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SHS of cast materials in the Mo–Al–C system

V. A. Gorshkov✉**, P. A. Miloserdov, D. Yu. Kovalev,**
O. D. Boyarchenko

Merzhanov Institute of Structural Macrokinetics and Materials Science of the Russian Academy of Sciences
8 Akademican Osip'yan Str., Chernogolovka, Moscow region 142432, Russian Federation

✉ gorsh@ism.ac.ru

Abstract. Materials based on molybdenum-aluminium-carbon compounds have a considerable potential for use under intense wear conditions at elevated temperatures. This paper presents the experimental results of self-propagating high-temperature synthesis of compounds within the Mo–Al–C system. By combining two processes: SHS of the elements and SHS-metallurgy, cast materials containing the Mo_3Al_2C, Mo_2C, Mo_3Al, and Mo_3Al_8 phases were obtained. The experiments used mixtures with compositions calculated according to the ratio $(1 - \alpha)(3MoO_3–8Al–C)/\alpha(3Mo–2Al–C)$, where α varied in the range from 0 to 1. The synthesis was carried out in a laboratory reactor of 3 L volume at an initial argon pressure of 5 MPa. The mass of the initial mixtures in all experiments was 20 g. The process of combustion was initiated by a 0.5 mm diameter molybdenum wire spiral by applying 28 V voltage to it. The resulting end products were studied by *X*-ray diffraction and local microstructural analysis. A significant influence of the ratio of the initial reagents on the synthesis parameters, phase composition, and microstructure of the target products was established. Introduction into the high-exothermic mixture $3MoO_3$–8Al–C inert “cold” mixture 3Mo–2Al–C leads to an increase in the content of carbide phases in the ingots. The possibility of obtaining cast materials based on the triple phase Mo_3Al_2C, the maximum content of which is 87 wt. % at the content of the “cold” mixture in the charge $\alpha = 0.4$ is shown. The presence of secondary phases of molybdenum carbide (Mo_2C) and molybdenum aluminides (Mo_3Al_8, Mo_3Al) in the final products is due to a change in the composition of the initial mixture caused by the ejection of components during combustion and insufficient existence time of the melt formed in the combustion wave.

Keywords: self-propagating high-temperature synthesis, Mo–Al–C system, microstructure, phase composition

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СВС литых материалов в системе Mo–Al–C

В. А. Горшков✉**, П. А. Милосердов, Д. Ю. Ковалев,**
О. Д. Боярченко

Институт структурной макрокинетики и проблем материаловедения им. А.Г. Мержанова РАН
Россия, 142432, Московская обл., г. Черноголовка, ул. Академика Осипьяна, 8

✉ gorsh@ism.ac.ru

Аннотация. Материалы на основе соединений молибдена с алюминием и углеродом обладают большим потенциалом для использования в условиях интенсивного износа при повышенных температурах. В настоящей работе представлены экспериментальные результаты самораспространяющегося высокотемпературного синтеза (СВС) соединений в системе Mo–Al–C. При совмещении двух процессов – СВС из элементов и СВС-металлургии – получены литые материалы, содержащие фазы Mo_3Al_2C, Mo_2C, Mo_3Al и Mo_3Al_8. В экспериментах использовали смеси с составами, рассчитанными согласно соотношению $(1 - \alpha)(3MoO_3–8Al–C)/\alpha(3Mo–2Al–C)$, где значение α меняли в интервале от 0 до 1. Синтез проводили в лабораторном реакторе объемом 3 л при начальном давлении аргона 5 МПа. Масса исходных смесей во всех экспериментах составляла 20 г. Процесс горения инициировали спиралью из молибденовой проволоки диаметром 0,5 мм путем подачи на нее напряжения 28 В. Полученные конечные продукты исследованы методами рентгенофазового и локального микроструктурного анализов. Установлено существенное влияние соотношения исходных реагентов на параметры синтеза, фазовый состав и микроструктуру целевых продуктов. Введение в высокоэкзотермическую смесь $3MoO_3$–8Al–C инертной «холодной» смеси 3Mo–2Al–C приводит к увеличению содержания карбидных фаз в слитках. Показана возможность получения литых материалов на основе тройной фазы Mo_3Al_2C, максимальное содержание которой составляет 87 мас. %, при количестве «холодной» смеси в шихте, соответствующей $\alpha = 0,4$. Присутствие в конечных продуктах вторичных фаз карбида молибдена (Mo_2C) и алюминидов молибдена (Mo_3Al_8, Mo_3Al) обусловлено изменением состава исходной смеси вследствие выброса компонентов при горении и недостаточным временем существования расплава, формирующегося в волне горения.

Ключевые слова: самораспространяющийся высокотемпературный синтез, система Mo–Al–C, микроструктура, фазовый состав

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Introduction

There are several binary compounds in Mo–Al system – $MoAl_3$, Mo_3Al_8, MoAl, and Mo_3Al [1]. These compounds, given their high melting points and mechanical properties, are promising materials for use under high temperature and intense wear conditions [2–4]. In Al–C system, the aluminum carbide phase Al_4C_3 is stable. Al_4C_3 particles finely dispersed in the aluminum matrix reduce the creep tendency of the material, especially in combination with silicon carbide particles. Aluminum carbide can be used as an abrasive material in high-speed cutting tools [5; 6]. In the Mo–C system, molybdenum and carbon form carbides Mo_2C and MoC_{1-x}. Among these, molybdenum carbide Mo_2C has the most widespread use in practice. It is used as a catalyst in hydrodesulfurization reactions, in dry reforming of methane, for the decomposition of hydrazine, and in regulators of small rocket engines [7–9]. Various methods are used for the synthesis of molybdenum carbide: carbothermal reduction of molybdenum (VI) oxide with graphite in an inert medium, electrochemical synthesis, melting with graphite, reduction of oxide using a methane–hydrogen mixture or other carbon sources [10–12].

Among the ternary molybdenum aluminum carbon compounds, the Mo_3Al_2C compound, a superconductor with a transition temperature of ~9 K, is of greatest interest. In the studies [13–16], this compound was obtained by the arc and high-frequency melting at high pressures (up to 10 GPa). Its transport, magnetic, and thermodynamic properties were investigated. The bulk modulus of elasticity is estimated at 221 GPa. Known methods for obtaining Mo_3Al_2C are inefficient and energy-intensive. A one-stage method – self-propagating high-temperature synthesis (SHS) – is a promising method for obtaining such compounds. It requires practically no electricity, exhibits high capacity and is environmentally friendly [17; 18].

The SHS method allowed to obtain a large number of binary and ternary element compounds [19–22]. One of the technological trends of application of self-propagating high-temperature synthesis is SHS-metallurgy, which allows to obtain “cast” materials by the complete melting of components in a combustion wave. A specific feature of the process is the use of mixtures consisting of metal oxides, a reducing metal (Al), and carbon. At a certain ratio of reagents, the combustion temperature exceeds the melting point of the initial reagents and final products. As a result, the product during synthesis is formed in the liquid state. Under the action of gravity, the heavy metal-like and light oxide phases of the formed products are separated. Cast materials based on binary and ternary compounds (MAX phases) in the systems: Cr–Al–C [23–25], Nb–Al–C [26; 27], and V–Al–C [28] obtained by SHS-metallurgy are of great interest. The mentioned studies demonstrate that the main synthesis parameter that determines the composition of the final products is the existence time of the melt, which depends on the combustion temperature of the initial mixture. To date, the authors of this paper have not identified any studies on the production of cast materials in the Mo–Al–C system by the SHS method.

The purpose of this paper was to study the possibility of obtaining cast products within the Mo–Al–C system by combining the methods of SHS from elements and SHS-metallurgy.

Materials and methods

Powders of MoO_3 (“Ch”, purity 99.9 %), Al (ASD-1, purity 99.2 %, particle size $d < 30$ μm), Mo (PM-M, 99.9 %, $d < 10$ μm), and graphite (PG, 99.2 %, $d < 400$ μm) were used as initial reagents. In the experiments, stoichiometric mixtures were used as base mixtures; their composition was calculated from a combination of two chemical reactions:

$$3MoO_3 + 8Al + C = Mo_3Al_2C + 3Al_2O_3, \qquad (I)$$

$$3Mo + 2Al + C = Mo_3Al_2C. \qquad (II)$$

The mass ratio of the mixtures $\alpha = \frac{M_{II}}{M_I}$ varied in the range from 0 to 1, where M_I and M_{II} are the masses of the mixtures calculated from reactions (I) and (II), respectively.

The experiments revealed that the combustion of the mixture, the composition of which was calculated according to reaction (I), is characterized by a high rate and a strong ejection of reagents and synthesis products from the reaction vessel. The mixture, the composition of which was calculated according to reaction (II), does not burn and, when added to the first mixture, behaves as a "reactive" inert component. The ratios of the initial compounds in reactions (I) and (II) remained constant in all experiments.

Before mixing, the powders were dried for 3 h at a temperature of 60 °C. The charge was prepared by manually stirring it in a porcelain mortar. The bulk density reaction mixture was placed in a 20 mm in diameter and 50 mm high quartz mold (Fig. 1, *a*). The weight of the initial mixtures in all experiments was 20 g. The syntheses were carried out in a 3 L reactor (Fig. 1, *b*) in an argon atmosphere at an initial pressure of 5 MPa according to the procedure described in work [23]. The reaction was initiated with a 0.5 mm diameter molybdenum wire coil. The combustion process was recorded using a video recorder. The combustion rate was calculated from the process video by measuring the time of passage of the combustion wave along the height of the sample. The parameters of synthesis were determined from the following values:

$\eta_1 = \frac{M_{ing}}{M_0}$ – the ratio of the product yield in the ingot and the weight of the mixture;

$\eta_2 = \frac{M_{ing}}{M_{ing}^{calc}}$ – the completeness of the reaction, the ration of the product yield in the ingot relative and its calculated value;

$\eta_3 = \frac{M_0 - M_k}{M_0}$ – mass loss during combustion due to the ejection of components from the reaction vessel,

where M_{ing} – the weight of the target product (ingot), M_{ing}^{calc} – the calculated weight of the ingot, M_0 – the mass of the initial mixture, M_k – the total mass of products after burning.

The end products of the synthesis were studied using *X*-ray diffraction (XRD) and microstructural analysis. The methods for studying the obtained products are described in more detail in the authors' previous works [25].

Results and Discussion

The samples after synthesis consist of two ingots that are easily separated mechanically. The material in the upper part of the sample, according to the XRD results, is mainly an α-Al_2O_3 phase. At the bottom, an ingot (target product) with a characteristic metallic sheen is formed (Fig. 1, *c*). The formation of an ingot

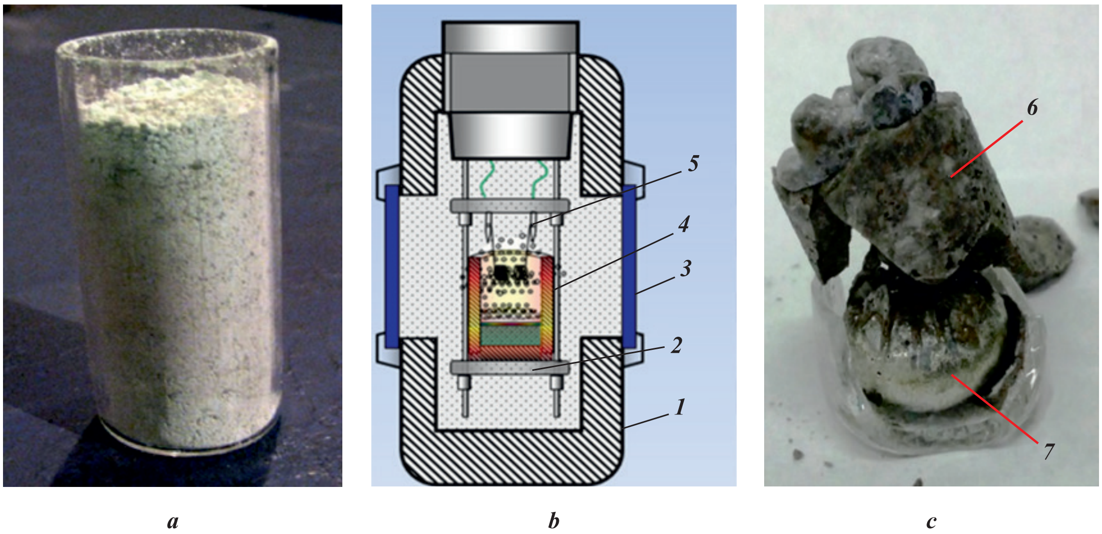

Fig. 1. Quartz crucible with a charge (***a***), reactor diagram (***b***), and final products (***c***)
1 – body, ***2*** – substrate, ***3*** – inspection window, ***4*** – charge, ***5*** – initiating coil, ***6*** – oxide layer, ***7*** – "metal" ingot

Рис. 1. Кварцевый тигель с шихтой (***a***), схема реактора (***b***) и конечные продукты (***c***)
1 – корпус, ***2*** – подложка, ***3*** – смотровое окно, ***4*** – шихта, ***5*** – инициирующая спираль, ***6*** – оксидный слой, ***7*** – «металлический» слиток

indicates that in the combustion wave a liquid phase was formed, i.e. the synthesis temperature is higher than the melting temperature of the initial components and the resulting products. Different specific masses of the final products lead to their separation due to gravity – the heavy "metal" phase settles at the bottom of the crucible, whereas the light oxide phase forms at the top.

The values of the combustion rate of the initial mixtures and the pressure increase in the reactor depending on the value of α are shown in Fig. 2, *a*. As α increases, these parameters decrease. When a "cold" mixture, the composition of which is determined from reaction (II), is added to a highly exothermic mixture, the composition of which is determined from reaction (I), part of the heat released as a result of a reaction (I) is spent on its melting, which leads to a decrease in the combustion rate and pressure increase in the reactor. Figure 2, *b* shows the dependences of the reaction completeness (η_2), product yield (η_1), and spread of combustion products (η_3) on α. The parameters η_1 and η_2 increase in the range of α from 0 to 0.4, then there is a gradual decrease. The values of η_3 decrease monotonically over the entire range of α.

The maximum product yield ($\eta_1 = 67$ %, $\eta_2 = 93$ %) is observed at α = 0.4 (Fig. 2, *b*). Yield is affected by two competing factors: reagent spread and combustion temperature. An increase in the amount of the "cold" mixture, on the one hand, reduces the ejection of reagents from the vessel and, accordingly, increases η_1, and, on the other hand, it reduces the combustion temperature, which leads to a decrease in η_2 due to a reduction in the time spent by the product in the liquid state, where oxide and "metal" phases spatially separate. *X*-ray phase analysis of the products (Figs. 3–5) obtained by combustion of mixtures with different α showed that as a result of synthesis, a multiphase material is formed, which includes Mo_3Al_2C, Mo_2C, Mo_3Al, and Mo_3Al_8. The quantitative ratio of the phases depends on the composition of the initial charge (see Table).

The combustion of mixtures of compositions *1* and *2* is non-stationary with a non-linear front and a considerable ejection of material from the crucible. *X*-ray phase analysis of the obtained products (see Fig. 3) revealed that as a result of synthesis, materials with a high content of molybdenum aluminides (Mo_3Al and Mo_3Al_8), more than 65 %, are formed, while the total content of carbide phases (Mo_3Al_2C and Mo_2) does not exceed 35 %. The low content of the latter is apparently associated with a deficit of carbon due to its ejection from the crucible in the form of particles or gaseous oxides. In the combustion wave, the mixture of initial reagents undergoes a number of physical and chemical

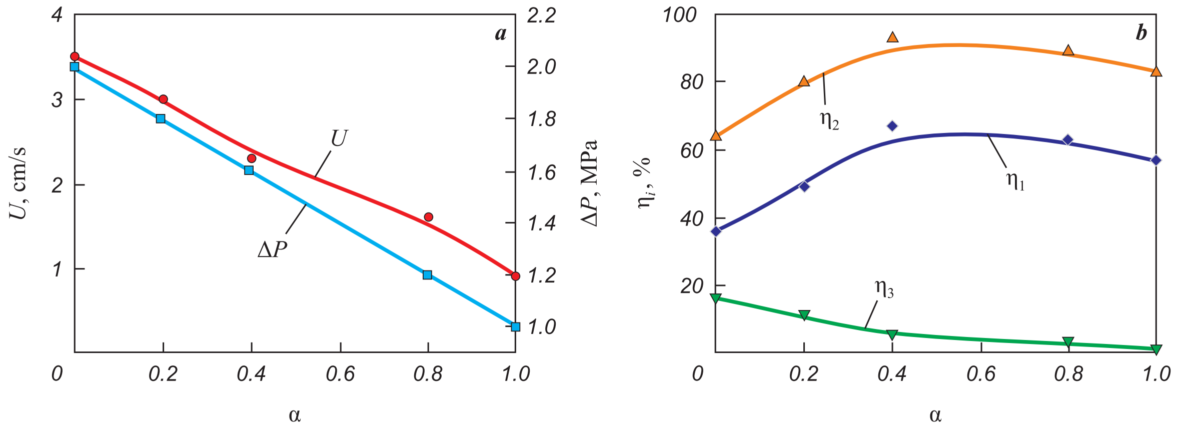

Fig. 2. Influence of α on the combustion rate and pressure increase in the reactor (***a***) and on the synthesis parameters (***b***)

Рис. 2. Влияние показателя α на скорость горения и прирост давления в реакторе (***a***), а также на параметры синтеза (***b***)

Phase composition of final products (wt. %)

Фазовый состав конечных продуктов (мас. %)

Composition No.	The proportion of mixture II, wt. %	α	Mo_3Al_2C ($P4_132$)	Mo_3Al ($Pm3n$)	Mo_3Al_8 ($C2/m$)	Mo_2C ($Pbnc$)
1	0	0	24.3	57.3	9.3	9.1
2	16.7	0.2	32.0	39.5	6.1	22.0
3	28.6	0.4	87.0	5.1	2.3	5.4
4	44.4	0.8	69.0	0.9	13.6	16.9
5	50.0	1.0	77.7	1.9	3.1	19.0

transformations. In the heating zone, aluminum and molybdenum oxide melt, forming a liquid-phase medium with distributed carbon particles. In the zone of chemical transformation, aluminum and carbon interact with molybdenum oxide:

$$MoO_3 + 2Al \rightarrow Mo + Al_2O_3, \qquad \text{(III)}$$

$$MoO_3 + xC \rightarrow MoC_x + CO(CO_2)\uparrow. \qquad \text{(IV)}$$

As a result, part of the carbon escapes from the reaction zone in the form of a gas, causing its deficiency in the system. The higher the combustion temperature of the mixture, the greater the likelihood of carbon participation in the redox reaction (IV). The material obtained as a result of the combustion of mixture *1* contains phases of Mo_3Al–Mo_3Al_8 intermetallic compounds forming an eutectoid, as well as Mo_3Al_2C and Mo_2C (see Fig. 3).

The introduction of a "cold" mixture into the charge leads to an increase of the content of carbide phases in the product while the proportion of molybdenum aluminides decreases (see table). The maximum content (87 %) of the ternary compound Mo_3Al_2C in the material was obtained at $\alpha = 0.4$ (see Fig. 4).

The phase composition of the combustion products of mixture *3* is practically equilibrium and is in the three-phase region Mo_3Al_2C–Mo_2C–Mo_3Al. An increase

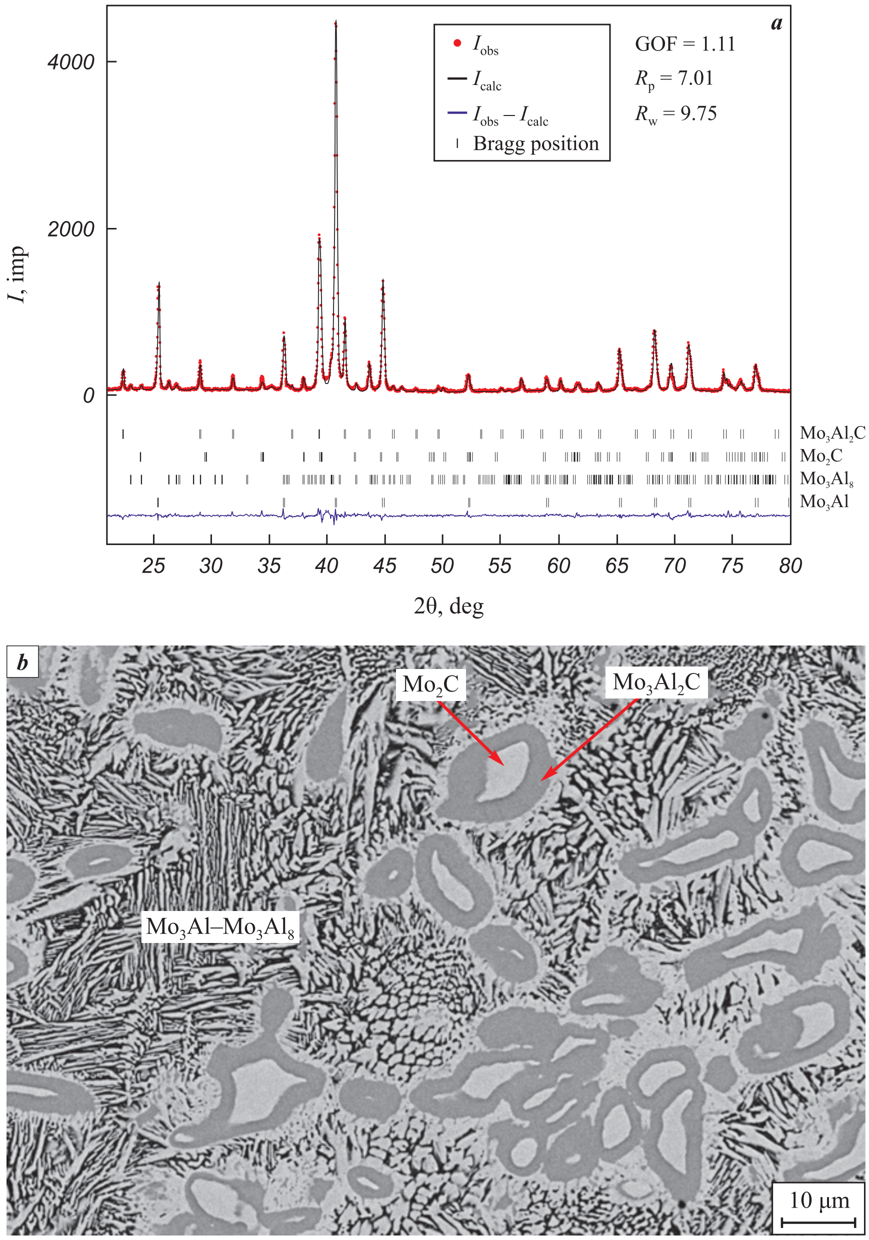

Fig. 3. Diffraction pattern (***a***) and microstructure (***b***) of the combustion product of mixture *1*

Рис. 3. Дифрактограмма (***a***) и микроструктура (***b***) продукта горения смеси *1* of the "cold" mixture content in the charge over the variation range $\alpha = 0.8 \div 1.0$ leads to an increase in the content of Mo_2C in the product while reducing the content of Mo_3Al_2C and molybdenum aluminides (see Table and Fig. 5).

The studies show that when a "cold" mixture is added to the charge, the combustion rate, pressure increase, and mass loss (η_3) decrease over the entire range of α (from 0 to 1.0). At the same time, in the range of $\alpha = 0 \div 0.4$, there is an increase in the target product yield into an ingot, whereas at $\alpha > 0.4$, there is a decrease. The target product yield into the ingot is affected by two competing factors: the content in the charge of the mixture (II), consisting of the target elements (Mo, Al, C) passing into the ingot as compounds, and the combustion temperature. With an increase in the amount of the "cold" mixture, on the one hand, the proportion of elements of the target phase in the charge increases and, accordingly, η_1 and η_2 increase, and, on the other hand, the combustion temperature decreases, which leads to a decrease in η_1 and η_2 due to a decrease in the residence time of the product in a liquid state, when there is a spatial separation of the oxide and "metal" phases. The influence of the first factor prevails at $\alpha = 0 \div 0.4$, whereas at $\alpha > 0.4$, prevails the influence of the second factor.

A single-phase product containing only Mo_3Al_2C in accordance with reactions (I) and (II) was not obtained due to several reasons. Obviously, the above scheme of reactions, used for the calculation of the equilibrium

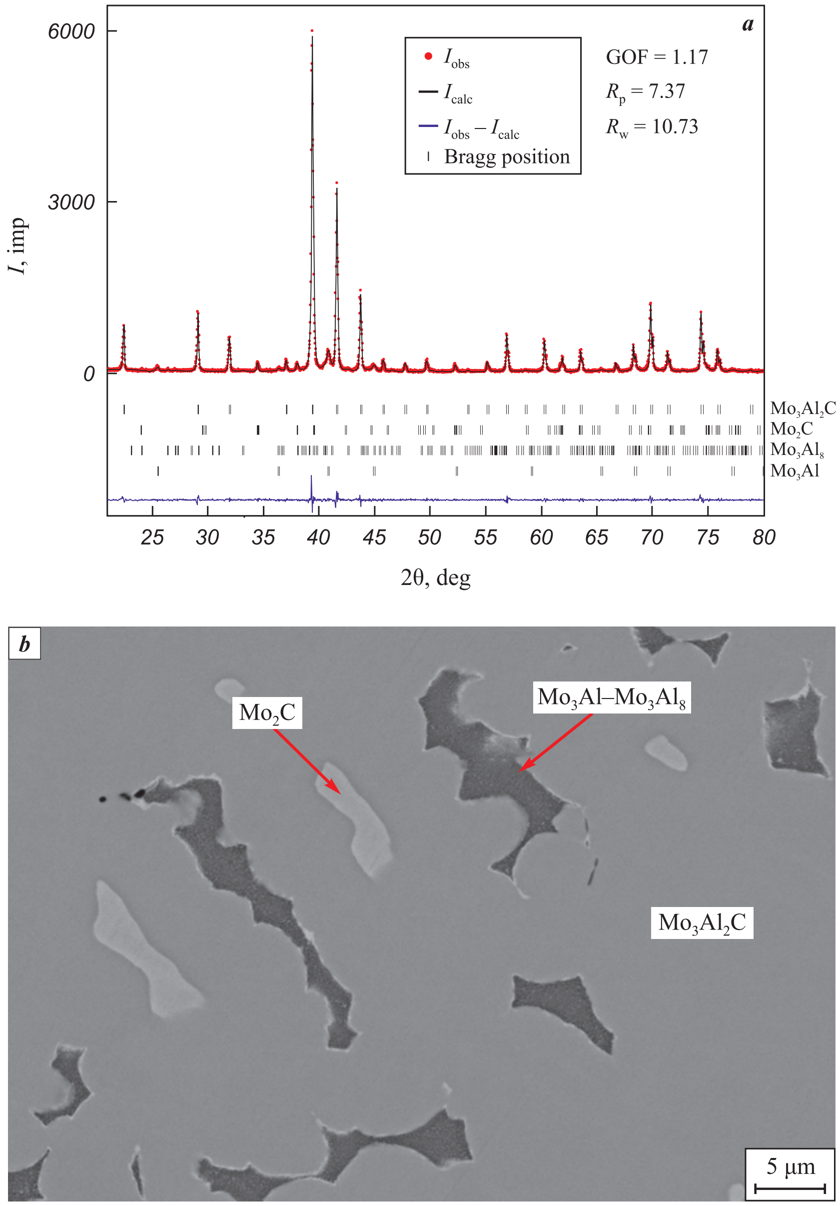

Fig. 4. Diffraction pattern (***a***) and microstructure (***b***) of the combustion product of a mixture *3*

Рис. 4. Дифрактограмма (***a***) и микроструктура (***b***) продукта горения смеси *3*

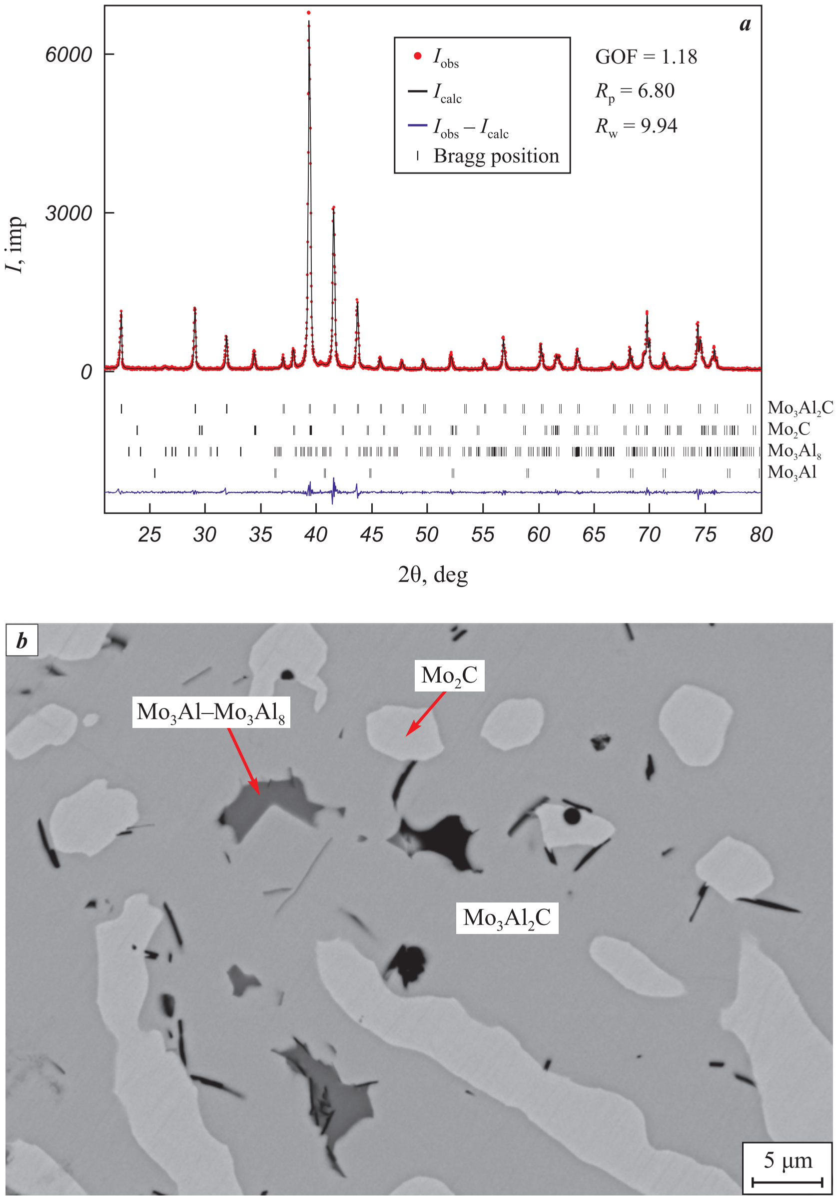

Fig. 5. Diffraction pattern (***a***) and microstructure (***b***) of the combustion product of mixture *5*

Рис. 5. Дифрактограмма (***a***) и микроструктура (***b***) продукта горения смеси *5*

composition of the target ternary phase Mo_3Al_2C, does not reflect all the interactions actually occurring in the multiphase system during SHS. *X*-ray diffraction phase analysis of the synthesized material showed that its phase composition differs from the calculated one. This indicates that the processes occurring in the liquid phase formed in the combustion wave and during its rapid crystallization lead to the formation of a nonequilibrium composition of the product. In addition, the combustion process is accompanied by the ejection of components as a result of the reaction (IV). It is quite likely that the resulting material is depleted in both carbon and aluminium. This is indirectly confirmed by the phase composition of mixture *5*, which is practically in the two-phase region Mo_3Al_2C–Mo_2C.

The analysis of microstructural analysis data suggests the following mechanism of phase formation of the final product. The phase composition of the ingot is formed as a result of a series of phase transformations. First, refractory Mo_2C carbide grains crystallize at a temperature of about 2500 °C. As a result, a Mo–Al melt is formed, which surrounds the Mo_2C grains. Then, during cooling in the temperature range of 2500–1720 °C, Al from the melt and Mo_2C interact, which leads to the formation of a carbide grain of the Mo_3Al_2C phase on their surface in a ring pattern (see Fig. 3). The growth of the layer is caused by the diffusion of Al from the Mo–Al melt through the Mo_3Al_2C layer into the Mo_2C grain. At temperatures below 1720 °C, the intergranular melt crystallizes with the formation of

Mo_3Al and MoAl. Then, at a temperature of 1467 °C, the MoAl phase undergoes the eutectoid transformation MoAl → Mo_3Al_8 + Mo_3Al [1]. Thus, the formation of a multiphase cast material is explained by the multistage nature of its formation and the rapid cooling of the melt.

Conclusion

Cast materials containing the phases Mo_3Al_2C, Mo_2C, Mo_3Al, and Mo_3Al_8 were obtained by the method of self-propagating high-temperature synthesis, combining two modes of the process – SHS from elemental powders and SHS-metallurgy. A significant effect of the ratio of reagents in the initial mixtures on the parameters of the combustion process, microstructure, and phase composition of the products was found. Introduction into the high-exothermic mixture $3MoO_3$–8Al–C inert “cold” mixture 3Mo–2Al–C leads to an increase in the content of carbide phases in the ingots. The maximum content (~87 wt. %) of the ternary phase Mo_3Al_2C was obtained at $\alpha = 0.4$. The presence of carbide Mo_2C and molybdenum aluminides Mo_3Al_8, and Mo_3Al in the final products is due to a change in the stoichiometric composition of the initial charge caused by the ejection of components during the combustion and insufficient existence time of the melt, which leads to the formation of a non-equilibrium composition of the product.

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Information about the Authors

Vladimir A. Gorshkov – Dr. Sci. (Eng.), Leading Researcher of the Laboratory of SHS melts and cast materials of Merzhanov Institute of Structural Macrokinetics and Materials Science of the Russian Academy of Sciences (ISMAN)
ORCID: 0000-0001-8845-4717
E-mail: gorsh@ism.ac.ru

Pavel A. Miloserdov – Cand. Sci. (Eng.), Senior Researcher of the Laboratory of SHS melts and cast materials of ISMAN
ORCID: 0000-0002-2587-0067
E-mail: yu_group@ism.ac.ru

Dmitrii Yu. Kovalev – D.Yu. – Dr. Sci. (Phys.-Math.), Head of the Laboratory of *X*-ray investigation of ISMAN
ORCID: 0000-0002-8285-5656
E-mail: kovalev@ism.ac.ru

Сведения об авторах

Владимир Алексеевич Горшков – д.т.н., вед. науч. сотрудник лаборатории «Жидкофазные СВС-процессы и литые материалы», Институт структурной макрокинетики и проблем материаловедения им. А.Г. Мержанова РАН (ИСМАН)
ORCID: 0000-0001-8845-4717
E-mail: gorsh@ism.ac.ru

Павел Александрович Милосердов – к.т.н., ст. науч. сотрудник лаборатории «Жидкофазные СВС-процессы и литые материалы», ИСМАН
ORCID: 0000-0002-2587-0067
E-mail: yu_group@ism.ac.ru

Дмитрий Юрьевич Ковалев – д.ф.-м.н., заведующий лабораторией рентгеноструктурных исследований, ИСМАН
ORCID: 0000-0002-8285-5656
E-mail: kovalev@ism.ac.ru

Ol'ga D. Boyarchenko – Cand. Sci. (Phys.-Math.), Researcher of the Laboratory of materials science of ISMAN
ORCID: 0000-0002-7543-7608
E-mail: olgaboyarchenko@gmail.com

Contribution of the Authors

V. A. Gorshkov – formation of the main concept, setting the goal and objectives of the study, conducting experiments, preparing the text, formulating conclusions.
P. A. Miloserdov – conducting the calculations, conducting experiments, processing research results, preparing the text of the article.

D. Yu. Kovalev – *X*-ray phase analysis, processing of research results.
O. D. Boyarchenko – microstructural analysis, processing of research results.

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Ольга Дмитриевна Боярченко – к.ф.-м.н., науч. сотрудник лаборатории физического материаловедения, ИСМАН
ORCID: 0000-0002-7543-7608
E-mail: olgaboyarchenko@gmail.com

Вклад авторов

В. А. Горшков – формирование основной концепции, постановка цели и задачи исследования, проведение экспериментов, подготовка текста, формулировка выводов.
П. А. Милосердов – проведение расчетов, проведение экспериментов, обработка результатов исследований, подготовка текста статьи.
Д. Ю. Ковалев – проведение рентгенофазового анализа, обработка результатов исследований.
О.Д Боярченко – проведение микроструктурного анализа, обработка результатов исследований.

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