure reactor: 1 is the vacuum pump, 2 is the transformer, 3 is the amperemeter, 4 is the upper reactor head; 5 is the bottom reactor head; 6 is the tubular heating furnace, 7 is the thermocouple, 8 is the specimen, 9 is the reactor body, 10 is the pressure gage, 11 are the inlet and outlet gate valves, 12 is the argon bulb, 13 is the unit of the LTR-U-1 data collection system, 14 is the computer

sophisticated equipment; first of all, this bears on nitride, boride, and carbide ceramic materials [8, 10, 11].

2. Materials and methods

The specimens were made from a charged material with magnesium powder (100-200 µm particles), boron oxide (50-100 µm particles), and graphite (0,5-1,0 µm particles) with a carbon content of 60-99,9 %. The specimens were molded on a Carver laboratory extruder at an effort of five tons and their sizes were diameter $d = 20$ mm, and height $h = 25$ mm. The specimens were placed in a SNOL electrical laboratory furnace, and the synthesis was conducted at 700 °C. The combustion temperature was measured using an Ircon Ultrimax Plus UX10P pyrometric temperature gage with a working range from 600 to 3000 °C. The respective error in measuring temperatures up to 1500 °C and from 1500 to 2000 °C was $\pm 0,5$ and ± 1 % respectively.

In addition, the SHS of the specimens was conducted in an inert medium in a constant-pressure reactor. The prepared specimens were placed there for synthesizing high-refractory boron-containing composites at an argon pressure of 1 MPa. The tubular baking furnace placed in the reactor serves to expand the concentration limits of synthesis and allows heating the test specimen up to 1000 °C. The layout of the pressure of the reactor is shown in Figure 1.

After the combustion process was initiated, the specimen temperature was recorded with the help of a computer and special-purpose software that read the data in real-time mode from BP5/20 tungsten rhenium thermocouples with a brazing seam thickness of 200 µm.

The source components were mechanically activated in a high-energy planetary centrifugal mill Pulverizette 5. The back plate rotation speed was 380 rpm. The relation of the ground powder mass to the grinding balls was 1:2.

The X-ray phase analysis of the specimens was made on a DRON-4M diffractometer on exposure to cobalt Ka radiation in range $2\theta = 10° - 70°$. The morphology of the specimens was analyzed by scanning electron microscopy (SEM) on a US-made microscope FEI QUANTA 3D 200i.

3. Results and discussions

An important parameter of SHS systems is combustion temperature. It eventually affects the quality of synthesized products and must be high enough to ensure an intensive heterogeneous reaction. The adiabatic temperature of combustion should be higher than the melting point of at least one of the components [22, 23].

Thermodynamic analysis allows calculating the adiabatic combustion temperature of exothermal mixtures (T_{ad}) and the phase composition of the equilibrium products of SHS. The temperature and the equilibrium composition of SHS products are calculated with the help of software algorithms of searching for systems minimal free energy [15, 23].

The calculation of adiabatic temperatures and SHS products proper for the B_2O_3-Al-C system (where C is for graphite) was made in FactSage [15].

For the dependence of the adiabatic combustion temperature on the content of aluminum in the B_2O_3-Mg-C system at $T_0 = 700\,°C$ as is shown in Figure 1. It is seen from Figure 1 that at a magnesium content of 20-22 % wt. in the charged material the maximal SHS temperature is 1767 °C. At a magnesium content in the system of 22-40 % wt; this temperature does not change and is equal to 1764,4 °C.

The system's theoretic combustion temperature of 1767 °C allows concluding that SHS is possible in this system. The phase formation and structuring of material occur exactly at the maximal temperature attained in SHS systems. The thermodynamic calculation provides an idea of the actual combustion temperature [13, 14, 15].

For the dependence of the potential phase composition of SHS products on the content of magnesium in the B_2O_3-Mg-C at $T_0 = 700\,°C$ see Figure 2.

It follows from the diagram and the calculations in the FactStage software that the content of boron carbide in the synthesized products increases with an increase in the amount of magnesium and reaches the maximum of 18 % wt. Magnesium oxide is also the main phase in SHS products and has a melting point of 2800 °C. An increase in the content of magnesium in the source components results in the high amount of magnesium oxide in SHS products. In addition, $Mg_2B_2O_6$ is formed whereas different magnesium borate varieties may form at 800-850 °C [6]. It should be noted that small amounts of CO form at a magnesium content of 26 to 40 % wt. in the charged material; however, there is still some amount of unreacted carbon that may enter into reaction as follows:

$$B_2O_3 + 3\,CO = 2\,B + 3\,CO_2 \tag{1}$$

$$4\,B + C = B_4C \tag{2}$$

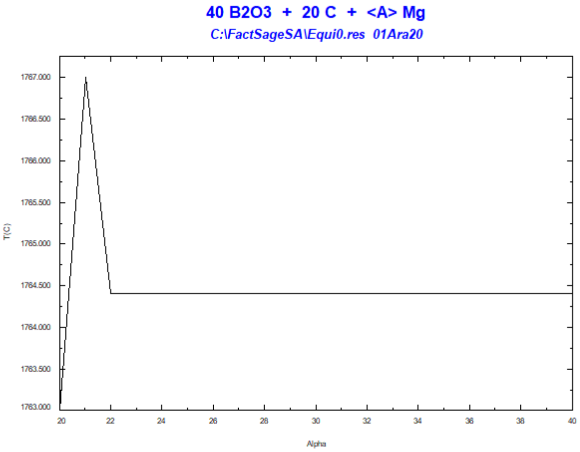


Figure 2: Dependence of the adiabatic combustion temperature on the content of magnesium in the B_2O_3-Mg-C system at $T_0 = 700\,°C$

The SHS tests were performed in the air at the initial room temperature, in the muffle roaster at $T_0 = 700\,°C$, and in argon in the pressure reactor. The room-temperature SHS in the B_2O_3-Mg-C and B_2O_3-Al-C systems did not provide any results. It is known from Merzhanov's empirical criterion that the adiabatic temperature (Tad) must be high enough to ensure a reaction in combustion mode without preheating [11].

The optimal proportion of the source components, defined by experiment, taking into account the calculated tentative equilibrium composition of the specimens and the adiabatic temperature, was 25 % of Mg to 30 % of B_2O_3 to 45 % of C. When the SHS test was conducted in the muffle roaster at

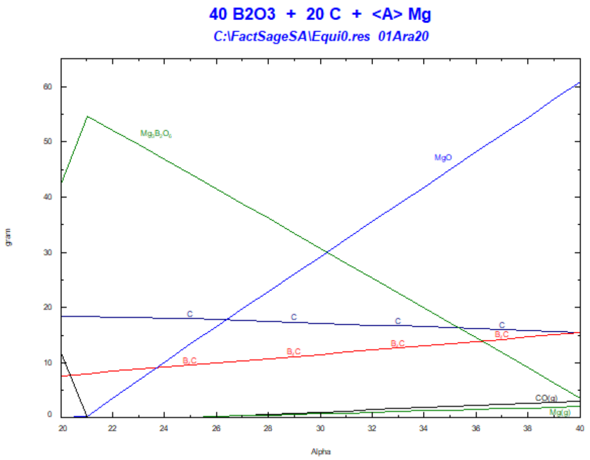


Figure 3: Dependence of the possible phase composition of SHS products on the content of magnesium in the B_2O_3-Mg-C system at T_0 = 700 °C

various initial heating temperatures, the specimens would not spontaneously ignite at $T < 700$ °C, whereas the magnesium-induced combustion made the specimens flowed, probably, because of high temperatures. The optimal temperature for starting SHS in the muffler roaster is 700 °C at which the synthesis was performed in its entirety while maintaining the initial shape of the specimens.

As seen from Figure 4 with the thermogram of the SHS of the $Mg-B_2O_3-C$ system, the maximal combustion temperature is 1600 °C.

According to thermodynamic calculations, the adiabatic temperature of combustion for $Mg-B_2O_3-C$ is 1760 °C. Actually, the combustion temperatures measured by a specific method are usually lower than the calculated temperatures. This is due to the fact that, in actual conditions, SHS specimens exhibit an intense heat and mass exchange with the environment. In addition, an SHS system often contains synthesized products with phase transitions to Tad. These transitions usually are usually attended by energy costs, which is a thermally destabilizing factor for SHS systems.

The synthesis by combustion usually begins with the melting of boron oxide at 450 °C, followed by the melting of magnesium at 650 °C, which corresponds to the phase transition plateau in the Figure. The rising temperature is related to the exothermal reactions of SHS (Figure 4). The possible stages of the reactions involved in the formation of B_4C-MgO are recorded as

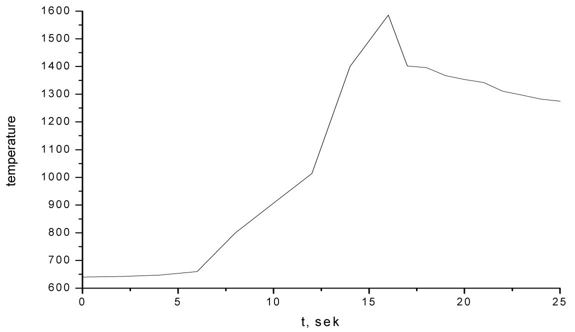


Figure 4: Thermogram of the $Mg-B_2O_3-C$ system

$$B_2O_3 + 3\,Mg = 2\,B + 3\,MgO, \tag{3}$$

$$B_2O_3 + 3\,CO = 2\,B + 3\,CO_2, \tag{4}$$

$$4\,B + C = B_4C. \tag{5}$$

It is known that the MA of charged materials before SHS in the planetary mill significantly reduces the initial temperature of the exothermal interactions among the components of the mixture, cuts the period of synthesizing the final product, and leads to a more complete extent of reaction. Mechanical activation allows accelerating chemical reactions by improving the mixture reactivity [17, 22]. To study the influence of the preliminary MA of the source components on the phase composition of SHS products, the mechanical activation was carried out for five, seven, and ten minutes. It was established that it is inexpedient to continue the MA for more than ten minutes because the extended MA results in the loss of the system's accumulated energy due to the agglomeration of the powder mixture particles.

For the percentages of the products of the SHS of the $Mg-B_2O_3-C$ system in argon in the reactor at the $Mg-B_2O_3-C$ ratio of 25 to 30 to 45 % see Table 1.

It is seen from Table 1 that the preliminary MA increases the content of boron carbide in the reaction products. The formed boron carbide,

Table 1: Phase composition of the products of the SHS system with the respective content of Mg, B_2O_3 , and C of 25, 30, and 45 % at T_0 = 700 °C

MCA duration (min)	Content of SHS products (%)			
	MgO	$Mg_3B_2O_6$	B_4C	Mg_2C_3
10	51,9	9,3	37,3	1,5
7	72	7	17,4	3,5
5	75,4	7,8	13,5	3,3
-	69,9	9,5	10,6	10

magnesium oxide, and magnesium carbide are high-temperature bondings.

To compare the conditions of synthesizing boron carbide ceramic materials by combustion, an SHS was carried out in an inert medium in a high-pressure plant (Figure 1). In the earlier study of the influence of the argon pressure on the parameters of SHS the argon pressure in the reactor was varied from 0,5 to 3 MPa. The tests showed that the optimal pressure for SHS was 1 MPa [24, 25]. It is obvious that a rise in the reactor pressure increases the heat losses during SHS and reduces the temperature in the system, which affects the final product yield.

For the results of the XRF of the products of the SHS of the $Mg-B_2O_3-C$ system in argon in the reactor at a pressure of 1 MPa see Table 2. The furnace was heated up to 850 °C. The components ratio in the system was 25 to 30 to 45 %.

Table 2: Phase composition of the products of the SHS system with the respective content of Mg, B_2O_3 , and C of 25, 30, and 45 %

MCA duration (min)	Content of SHS products (%)				
	B_4C	$Mg_3B_2O_6$	MgO	B_2O_3	C
10	57,2	26,2	16,6	-	-
7	41,1	24,7	34,2	-	
5	38,8	37,5	19,6	3,4	0,7
-	29,7	39,1	26,1	4,2	0,9

It follows from Table 2 that the synthesis in the inert medium encourages an increase in the output of boron carbide in SHS products. That said, preliminary MA increases the boron carbide content in the reaction products. The optimal duration of preliminary MA is 10 minutes. As a result of the preliminary MA of the charged material, the source boron oxide and graphite in the reaction products remain unreacted, which is due to an expansion of the unit surface area and an increase in the reactivity of the mixtures.

In addition, the conditions for the SHS of the composites in the B_2O_3-Al-C system were studied. The thermodynamic calculations of the products of the SHS were made for this system.

For the dependence of the adiabatic combustion system on the content of Al in the B_2O_3-Al-C at T_0 = 700 °C see Figure 5.

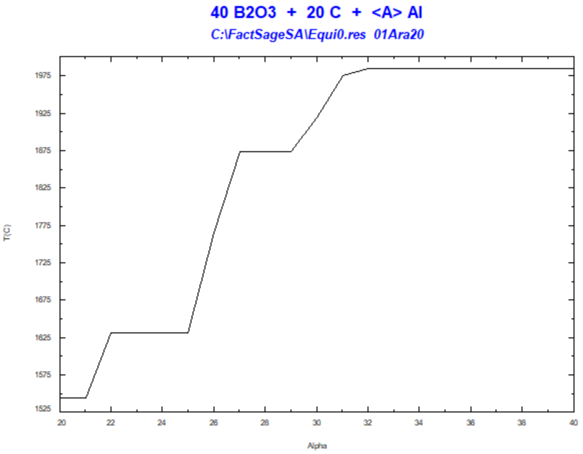


Figure 5: Dependence of the adiabatic combustion system on the content of Al in the B_2O_3-Al-C at T_0 = 700 °C

It is seen from Figure 5 that the maximal SHS temperature at an aluminum content of 32 to 40 % wt. in the charged material is 1980 °C. The adiabatic temperature growth curve exhibits plateaus at the content of aluminum from 22-25 to 27-29 % wt., which can be explained by phase transitions during SHS. The dependence of the phase composition of SHS products on the content of Al in the B_2O_3-Al-C at T_0 = 700 °C is shown in Figure 6.

According to the diagram in Figure 6 and the data of the calculations in FactStage, the boron carbide content in the synthesized products increases with an increase in the content of aluminum and reaches 17 % wt. Aluminum carbide forms at an Al content of more than 31 % wt. in the charged material. An increase in the content of aluminum in the source charged material increases the amount of aluminum oxide in SHS products. It should be noted that, if the Al content in the charged material ranges from

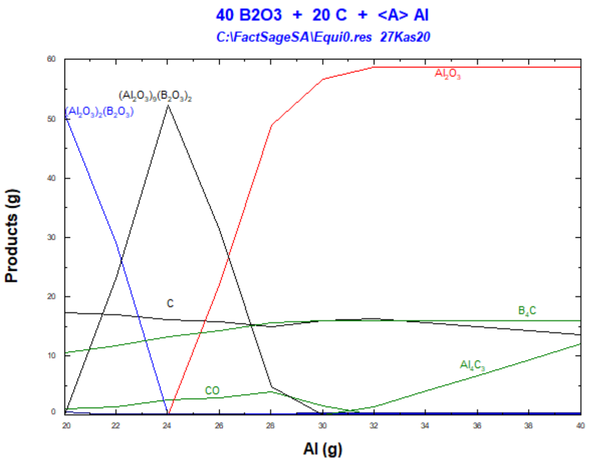


Figure 6: Dependence of the phase composition of SHS products on the content of Al in the B_2O_3-Al-C

20 to 31 % wt., the products of SHS may appear to contain small amounts of carbon dioxide.

It is established that the optimal composition of the source components includes 30 % of B_2O_3, 30 % of Al, and 40 % of C. The optimal fraction of Al in the charge material is 30 % wt. because more than 35 % of Al in the reaction products remain unreacted and the specimen melts away and does not hold shape. According to the thermodynamic calculation data, the highest boron carbide content is also assumed at a boron carbide content of 30 % wt. The initial baking furnace temperature at which the SHS preserves the specimen shape is 800 °C, which is higher than for the B_2O_3-Mg-C with a more active reducing agent.

Table 3: Phase composition of the SHS products of the B_2O_3-Al-C system at T_0 = 800 °C

MCA duration (min)	Content of SHS products (%)				
	Al_2O_3	B_4C	Al	$AlBO_5$	AlB_{10}
5	72,6	8,7	-	13,4	8,7
7	84,1	10,3	-	6,6	7,4
10	80,6	13,1	-	5,6	6,3
-	73,7	5,6	2,5	6,2	15,5

For the phase composition of the products of the SHS of B_2O_3-Al-C in the muffle roaster at initial temperature T_0 = 800 °C see Table 3. In the course of the SHS, aluminum reduces boron oxide, which results in the formation of boron carbide, alumina, and aluminum boride. In this case, the content of boron carbide in combustion products increases from using preliminary MA.

For the information on the products of the SHS performed for the Al + B_2O_3 + C system in argon in the reactor at a pressure of 1 MPa see Table 4. During the SHS the baking roaster heated to 850 °C.

Table 4: Combustion products of the B_2O_3-Al-C

MCA duration (min)	Content of SHS products (%)			
	B_4C	Al_2O_3	AlB_{10}	C
10	45,4	42,5	12,1	-
5	44,1	38,7	15,6	0,6
-	37,2	50,4	11,2	1,2

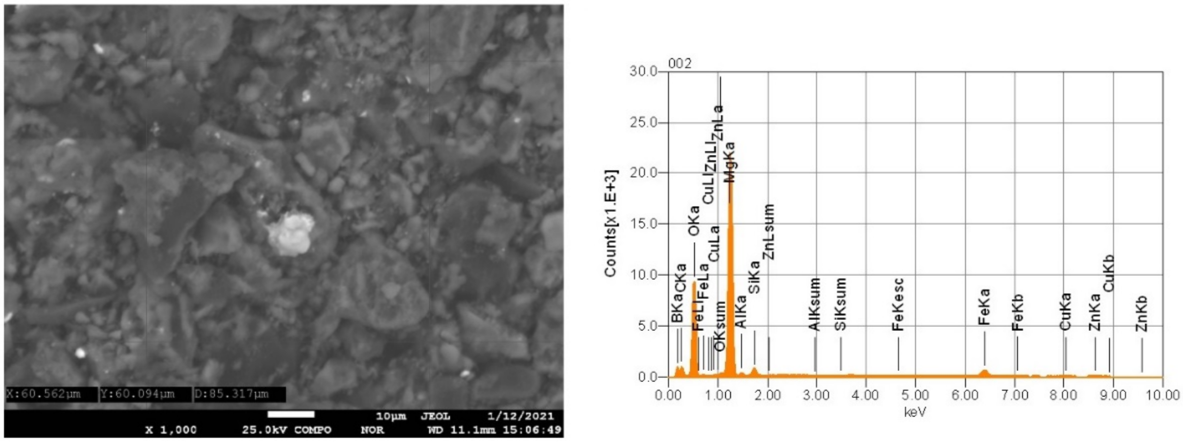
The results from Table 4 allow concluding that there was an increase in the boron content in the combustion synthesis products. The preliminary MA of the charged material significantly increases the boron carbide output.

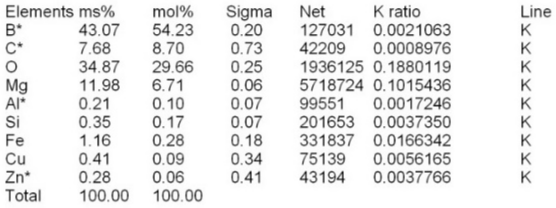
The formation of the phase and chemical composition as well as of the crystalline structure and microstructure of SHS products are complex interrelated processes. It is very important to study them during SHS for the properties of a material are determined by its structure. The electron microscopic study of the morphology of the SHS products was conducted in an inert medium for systems $Mg-B_2O_3-C$ (Figure 7) and B_2O_3-Al-C (Figure 8).

The morphology of the combustion products is represented by crystalline structures with an average size of 1-10 µm. The contrast among the parts of the image indicates the presence of at least three compounds. The high content of boron and carbon showed by the microanalysis confirms the results of the X-ray phase analysis of the SHS products.

4. Conclusion

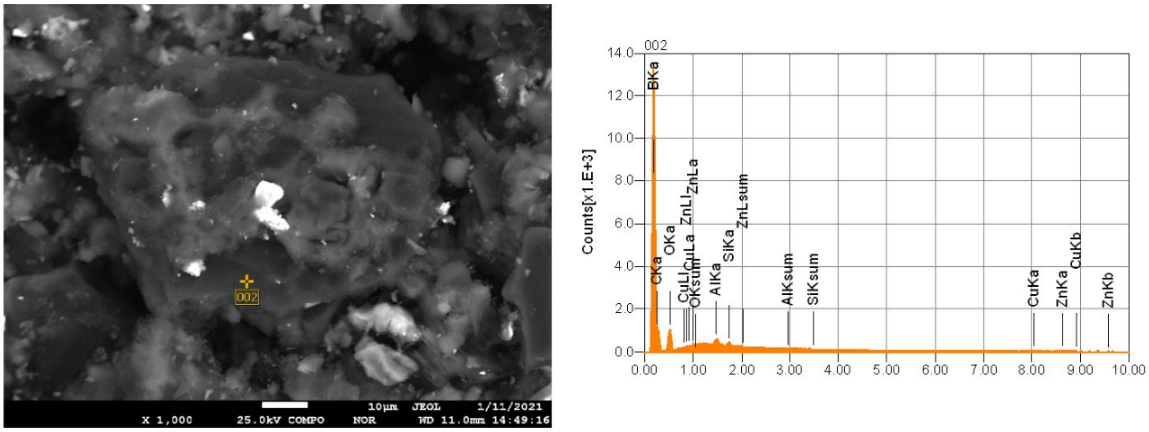
The thermodynamic calculation of the supposed phase composition of SHS products and the adiabatic combustion temperature of the B_2O_3-Mg-C and B_2O_3-Al-C systems have been made. The tests have helped determine the optimal conditions for SHS. The influence of the initial temperature on the complete course of SHS has been determined. It has been established that the initial room

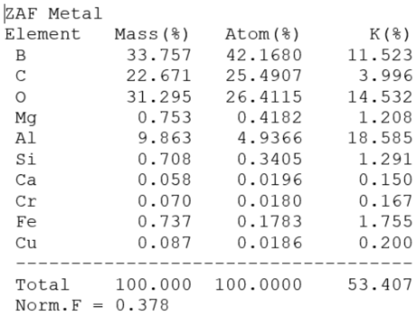




Elements	ms%	mol%	Sigma	Net	K ratio	Line
B*	43.07	54.23	0.20	127031	0.0021063	K
C*	7.68	8.70	0.73	42209	0.0008976	K
O	34.87	29.66	0.25	1936125	0.1880119	K
Mg	11.98	6.71	0.06	5718724	0.1015436	K
Al*	0.21	0.10	0.07	99551	0.0017246	K
Si	0.35	0.17	0.07	201653	0.0037350	K
Fe	1.16	0.28	0.18	331837	0.0166342	K
Cu	0.41	0.09	0.34	75139	0.0056165	K
Zn*	0.28	0.06	0.41	43194	0.0037766	K
Total	100.00	100.00				

Figure 7: SEM photos of the SHS products of the $Mg-B_2O_3-C$ system





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ZAF Metal
Element    Mass(%)   Atom(%)       K(%)
 B          33.757   42.1680     11.523
 C          22.671   25.4907      3.996
 O          31.295   26.4115     14.532
 Mg          0.753    0.4182      1.208
 Al          9.863    4.9366     18.585
 Si          0.708    0.3405      1.291
 Ca          0.058    0.0196      0.150
 Cr          0.070    0.0180      0.167
 Fe          0.737    0.1783      1.755
 Cu          0.087    0.0186      0.200
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 Total     100.000  100.0000     53.407
 Norm.F = 0.378
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Figure 8: SEM photos of the SHS products of the B_2O_3-Al-C

temperature makes the synthesis by combustion impossible. When placed to the muffle roaster at

$T = 700$ and 800 °C, the B_2O_3-Mg-C and the B_2O_3-Al-C systems self-ignite and retain their initial form after the advancing of the combustion wave. In the case of SHS, the respective specimens also retain their form in the inert medium at a reactor argon pressure of 1 MPa.

In addition, the optimal initial proportions of the SHS products in the systems have been determined: 25 % of Mg to 30 % of B_2O_3 to 45 % of C and 30 % of B_2O_3 to 30 % of Al to 40 % of C, respectively. The influence of the duration of preliminary mechanical activation (MA) of the reactive mixture on the phase composition of ceramic composites has been studied. It has been shown that this activation increases the boron carbide content in the combustion products increases.

According to the X-ray phase analysis of the B_2O_3-Mg-C system, the SHS products contain mostly the phases of boron carbide and MgO and unreduced B_2O_3 reacts with MgO to form $Mg_3B_2O_6$. The composition of the SHS in the B_2O_3-Al-C system is represented by boron carbide, alumina, and aluminum boride. The morphology of carbide impurities in the sintered composites is shown.

Thus it is proven that boron carbide composites with high-refractory compounds, such as alumina and magnesia, can be made by SHS in the muffle roaster at $T = 700$ and 800 °C and in the reactor argon at $P = 1$ MPa. It has been established that to ensure the maximal content of boron carbide in SHS products, it is more preferable to make them with magnesium as a reducing agent, not with aluminum. It has also been established that the preliminary MCA of the source components and the implementation of SHS in the reactor at an argon pressure of 1 MPa allow achieving the highest boron carbide output in the SHS products.

5. References

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