

## PHASE COMPOSITION AND STRUCTURE OF POWDER MATERIALS OF THE Ti–Al–B/TiB<sub>2</sub> SYSTEM AFTER VACUUM SINTERING AND HIGH-TEMPERATURE SYNTHESIS

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*The structure and phase composition of powder materials of the Ti–Al–B system formed in the process of vacuum sintering and synthesis in the mode of high temperature self-propagating synthesis (SHS) are considered depending on the combination of components in the form of elementary powders (Ti, Al and B) and using the finished titanium diboride (TiB<sub>2</sub>) compound. The proportions of the components were calculated in such a way that the number of interacting elements was sufficient to form a two-phase TiAl<sub>3</sub> + TiB<sub>2</sub> composition. When sintering the Ti + Al + TiB<sub>2</sub> mixture, the diboride is retained, but the presence of TiB is noted as a result of the redistribution of boron due to its migration into free titanium. It was discovered that sintering of compacts from the mixture based on elemental powders (Ti, Al and B) occurs under conditions of high exothermic effect, as a result of which the samples were destroyed. This made it possible to use this mixture under conditions of high-temperature synthesis in combustion mode. As a result of both vacuum sintering and SHS compacting, aluminide TiAl<sub>3</sub> and titanium diboride (TiB<sub>2</sub>) are mainly formed from a mixture of elemental powders (Ti, Al and B). In this case, some transition phases can be observed. It is shown that after the synthesis of the Ti + Al + B mixture, it is possible to obtain a powder product from which compacts are well sintered while maintaining their shape with a slight shrinkage.*

**Keywords:** Titanium aluminides, titanium borides, reaction sintering, porosity, structure, phase composition.

### INTRODUCTION

Metal matrix composites (MMCs) are of interest in various industries due to their physical, mechanical, and chemical properties. The main feature of these materials is a unique combination of hardness, strength, ductility, and good resistance to wear and oxidation at high temperatures. The MMC combines various groups of matrix material and strengthening phase. The matrix can be made of aluminum, titanium, or their compounds, other metals and their alloys. The most commonly used strengthening phases are titanium carbides, silicides, and borides.

Ti–B, Ti–Al and Ti–Al–B systems have been studied for many years both from the viewpoint of studying fundamental physical and chemical processes and in the context of specific applications in mechanical engineering, transport, and chemical industry. They have gained even greater popularity due to the development of new technological areas related to additive manufacturing.

The studies involved both aluminum-matrix compositions with inclusions of titanium borides and titanium-matrix composites. Many works are devoted to titanium matrix composites due to their high specific strength, high specific modulus, and durability at high temperatures [1–6]. In particular, Ti–Al alloys are used in the aerospace, automotive, and turbine industries due to their significant high-temperature ductility and heat resistance, corrosion

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resistance, and low density [4–7]. The system is distinguished by the variety of existing phases:  $\alpha$ -Ti,  $\beta$ -Ti,  $\alpha$ -Ti<sub>3</sub>Al and  $\gamma$ -TiAl, TiAl<sub>2</sub>, and TiAl<sub>3</sub> compounds. The formation of the TiAl<sub>3</sub> phase, which has the lowest free energy of formation in this system, is energetically and thermodynamically more advantageous in the Ti–Al system. According to various sources, its formation enthalpy is about –146.5 kJ/mol. The TiAl<sub>3</sub> compound has excellent strength, lowest density and high oxidation properties. But the use of Ti–Al alloys is limited by the fragility of the Ti–Al compounds at room temperature.

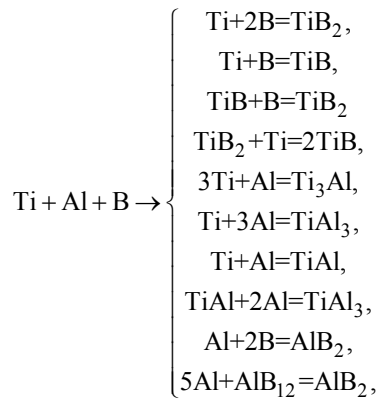
Boron and its compounds are often used as a modifying additive in Ti–Al based alloys [5–10]. In alloys based on  $\alpha$ -Ti with low aluminum content, it is most often preferred to add ready-made titanium diboride (TiB<sub>2</sub>) to avoid unwanted exothermic reactions [8]. As it is known, the Ti–Al–B system is a ternary system with interacting components. The nature of the interaction of element pairs (Ti–Al, Ti–B, and Al–B) determines the form of their classical equilibrium state diagrams of binary systems [11–12]. Since all elements form chemical compounds with each other to one degree or another, there is a problem of predicting the final phase composition of the resulting composite based on this ternary system. In this case, it is necessary to take into account possible cross-reactions, their likelihood of occurring in the presence of a third component, and the stability of the new compounds formed under conditions of competing diffusion-reaction processes. The set of possible reactions is reduced in the case of using ready-made titanium diboride (TiB<sub>2</sub>) powder, since this compound is very refractory, and there is no free boron at the first stage of sintering. The dominant process will be the liquid-phase interaction of titanium with aluminum with the formation of intermetallics. At the same time, some degree of degradation of TiB<sub>2</sub> diboride is allowed due to the diffusion redistribution of boron in the presence of free titanium [13].

As is well known, boron actively interacts with titanium; their system is well studied and underlies many theoretical aspects of the high temperature self-propagating synthesis (SHS) [14]. A number of compounds TiB, TiB<sub>2</sub>, Ti<sub>3</sub>B<sub>4</sub> are also formed in the Ti–B system [11]. The most well-known and in demand are titanium boride (TiB) and diboride (TiB<sub>2</sub>). The existence of a whole series of unstable boride phases is assumed [15–16], but their synthesis, as a rule, occurs under nonequilibrium SHS conditions, and the region of the existence is very limited. The mutual solubility of Ti and B in the solid state is practically non-existent. The direct interaction of titanium and boron during preheating can occur in the SHS mode with the formation of titanium diboride at the initial stage:  $\text{Ti} + 2\text{B} \rightarrow \text{TiB}_2$ . The Gibbs energy and the formation enthalpy of titanium diboride are lower than those of titanium monoboride (for solid-phase reactions  $\Delta H = -293$  and  $-160$  kJ/mol, respectively) [16]. In the presence of excess boron, it is energetically favorable to convert TiB into TiB<sub>2</sub> in accordance with the chemical reaction  $\text{TiB} + \text{B} = \text{TiB}_2$ . When there is an excess of titanium in the system, the chemical reaction  $\text{TiB}_2 + \text{Ti} = 2\text{TiB}$  becomes more favorable.

In the Al–B system, aluminum also interacts with boron to form diboride AlB<sub>2</sub> and dodecaboride AlB<sub>12</sub> amid a very low mutual solubility of aluminum and boron in the solid state. But for the synthesis of aluminum borides, the amount of boron must be large enough, and the titanium content insignificant, so that these interaction reactions are not blocked by the titanium.

The interaction between elements in the Ti–Al, Ti–B, and Al–B systems is highly exothermic in nature with different amounts of heat generated. This allows the use of powder metallurgy methods such as the high temperature self-propagating synthesis (SHS), mechanical activation, spark plasma sintering (SPS), hot isostatic pressing (HIP), etc. [17–19]. For some technological tasks, sintering of compacts from powder compositions based on the Ti–Al–B system in a vacuum or in a protective/inert environment is used [20]. Under vacuum reaction sintering conditions, it is impossible to unambiguously predict the structure formation of compacts from the Ti + Al + B powder mixture. In this case, it is more convenient to focus on those expected reactions that can occur as a result of heating the powder material. At the selected ratios of components under the conditions of reaction sintering of Ti + Al + B powder mixtures, serial-parallel chains of reactions can be realized, as a result of which not only the energy contribution to the general state of the sintered compacts changes at each stage, but also their phase pattern.

If mixtures of elemental powders (Ti, Al, and B) are considered, then the most likely reactions can be represented as follows:



where the formation and existence of some compounds can occur in a very narrow time and temperature ranges. It is possible that current thermodynamic conditions may be such that some compounds will not be formed at all. Only the final set of phases and their distribution will be observed in the sintered material. Thus, it is difficult to predict the structural-phase state of composites from the Ti + Al + B mixture during alloy formation. Understanding how reaction-diffusion processes are implemented in a given powder system at certain component ratios is a key point for controlling the structure in further use of these composites. A more detailed study of the features of the component interaction in powder compositions based on the Ti–Al–B system under certain technological conditions makes these studies relevant.

The purpose of this work is to study experimentally the structure formation under vacuum sintering conditions and SHS mode of powder materials of the Ti–Al–B system with different combinations of components and identical ratios of the elements (Ti, Al, and B). Boron was added to a mixture of titanium and aluminum powders, both in the form of an elemental powder and a powder of the finished titanium diboride (TiB<sub>2</sub>) compound.

## EXPERIMENTAL MATERIAL AND PROCEDURE

The powder mixtures based on the Ti–Al–B system were selected for the study, where two options for combining components were used: a) a mixture of the titanium powder, aluminum powder, and industrial titanium diboride (TiB<sub>2</sub>) powder and b) a simple mixture of elemental titanium, aluminum, and boron powders. In both cases, the amount of powder components used was calculated in such a way that the total number of elements was sufficient to form a metal matrix composite with a potential equimolar ratio of the synthesized TiAl<sub>3</sub> + TiB<sub>2</sub> phases. The percentage distribution of components in the considered mixture options is presented in Table 1.

Grade TPP-8 titanium powders (main fraction with  $d < 160 \mu\text{m}$ ) (AVISMA, Berezniki), PA-4 aluminum powders ( $d < 100 \mu\text{m}$ ), grade A amorphous boron ( $d < 10 \mu\text{m}$ ), and class H boron diboride ( $d < 20 \mu\text{m}$ ) were the initial materials for the reaction mixtures. The morphology of the powders is shown in Figure 1.

The powder mixtures were pressed into cylindrical specimens with a diameter of 10 mm, height of  $10^{-15}$  mm, and residual porosity of 25 to 30% after mixing in an axial mixer. The additive method was used to calculate the porosity and compact density (theoretical) of the selected compositions. The data from the initial components were used for raw compacts, and the data from X-ray diffraction analysis with the actual phase distribution were used after vacuum sintering. The specimens were sintered in a vacuum furnace at 1000–1300°C with a holding time of 60 min. The holding time was determined based on the condition of guaranteed completion of potential reactions in the Ti–Al–B system. The studies were carried out on the implementation of the SHS mode under conditions of layer-by-layer combustion in an argon environment, where compressed specimens with a diameter of 30 mm and a height of 40 mm were used, since the powder mixture of pure Ti + Al + B components is an exothermic system.

The Ti–Al–B composite powders were studied using the equipment of the Shared Use Center “Nanotech” of the ISPMS SB RAS by the methods of X-ray diffraction analysis using CuK $\alpha$  radiation with a DRON-8 diffractometer (Burevestnik, Russia), optical metallography (AXIOVERT-200MAT, Zeiss, Germany), and scanning electron microscopy (EVO 50, Zeiss, Germany).

TABLE 1. Ti–Al–B System Powder Compositions

Target composition with planned ratio of synthesized phases		Components, wt.%			
		Ti	Al	B	TiB <sub>2</sub>
TiAl <sub>3</sub> + TiB <sub>2</sub>	Option 1	24.2	40.8	–	35.0
	Option 2	48.3	40.8	10.9	–

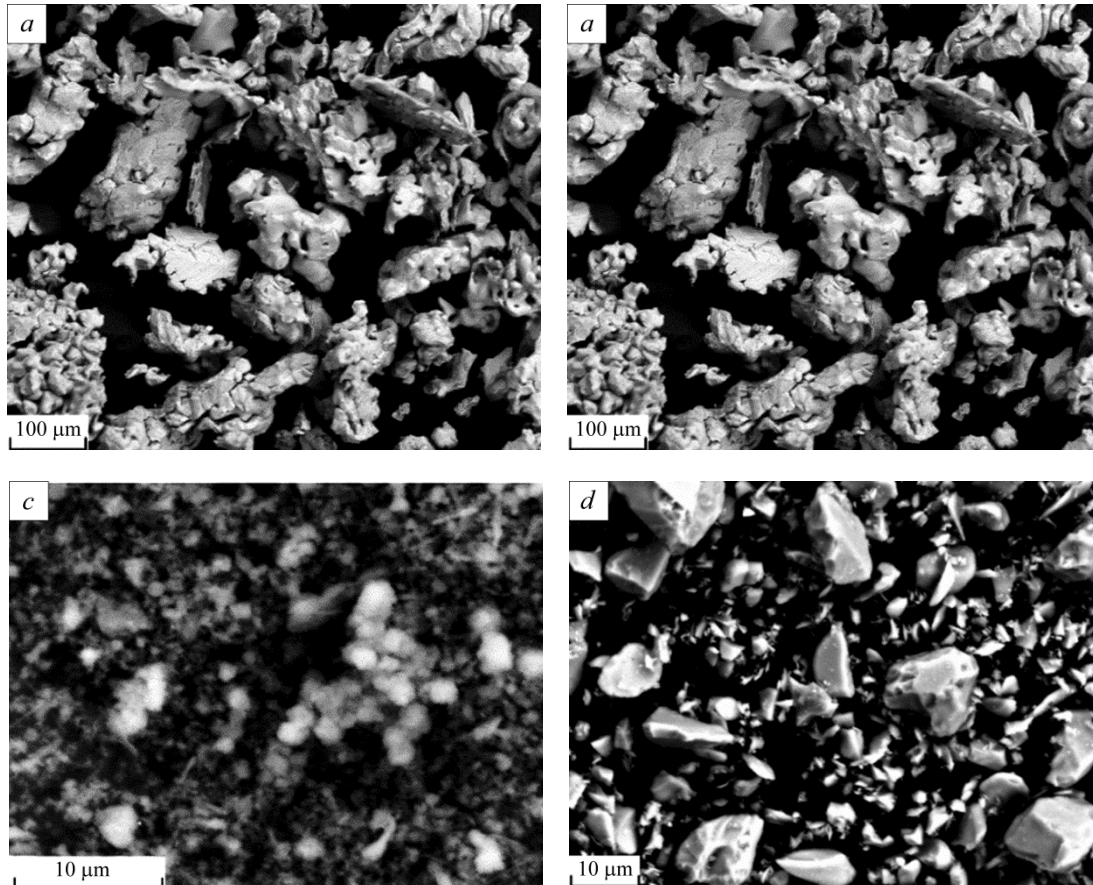


Fig. 1. Morphology of the initial powders including TPP-8 titanium (a), PA-4 aluminum (b), amorphous grade A boron (c), and class H boron diboride powders (d).

## RESULTS AND DISCUSSION

The use of the titanium diboride powder in the Ti + Al + TiB<sub>2</sub> mixture with a component ratio corresponding to the intended synthesis of the two-phase TiAl<sub>3</sub> + TiB<sub>2</sub> composite avoids the additional energy contribution from the reaction of the Ti–B system. At the same time, liquid-phase alloy formation occurs in another pair of the Ti–Al components during the sintering process. The results of the interaction of titanium diboride and the aluminum-titanium matrix under vacuum sintering at 1000°C are illustrated by Figs. 2 and 3. The formed structure of sintered specimens is characterized by high residual porosity, reaching up to 45%, and its heterogeneous distribution over the volume. The reaction-diffusion processes in the mixture of titanium, aluminum, and boron diboride with a large amount of aluminum lead to a noticeable volumetric growth of compacts. As the microstructure of the sintered sample shows (Fig. 2a), the

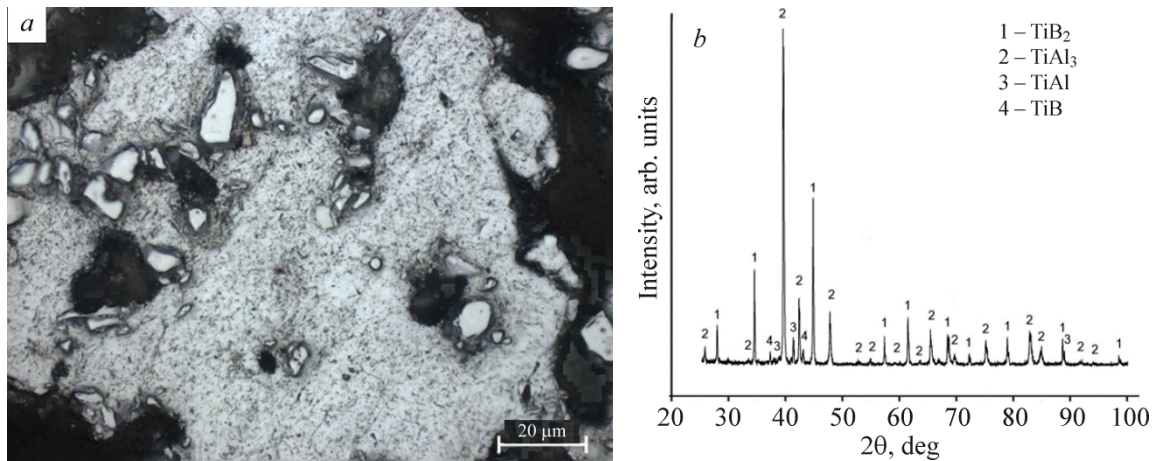


Fig. 2. Optical image of the microstructure (*a*) and X-ray diffraction pattern (*b*) of the samples from the Ti + Al + TiB<sub>2</sub> mixture sintered in vacuum at  $T = 1300^{\circ}\text{C}$ .

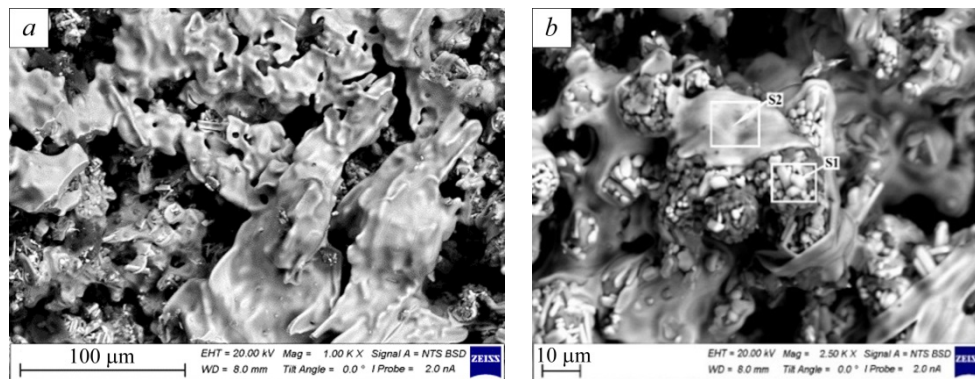


Fig. 3. SEM image at different magnifications ( $\times 1000$  (*a*) and  $\times 2500$  (*b*)) and distribution of the Ti and Al elements in the samples from the Ti + Al + TiB<sub>2</sub> mixture sintered in vacuum at  $T = 1300^{\circ}\text{C}$ .

titanium diboride particles generally retained their morphology without formation of noticeable diffusion layers with the titanium-aluminum matrix.

In this case, the liquid-phase interaction of the metal matrix components corresponded to the standard behavior of the components in the Ti–Al powder system. According to the Ti–Al phase diagram, there is a large set of phases in the system, which includes  $\alpha$ -Ti,  $\beta$ -Ti, and  $\alpha_2$ -Ti<sub>3</sub>Al and  $\gamma$ -TiAl, TiAl<sub>2</sub>, and TiAl<sub>3</sub> compounds and a large amount of limitedly disordered  $\beta$ -solid solution. Since the amount of aluminum powder sufficient to form TiAl<sub>3</sub> aluminide was used in the mixture, this intermetallic compound was found in the final structure of the matrix (Fig. 2*b*) along with the original TiB<sub>2</sub> particles. According to X-ray phase analysis, in addition to the main planned phases TiAl<sub>3</sub> (up to 62.0 vol.%) and TiB<sub>2</sub> (up to 33.0 vol.%), traces of the intermediate compound TiAl (up to 2 vol.%) and TiB monoboride (up to 3 vol.%) were also found in the sintered compact. A large amount of aluminum in the powder mixture ensured the formation in a large volume and long-term existence of the liquid phase, which surrounded the diboride particles (Fig. 3*a*). However, the wettability of TiB<sub>2</sub> by the liquid phase is insufficient to have a noticeable effect on solid particles, although boron diffusion with the formation of titanium monoborides is possible. The distribution map of Ti and Al elements in individual scanning areas shows that the migration of aluminum into TiB<sub>2</sub> particles is also not excluded (Fig. 3*b* and Table 2).

TABLE 2. Element Distribution Map

At.%	Al	Ti
Spectrum 1	15.18	84.82
Spectrum 2	37.23	62.77

As mentioned above, the interaction between elements in the Ti–Al, Ti–B, and Al–B systems is exothermic in nature with different amounts of generated heat. Under conditions of vacuum reaction sintering, it is impossible to predict unambiguously the behavior of compacts made of the powder mixture based on the Ti–Al–B ternary system. In the case of a similar proportion of elements, but using individual powders of titanium, aluminum, and boron (Ti + Al + B), sintering of compacts in a vacuum furnace already at 1000°C leads to destruction of the specimens as a result of intense heat generation with loss of their original shape. The exothermic reactions are ensured by not only the interaction of aluminum and titanium, but also titanium and boron. The direct interaction of titanium and boron during heating can proceed in the high-temperature synthesis mode with the formation of titanium diboride  $\text{Ti} + 2\text{B} \rightarrow \text{TiB}_2$  at the initial stage.

In the Ti + Al + B mixture, this process occurs amid other competing reactions:  $3\text{Ti} + \text{Al} \rightarrow \text{Ti}_3\text{Al}$ ,  $\text{Ti} + \text{Al} \rightarrow \text{TiAl}$ , and  $\text{Ti} + 3\text{Al} \rightarrow \text{TiAl}_3$ . Since all processes in the powder systems take place under nonequilibrium conditions with the mutual influence of competing and accompanying reactions, a gradual formation of some phases is possible during the sintering process followed by their decomposition and transition to other phases. In this case, the volume of the formed liquid phase and its lifetime were quite large. This promoted intensive alloy formation followed by recrystallization (Fig. 4). As a result,  $\text{TiAl}_3$  (up to 41.2 vol.%) and  $\text{TiB}_2$  (up to 41.9 vol.%) phases were mainly formed. The nonequilibrium phases based on  $\text{Ti}_2\text{Al}_5$  (up to 14.6 vol.%) and residues of unreacted titanium up to 2.3 vol.% were also recorded (Fig. 4c).

The highly exothermic behavior of the titanium, aluminum, and boron powder mixture in a proportion corresponding to the target composition  $\text{TiAl}_3 + \text{TiB}_2$  allowed applying the SHS method in the layer-by-layer combustion mode. As a result, a brittle sinter was formed that was easily crushed into small fractions. The products of this reaction are shown in Fig. 5, where the internal structure of the fragments of the synthesized powder product is close to the microstructure of the vacuum-sintered compact from the Ti + Al + B mixture.

The X-ray phase analysis (Fig. 5c) showed that as a result of high-temperature synthesis in the layer-by-layer combustion mode of compacts from the Ti + Al + B mixture, a nonequilibrium structure is formed from the reaction products of titanium, aluminum, and boron with residues of unreacted titanium the volume of which is no more than 1 %. The basis of the synthesized powder material was the  $\text{TiAl}_3$  (about 66.3 vol.%) and  $\text{TiB}_2$  (up to 33.7 vol.%) phases. The difference from the product sintered from the same mixture shows that the vacuum sintering mode during the volumetric heating of the compact actually turned into a thermal explosion when the liquid phase appeared preventing the reaction from being completed. The layer-by-layer combustion in the SHS mode made it possible to gradually involve the components in the reactions, which allows fully completing them.

The synthesized powder product, after crushing and sifting out the fine fraction ( $d < 50 \mu\text{m}$ ), was pressed into samples for secondary heat treatment under vacuum sintering conditions at 1300°C. The results of this sintering are shown in Fig. 6, where a more homogeneous structure is observed compared to the first version of the Ti + Al +  $\text{TiB}_2$  mixture. It can also be noted that the two-stage heat treatment of the simple Ti + Al + B mixture (the second option including the primary SHS process, crushing, and sifting out the powder product of the corresponding fraction, and pressing followed by sintering) mainly retains the same two-phase structure close to the target qualitative  $\text{TiAl}_3 + \text{TiB}_2$  composition as with simple vacuum sintering of this mixture.

However, the morphology of boride and intermetallic grains is slightly different (Fig. 6a). The dynamic SHS process prevented the formation of extended needles of boride inclusions. The element distribution map (Table 3) demonstrates the presence of aluminum and titanium in local zones in accordance with the formed phase composition, where the S1 part of the spectrum is mainly occupied by  $\text{TiAl}_3$  aluminide with small inclusions of formed  $\text{TiB}_2$ . The spectrum of the S2 region can be considered as a particle of titanium diboride with a small inclusion of  $\text{TiAl}_3$  aluminide.



TABLE 3. Element Distribution Map

At.%	Al	Ti
Spectrum 1	57.40	42.60
Spectrum 2	17.79	82.21

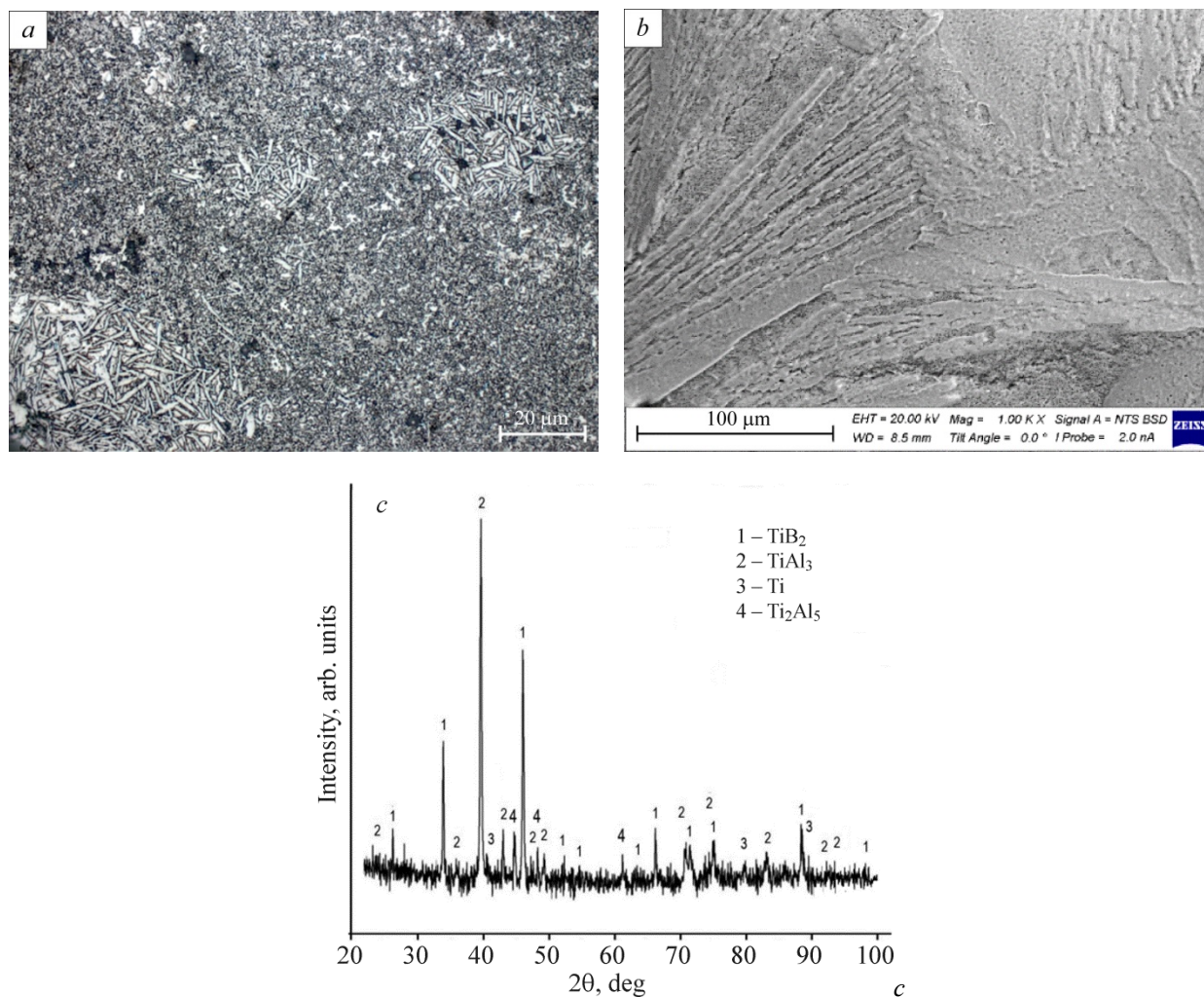


Fig. 4. Optical (a) and SEM images (b) of the microstructure and X-ray diffraction pattern (c) of specimens from the Ti + Al + B mixture sintered in vacuum at  $T = 1000^{\circ}\text{C}$ .

In contrast to direct sintering of compacts from the Ti + Al + B mixture, compacts from the synthesized powder not only retained their shape, but also underwent shrinkage of up to 5% during vacuum sintering. At the same time, this shrinkage was not sizable enough to reduce significantly the residual porosity. This can be explained by the fact that the formed rigid frame of refractory particles did not lend itself well to solid-phase sintering.

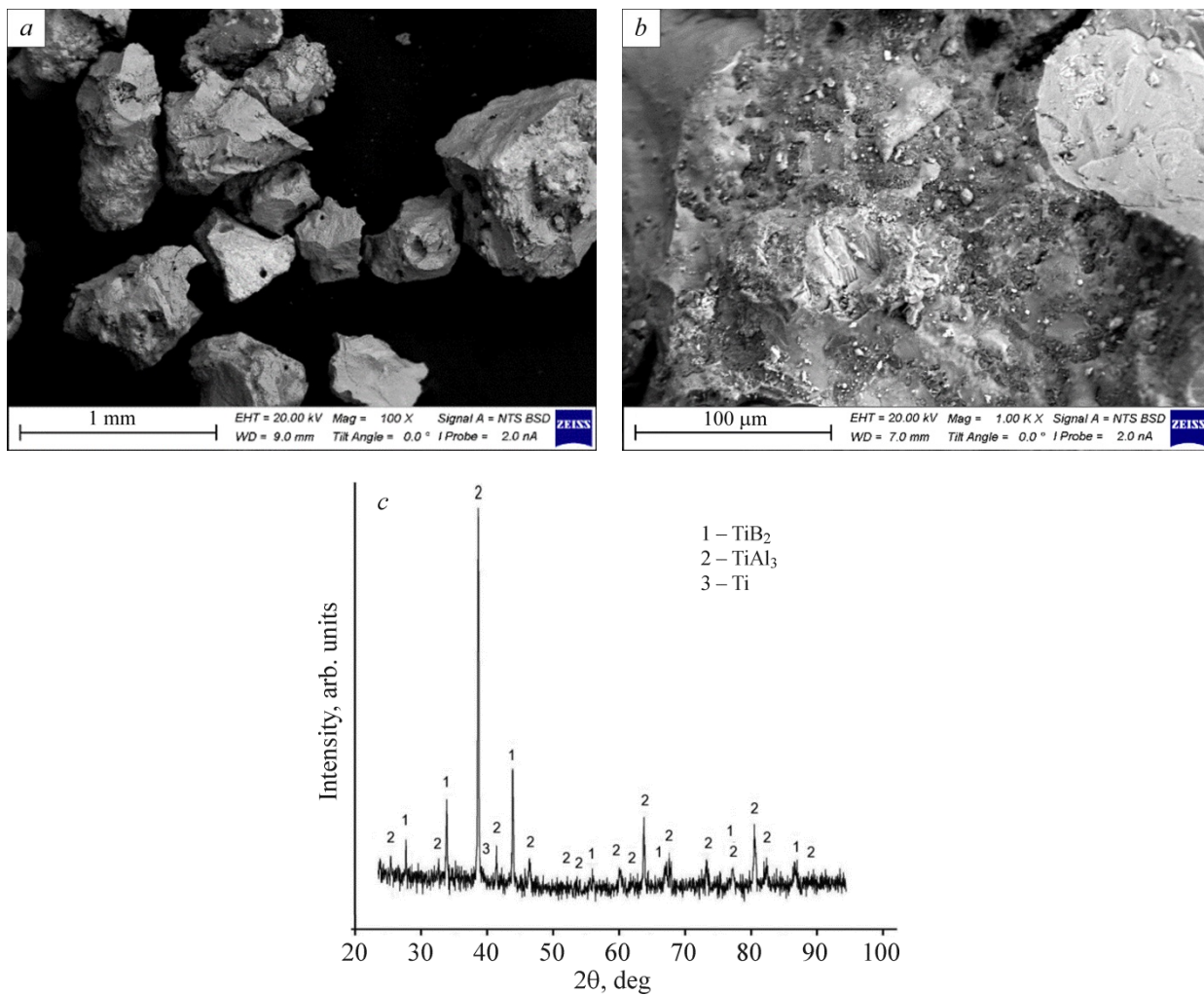


Fig. 5. Morphology (a), optical image of the microstructure (b), and X-ray diffraction pattern (c) of the powder product obtained by the SHS method from the Ti + Al + B mixture.

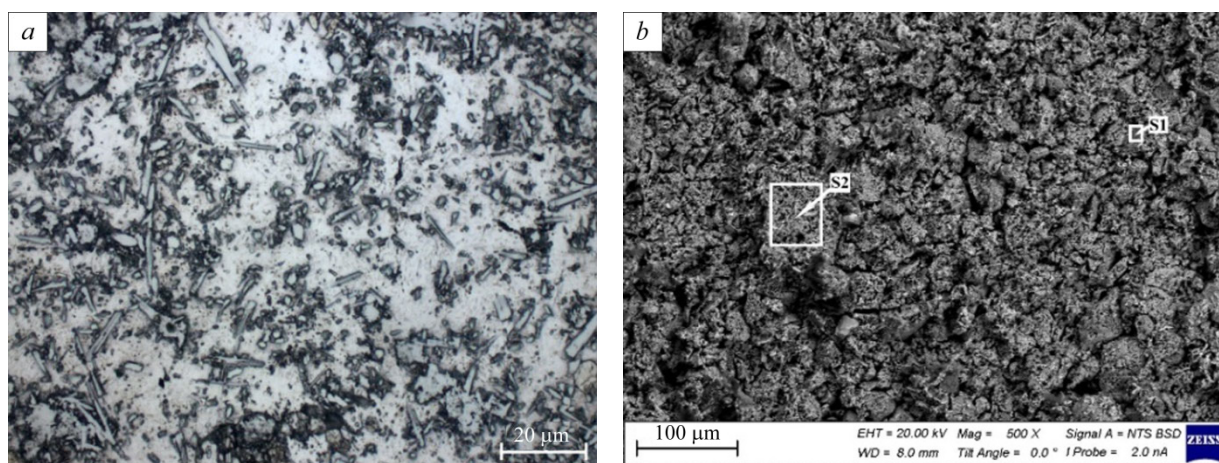


Fig. 6. Optical (a) and SEM images (b) of the microstructure with the distribution of Ti and Al elements in individual areas.



## CONCLUSIONS

Vacuum sintering and SHS of powder mixtures of the Ti–Al–B system with different methods of introducing boron in the form of the TiB<sub>2</sub> compound or amorphous boron powder at the same ratio of elements in order to obtain the two-phase composite TiAl<sub>3</sub> + TiB<sub>2</sub> showed that in the case of the Ti + Al + TiB<sub>2</sub> powder mixture, partial migration of boron atoms from the refractory TiB<sub>2</sub> compound can occur. As a result, a small amount of titanium monoboride TiB is formed. In the presence of TiB<sub>2</sub>, the titanium and aluminum powder components react with each other according to selected ratios to form TiAl<sub>3</sub> aluminide, but some of their reaction products remain in the form of monoaluminide TiAl.

When using amorphous boron powder in the Ti + Al + B mixture with a similar calculated element contents, vacuum sintering at 1000°C leads to the destruction of compacts from this mixture. The total exothermic effect from cross-reactions between titanium and boron as well as between titanium and aluminum actually transformed the vacuum sintering state into a thermal explosion. As a result, in addition to the planned TiAl<sub>3</sub> and TiB<sub>2</sub> phases, nonequilibrium phases based on Ti<sub>2</sub>Al<sub>5</sub> and residues of unreacted titanium were discovered. The Ti + Al + B mixture released as a result of the reaction interaction of the components in the powder mixture turned out to be sufficient to implement the SHS process in the layer-by-layer combustion mode. The powder material obtained in this way mainly consisted of the calculated TiAl<sub>3</sub> and TiB<sub>2</sub> phases with small titanium residues (less than 1 vol.%). The vacuum sintering of compacts from the resulting SHS powder with a two-phase structure retained the phase composition with a slight correction of the volume content due to solid-phase homogenizing sintering. As a result, the samples maintained their shape and shrank slightly, thereby reducing the residual porosity.

## COMPLIANCE WITH ETHICAL STANDARDS

### Author contributions

E.N.K.: conceptualization, methodology, original draft preparation, design of the experiments, formal analysis, supervision, discussion of the results, and editing; V.V.K.: investigation, formal analysis, and discussion of the results. All authors have read and agreed to the published version of the manuscript.

### Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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### Financial interests

The authors have no relevant financial or non-financial interests to disclose.

### Institutional review board statement

Applicable.

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