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# DETERMINING THE PATTERN OF DIRECTION AND DISTRIBUTION OF INTERMETALLIC PHASE IN THE EUTECTIC OF THE WELD MATERIAL AFTER ELECTRON-BEAM WELDING OF TITANIUM AND NIOBIUM ALLOYS

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object was material of the weld. The nature of changes in the microstructure of the weld material, which are caused by changes in the supplied energy, alloying elements and heat removal from the melt area, was investigated. Welding was performed with an electron beam at  $U_{acc}=60 \ kV$ ,  $I_{eb}=90 \ mA$ , with an elliptical sweep of 3×4 mm. The speed of electron beam movement  $v_{eb}$  was varied from 7 to  $15 \text{ mm} \cdot \text{s}^{-1}$ . The temperature of the experimental welded samples  $T_0$  was varied from 300 K to 673 K. Ti-TiB alloy (a microcomposite alloy with reinforcing TiB fibers) was welded with Ti-TiB alloys, T110, and with niobium. One of the tasks of welding this alloy was to preserve and optimize the structure of this type in the weld. Grinding of boride fibers, loss of their initial orientation, and formation of a dendritic or cellular microstructure was observed in the weld.

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This paper reports a study whose

Using the methods of raster electron microscopy and micro-X-ray spectral analysis, the microstructure of the weld material was investigated and the dimensional characteristics of TiB fibers under different welding conditions were determined. The analysis of changes in the microstructure of the weld material, the average length a and the thickness e of the boride fibers in the material of the joints made at different velocities of electron beam movement and initial temperatures T0 was carried out. It was established that the growth of the ratio e/afrom 0.04-0.07 to 0.1-0.27 is accompanied by significant changes in the microstructure and the mechanism of formation of eutectic phases.

It is shown that the process that determines the formation of the microstructure of the weld material was the eutectic breakdown with the determining influence of the temperature gradient, crystallization rate, supercooling, concentration inhomogeneities, and alloying impurities

Keywords: electron beam welding, microcomposite alloy, eutectic decay, fiber size, weld

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# 1. Introduction

For alloys of titanium, niobium, and other metals, the existence of eutectics in the state diagrams and, accordingly, in the microstructures is quite characteristic. The presence of eutectic phase formations can both improve and worsen mechanical properties. This is typical for welds formed both during welding of homogeneous metals and during welding of non-homogeneous metals. One of the examples of the positive use of eutectic phase formations is the creation of so-called natural composite materials [1], on the basis of which structural materials with increased operational characteristics have already been designed. Alloys based on Ti and B have not yet found wide industrial application but show promises subject to their standardization, serial production, and the availability of technological recommendations, in particular regarding welding.

The execution of welded joints as a result of intensive thermal energy supply to the welded joint usually leads to the formation of a weld seam from a melt with a changing elemental composition, with rapid crystallization under conditions of intensive heat removal. When a eutectic transformation occurs in the weld material, the formation of microstructures of various types is possible. At the same time, the formation of a microcomposite structure is a positive factor for the mechanical properties of the weld material. To obtain microcomposite materials, conditions of directional crystallization are used, which differ significantly from the conditions of electron beam welding and other fusion welding techniques. The lack of studies of directional crystallization in the weld causes the uncertainty of the patterns of growth of eutectic phases in the Ti-TiB alloy during fusion welding.

After eutectic breakdown under the conditions of crystallization in Ti-TiB, T110, and niobium alloys in the weld after electron beam welding, the microstructure is one of the determining factors of the operational properties of the welded joint. The characteristics of the microstructure, the direction and distribution of the intermetallic phase, can be selected as parameters of the structural analysis and will serve as an important factor for the selection of technological welding parameters. Carrying out research on determining the features of the eutectic transformation in the Ti–B system with the formation of a microfibrous structure under conditions of imbalance of the welding process will provide a scientific contribution to the understanding of the nature of the growth of intermetallics under such conditions.

The prospects of microcomposite materials such as Ti-TiB alloys for industrial production, the need to weld them with titanium alloys and refractory materials, predetermine the relevance of research on determining the patterns of formation of the structure of the weld material.

### 2. Literature review and problem statement

Many alloys of Ti, Nb, and other structural materials, which contain elements with limited solubility, are characterized by eutectic decomposition. During such decay, a structure with alternating two phases is formed in the microstructure of the material (or in individual grains), one of which is the base, and the second is plates or fibers in the base of the metal [1]. Eutectic alloys are widely known and used in the industry. A significant part of such alloys has eutectic grains in the microstructure mixed with grains of other phases. More promising in terms of properties are materials for which the presence of intermetallic crystals formed during the eutectic transformation is a characteristic feature of the entire volume of the material. Study [2] shows the possibility of predicting the formation of regions of a separate solid solution and composite phases. For the first time, a phenomenological method of analyzing binary phase diagrams for predicting reinforcing phases is presented. The disadvantage of the study for practical application is the impossibility of taking into account in this method non-equilibrium conditions that are realized during electron beam welding. The method also does not provide the possibility of taking into account existing alloying additives. In work [1], a review of publications on the conditions for obtaining microcomposite materials based on metals, boron, carbon, and silicon was performed. The work shows that the main requirements for obtaining natural composite materials are the creation of crystallization conditions under a temperature gradient and high purity of materials. In particular, paper [3] shows the fundamental importance of directed crystallization of the intermetallic phase under conditions of eutectic decomposition for obtaining a microcomposite material. In work [4], on various eutectic systems, the fundamental importance of the existence of a temperature gradient and its level for directional crystallization of borides is shown, and in [5], the procedure for manufacturing a Ti-TiB microcomposite alloy under conditions of such a gradient is given. The disadvantage of the work for practical use is the lack of key parameters of the developed technical process.

Work [4], on the example of the Ti-TiB alloy, and study [6] for the two-phase titanium alloy VT25U (Ti – 6.8 %, Al – 2.1 %, Sn – 2 %, Zr – 3.5 %, Mo – 0.8 %, W – 0.2 %) with 8 vol. % TiB and 2 vol. % TiC, show that the orientation of the intermetallic phase, which plays the role of microcomposite fibers, is a rather significant factor in determining the level of mechanical properties of Ti-TiB alloys. At the same time, it was established that the material with an oriented structure of reinforcing fibers can be inferior in terms of mechanical characteristics to an alloy with a disoriented but much more dispersed one.

Among the factors that allow changing the nature of the microstructure of microcomposites, supercooling [1], which causes an increase in dispersion, has been identified. For the Ti-TiB alloy, the influence of this factor remained undetermined. As for the conditions for achieving high mechanical characteristics of microcomposites, they are defined in works [7, 8] as follows:

 the structure of the initial workpiece should be as dispersed as possible;

- the microfiber (reinforcing) phase must be sufficiently plastic and have a sufficiently large volume fraction (>20 %).

It should be noted that methods of directional crystallization, which are based on Bridgman's technique or similar ones [9], are quite well-known and widely used in aircraft construction, in particular for the manufacture of engine turbine blades [7, 8]. These methods make it possible to form a coarse-grained columnar oriented structure in eutectic alloys. Such methods were not used to form the weld seam, which is due to the lack of technical solutions for their implementation. Achieving this type of structure during fusion welding is promising; at the same time, paper [8] states that the creation of a fine-grained non-oriented structure in composite alloys for turbine blades can compete in terms of mechanical properties with a columnarly oriented but coarse-grained structure.

Approximate technological parameters for the production of microcomposite materials are the crystallization rate  $-15-50 \ \mu m \cdot s^{-1}$  and the temperature gradient -80-200 degrees·cm<sup>-1</sup>, high purity of eutectic materials [10]. They differ significantly from the conditions of weld formation: the crystallization rate is  $100-500 \ \mu m \cdot s^{-1}$  and the temperature gradient is 400-800 degrees·cm<sup>-1</sup> due to the presence of alloying impurities. At the same time, the laws of directional crystallization in eutectic alloys can be used in welding both eutectic alloys themselves and metals, the mixing of which in the weld leads to the formation of eutectics.

The first of the well-known cycles of research on welding features of natural composite materials is a set of research on electron beam welding of the Ti-TiB alloy [11]. The main results of the study were the definition of technological modes

of welding Ti-TiB alloy with Ti-TiB alloys and  $(\alpha+\beta)$  titanium alloy T110. It was experimentally proven that alloys with TiB microfibers oriented across the axis of mechanical load are prone to brittle failure. It was established that during the formation of the weld, the source material undergoes changes that lead to the grinding of boride fibers. Studies into the features of the elemental composition of reinforcing boride fibers [12] have shown that the crystallization of the weld pool occurs under conditions of intense cooling through an incomplete eutectic transformation with the formation of metastable phase formations. Performing thermal annealing [13] allows reducing the thermodynamic imbalance of the structural state and phase composition in the weld. The disadvantage of these works is the lack of analysis of the influence of alloying elements on the structure and properties of the weld material.

Despite the fact that in most works tackling directional crystallization in eutectic alloys, it is recommended to perform eutectic decomposition under conditions approaching equilibrium, in work [14] a microcomposite alloy based on titanium and TiB with TiC was successfully executed by electrospark sintering. Such conditions are much closer to the conditions of electron beam welding.

The technological modes of electron beam welding of the Ti-TiB alloy with the Ti-TiB and T110 alloys established in [11] provide satisfactory mechanical characteristics of the welded joint. At the same time, the regularities regarding the possibilities of regulating the direction and distribution of the reinforcing fiber of the intermetallic phase remained undetermined. Obtaining satisfactory mechanical characteristics of the welded joint of the Ti-TiB alloy with the Ti-TiB and T110 alloys [11] was due to the fine-grained structure of the weld material. This work did not determine the characteristics of the distribution and direction of TiB fibers in the weld work.

Thus, the state of completed research shows that the recommended limits of the crystallization rate, temperature gradient, and defined elemental composition can provide conditions for the production of microcomposite materials. These conditions vary widely for different materials [1]. During electron beam welding, the crystallization of the weld material occurs at a higher speed, with more intensive heat removal. The formation of a highly dispersed micro-composite structure of the weld material, which is atypical for structural materials of this type, necessitates additional research into its features.

This allows us to state that conducting a study into the influence of the weld formation conditions on the direction and distribution of the intermetallic phase in its material is expedient both from the point of view of obtaining scientific results and for solving practical tasks of optimizing welding modes.

### 3. The aim and objectives of the study

The purpose of this work is to establish the main factors that determine the direction and distribution of the intermetallic phase in the weld, which is formed under the conditions of electron beam welding of the eutectic Ti-TiB alloy, T-110 alloy, and niobium. This will make it possible to determine the mechanism of structure formation in the weld material, which will become the basis for choosing the recommended technological modes of welding. To achieve the goal, the following tasks were set:

 to conduct a comparative analysis of the direction and distribution of TiB fibers in the weld depending on the conditions of heat supply and removal to the crystallization area;

– to investigate the influence of the elemental composition of the weld material on the orientation and distribution of the boride phase in it.

# 4. The study materials and methods

The object of our research was the weld material of Ti-TiB alloy with titanium alloys and niobium.

The research hypothesis assumed the possibility of changing the microstructure of the Ti-TiB alloy weld with titanium alloys and niobium by adjusting the conditions of eutectic decomposition during crystallization.

During the experiments, the assumption was the constancy of the parameters, the change of which was not expected under the conditions of the study, and the absence of influence on the microstructure of the weld seam by factors that were not controlled in the experiments.

The basic studied materials in the experiments performed in this work were titanium alloys Ti-TiB, T-110, and technically pure niobium.

To obtain the Ti-TiB alloy, Ti powders were sintered (grade PTK-1 TU 14-22-57-92, fraction 45–100  $\mu$ m>85 %, chemical composition: wt.%: N – 0.07 %, C – 0.05 %, H – 0.35 %, Fe – 0.35 %, Si – 0.10 %, Ca – 0.08 %, Cl – 0.003 %, Ti – in the residue) and TiB<sub>2</sub> (fraction ~5  $\mu$ m, chemical composition (TU 113-07-11.040-89): Ti~70 %, B~30 %, Fe<0.05 %, and C<0.1 %). Before sintering, the powders were mixed with Ti – 95 % and TiB<sub>2</sub> – 5 %, pressed at *P*=0.65 GPa and annealed in the temperature range of the  $\beta$  region (sintering start temperature 1000 °C, heating at a rate of 0.03 deg/s to 1200 °C, 3 hours, under a pressure of 10 Pa).

The further procedure of making a blank for Ti-TiB alloy samples is described in [15]. After mechanical processing of the received ingot with removal of a layer of 2.5 mm, repeated deformation processing was carried out on a rolling mill 500/350 "Skoda" with a degree of plastic deformation  $\varepsilon$ =20% to a final thickness of the workpiece of 10 mm. Cutting of the workpiece to the size of 50×100×10 mm was performed by water-abrasive cutting. The cut ends of the samples were ground to the arithmetic average deviation of the surface profile  $R_a < 3.2 \ \mu$ m.

The microstructure of the Ti-TiB alloy is shown in Fig. 1. The niobium used corresponded to the grade SB-1, TU 48-4-337-75 (Nb – 98.8 %, Zr – 0.9 %, W – 0.1 %, Mo – 0.1 %, other impurities – 0.1 %). The experimental samples were made in the form of a strip with a width of 10 mm, a thickness of 2.0 mm, and a length of 100 mm, which was polished to  $R_a$ <3.2 µm on the welded surface of 100×10 mm. The microstructure of niobium is shown in Fig. 2.

The composition of the  $(\alpha+\beta)$  titanium alloy T110 used in the research corresponded to: Al – 3.5 %, Nb – 3.0 %, Fe – 2.5 %, V – 1.9 %, Mo – 1.4 %, Zr – 1.3 %, Si – 0.1 %, Ti - remaining. The material was obtained in the form of a sheet with a thickness of 10 mm, from which, similarly to the Ti-TiB alloy, samples of  $50 \times 100 \times 10$  mm were made. The microstructure of the T110 alloy is shown in Fig. 3.

Experimental welded samples from Ti-TiB alloy, joined with Ti-TiB, T110, and niobium alloys, were executed by electron beam welding. Samples measuring 50×100×10 mm

or  $2 \times 100 \times 10$  mm were connected to each other on a plane of  $100 \times 10$  mm. The following welding modes were used:  $U_{acc}=60$  kV,  $I_{eb}=90$  mA. The speed of the web electron beam was varied in the experiments. The beam sweep during welding of titanium alloys was elliptical,  $3 \times 4$  mm. Electron beam welding was performed on a UEL-144 welding machine (UEL-144, Pilot Paton Plant, Ukraine).



Fig. 1. Microstructure of experimental samples of Ti-TiB alloy in the initial state



Fig. 2. Microstructure of experimental samples of niobium in the initial state



Fig. 3. Microstructure of experimental samples of T110 alloy in the initial state

Microstructure analysis of experimental samples was performed on a JSM-840 scanning electron microscope (JEOL Ltd., Japan) with an INCA 450 microanalysis system (Oxford Instruments, USA). 5. Results of the study of changes in the microstructure of the weld material depending on the welding conditions and the type of materials to be welded

# 5. 1. Determination of directionality and distribution of fibers of the Ti-B phase in the weld joint of Ti-TiB alloys

The properties of the weld are mainly determined by the characteristics of the applied energy, the characteristics of the materials to be joined, and the conditions of crystallization of the weld material. During welding of Ti-TiB alloy samples, the energy supplied by the electron beam  $E_w$  can be estimated by the formula:

$$E_w = U_{acc} I_{eb} l / v_{eb}, \tag{1}$$

where l is the length of the weld,  $E_w$  is the energy supplied by the electron beam,  $U_{acc}$  is the accelerating voltage;  $I_{eb}$  is the welding current,  $v_{eb}$  is the speed of the electron beam.

This can be considered a rough estimate but for the comparison of welding conditions with a changing speed of the electron beam movement and constancy of other welding parameters, it is sufficient to determine the relative energy parameter of the added energy  $\Psi_{ij}$ :

$$\Psi_{ij} = E_{wi} / E_{wj} = v_{ebj} / v_{ebi}, \qquad (2)$$

where i, j are, respectively, the indices of the compared experimental samples.

During the welding of samples of the Ti-TiB alloy with each other, the speed of the electron beam movement varied from 7 mm·s<sup>-1</sup> to 13 mm·s<sup>-1</sup>. Under all modes, the quality of the welded joint was satisfactory, but the structure of the weld material changed slightly. Fig. 4–7 show the microstructures of the weld material in the Ti-TiB alloy obtained under the conditions of different speeds of electron beam movement and initial temperatures. These microstructures reflect typical changes characteristic of the distribution of boride fibers, depending on speed  $v_{eb}$  and the initial temperature of the welded samples  $T_0=300$  K and  $T_0=673$  K.

The analysis of microstructures gives reason to believe that at all welding parameters, a microcomposite structure with a much higher dispersion is formed in the weld material, compared to the welded material of Fig. 1. The length, thickness, and ratio of thickness to length of TiB microfibers were used to compare dimensional characteristics.

Table 1 gives the size characteristics of boride fibers formed in the weld material depending on speed  $v_{eb}$  and the initial temperature  $T_0$ .

At a sufficiently high level of applied energy ( $v_{eb}$ =7 mm·s<sup>-1</sup> and 10 mm·s<sup>-1</sup>), the formation of a structure (Fig. 4, 5) with maximally long and thin boride fibers is observed Table 1. When the speed of movement of the electron beam  $v_{eb}$  increased to 13 mm·s<sup>-1</sup>, an increase in both the average length a, and thickness e of boride fibers was observed (Fig. 7) with a slight increase in e/a. Increasing the initial temperature  $T_0$  of the welded samples to 673 K led to a significant decrease in the length of the TiB fibers as their thickness increased (Fig. 6). In all variants of weld formation, TiB fibers formed a structure of dendritic type branches, the features of which were studied in [12].



Fig. 4. Microstructure of the weld material of the experimental samples in the Ti-TiB alloy, obtained at the speed of the electron beam movement  $v_{eb}$ =7 mm·s<sup>-1</sup> and the initial temperature  $T_0$ =300 K



Fig. 5. Microstructure of the weld material of the experimental samples in the Ti-TiB alloy obtained at the velocity of the electron beam movement  $v_{eb}$ =10 mm·s<sup>-1</sup> and the initial temperature  $T_0$ =300 K



Fig. 6. Microstructure of the weld material of the experimental samples in the Ti-TiB alloy, obtained at the speed of the electron beam movement  $v_{eb}$ =13 mm·s<sup>-1</sup> and the initial temperature  $T_0$ =673 K



Fig. 7. Microstructure of the weld material of the experimental samples in the Ti-TiB alloy, obtained at the speed of the electron beam movement  $v_{eb}$ =13 mm·s<sup>-1</sup> and the initial temperature  $T_0$ =300 K

It should be noted that no integral orientation of TiB fibers relative to the plane of the welded joint was found. In a number of branches of the dendritic structure of the distribution of TiB fibers, a common directionality of boride fibers was observed, but the different orientation of the branches themselves determined the absence of integral directionality.

#### Table 1

The average length a and thickness e of boride fibers in the weld material of the Ti-TiB alloy joint made at different velocities of electron beam movement  $v_{eb}$  and initial temperatures  $T_0$ 

$v_{eb}$ , mm·s <sup>-1</sup>	<i>T</i> <sub>0</sub> , K	ę, µm	a, µm	ę/a,
7	300	0.3	7	0.04
10	300	0.2	5	0.04
13	673	1	8	0.13
13	300	0.8	15	0.05

# 5. 2. Determination of the effect of the elemental composition of the material of the weld joint of the T110 alloy and niobium with the Ti-TiB alloy on the orientation and distribution of boride phase in it

The difference in the structure of the weld material of the connection of Ti-TiB alloys with T110 from the connection of Ti-TiB alloy with Ti-TiB is due to the elemental composition of the T110 alloy, which contains Al – 3.5 %, Nb – 3.0 %, Fe – 2.5%, V – 1.9 %, Mo – 1.4 %, Zr – 1.3 %. The microstructure of the material in the central regions of the Ti-TiB and T110 alloy weld obtained at different speeds of electron beam movement and initial temperatures is shown in Fig. 8–12. The composition of the alloying elements in the weld ranged from Al – 0.42 %, Nb – 0.13 %, Fe – 0.40 % in the area near the contact of the weld with the T110 alloy to Al – 0.11 %, Nb – 0.04 %, Fe – 0.10 % in area near the contact of the weld seam with the Ti-TiB alloy.

At a speed  $v_{eb}=7 \text{ mm}\cdot\text{s}^{-1}$ , the structure and distribution of TiB fibers (Fig. 8) have a character similar to the material of the weld joint of Ti-TiB alloys at a speed  $v_{eb}$ =13 mm·s<sup>-1</sup> (Fig. 10, 11). Decreasing the speed of electron beam movement to  $v_{eb}=10 \text{ mm} \cdot \text{s}^{-1}$  leads to the formation of a weld with a microstructure of the material, which is characterized by a cellular structure with the placement of the boride phase at the boundaries of the cells (Fig. 9). At the maximum velocity  $v_{eb}$ =13 mm·s<sup>-1</sup>, the propensity of the weld material to form a cellular type microstructure with placement of boride fibers at the intercellular boundaries remains both at the initial temperature  $T_0=300$  K (Fig. 11) and at  $T_0=673$  K (Fig. 10). At the same time, from the analysis of the given microstructures, it can be seen that the characteristics of boride fibers depend significantly on both the speed  $v_{eb}$  and the temperature  $T_0$ . The measurement characteristics of boride fibers in the material of the Ti-TiB and T110 alloy weld, obtained at different speeds of electron beam movement and initial temperatures, are given in Table 2.

Al, Nb, and Fe impurities, which got into it from the T110 alloy, were registered in the weld by X-ray microspectral analysis. In other respects, the conditions for the formation of the T110 alloy weld with the Ti-TiB alloy do not differ from the conditions for welding the Ti-TiB alloy with the same alloy – this indicates that the presence of alloying impurities in the weld material significantly changes its microstructure. Not only the nature of the microstructure changes but also its dependence on the velocity of beam  $v_{eb}$  and the initial tem-

perature  $T_0$ . The smallest differences in the microstructure of the weld materials are observed in the joints of Ti-TiB alloys and Ti-TiB and T110 alloys at the beam speed  $v_{eb}=7$  mm·s<sup>-1</sup>. Under other modes of welding Ti-TiB and T110 alloys, a change in the nature of the distribution of boride fibers is observed, which changes from dendritic to cellular.



Fig. 8. Microstructure of the material of the weld joint of experimental samples made of Ti-TiB and T110 alloys, obtained at the speed of the electron beam movement  $v_{eb}$ =7 mm·s<sup>-1</sup> and the initial temperature  $T_0$ =300 K



Fig. 9. The microstructure of the material of the weld joint of the experimental samples made of Ti-TiB and T110 alloys, obtained at the speed of the electron beam movement  $v_{eb}$ =10 mm·s<sup>-1</sup> and the initial temperature  $T_0$ =300 K



Fig. 10. Microstructure of the material of the weld joint of the experimental samples made of Ti-TiB and T110 alloys, obtained at the speed of the electron beam movement  $v_{eb}$ =13 mm·s<sup>-1</sup> and the initial temperature  $T_0$ =673 K

For electron beam welding of experimental samples of the Ti-TiB alloy with niobium, the following welding mode was used:  $U_{acc}$ =60 kV,  $I_{eb}$ =90 mA;  $v_{eb}$ =15 mm·s<sup>-1</sup> ( $T_0$ =300 K). This mode was recommended for welding the Ti-TiB alloy with refractory metals [13], it provided a welded joint with a seam in which metallographic analysis did not reveal existing defects. Studies of the microstructure of the weld material showed that a fine-grained eutectic structure was formed in the central

region of the weld (Fig. 12): a solid solution of Nb in Ti with a fiber-like phase of TiB. The nature of the distribution of boride fibers is dendritic. In the zone of contact of the melt, which was formed as a result of bringing the energy of the electron beam to the Ti-TiB–Nb junction, with unmelted materials on both sides, a greater fine dispersion is characteristic (Fig. 13, 14). At the same time, the minimum niobium content recorded in the weld material was recorded by X-ray microspectral analysis as 7 at. %, and the maximum at 10 at. %. This indicates the predominant influence of the turbulent rather than the diffusion component of the saturation of the weld material with niobium.

The formation of ternary phases was not recorded, only titanium was recorded in boride grains of metals. It should be noted that in the presence of fibers longer than 30  $\mu$ m (with an average fiber thickness of ~0.8  $\mu$ m), their average length is 8  $\mu$ m. The predominant orientation of TiB fibers in most cases is related only to the direction of dendritic branches, which, in turn, do not have a predominant orientation in the central part of the weld (Fig. 12).



Fig. 11. Microstructure of the material of the weld joint of experimental samples made of Ti-TiB and T110 alloys, obtained at the speed of the electron beam movement  $v_{eb}$ =13 mm·s<sup>-1</sup> and the initial temperature  $T_0$ =300 K

#### Table 2

The average length a and thickness  $\varphi$  of boride fibers in the material of the weld joint of Ti-TiB and T110 alloys made at different velocities of electron beam movement  $v_{eb}$  and initial temperatures  $T_0$ 

$v_{eb}$ , mm·s <sup>-1</sup>	<i>T</i> <sub>0</sub> , K	ę, μm	a, μm	ę/a,
7	300	0.5	8	0.06
10	300	0.8	3	0.27
13	673	0.6	8	0.07
13	300	0.4	4	0.1

On the border of the niobium weld and the Ti-TiB alloy weld. In the contact region of the "welded seam" – "Ti-TiB alloy" (Fig. 13), the formation of a transition zone of thermal influence with a width of ~60  $\mu$ m is observed, which is characterized by the partial melting of the primary phases of TiB with the formation of fine-fiber eutectics, the absence of niobium.

The formation of TiB fibers with an average thickness of 0.7  $\mu$ m and a length of 5–8  $\mu$ m (¢/a=0.09) is characteristic of the weld material (in the melt zone) in contact with the unmelted Ti-TiB alloy.

For the zone of contact of the melt of the weld material with niobium, the formation of the zone of thermal influence was not detected. Along with this, there are certain features of the structure and distribution of the boride phase. This region (Fig. 14), as well as the central region of the weld, is characterized by a dendrite-like structure of TiB fiber distri-

bution. Most of the TiB fibers themselves have a short length of  $\sim 4-5 \,\mu\text{m}$  (in the presence of individual fibers with a length of  $10-15 \,\mu\text{m}$ ) with a thickness of  $\sim 1 \,\mu\text{m}$  (e/a=0.23).



Fig. 12. Microstructure of the Ti-TiB alloy and niobium compound material in the central region of the weld



Fig. 13. Microstructure of the Ti-TiB alloy and niobium compound material in the area of contact of the weld with the Ti-TiB alloy



Fig. 14. Microstructure of the material of the connection of the alloy Ti-TiB and niobium in the area of contact of the weld with niobium

A feature of the microstructure of the Ti-TiB alloy and niobium joint material in the area of contact of the weld with niobium (Fig. 14) is the characteristic orientation of the grains of the metal phase in the direction perpendicular to the contact line. Such an orientation is not observed in the central region (Fig. 12), and in the region of contact of the weld seam with the Ti-TiB alloy, a partial orientation of the TiB microfibers in the direction perpendicular to the contact line is observed (Fig. 13).

# 6. Discussion of results of changes in the microstructure of the weld depending on the welding conditions and the materials to be welded

Analyzing our results, it is necessary to take into account not only the processes of eutectic crystallization of the weld material but also the formation of the melt in this area. The Ti-TiB alloy in its initial state contains TiB fibers with a thickness of  $2-4 \,\mu\text{m}$  and a length of  $15-50 \,\mu\text{m}$ . During the formation of the melt zone, the influence of the electron beam causes a certain mixing of the liquid and the development of diffusion processes, which contribute to the reduction of concentration inhomogeneities.

Under such conditions, the distribution of boron in the melt will be more uniform under conditions of higher energy supply by the electron beam. At the same time, a sufficient number of boride phase nuclei and a longer time for their growth may occur per unit area, which is typical for the conditions of  $v_{eb}=7 \text{ mm}\cdot\text{s}^{-1}$ . At the same time, a large number of growing fibers reduces the concentration of boron in the melt and limits the growth of a new crystal, which is especially important under conditions of rapid crystallization of the weld. It is known that an increase in the crystallization rate contributes to a decrease in the size of the eutectic fibers, which is probably observed under the conditions of  $v_{eb}=10 \text{ mm}\cdot\text{s}^{-1}$ . With a further increase in the speed of movement to  $v_{eb}$ =13 mm·s<sup>-1</sup>, the homogeneity of the distribution of boron in the liquid of the weld pool may not be sufficient, which will lead to an increase in the size of boride fibers in places of higher concentration of boron. The hypothesis of such a mechanism of influence of the supplied energy (1) at different speeds of electron beam movement looks plausible but needs to be confirmed by carrying out the distribution of boron in the weld material by Auger spectroscopy, which is sensitive for determining the concentration of light elements.

The similarity of the growth mechanisms of borides under the above-mentioned conditions confirms the closeness of the e/a characteristics of the reinforcing fibers (Table 1). With preheating of the welded materials, this indicator increases sharply, which indicates a significant change in the conditions of their growth. In this case, two factors that may cause such changes should be noted. An increase in the temperature of the welded materials reduces the intensity of heat removal (reduces the rate of crystallization) and the temperature gradient, which contribute to the formation of the TiB fiber phase [1, 4, 6, 10]. A decrease in the parameters that contribute to directional growth and the formation of fibers will cause the intensification of lateral growth.

On the other hand, after the crystallization of the weld material, in the case of the presence of residual boron in certain areas of the titanium matrix (which is likely due to insufficient uniformity of its distribution in the melt at  $v_{eb}$ =13 mm·s<sup>-1</sup>), the eutectoid growth of TiB fibers, which was established in work [12], may continue. Under such conditions, growth will be determined by diffusion processes in the solid state, which require elevated temperatures. When the growth mechanism of the intermetallic phase changes, the predominant crystallographic direction of TiB crystal

growth may change [1], which explains the growth of both the thickness of microfibers (Fig. 6) and the ratio of the thickness and length of TiB crystals (Table 1).

The microstructure of the material of the weld joint of Ti-TiB and T110 alloys changes quite complexly depending on the web speed. At a velocity of  $v_{eb}$ =7 mm·s<sup>-1</sup>, the microstructure (Fig. 8) and the ¢/a ratio (Table 2) differ little from the microstructure (Fig. 4) and the ¢/a ratio for the connection of Ti-TiB alloys (Table 1). This gives reason to expect that the crystallization process at the velocity  $v_{eb}$ =7 mm·s<sup>-1</sup> has a similar character.

At the same time, at a speed  $v_{eb}=10 \text{ mm}\cdot\text{s}^{-1}$ , the microstructure acquires other features (Fig. 9), which are not observed at other speeds of the electron beam. The formation of cluster formations by titanium boride at the border of cells formed by titanium with alloying impurities gives reason to believe that changes in the mechanism of eutectic decomposition during crystallization occur during welding of these alloys. This is particularly evidenced by the change in structure from dendritic (Fig. 8) to cellular type in the weld material (Fig. 9).

It should be noted that such changes in the structure from dendritic to cellular type are also characteristic of the weld material obtained at a beam speed  $v_{eb}$ =13 mm·s<sup>-1</sup> (Fig. 10, 11). Explaining such facts, it should be emphasized that the ability of alloying impurities to change the mechanism of eutectic growth was noted in [1]. They lead to the stimulation of the cellular growth of the eutectic phase. Under conditions of rapid crystallization, the sufficient concentration of boron in the zone of crystal growth is the limiting factor for the growth of boride by the cellular mechanism [1], which is necessary for changing the growth mechanism. As it was shown in the previous review, it is when the beam speed  $v_{eb}$ =10 mm·s<sup>-1</sup> is reached that concentration inhomogeneities appear, which can ensure cellular growth initiated by the presence of alloying impurities.

A further increase in the speed to  $v_{eb}$ =13 mm·s<sup>-1</sup> can increase the concentration heterogeneity of boron, which will lead to an increase in local supercooling of the boron-saturated part of the alloy and, as a result, to the formation of fibers of smaller sizes (Fig. 11). Such an effect of supercooling of eutectics is indicated in [1].

Preheating to 673 K of the welded samples leads to an increase in the crystallization period, which determines the size of the growing crystal. This is exactly what is observed (Fig. 10) during the preliminary heating of welded Ti-TiB and T110 alloys. TiB microfibers in this case have the maximum length (Table 2).

Thus, a set of factors that are capable of multidirectional influence on crystal growth under conditions of eutectic decay can significantly change the microstructure of the material of the weld joint of Ti-TiB and T110 alloys with small changes in the supplied energy and initial temperature.

In all cases of welding of Ti-TiB alloys with titanium alloys, there is no integral orientation of boride fibers in the weld material. Only a local common orientation of microfibers is observed in the zones of their growth at the borders of cells with a cellular type of structure (Fig. 9–11) and in branches of the dendritic type with a dendritic growth mechanism (Fig. 4–8). Such types of structure are typical for the conditions of rapid crystallization of the eutectic melt in the presence of a temperature gradient [1]. The possibility of creating conditions for the formation of uniformly oriented microfiber or coarse-grained columnar microstructure in the weld and the consequences of its formation for mechanical characteristics should be further investigated. It was established that a change in the speed of beam  $v_{eb}$  in a fairly narrow range from 7 mm·s<sup>-1</sup> to 13 mm·s<sup>-1</sup>, an increase in the initial temperature from 300 K to 673 K and the introduction of alloying additives into the weld, the concentration of which in total does not exceed 1 at. % are able to significantly change the microstructure of the seam. This gives reason to recommend that when welding the Ti-TiB alloy with alloyed titanium alloys, one should control compliance with the established technological regimes, and when changing the regimes or composition of the alloy – control the structural features and mechanical characteristics of the welded joint. In the case of electron beam welding of Ti-TiB alloys, such a high sensitivity of the microstructure to the applied energy and initial temperature was not found.

As a result of the study of the features of TiB phase distribution in the weld seam of the Ti-TiB alloy with niobium, no significant differences from the microstructures characteristic of the welding of the TiB alloy itself were found. The dendritic character of the distribution of microfibers, the ratio of thickness and length of TiB crystals is close to the values given in Table 1 gives reason to believe that the high solubility of niobium in titanium ensures the preservation of the crystallization mechanism of the weld material characteristic of the Ti-TiB alloy.

The directional orientation of the boride fibers at the boundary of the weld seam and the Ti-TiB alloy (Fig. 13) can be caused by the temperature gradient that occurs under the conditions of heat transfer from the weld seam to the unmolten metal. Such an influence of the temperature gradient was noted in most works on directional crystallization [1, 3-8, 10]. The lack of such an orientation of microfibers at the weld boundary in titanium joints remains a question.

A feature of the structure of the material of the contact area of the weld with niobium is the presence of a sufficiently distinct orientation not of TiB fibers but of grains of a solid solution of niobium in titanium (Fig. 14). This may be a sign of significant supercooling of the melt near niobium, which is associated with an increase in the melting temperature of the solid solution of niobium in titanium with an increase in niobium concentration and the occurrence of competing crystallization processes of both eutectic phases under such conditions. The temperature gradient necessary for directional crystallization is provided by heat removal to niobium. The obtained results regarding the detection of directional grains of eutectic phases at the weld boundary provide prospects for achieving directional crystallization during eutectic decomposition in the weld of Ti-TiB type alloys, which requires devising a special procedure.

Our results are important for the welding of all microcomposites, but for a wide range of such materials, they are important only from the point of view of understanding the mechanism of eutectic decay during weld crystallization. The high sensitivity of structure formation to the technological parameters of welding and the elemental composition of welding materials determines the need to optimize the technological modes of welding for each pair of materials to be joined.

During the analysis of the obtained experimental results regarding the distribution of boron in the weld material, only metallographic analysis was used, and the results of other experiments were taken into account. It is necessary to use Auger spectroscopy, which can be implemented in subsequent studies.

It should be noted that the established high sensitivity of structure formation to the technological parameters of welding and the elemental composition of welding materials gives reason to believe that there are limitations of the current research. The regularities determined in the experiments and the corresponding conclusions regarding the results are established within the limits of the welding modes and materials with the specified initial structure and elemental composition used in the work.

The development of this study involves the implementation of work in the direction of expanding the range of welded microcomposite materials, the use of such materials as intermediate inserts to prevent the formation of intermetallic layers. A separate promising area of research is the achievement of a columnar unidirectional structure in the weld material, which is possible under the conditions of a stable temperature gradient, a low crystallization rate, and the necessary concentration of eutectic-forming elements.

### 7. Conclusions

1. For the structure of the Ti-TiB alloy weld, which is formed under the conditions of eutectic dissolution of the melt, there is a characteristic dependence on the speed of movement of the electron beam  $v_{eb}$  and the initial temperature  $T_0$ . Changing these parameters in the interval v  $v_{eb}$ =7–13 mm·s<sup>-1</sup>,  $T_0$ =300–673 K makes it possible to adjust the thickness and length of TiB fibers in the Ti-TiB eutectic. 2. The presence of alloying impurities Al, Nb, Fe in the

T110 alloy determines the mechanism of eutectic disintegra-

tion of the Ti-TiB alloy in the weld seam of their connection made by an electron beam at different speeds  $v_{eb}$  of movement of the electron beam and initial temperatures  $T_0$ . Changing these parameters in the interval  $v_{eb}$ =7–13 mm·s<sup>-1</sup>,  $T_0$ =300–673 K makes it possible not only to adjust the thickness and length of TiB fibers in the Ti-TiB eutectic but also change the dendritic mechanism of crystal growth to a cellular one.

# **Conflicts of interest**

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study and the results reported in this paper.

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# Data availability

The data will be provided upon reasonable request.

#### References

- Tikhonovskiy, M. A. (2004). Issledovanie napravlennykh fazovykh prevrascheniy i razrabotka mikrokompozitnykh materialov v NNTs KhFTI. Voprosy atomnoy nauki i tekhniki, 6, 115–127. Available at: https://vant.kipt.kharkov.ua/ARTICLE/VANT\_2004\_6/ article\_2004\_6\_115.pdf
- Qi, J., Cheung, A. M., Poon, S. J. (2019). High Entropy Alloys Mined From Binary Phase Diagrams. Scientific Reports, 9 (1). doi: https://doi.org/10.1038/s41598-019-50015-4
- Andrievskaya, N. F., Kapcherin, A. S., Tikhonovskiy, M. A. (1980). Morfologicheskie perekhody v evtekticheskikh kompozitakh. Fizika prochnosti kompozitsionnykh materialov. Leningrad: Izd-vo LFTI, 42–44.
- Gaisin, R. A., Imayev, V. M., Imayev, R. M. (2017). Effect of hot forging on microstructure and mechanical properties of near α titanium alloy/TiB composites produced by casting. Journal of Alloys and Compounds, 723, 385–394. doi: https://doi.org/10.1016/ j.jallcom.2017.06.287
- Grigorenko, G. M., Akhonin, S. V., Loboda, P. I., Grigorenko, S. G., Severin, A. Yu., Berezos, V. A., Bogomol, Yu. I. (2016). Structure And Properties Of Titanium Alloy, Alloyed With Boron, Produced By The Method Of Electron Beam Remelting. Electrometallurgy Today, 1, 21–25. doi: https://doi.org/10.15407/sem2016.01.03
- Gaisin, R. A., Imayev, V. M., Imayev, R. M. (2018). Microstructure and Mechanical Properties of a Near-α-Titanium-Alloy/TiB Composite Prepared in situ by Casting and Subjected to Deformation and Heat Treatment. Physics of Metals and Metallography, 119 (9), 907–916. doi: https://doi.org/10.1134/s0031918x18090041
- Bondarenko, Yu. A., Kablov, E. N. (2002). Napravlennaya kristallizatsiya zharoprochnykh splavov s povyshennym temperaturnym gradientom. Metallovedenie i termicheskaya obrabotka metallov, 7, 20–23.
- Kablov, E. N., Lukin, V. N. (2008). Intermetallidy na osnove titana i nikelya dlya izdeliy novoy tekhniki. Avtomaticheskaya svarka, 11, 76–82. Available at: http://dspace.nbuv.gov.ua/bitstream/handle/123456789/100041/09-Kablov.pdf?sequence=1
- 9. Bagdasarov, Kh. S. (2004). Vysokotemperaturnaya kristallizatsiya iz rasplava. Moscow: FIZMATLIT, 160. Available at: https://www.studentlibrary.ru/book/ISBN5922104829.html
- Alikhanov, V., Khudoyan, M. V. (2019). Directionally crystallized eutectics obtaining method. University News. North-Caucasian Region. Technical Sciences Series, 1, 77–81. doi: https://doi.org/10.17213/0321-2653-2019-1-77-81
- 11. Loboda, P., Zvorykin, C., Zvorykin, V., Vrzhyzhevskyi, E., Taranova, T., Kostin, V. (2020). Production and Properties of Electron-Beam-Welded Joints on Ti-TiB Titanium Alloys. Metals, 10 (4), 522. doi: https://doi.org/10.3390/met10040522
- Loboda, P., Zvorykin, V., Zvorykin, C., Vrzhyzhevskyi, E., Taranova, T., Kostin, V., Zvorykin, L. (2023). Features of the welded seam material crystallization in Ti-TiB alloy under electron-beam welding conditions. Mechanics and Advanced Technologies, 7 (1), 36–42. doi: https://doi.org/10.20535/2521-1943.2023.7.1.277544
- Loboda, P., Zvorykin, C., Zvorykin, V., Vrzhyzhevskyi, E., Taranova, T., Kostin, V. (2020). Structural regularities of welded seam between Ti-TiB and vanadium with 12X18H10T interlayer by using electron beam welding. Mechanics and Advanced Technologies, 2 (89). doi: https://doi.org/10.20535/2521-1943.2020.89.211400
- Hu, Z.-Y., Peng, H.-C., Zhang, Z.-H., Song, P., Chen, M., Ding, Y.-S. et al. (2021). Influence of the sintering temperature on the microstructure, mechanical properties and densification characteristics of (TiB + TiC)/TC4 composite. Materials Research Express, 8 (12), 126517. doi: https://doi.org/10.1088/2053-1591/ac40b7