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Features of SHS of multicomponent carbides

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Abstract: Combustion of powders of transition metals of titanium PTS (average particle size 57 μm), zirconium PCRK-1 (12 μm), tantalum Ta PM-3 (8 μm), hafnium GFM-1 (4 μm), niobium NBP-1a (21 μm) with carbon black grade P-803 dispersion 1—2 μm was studied. The combustion process of the compressed samples (mass 2.5—6.9 g, height 1.2—1.7 cm, relative density 0.55—0.61) was performed in an inert argon medium at a pressure of 760 mmHg in the constant pressure chamber. Combinations were studied, Me1 + Me2 + Me3 + Me4 + 4C, Me1 + Me2 + Me3 + Me4 + Me5 + 5C. XRD patterns of the mixtures were recorded on a DRON-3M diffractometer (CuK_α-radiation). Combustion product sections were studied using a LEO 1450 VP scanning electron microscope (Carl Zeiss, Germany). The fractional composition and particle size distribution of the mixture were determined according to standard procedure using a Microsizer-201C laser particle size analyzer. Combustion velocity, elongation of samples, phase composition of products were determined. The maximum combustion temperature of the mixture (Ti + Hf + Zr + Nb + Ta) + 5C was measured experimentally for the first time. The morphology and microstructure of the reaction products were also observed. Combustion products of mixtures (Ti + Zr + Nb + Ta) + 4C and (Ti + Zr + Nb + Hf) + 4C contain high entropy carbides, which are solid solutions with the same structural type B1 (space group Fm-3m) and having different cell parameters. Product samples of mixtures (Ti + Zr + + Hf + Ta) + 4C and (Ti + Hf + Zr + Nb + Ta) + 5C contain high entropy and medium entropy carbides, also representing solid solutions with the same structural type B1 (space group Fm-3m). The results of this work can be used in the production of high-entropy and medium-entropy multicomponent carbides.

Keywords: SHS, combustion, high-entropy multicomponent carbides, medium entropy carbides, high-entropy ceramics, transition metals, refractory metals.

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Особенности СВС многокомпонентных карбидов

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Аннотация: Изучено горение порошков переходных металлов: титана марки ПТС (средний размер частиц 57 мкм), циркония ПЦРК-1 (12 мкм), тантала Та ПМ-3 (8 мкм), гафния ГФМ-1 (4 мкм), ниобия НБП-1а (21 мкм) — с сажей марки П-803 дисперсностью 1—2 мкм. Процесс горения спрессованных образцов (масса 2,5—6,9 г, высота 1,2—1,7 см, относительная плотность 0,55—0,61) осуществляли в инертной среде аргона при давлении 760 мм рт. ст. в камере постоянного давления. Исследовали комбинации Me1 + Me2 + Me3 + Me4 + 4C и Me1 + Me2 + Me3 + Me4 + Me5 + 5C. Рентгенограммы смесей регистрировали на дифрактометре «Дрон-3М» (CuK_α-излучение). Шлифы продуктов горения изучали на сканирующем электронном микроскопе LEO 1450 VP (Carl Zeis, Германия). Фракционный состав и распределение частиц смеси по размеру устанавливали по стандартной методике на лазерном анализаторе размера частиц «Микросайзер-201С» (РФ). Определяли скорость горения, удлинение образцов, фазовый состав продуктов. Впервые экспериментально измерена максималь-

ная температура горения смеси (Ti + Hf + Zr + Nb + Ta) + 5C. Также наблюдали за морфологией и микроструктурой продуктов реакции. Продукты горения смесей (Ti + Zr + Nb + Ta) + 4C и (Ti + Zr + Nb + Hf) + 4C содержат высокоэнтропийные карбиды, представляющие собой твердые растворы с одинаковым структурным типом B1 (пространственная группа Fm-3m) и обладающие различными параметрами ячейки. Образцы продуктов смесей (Ti + Zr + Hf + Ta) + 4C и (Ti + Hf + Zr + Nb + Ta) + 5C содержат в составе высокоэнтропийные и среднеэнтропийные карбиды, также представляющие собой твердые растворы с одинаковым структурным типом B1 (пространственная группа Fm-3m). Результаты данной работы могут найти применение при получении высокоэнтропийных и среднеэнтропийных многокомпонентных карбидов.

Ключевые слова: СВС, горение, высокоэнтропийные многокомпонентные карбиды, среднеэнтропийные карбиды, высокоэнтропийная керамика, переходные металлы, тугоплавкие металлы.

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Introduction

The combustion of transition metals with carbon was the subject of scientific research as recently as several decades ago [1—5].

Self-propagating high-temperature synthesis (SHS) in Zr + C, Ta + C and Hf + C systems was first implemented by A.G. Merzhanov and I.P. Borovinskaya [1].

In work [2], it has been demonstrated that the combustion process in Nb + C system is initiated in a much more complicated way compared to Zr + C system, and the combustion limit for Nb in Nb + Zr + + C system was 0.7 at. fr. Upon a higher content of niobium, the samples did not flare up at any concentration of carbon. It is noted by the authors [3] that the combustion parameters of Zr + C system depend heavily on the grade of powders being used. In work [4], the substantial impact of impurity gas release on the combustion of Ta + C system was established, which is expressed in the dependence of the combustion rate on the pressure of inert gas, as well as in the elongation and delamination of the sample during combustion. Besides, the combustion at a sample diameter of $d > 1$ cm was implemented successfully [4]. There was no combustion in Ta + C system at $d \leq 1$ cm. The considerable elongation of the compressed samples of Ti + C mixture during combustion was also noted earlier [5]. In work [6], it is mentioned that combustion in Ta + C system is possible at temperatures below the melting temperature, upon which it is necessary to use metal powders with a submicron particle size. The authors [7, 8] determined the influence of the particle size of the titanium powder used on the combustion rate of samples made from a Ti + C mixture.

The synthesis and study of the properties of high-entropy alloys (HEAs), which represent a new class of metallic materials, have been a popular and promising line of scientific research in recent times. HEAs include compounds containing several (usually 5) metals and forming a single-phase solid solution [9]. The works devoted to the production and study of medium entropy alloys, containing 3 metals, appear [10]. The mechanical properties of medium entropy alloys can exceed the properties of HEAs [11, 12].

The works devoted to the production of high-entropy ceramics [13, 14] and, in particular, high-entropy carbides (HECs) [15—21] have been published relatively recently. HECs were obtained by mechanical alloying in ball mills and spark plasma sintering.

The combustion of powders of transition metals with carbon black in Me1 + Me2 + Me3 + Me4 + 4C (4 metals with carbon, 5 combinations) and Me1 + Me2 + + Me3 + Me4 + Me5 + 5C (5 metals with carbon, 1 combination) combinations, where Mei is Ti, Hf, Nb, Zr, Ta, was studied. The objective of obtaining a high-entropy metal carbide by using SHS was set.

Research methods

The powders of titanium PTS (average particle size 57 μm), zirconium PCRK-1 (12 μm), tantalum Ta PM-3 (8 μm), hafnium GFM-1 (4 μm), niobium NBP-1a (21 μm) and carbon black grade P-803 dispersion 1—2 μm were used as initial materials. The *X*-ray phase analysis (XRD) of initial metals revealed that all powders are single-phase, except for Hf, which exhibits $HfH_{1.63}$ phase impurity.

The initial powders were thoroughly mixed in a porcelain mortar in the required weight proportions for the purpose of obtaining Me + C mixtures. Me1 + Me2 + + Me3 + Me4 + 4C (4 metals with carbon, 5 combinations) and Me1 + Me2 + Me3 + Me4 + Me5 + 5C (5 metals with carbon, 1 combination) mixtures, where Mei is Ti, Hf, Nb, Zr, Ta, were mixed in a ball mill for 2 h.

The combustion process of the compressed samples (mass 2.5—6.9 g, height 1.2—1.7 cm, relative density 0.55—0.61) was carried out in an inert argon medium at a pressure of 760 mmHg in a constant pressure chamber [22]. The process was recorded on the camera through a viewing glass. The combustion of samples was initiated from the upper end with an ignite tablet consisting of Ti + 2B by means of heated tungsten coil, which ensured stable ignition conditions. Upon stop motion viewing of video records, the combustion rate of the samples was determined. The maximum combustion temperature was measured with a tungsten-rhenium thermocouple.

The measurements of combustion rate, maximum combustion temperature and relative elongation of the samples had an error within 10 %.

Thermodynamic calculations were performed using THERMO software — http://www.ism.ac.ru/thermo/.

Combustion product sections were studied using a LEO 1450 VP scanning electron microscope (Carl Zeiss, Germany). To obtain the sections, combustion product powders were impregnated with epoxy resin diluted with acetone to reduce viscosity. Metallographic sections were made after the resin hardened.

The *X*-ray phase analysis (XRD) of initial powders and combustion products was performed with DRONE-3M diffractometer (the RF) upon copper radiation being in the range of angles 2θ from 20 to 80°. The obtained data were analyzed using PDF-2 database [14].

The fractional composition and particle size distribution of the mixture were determined according to a standard procedure using a Microsizer-201C laser particle size analyzer (the RF). The measurement error did not exceed 1.2 %.

Results and discussions

The values of combustion rates, elongation of samples during the SHS, as well as the phase composition of the products of synthesis of metal powders with carbon black in various combinations are provided in the table.

Combustion rate, elongation of samples, phase composition of products, and adiabatic temperature of SHS of metal powders with carbon black

Скорость горения, удлинение образцов, фазовый состав продуктов и адиабатическая температура СВС порошков металлов с сажей

System	Combustion rate, cm/s	Composition of the products	Elongation of the sample during combustion, %	Adiabatic combustion temperature, K
Ti + C	1.3	TiC	94	3289
Ta + C				2721
Hf + C				3899
Zr + C	1.0	ZrC	224	3777
Nb + C				2835
(Ti + Hf + Zr + Nb + Ta) + 5C	0.24	[Ti,Hf,Zr,Nb,Ta]C, [Ti,Hf, Zr,Ta]C, [Ti,Hf,Ta]C	181	3290
(Ti + Zr + Nb + Ta) + 4C	0.35	[Ti,Zr,Nb,Ta]C	96	3180
(Ti + Zr + Nb + Hf) + 4C	0.35	[Ti,Hf,Zr,Nb]C	331	3309
(Ta + Hf + Zr + Nb) + 4C				3360
(Ti + Hf + Nb + Ta) + 4C				3286
(Ti + Zr + Hf + Ta) + 4C	0.55	[Ti,Hf,Zr,Ta]C, [Ti,Hf,Ta]C	213	3290

Among Me + C systems, the SHS without preheating was initiated in samples compressed from Ti + C and Zr + C mixtures. Samples of other mixtures (Nb + C, Hf + C, Ta + C) did not burn at room temperature under the present experimental conditions (the component particle size and the sample size). The values of the combustion rates of samples of Ti + C and Zr + C mixtures appeared to be close to: 1.3 and 1.0 cm/s (see the table). Due to the release of impurity gas, the samples often elongate during the combustion [23, 24]. The samples of Zr + C mixture elongated significantly compared with the samples of Ti + C mixture (for comparison, 224 and 94 %, respectively), which is similar to the results obtained earlier upon the combustion of mixtures of Zr and Ti powders with boron [14]. According to XPhA results, the combustion products of Me + C systems exhibited the reflections of the only carbide phase MeC (ZrC and TiC, respectively).

Among the mixtures containing 4 metals with carbon (where Me is Ti, Hf, Nb, Zr, Ta), the combustion process without preheating was successfully conducted (and the sample burned completely) in the systems containing Ti and Zr, i.e. (Ti + Zr + Nb + Ta) + 4C, (Ti + Zr + Nb + Hf) + 4C, (Ti + Zr + Hf + Ta) + 4C. The combustion of samples compressed from (Ti + Hf + + Nb + Ta) + 4C mixture could not be initiated without preheating. The samples of (Ta + Hf + Zr + Nb) + 4C mixture did not burn out completely. The design adiabatic combustion temperature of the compositions varied in the range of 3180 to 3360 K. Upon that, the samples of (Ti + Zr + Hf + Ta) + 4C mixture appeared to be the fastest burning ones as their combustion rate was 0.55 cm/s. The samples of (Ti + Zr + Nb + Ta) + 4C and (Ti + Zr + Nb + Hf) + 4C mixtures had the same combustion rate of 0.35 cm/s.

According to XRD results, the products of synthesis of (Ti + Zr + Nb + Ta) + 4C and (Ti + Zr + Nb + + Hf) + 4C slow burning systems contain 3 carbide phases based on solid solutions with the same structural type B1 (spatial group Fm-3m), having different cell parameters (Fig. 1).

Phase *1* is the closest to HfC, phase *2* is the closest to ZrC, phase *3* is the closest to NbC or TaC in cell parameter (see below). The combustion products of the faster burning system (Ti + Zr + Hf + Ta) + 4C already contain 4 carbide phases, are distinguished by their cell parameters (see Fig. 1), where phase *4* is the closest to phase $TaTiC_2$ by its cell parameter. Below are the unit cell parameters of the synthesized

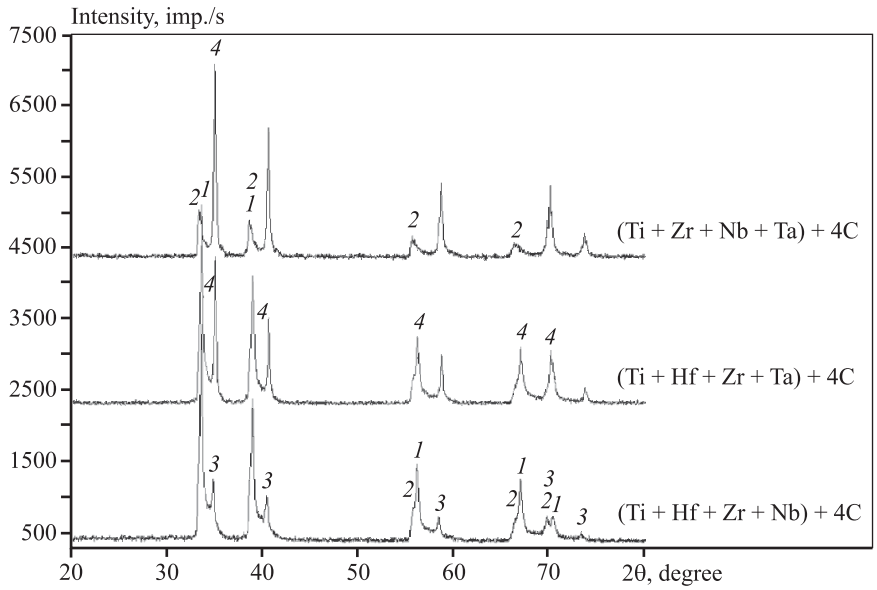

Fig. 1. XRD patterns of products of synthesis of Me1 + Me2 + Me3 + Me4 + 4C mixtures (where Me is Ti, Ta, Zr, Hf, Nb)

1—4 — FCC phases; *a*, Å = 4.6360 (***1***), 4.6737 (***2***), 4.4748 (***3***), 4.4526 (***4***)

Рис. 1. Рентгенограммы продуктов синтеза смесей Me1 + Me2 + Me3 + Me4 + 4C (Me — Ti, Ta, Zr, Hf, Nb)

1—4 — ГЦК-фазы; ***a***, Å = 4,6360 (***1***), 4,6737 (***2***), 4,4748 (***3***), 4,4526 (***4***)

solid solutions and the cell parameters of monocarbides (*a*), Å:

NbC	4.4698
HfC	4.6377
ZrC	4.6930
TaC	4.4547
TiC	4.3274
Phase *1*	4.6360
Phase *2*	4.6737
Phase *3*	4.4748
Phase *4*	4.4526

To identify metals in carbide phases, sections were made of product samples and local elemental analysis was performed (Fig. 2). According to XPhA and elemental analysis results, the combustion products of (Ti + Zr + Nb + Ta) + 4C and (Ti + Zr + Nb + Hf) + 4C slow-burning systems contain the phases of high-entropy carbides ([Ti,Zr,Nb,Ta]C and [Ti,Hf,Zr,Nb]C, respectively), differing in the ratio of components (Fig. 2, *a*, *b*). The products of a faster burning system (Ti + Zr + Hf + Ta) + 4C include medium entropy carbides [Ti,Hf,Ta]C, containing 3 metals (Fig. 2, *c*), along with high-entropy carbides [Ti,Hf,Zr,Ta]C.

It appears that considerably high combustion rate of the sample made of (Ti + Zr + Hf + Ta) + 4C mixture (0.55 cm/s) and its small size result in a lack of time for the homogenization of the product.

Similar to the samples of Me + C mixtures, the samples made of Me1 + Me2 + Me3 + Me4 + 4C mixtures grew during combustion and dispersed into separate fragments due to impurity gas release. Upon that, the minimum elongation was observed during the combustion of samples made of a mixture without haf-

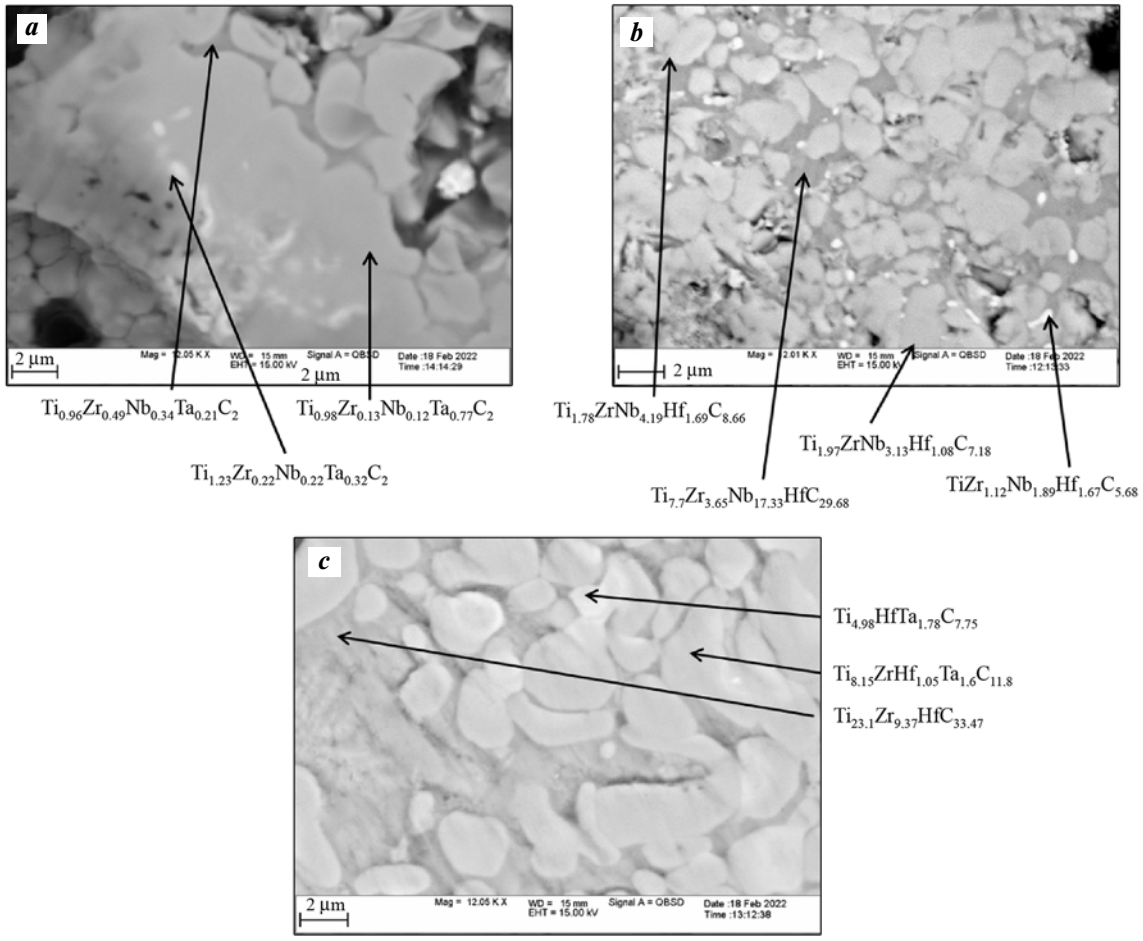

Fig. 2. Microstructure of combustion products of samples of Ti + Zr + Nb + Ta + 4C (***a***), Ti + Hf + Zr + Nb + 4C (***b***) and Ti + Zr + Hf + Ta + 4C (***c***) mixtures

Рис. 2. Микроструктура продуктов горения образцов из смесей Ti + Zr + Nb + Ta + 4C (***a***), Ti + Hf + Zr + Nb + 4C (***b***) и Ti + Zr + Hf + Ta + 4C (***c***)

nium — (Ti + Zr + Nb + Ta) + 4C, i.e. 96 % (see the table). The samples made of mixtures containing hafnium ((Ti + Zr + Nb + Hf) + 4C and (Ti + Zr + Hf + + Ta) + 4C) elongated much more, namely 331 and 213 %, respectively. This suggests that hafnium, containing hydride $HfH_{1.63}$, results in an increase in impurity gas release during the combustion of samples.

The photograph of the microstructure of the combustion products of Ti + Hf + Zr + Nb + 4C mixture, as well as the element distribution map are presented in Fig. 3. One can see that all the elements contained in the product are quite uniformly distributed over the cross-sectional area of the section.

Combustion of Me1 + Me2 + Me3 + Me4 + Me5 + 5C mixture

The sample made of (Ti + Hf + Zr + Nb + Ta) + + 5C mixture burned out completely without preheating. Upon that, its combustion rate appeared to be lower than the one of burned samples made of Me + C and Me1 + Me2 + Me3 + Me4 + 4C mixtures and amounted to only 0.24 cm/s. The design adiabatic combustion temperature of (Ti + Hf + Zr + Nb + Ta) + 5C mixture amounted to T_{ad} = 3290 K, which coincides with T_{ad} for the fastest burning mixture of 4 metals with carbon (Ti + Zr + Hf + Ta) + 4C (see the table).

According to XPhA results, the composition of combustion products of (Ti + Hf + Zr + Nb + Ta) + 5C mixture, exhibit three carbide phases based on solid solutions with the same structural type B1 (spatial group Fm3m), having different cell parameters (Fig. 4), in the same way as for mixtures of 4 metals with carbon. The first phase is the closest to HfC, the second phase is the closest to ZrC, and the third phase is the closest to NbC or TaC in cell parameter (the same has been specified above).

The elemental analysis of sections of combustion products allowed to identify these carbide phases (Fig. 5). They turned out to be high-entropy carbide [Ti,Hf,Zr,Ta]C, containing 4 metals, and medium entropy carbide [Ti,Hf,Ta]C, containing 3 metals, there are also the traces of high-entropy carbide [Ti,Hf, Zr,Nb,Ta]C, including all 5 metals.

Similar to the samples of previous systems (containing 1 or 4 metals), a sample made of (Ti + Hf + Zr + + Nb + Ta) + 5C mixture significantly elongated and dispersed into separate fragments during combustion. The relative elongation of the sample accounted for 181 %,

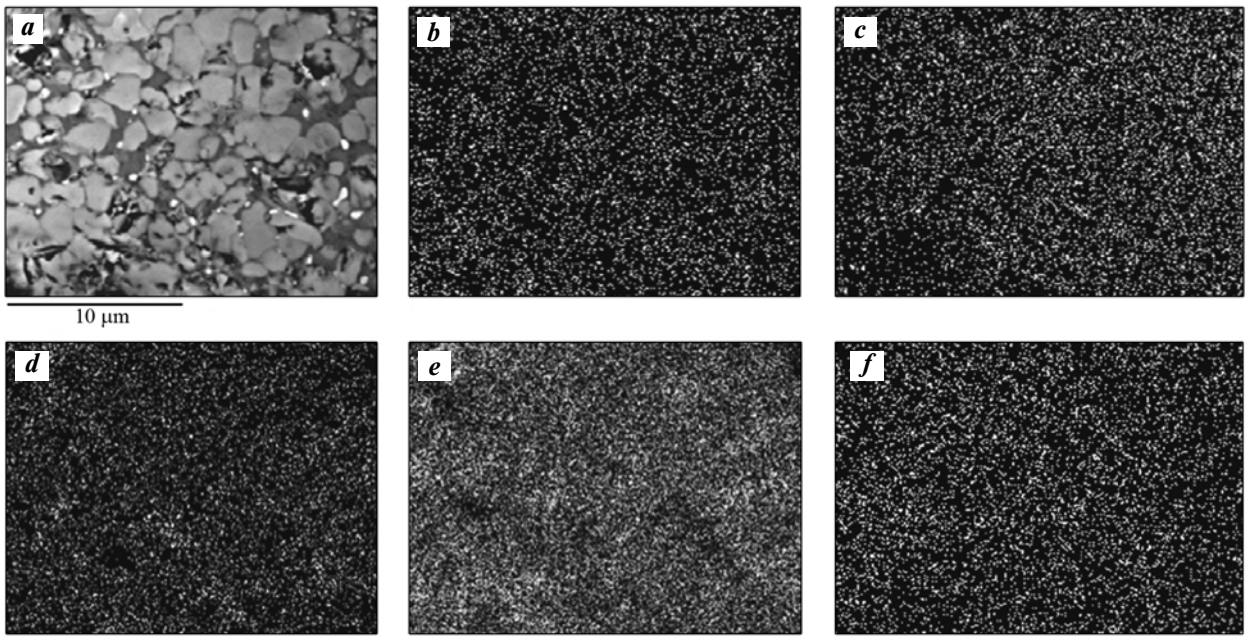

Fig. 3. Microstructure of combustion products of Ti + Hf + Zr + Nb + 4C mixture (***a***) and element distribution map (***b—f***)
b — C, ***c*** — Ti, ***d*** — Zr, ***e*** — Nb, ***f*** — Hf

Рис. 3. Микроструктура продуктов горения смеси Ti + Hf + Zr + Nb + 4C (***a***) и карта распределения элементов (***b—f***)
b — C, ***c*** — Ti, ***d*** — Zr, ***e*** — Nb, ***f*** — Hf

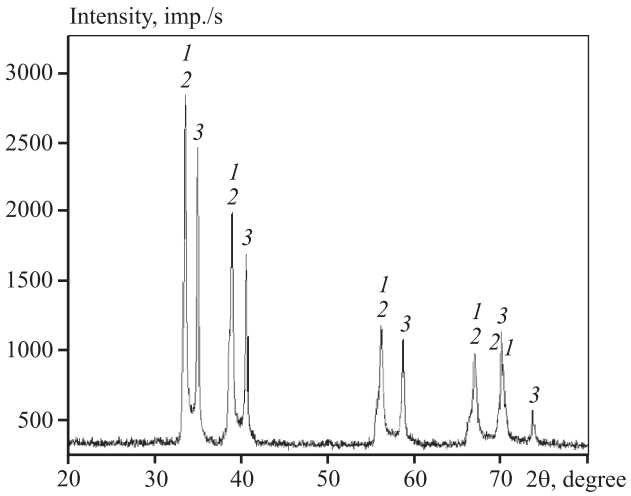

Fig. 4. XRD of products of synthesis of Me1 + Me2 + Me3 + Me4 + Me5 + 5C mixture (where Me is Ti, Ta, Zr, Hf, Nb)
1—3 — FCC phases; *a*, Å = 4.6360 (***1***), 4.6737 (***2***), 4.4748 (***3***)

Рис. 4. Рентгенограмма продуктов синтеза смеси Me1 + Me2 + Me3 + Me4 + Me5 + 5C (Me — Ti, Ta, Zr, Hf, Nb)
1—3 — ГЦК-фазы; *a*, Å = 4,6360 (***1***), 4,6737 (***2***), 4,4748 (***3***)

which falls within the interval between maximum and minimum elongation values for the studied systems containing 1 or 4 metals.

Considerable elongation of the sample during combustion made it difficult to determine maximum combustion temperature by means of a thermocouple. To measure the maximum combustion temperature, experiments were carried out on the combustion of compressed samples. The sample was pre-compressed, and that limited its elongation. As a result, it decreased by an order of magnitude (15 %) that allowed to measure the maximum combustion temperature, the value of which was 1950 °C. The measured maximum combustion temperature appeared to be significantly lower than the design adiabatic one, probably due to the fact that the heat loss is not taken into account during the calculation. At such a combustion temperature, only two of five initial metals (Ti and Zr) melt in the combustion wave. The rest of the metals (Hf, Nb, Ta) have a melting temperature higher than the experimentally measured maximum combustion temperature of (Ti + Hf + Zr + Nb + Ta) + + 5C mixture, below are the melting temperatures of the components of the mixture, °C:

Zr	1855
Ti	1668
Hf	2227
Nb	2468
Ta	3017

Furthermore, the compression of the sample and a decrease by an order of magnitude of its growth resulted in a combustion rate increase by a several-fold factor, namely from 2.4 to 8.8 mm/s, which correlates well with a pioneer work on the study of the compression of samples by combustion rate [25]. As it is shown in works [23, 24], the elongation of the sample during combustion occurs behind the combustion front due to impurity gas release. In the case of compression of the sample, hindering its elongation, the pressure of these gases, being released behind the front during combustion, increases. According to the findings of the con-

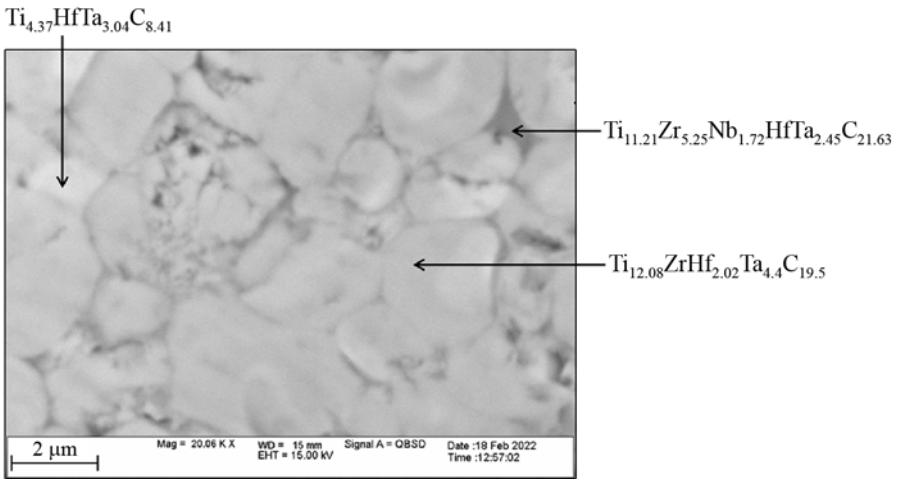

Fig. 5. Microstructure of combustion products of the sample made of Ti + Zr + Nb + Ta + Hf + 5C mixture

Рис. 5. Микроструктура продуктов горения образца из смеси Ti + Zr + Nb + Ta + Hf + 5C

vective-conductive combustion model, this results in an increase in the propagation velocity of the combustion front [26].

It is further planned to obtain compact samples of high-entropy carbides from the combustion products of mixtures of 4 metals with carbon and 5 metals with carbon by SPS method.

Conclusion

The combustion and composition of the products of Me + C, Me1 + Me2 + Me3 + Me4 + 4C (4 metals with carbon in 5 combinations) and Me1 + Me2 + Me3 + + Me4 + Me5 + 5C (5 metals with carbon, 1 combination) mixtures, where Me*i* is Ti, Hf, Nb, Zr, Ta, were studied.

Multicomponent carbides, belonging to the class of high-entropy compounds, were synthesized in the combustion mode.

Among the mixtures of 4 metals with carbon, combustion was achieved in the samples containing Ti and Zr.

The measured maximum combustion temperature of (Ti + Hf + Zr + Nb + Ta) + 5C mixture was 1950 °C.

The conclusions of the work can be used in obtaining new materials such as high-entropy and medium entropy carbides.

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