Self-Propagating High-Temperature Synthesis
Самораспространяющийся высокотемпературный синтез

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Research article
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Fabrication of (Ti–Al–Si)/(Ti–C)/Ti – layered alloy by SHS pressing

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Abstract. A metal–carbide–intermetallic material based on combustion products of the layer system (Ti–Al–Si)/(Ti–C)/Ti was for the first time obtained with the help of self-propagating high-temperature synthesis (SHS) combined with pressing. Exothermic synthesis from elementary powders was carried out at a pressure of 10 MPa, and pressing of the hot synthesis product was carried out at a pressure of 100 MPa. It has been shown that SHS pressing contributes to the formation of permanent joints of «metal/carbide/intermetallic» layers. The main features of microstructure formation, phase composition, and strength properties of transition zones at the boundary between reacting SHS compositions, Ti–C and Ti–Al–Si and Ti-metal substrate are investigated. It is shown that during SHS reaction, a homogeneous microstructure of Ti–C and Ti–Al–Si layers with an insignificant content of cracks and pores is formed. The thickness of the transition zone between the layers was at least 15 μm. The main phase formed in the combustion product of Ti–Al–Si layer is, according to the results of *X*-ray phase analysis, triple phase $Ti_{20}Al_3Si_9$, the content of which, calculated by the Rietveld method, was at least 87 wt. %. In addition, the combustion product contains a secondary phase of Ti_3Al in the amount of 13 wt. %. The energy dispersion analysis revealed that diffusion of aluminium through the titanium carbide layer into the titanium substrate to a depth of approx. 30 μm is observed. Microhardness value of the combustion product of Ti–Al–Si layer was about 10 GPa. The rectilinear nature of crack propagation in the synthesized combustion product of Ti–Al–Si layer, as well as the Palmquist crack resistance coefficient varying within 5.1–5.7 MPa·m$^{1/2}$, indicate the fragility of the material.

Keywords: layered material, intermetallic compound, SHS-pressing, Ti–Al–Si

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Получение слоевого (Ti–Al–Si)/(Ti–C)/Ti сплава методом СВС-прессования

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Аннотация. Методом самораспространяющегося высокотемпературного синтеза (СВС), совмещенного с прессованием, впервые получен металло-карбидно-интерметаллидный материал на основе продуктов горения слоевой системы

(Ti–Al–Si)/(Ti–C)/Ti. Экзотермический синтез из элементарных порошков осуществляли при давлении 10 МПа, а прессование горячего продукта синтеза – при давлении 100 МПа. В работе продемонстрировано, что в результате СВС-прессования формируется неразъемное соединение слоев «метал/карбид/интерметаллид». Исследованы основные особенности формирования микроструктуры, фазовый состав и прочностные свойства переходных зон на границе между реагирующими СВС-составами Ti–C и Ti–Al–Si и Ti-металлической подложкой. Показано, что в процессе СВС-реакции формируется однородная микроструктура слоев Ti–C и Ti–Al–Si с незначительным содержанием трещин и пор. Толщина переходной зоны между слоями составила не менее 15 мкм. Основной фазой, формирующейся в продукте горения слоя на основе Ti–Al–Si, является, по результатам рентгенофазового анализа, тройная фаза $Ti_{20}Al_3Si_9$, содержание которой, посчитанное по методу Ритвельда, составило не менее 87 мас.%. Кроме того, в продукте горения присутствует вторичная фаза Ti_3Al в количестве 13 мас.%. Результаты энергодисперсионного анализа показали, что наблюдается диффузия алюминия сквозь слой карбида титана в титановую подложку на глубину ~30 мкм. Значение микротвердости продукта горения слоя на основе Ti–Al–Si составило около 10 ГПа. Прямолинейный характер распространения трещин в синтезированном продукте горения слоя Ti–Al–Si, а также варьирующийся в пределах 5,1–5,7 МПа·м$^{1/2}$ коэффициент трещиностойкости по Палмквисту говорят о хрупкости материала.

Ключевые слова: слоевой материал, интерметаллид, СВС-прессование, Ti–Al–Si

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Introduction

Obtaining layered systems and permanent joints of various metal-intermetallic materials constitutes a significant task for modern industry, particularly for aerospace applications, taking into account the unique combination of physical and mechanical characteristics of metal-intermetallic layered composites [1]. Material microstructure development, oriented towards a specific set of structural and functional characteristics, represents an urgent task in materials science. Ti–Al titanium aluminides can be distinguished among a variety of intermetallic compounds, which need to increase their temperature resistance to oxidation and deformation [2–4]. Titanium-based alloys with other light elements (Mg, Si etc.) appear to be highly promising for high-temperature applications in a variety of industries, particularly for use as protective coatings. Silicon appears to be a compelling choice for a reinforcing component (Ti_5Si_3) in a TiAl-based composite and also positively affects the resistance of titanium and its alloys to high-temperature oxidation [5; 6].

Since the well-known methods for producing the required materials (hot isostatic pressing [7] and spark plasma sintering [8]) are costly and technically demanding, it is essential to find new, technologically simplified methods for their production. Self-propagating high-temperature synthesis (SHS) may be a promising way of solving the problem of obtaining metal-intermetallic layered materials [9]. During the SHS process, a large amount of heat released can be used not only for further processing of the material or formation of its structure, but also as a source of additional heat for joining (welding, repairing) heterogeneous materials and applying coatings [10; 11]. For instance, Ti–Al–Si alloys were produced by SHS in work [12] upon the interaction of titanium, silicon, and AlSi30 alloy followed by the addition of alloying elements. A method for the production of Ti–Al–Si alloys with an aluminum content ranging from 8 to 20 %[1] and a silicon content of 10–20 % has been elaborated in works [13–15]. Combining the SHS method and pressing may be used to produce layered and graded carbide-hardened materials, as well as permanent joints of heterogeneous materials and protective coatings. The SHS-pressing method was used to obtain NiAl–Ni layer compositions [16] and multilayer systems “hard alloy–intermetallic–metal” [17].

In this paper, the features of the formation of microstructure and strength properties of transition zones at the boundary between reacting SHS compounds and Ti-substrate in a layer system (Ti–Al–Si)/(Ti–C)/Ti have been investigated.

Experimental procedures

The following metal powders were used in the experiment: Si (semiconductor silicon, solar-grade, ~100 μm, at least 99 %), Ti (PTM, <100 μm, 99.2 %), and Al (ASD-4, ~10 μm, 99.20 %) to obtain a powder reaction mixture of 74.1Ti–6.3Al–19.6Si (%); incendiary mixture Ti/C (black) (50/50 %); titanium foil (Ti) of 200 μm thick. The mass ratio of layers 1-layer/2-layer/Ti-substrate was approximately ~90/8/2 %. Composition of the reaction mixture based on Ti–Al–Si was chosen to obtain the phase $Ti_{20}Al_3Si_9$. The initial powder blank of the 1st layer was obtained by dry mixing in a mortar, followed by pressing of cylindrical samples 30 mm in diameter and 16 mm in height with a relative density of 0.6. The compressed samples were placed in a reaction compression mold (Fig. 1), pre-mounted on a Ti substrate.

[1] Here in after – wt. %.

To obtain a nonporous material, SHS pressing technique described in [18; 19] has been used, while exothermic synthesis was carried out at a pressure of 10 MPa, and pressing of the synthesis product was carried out at a pressure of 100 MPa. The exposure time under load was 3 s.

The microstructure of the synthesized alloy was studied using an "Zeiss Ultra plus 55" field emission scanning electron microscope. *X*-ray phase analysis (XRD) was performed on a DRON-3 diffractometer using CuK_α radiation. Microhardness (H_μ) was measured on a PMT-3 hardness tester using the Vickers method (indentation of a tetrahedral diamond pyramid with a load of 100 g). Crack formation was studied by the method of indentation by the Vickers diamond pyramid *HV* at a higher load of up to 30 kg.

Results and discussion

Preliminary thermodynamic calculations performed in the software "Thermo"[2] clearly revealed that the largest thermal effect is observed in the layer based on the Ti–C system for which the adiabatic combustion temperature was 2617 °C and the enthalpy of formation was 176 kJ/mol. During the combustion of Ti–C reaction composition, the melting of the surface layer of the titanium substrate ($t_{\text{Ti}}^{\text{melt}}$ = 1670 °C) and the formation of the Ti/TiC transition zone is most likely to occur. Adiabatic combustion temperature of Ti–C is much higher than combustion temperature of Ti–Al–Si layer, equal to 1259 °C [15] which also affects the diffusion interaction and formation of a transition zone between Ti–C and Ti–Al–Si layers and provides a strong interpase connection between the Ti-substrate and the Ti–C carbide layer.

Figure 2 represents the microstructure and element distribution map of Ti, Al, Si and C in the synthesised alloy. A firm contact between the layers with the absence of any defects (pores, cracks) has been formed. This indicates a high quality of diffusion interaction of the elements between the layers.

As shown by XRD (Fig. 3), the 1st layer conforms to an alloy based on the main phase $Ti_{20}Al_3Si_9$ (PDF 01-079-2701) with a hexagonal close-packed (HCP) lattice; furthermore, there is a secondary ordered phase Ti_3Al with a superstructure $D0_{19}$ (PDF 52-859), which exhibits a HCP latitude (spatial group *P*63/*mmc*). The content

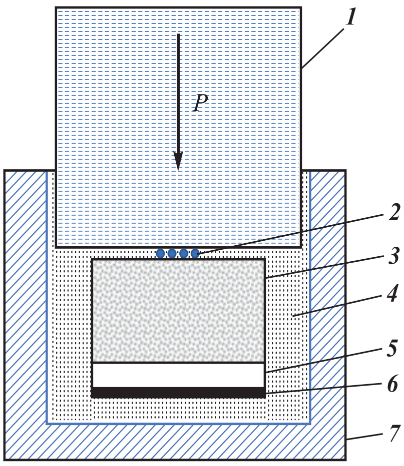


Fig. 1. Scheme of experiments on SHS pressing
1 – upper punch, ***2*** – igniting spiral W, ***3*** – Ti–Al–Si layer, ***4*** – SiO_2 heat insulator, ***5*** – Ti–C layer, ***6*** – Ti-substrate, ***7*** – mold

Рис. 1. Схема проведения экспериментов по СВС-прессованию
1 – верхний пуансон, ***2*** – поджигающая спираль W, ***3*** – слой Ti–Al–Si, ***4*** – теплоизолятор SiO_2, ***5*** – слой Ti–C, ***6*** – Ti-подложка, ***7*** – пресс-форма

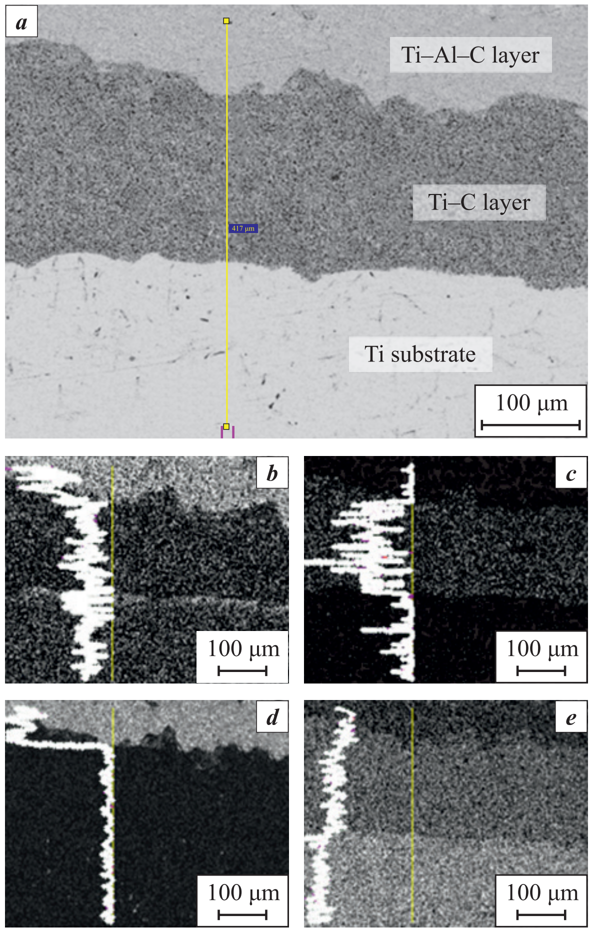


Fig. 2. Photo of the microstructure (***a***) and element distribution map in the synthesised alloy
b – Al, ***c*** – C, ***d*** – Si, ***e*** – Ti

Рис. 2. Фотография микроструктуры (***a***) и карта распределения элементов в синтезированном сплаве
b – Al, ***c*** – C, ***d*** – Si, ***e*** – Ti

[2] *Program for thermodynamics equilibrium calculations "THERMO"*. URL: http://www.ism.ac.ru/thermo (accessed: 15.02.2022).

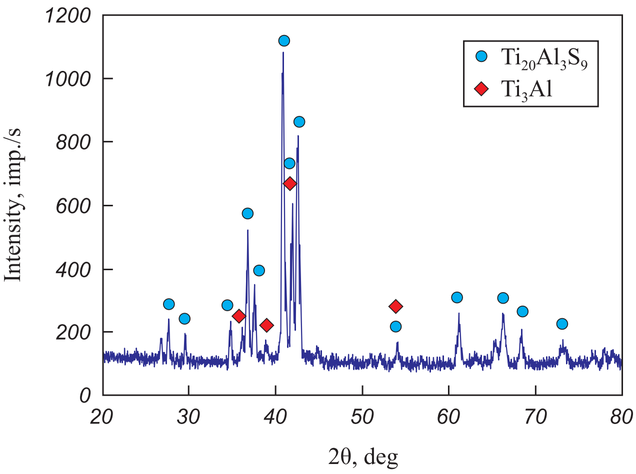
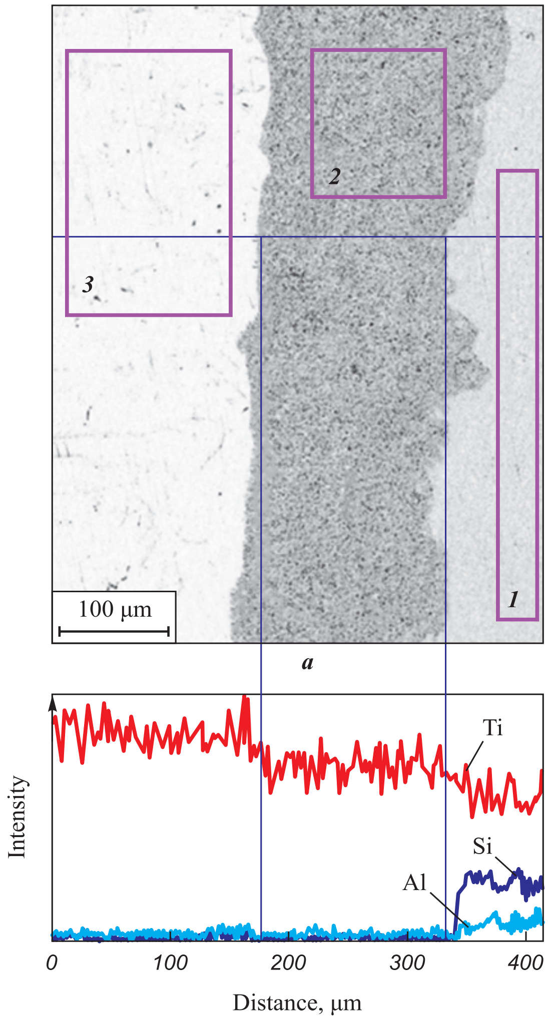


Fig. 3. *X*-ray data of a synthesized alloy based on the Ti–Al–Si system (1st layer)

Рис. 3. Данные РФА синтезированного сплава на основе системы Ti–Al–Si (1-й слой)



Range	C	Al	Si	Ti	Phase
1	3.92	8.08	26.21	61.78	$Ti_{20}Al_3Si_9$
2	39.23	1.99	0.46	58.32	$TiC_{0.66}$
3	6.16	0.27	0.27	90.60	Ti (substrate)

b

Fig. 4. Concentration profile of element distribution between the layers (***a***) and EDA data, wt. % (***b***)

Рис. 4. Концентрационный профиль распределения элементов между слоями (***a***) и данные ЭДА, мас. % (***b***)

of the main phase $Ti_{20}Al_3Si_9$ (calculated by the Rietveld method) was 87 %, the phase Ti_3Al – 13 %. According to the data obtained by energy-dispersive analysis (EDA), the 2nd layer conforms to the phase $TiC_{0.66}$ (cubic structure *Fm*3*m*), and the 3rd layer conforms to titanium in a substrate made of titanium foil. The transition zones between the layers do not exceed 10–15 µm.

According to the concentration profile of the element distribution between the layers (Fig. 4) a slight increase of aluminium concentration in the boundary area between Ti/TiC layers can be observed due to the melting of the titanium foil caused by heat release during reaction in the Ti–C layer and diffusion of aluminium into the titanium substrate through the Ti–C layer. Upon that, the depth of Al diffusion into the Ti substrate is rather shallow ~30 µm. Silicon concentration during transition from the Ti–Al–Si layer to Ti–C drops sharply and remains at zero values in the 2nd and 3rd layers.

Microhardness (H_μ) of each of the layers of the synthesized gradient material is presented in Table. The highest H_μ value (~12.3 GPa) corresponds to a Ti–C-based layer, the lowest is for a titanium substrate (4.1 GPa). Microhardness of Ti–Al–Si layer is ~10.1 GPa.

When an indenter is inserted at a load of more than 30 kg into a Ti–Al–Si-based layer, radial cracks are formed in the corners of the Vickers pyramid imprints in the area of maximum tension stresses (Fig. 5, *a*, *b*). The formation of main concomitant cracks and their

Microhardness values in layers of the synthesized material

Значения микротвердости в слоях синтезированного материала

Layer		Microhardness H_μ, GPa	Cracking resistance coefficient K_{1c}, MPa·m$^{1/2}$
No.	Composition		
1	Ti–Al–Si	10.1	5.1–5.7 (this work)
2	Ti–C	12.3	2.5–4.3 [24]
3	Ti-substrate	4.1	50–55 [25]

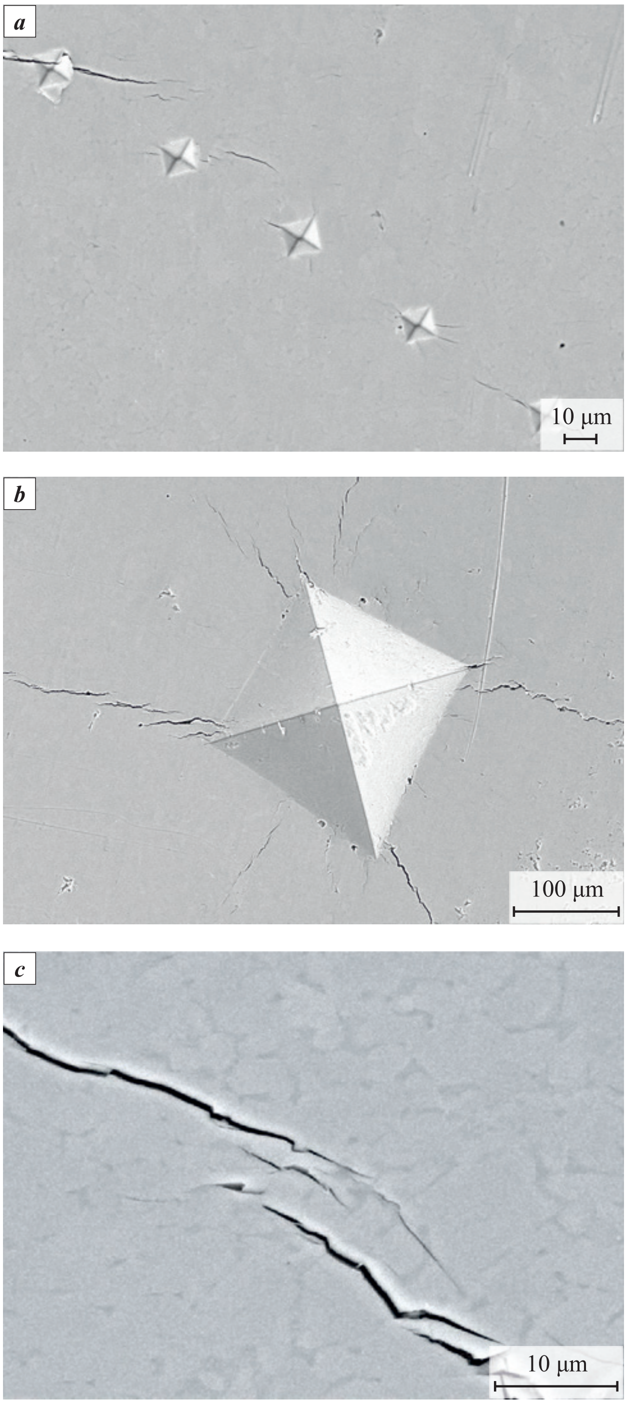


Fig. 5. Micrographics of indentation after measuring microhardness H_μ (***a***) and Vickers hardness *HV* (***b***), as well as an enlarged fragment of crack propagation in Ti–Al–Si layer (***c***)

Рис. 5. Микрофотографии отпечатков индентора после измерения микротвердости H_μ (***a***) и твердости по Виккерсу *HV* (***b***), а также увеличенный фрагмент распространения трещины в слое на основе Ti–Al–Si (***c***)

branching, as well as the fusion of cracks with microheterogeneities and structural defects are observed (Fig. 5, *c*). One of the reasons for this is that the micropores affect the crack propagation process. The rectilinear nature of crack propagation indicates a high fragility of the material. It is noteworthy that the cracks propagate both through and around the grains (Fig. 5, *c*). Calculated by the Palmquist method [20] for a Ti–Al–Si-based layer, the crack resistance coefficient is $K_{1c} = 5.1{\div}5.7$ MPa·m$^{1/2}$. The measurements were taken at an indenter load of 100 g using the formula:

$$K_{1c} = 0{,}0028\sqrt{HV}\sqrt{P}c^{-1},$$

where *P* – indentation load, *c* – total length of the crack from the indenter, mm.

The following results can be given to compare the resulting value of K_{1c}. According to work [21], the crack resistance coefficient of Ti–Al–Si-based alloys can reach values from 0.7 to 1.7 MPa·m$^{1/2}$, while Ti_5Si_3 silicide has K_{1c} = 7 MPa·m$^{1/2}$ [22]. It is noted in work [23] that materials based on Ti–Al_3Ti with a volume fraction of Al_3Ti phase equal to 86, 80, and 65 % are characterized by high crack resistance values at the level of 15, 23, and 29 MPa·m$^{1/2}$, respectively. The measurement results of K_{1c} depend on the size and direction of movement of cracks, pores, interphase transformations, and the magnitude of loads on the material.

Conclusion

A metal-carbide-intermetallic layer material based on (Ti–Al–Si)/(Ti–C)/Ti was synthesized by SHS pressing. The heat released as a result of SHS reactions in the layers and the subsequent pressing of the hot product ensured the required diffusion through the boundaries (Ti–Al–Si)/(Ti–C) and (Ti–C)/Ti and resulted in the formation of a solid permanent joint between the layers with a transition zone thickness between them about 10÷15 µm. Combustion product of Ti–Al–Si layer exhibits two phases: triple phase $Ti_{20}Al_3Si_9$ and Ti_3Al with a content of 87 and 13 wt. %, respectively. The microhardness of the synthesized combustion product of Ti–Al–Si layer was ~10.1 GPa, crack resistance coefficient $K_{1c} = 5.1{\div}5.7$ MPa·m$^{1/2}$. The obtained results can be used in the development of methods for applying protective coatings/layers to the surface of titanium products.

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