TiB / 30 wt.% Ti layered composite material obtained by free SHS compression on a Ti6Al4V titanium alloy

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Abstract. In this work, a layered composite material based on TiB / 30 wt.% Ti on a Ti6Al4V titanium alloy was obtained by free SHS compression. Powders of titanium and amorphous black boron were used as initial components. The structural features, phase composition, porosity and distribution of microhardness along the height of the layered material were studied. It was shown that, as a result of synthesis and subsequent high-temperature deformation, the following phases were formed in the layered composite material: TiB, TiB2, Ti0.76N. It was found that at a compression pressure of 50 MPa a layered composite material with a porosity of up to 1-2% is formed. It was found that the delay time before applying pressure affects the size of the diffusion zone in the range of 10-50 microns. Due to the formation of hardening phases in the composite material on Ti6Al4V, the microhardness of its surface increased from 2.4 to 11 GPa.

1. Introduction
Titanium fame is widely used in the aviation industry due to its low density, high strength and hardness, corrosion resistance and elevated temperatures [1-4]. These include Ti6Al4V alloy, which is used for the manufacture of large-sized welded and prefabricated aircraft structures. Today, there are many different methods aimed at improving the physicomechanical properties of Ti6Al4V titanium alloys and obtaining composite materials based on them [5-8]. Each of the production methods has its own characteristics and areas of application. A lot of work has been devoted to the production of layered composite materials and multi-layered based on TiB or TiC on the titanium alloy Ti6Al4V [9-11].

A promising method for producing layered composite materials (Layered composite material or LCM) on titanium alloys is free SHS compression [12, 13]. This method combines in one technological stage the synthesis of the initial powder components and their subsequent high-temperature deformation. Due to the selection of the composition of the initial components, the location of macro layers, technological and structural parameters of the process, it is possible to vary the structure and physico-mechanical properties of the resulting composite materials over a wide range. Herewith, taking into account that the synthesis and preparation of a compact composite material takes tens of seconds, this confirms the great advantages of the method used.
This article is about obtaining a layered composite material based on TiB / 30 wt.% Ti on a Ti6Al4V titanium alloy by free SHS compression and studying its structure, phase composition, and microhardness.

2. Objects and methods of research
To obtain LCM, a model composition with an excess titanium content was taken from the calculation that, after synthesis, a TiB-30 wt.% Ti compound is formed. From pre-mixed powders, 81.6 wt.% Titanium (> 99.1%, 45 μm) and 18.4 wt.% Boron (amorphous black boron, 99%, 5 μm) were formed into cylindrical blanks with a diameter of 30 mm, a height of 15 mm, and a relative density of 0.55 - 0.6 and placed them on a substrate of Ti6Al4V with dimensions of 30x30 mm and a thickness of 7 mm. After the initiation of combustion of the formed billet by a tungsten spiral, the passage of the combustion wave in the SHS mode, a delay time of 1 s, and a press plunger speed of 60 mm / s, the synthesized material was compressed with a force of 50 and 100 MPa. After compression and holding time under a pressure of 5 s, the sample was placed in a furnace for 2 h, followed by cooling at a temperature of 500 °C to relieve thermoelastic stresses. As shown by preliminary experiments at temperatures below 500 °C, due to thermoelastic stresses, cracking of the obtained samples occurs.

For the implementation of structural studies of synthesized powder materials the equipment of the Distributed Center for Collective Use of ISMAN was used: the high-resolution field-scanning electron microscope Carl Zeiss Ultraplus (Germany), the powder X-ray diffractometer ARL XTRA, and other certified methods and techniques.

3. Discussion
As a result of the experimental work, by the method of free SHS compression, LCM of 30x30 mm in size with a ceramic material height based on titanium boride of 2 and 5 mm were obtained, depending on the applied pressure. It was found that at a compression pressure of 100 MPa, the porosity in the material is practically absent, however, in this case, main cracks are observed at the points of its interaction with the Ti6Al4V substrate. The destruction of the material occurs during cooling due to the loss of its ductile properties and increased external pressure. The study of the structure of the obtained SCMs was further conducted for a compression pressure of 50 MPa.

Based on the XRD results, it was found that during the chemical interaction of the initial components of boron and titanium, the main phase is formed - titanium monoboride, as well as the titanium diboride phase. Because of titanium was taken in excess, in the material it acted as a matrix. Due to experiments in the open air and high chemical activity of excess titanium, an additional Ti0.76N phase was formed (cubic crystal lattice, a = 4.235 Å), which, having increased hardness values (up to 20 GPa), increases the mechanical characteristics of the LCM surface.

The synthesis of the studied material proceeds with the melting of titanium (the combustion temperature of the selected composition was 1800 °C), which in the liquid state, under the action of gravity and external pressure, fills the pores and lower layers, thereby depleting or enriching the volumes in the material. According to the Ti-B phase diagram, the crystallization of titanium monoboride occurs by peritectic transformation from a Ti-B melt, which leads to the fact that initially TiB2 grains remain due to lack of free titanium in the material and crystallize in the form of hexagonal structures with sizes less than 1 μm (Figure 1). As you approach the substrate, the proportion of titanium increases, and the number of grains of diboride and titanium boride decreases.
When compressed due to the contact of the surface of the synthesized material with the plunger of the press and the Ti6Al4V substrate, conductive heat loss occurs, which leads to faster cooling of the surface layers compared to the central part. This limits the residence time of the material at elevated temperatures and prevents grain growth. As a result of this cooling, finer titanium boride grains 1-10 μm in size are observed on the surface and next to the substrate in the obtained LCM, and as they approach the central part of the LCM, titanium boride grains grow to larger sizes up to 20 microns. In this case, the orientation of titanium boride grains along the direction of flow of the material during deformation is observed (Figure 2). The texture of the obtained material is also confirmed by the results of the XRD [13]. It was found that due to faster cooling of the material surface, the LCM surface has a porosity of up to 10-20%, which decreases as it approaches the central part to 1-2%. Due to the high-temperature shear deformation of the combustion products during SHS compression, the synthesized material moves into pores and defects, which reduces the porosity and defect formation of LCM.

Figure 1. Results of SEM LCM

Figure 2. Direction of material flow during deformation
Under the experimental conditions, the surface layer of the Ti6Al4V substrate also melts, and when external pressure is applied after the combustion wave passes through the sample, the molten titanium and aluminum interact (mix) with the synthesized material of the ceramic composite at the interface. In this case, a diffusion zone of 10–50 μm is formed, the presence of which leads to an improvement in the adhesive strength between the layers. The main influence on the size of the diffusion zone is exerted by the delay time before applying pressure. With an increase in the delay time, the size of the diffusion zone decreases due to cooling and loss of plastic properties of the molten material. The diffusion of molten aluminum and vanadium (which is in the solid state) into the ceramic composite is confirmed by the results of SEM and EDS (Figure 3). It was found that aluminum and vanadium, due to diffusion processes and external pressure, mixed with the molten components of the synthesized material. Thus, aluminum and vanadium in LCM were detected up to 50 μm from the surface of the titanium substrate. EDS revealed traces of silicon in the LCM due to the preparation of samples for analysis.

Microhardness measurements along the LCM height were carried out on a PMT-3 microhardness meter with an indenter load of 100 g. Measurements showed that microhardness values decrease from 9.5 to 2.4 GPa when approaching the substrate (Figure 4). On the surface, increased microhardness values are due to the presence of a titanium nitride phase. The zones in which the titanium diboride phase is contained have even higher microhardness values up to 11 GPa. These zones are located approximately 1-2 mm from the surface of the LCM. As the distance from the surface of the LCM to a distance of more than 4 mm, a sharper decrease in the microhardness occurs due to a decrease in the amount of solid phase in the composite. At the interface between the ceramic composite and titanium Ti6Al4V substrate, a transition zone is observed, after which the microhardness of the substrate is 2.4 GPa and does not change further.
Thus, on the basis of the work carried out, the formation of a layered composite material is shown due to the characteristics of the synthesis of the used composition of the initial titanium and boron reagents, as well as due to the high-temperature shear deformation realized under conditions of free SHS compression.

4. Conclusion
A layered composite material based on TiB / 30 wt.% Ti on a Ti6Al4V titanium alloy with a ceramic layer height of 2-5 mm, depending on the applied pressure, was obtained by free SHS compression. In the obtained material, zones enriched in titanium diboride and zones with the orientation of titanium boride whiskers along the direction of flow of the material are revealed.

It is shown that in the synthesis and subsequent high-temperature deformation of the material, depending on the conditions of the experiment, a diffusion zone of 10–50 μm is formed. The value of this zone is mainly influenced by the delay time before applying pressure. With an increase in the delay time, the size of the diffusion zone decreases due to cooling and loss of plastic properties of the molten material.

It has been established that due to the formation of titanium nitride and diboride phases in the material, the microhardness of the LCM surface increases to 11 GPa, which decreases to 2.4 GPa as it approaches the substrate. As a result, the microhardness of the surface of the titanium alloy increases by 4.5 times.

Acknowledgement
This work was supported by the Russian Science Foundation (project № 18-79-10254).

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