

Features of the welded seam material crystallization in Ti-TiB alloy under electron-beam welding conditions

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Received: 15 March 2023 / Accepted: 10 April 2023

Abstract. Natural metal composite materials represent a promising class of modern structural materials that need to be welded. Such materials can be welded by fusion, as has been established with the Ti-TiB alloy as an example. The enhanced operational properties of such materials are determined by the microstructure, which is characterized by the presence of microfibers of borides, carbides, or silicides in the metal matrix. To preserve the mechanical properties of materials in a welded joint, it is necessary to ensure the formation of reinforcing microfibers in the welded seam material. Determination of formation mechanism of boride microfibers, originated in the welded seam material, will become the basis for optimizing of fusion welding modes, in particular, electron beam welding mode.

The purpose of this study is the determination of formation mechanism of boride microfibers originated in the welded seam material. Two most probable variants of the formation mechanism are analyzed, which involve eutectic decomposition during crystallization from a liquid melt or eutectoid decomposition from a metastable crystallized alloy. The third version is a mixed variant of the two above-mentioned mechanisms.

In the article the results of metallographic analysis of features of boride phase distribution and an analysis of elemental composition of boride fibers based on local Auger electron spectroscopy are presented. The object of study was a Ti-TiB alloy joint obtained by electron-beam welding. The analysis factors were the features of size, orientation, and nature of the distribution of boride phase microfibers in different areas of the welded seam. The characteristic elemental composition of boride microfibers, which characterizes the correspondence to equilibrium phases, is also studied.

The degree of deviation of the ratio of boron and titanium in such a phase from the thermodynamically equilibrium in different layers of the material of the welded seam, formed by an electron beam in vacuum, is determined. The dependence of boride phase distribution under various conditions of heat exchange in the welded seam material on the side surfaces and in the central regions is established. It is shown that some of boride microfibers formed in the material of the welded seam are characterized by a deviation from the thermodynamically stable composition TiB_n ($n = 1$) to TiB_n ($n = 0.85$). The dendritic nature of boride microfibers distribution and the presence of meta-stable phase formations on Ti and B basis provide the grounds for proposing the predominant mechanism for the formation of structure of the welded seam material in the Ti-TiB alloy during crystallization.

An analysis of hypothetical variants of the formation mechanism of boride microfibers originated in the welded seam material showed that the formation of a dendritic type of structure is characteristic for the growth of crystals of a new phase in the liquid phase. Such growth is characterized by the formation of equilibrium phases. The presence of a significant amount of non-equilibrium boride phase in the welded seam indicates the residue of non-equilibrium boron in the titanium matrix and continuation of growing of boride fibers in the crystallized welded seam. A determined mechanism for formation of boride microfibers originated in the welded seam material is eutectic decomposition during crystallization from a liquid melt with the formation of TiB microfibers and further growth of such crystals due to eutectoid decomposition from a metastable crystallized Ti-TiB alloy. The results obtained make it possible to understand the mechanism of formation of a welded seam in welded natural-composite metal materials, which permits to develop the recommendations for optimizing the welding technology for such materials.

Ключевые слова: titanium alloys; titanium boride; micro-structure; mechanical properties; welded joint; electron-beam welding; heat treatment.

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1. Introduction

Welding of microcomposite alloys of the Ti-B system is successfully implemented by melting occurring in the welded joint area due to the heat supply by an electron beam [1–2]. The electron beam is the main factor influencing formation of structure and properties of the material in

this zone. The emergence of a steam-gas channel, which under conditions of single-pass welding is of a through nature, forms a liquid pool behind it, which crystallizes into a welded seam during the cooling. Under conditions of welding of titanium alloys, which are characterized by low thermal conductivity, the cooling occurs less intensively than in steels or aluminum alloys, however, it is accompanied by appearance of significant temperature gradients. This promotes the formation and growth of TiB reinforcing fibers during crystallization [3], and cooling through a heat sink into the materials to be welded causes traditional changes in phase decomposition that usually occur in eutectic alloys. Provided a narrow welded seam that meets the conditions: $\Delta < 0.3\lambda$ (where Δ is the welded seam width, mm, and λ is thickness of materials being welded, mm), and under the condition of a fast movement of thermal energy source, the features of such changes include refinement of granular structure, changes in liquation heterogeneity and a change in shape of macro and micromorphology of crystals [4].

2. Materials and Methods

The Ti-TiB alloy used in this study was obtained by the method described in [3]. After machining the ingot with removing a 2.5 mm layer, multiple deformation processing was performed on a Skoda 500/350 rolling mill with $\varepsilon = 20\%$ degree of plastic deformation. The final blank thickness for the experimental samples was 10 mm. The blanks for welding investigations were then extracted by water-jet cutting. The ends of the cut of the samples were ground to ensure parallel joint of the welded surfaces, so that the resulting surface roughness R_a was less or equal to 3.2 μm .

Samples for metallographic studies were prepared using a precision hydroabrasive complex KGA 2-R-2500 ("Rodent" Private Enterprise, Ukraine) by cutting in a plane perpendicular to the surface of the welded joint, followed by grinding and polishing, removing 0.5 mm. The experimental welded samples were also cut with water along the welded seam, displacing the water jet away from the welded seam in such a way that one of cut planes passed in the material near the center of the welded seam or near the heat-affected zone of the welded seam at its center.

To obtain the welded samples based on the Ti-TiB alloy, used for the investigations fulfilled in this work, fusion welding was performed by means of UL-144 electron-beam welding installation (UL-144, Pilot Paton Plant, Ukraine), which was carried out in a vacuum of 10^{-2} Pa.

When performing elemental and structural analysis of surfaces, the following methods of elemental and structural analysis of surfaces were used:

- high-resolution scanning electron microscopy using JSM-840 electron microscope (JEOL Ltd., Japan) with a Noran-Quest X-ray analysis system;
- local Auger electron spectroscopy of JAMP-9500F Auger microprobe (JEOL Ltd., Japan).

The microstructure of the Ti-TiB alloy, consisting of a Ti-matrix with reinforcing boride fibers, is shown in Fig. 1.

The quantitative ratio of the Ti and TiB phases in the experimental samples was controlled by quantitative X-ray phase analysis (by the RIR reference intensity method) and Auger spectral analysis, by which the quantitative ratio of boron and titanium content was determined.

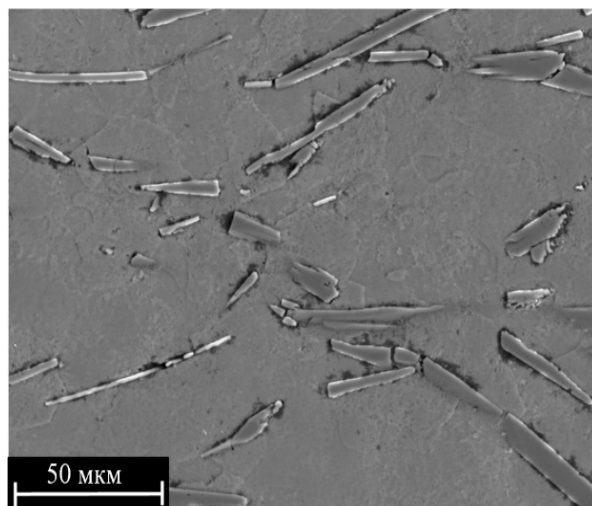


Fig. 1. Microstructure of the Ti-TiB alloy before welding [5]

Metallographic analysis is shown, that in the Ti-TiB welded material the TiB microfibers were evenly distributed throughout the volume of titanium matrix (α -Ti), their thickness was 2–7 μm (see Fig. 1), the length of fibers varied in the range from 8 μm to 70 μm . The thickness of the TiB fibers correlated with their length on average in a ratio of 1:6, however, the indicated ratio for different inclusions varied from 1:3 to 1:15.

3. Results and Discussion

Changes, occurring in the material during the welded seam formation owing to the electron beam moving, can have a significant effect on the mechanical properties of the welded joint, so it is necessary to analyze the features of structure and phase composition of the welded seam material.

Fig. 2 shows the microstructure of the welded seam material in the plane of section, approximately coinciding with the plane of joint of welded elements.

Metallographic analysis of the welded seam material showed, that in the material of the Ti-TiB welded seam, TiB microfibers were distributed in the form of dendritic clusters throughout the all volume of (α -Ti) titanium matrix, at that the thickness of microfibers was 1–2 μm and the fibers length was varied in the range from 3 μm up to 10 μm . The thickness of the TiB fibers correlated with their length on average in a ratio of 1:7, however, the indicated ratio for different inclusions varied from 1:4 to 1:12.

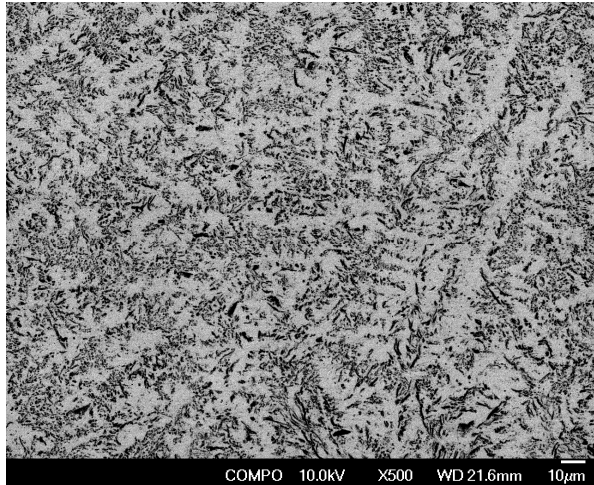


Fig. 2. Microstructure of welded seam material of the Ti-TiB alloy in the longitudinal section ($U_e = 60$ kV, $I_e = 90$ mA, $v_e = 10$ mm·s⁻¹)

The conducted experiments are characterized by the ratio $\Gamma / \lambda \sim 0.2$, according to [4]. This gives grounds to believe, that at $v_e = 10$ mm·s⁻¹ the weld pool cooling is fast. With slow cooling of liquid solution on the interfacial surfaces, its composition has time to even out, so the TiB and Ti phases are able to grow freely until they meet, forming a rough conglomerate of crystallites. On rapid cooling, the crystallizing TiB phases usually alternate, which leads to the formation of dispersed structure, typical for eutectic. It is believed [4] that eutectic crystallization occurs from crystallization centers with the formation of eutectic colonies or eutectic grains, characterized by different macro- and micromorphology. In the welded seam zone (see Fig. 2), the eutectic colony is formed by the Ti crystal and dispersed inclusions of TiB fibers. Nevertheless, under high supercooling conditions, the eutectic of Ti-TiB can consist of TiB fibers that are in contact with each other and mixed with irregularly shaped titanium matrix globules.

Upon cooling, after the melt reaches the temperature of crystallization beginning, the Ti-B alloy containing less than 48 at.% B (assuming an uniform distribution of components), corresponds to a liquid solution of titanium and boron with the TiB phase crystallizing in the melt. As is known [6], to start the melt crystallization, its overcooling ΔT from the phase equilibrium temperature is necessary. At that, the new phase nuclei originate by fluctuation way and grow or dissolve depending on the ratio of their effective nucleus radius to the critical radius r_{cr} of the nucleus. Critical radius r_{cr} corresponds to the value at which the free energy E_{fr} reaches its maximum and decreases with each change in the effective radius of nucleus and is determined by formula:

$$r_{cr} = \frac{2\eta T_{pe.}}{\Delta T \Omega} \quad (1)$$

where η is surface tension coefficient, J·m; $T_{pe.}$ – phase equilibrium temperature, K; ΔT – temperature subcooling, K; Ω – latent crystallization heat, J.

Under conditions of equilibrium in the TiB alloy in the liquid state, there are atomic clusters corresponding to the structure of crystals of stable or metastable phases. When the size of such a cluster exceeds the critical radius r_{cr} , it becomes the nucleus of a new phase. Accordingly, with significant supercooling, the number of clusters that become nuclei increases, which contributes to an increase in the number of grains and a decrease in their size. Provided, that the surface tension coefficient η , the phase equilibrium temperature $T_{pe.}$ and latent crystallization heat Ω are unchanged, an increase in temperature overcooling also contributes to the formation of metastable phases.

Changes in the structure, caused by rapid cooling, include: refinement of granular structure, change in liquation microheterogeneity, and crystal morphology shape change [4]. In the range of rates of transition to the solid state, which promote the cooperative growth of TiB, the acceleration of cooling leads to the development of a cellular substructure, to a change in the shape of segregation accumulations, and to an expansion of region of boron solubility in solid solutions. Since metastable intermediate phases often determine the type of segregation accumulation, their contribution to the formation of alloy properties can be significant.

In [6], the metastable intermediate phases are divided into new metastable phases and phases with limited metastability. New metastable phases (Ti₂B, Ti₂B₃) may not be present in the equilibrium state diagram and be metastable in the entire temperature-concentration region of the “titanium” – “boron” diagram. Phases with limited metastability are present in the stable equilibrium diagram, but due to the acceleration of cooling, they can expand the area of their existence, being metastable precisely in such increased boundaries. The acceleration of eutectic solidification during rapid cooling can also be accompanied by the crystallization of metastable intermediate phases. Since metastable intermediate phases often determine the type of colony, their contribution to the formation of alloy properties is often more significant. The level of metastability of these phases is different and is characterized by the difference between the free energies of formation of metastable phase formation and a mixture of stable phases of the same composition [6].

For the case of single-phase alloys crystallization, it is known [7], that supercooling depends on grain orientation, at that a change in the crystallization rate can lead to a change in the “favorable” direction of growth. A similar situation is also observed during the growth of two-phase eutectic grains [8]. An increase in heat removal from the crystallization zone can cause an increase in the growth rate and lead to a change in the direction of the predominant growth of the TiB phase; in this case, a change in the nature of its branching and the morphological appearance of the eutectic is likely.

For eutectic composites, of paramount importance is the question of limits within which the crystallization conditions can be changed while maintaining the compositional nature of TiB fibers growth. For a wide range of alloys subject to compositional growth, it has been established [9–11] that an increase in temperature gradient in a liquid leads to an expansion of the concentration region of compositional growth.

The crystallization rate has a more complex effect on the limits of compositional growth. It was shown in [8] that the boundaries of composite growth change at different cooling rates, depending on the form of growth of primary crystals with which the composite microstructure borders.

With an increase in crystallization rate of the Ti-TiB alloy, the boundary of compositional growth approaches the eutectic concentration of 8–9 at.% if the primary TiB crystals grow unfaceted and move away from the eutectic concentration of 8–9 at.% under the condition of faceted growth of primary crystals. According to the concept of competing growth [8], primary crystals will be absent in the welded seam microstructure if the temperature at their crystallization front of primary crystals is lower than the temperature at the growth front of the boride phase of the composite microstructure. The boundaries of the compositional growth of the eutectic phase correspond to the equality of these temperatures and are determined by their dependences on the crystallization rate, temperature gradient, and melt composition. Since the strengthening phase of TiB has a high temperature and melting entropy, it is recommended in [11] to use the highest possible crystallization rates to maximize the proportion of the fiber-like state of the strengthening phase by expanding the boundaries of its compositional growth.

With a controlled flow of phase transformations, a composite microstructure can also be formed through eutectoid decomposition, which occurs under conditions of a sharp temperature gradient, the source of which moves along the sample [12]. The eutectoid transformation occurs in the solid state, but resulting microstructure of the alloy is similar to the eutectic one.

The formation of the oriented structure of eutectoids has its own peculiarities [12–13], which were discovered in the study of the stationary decomposition of the high-temperature phase in the case of a temperature gradient of 200–600 deg·cm⁻¹, which is close to the case of electron-beam welding of the Ti-TiB alloy. These features are as follows [8]:

1. A microstructure oriented in the direction of heat removal is formed in the case of a temperature gradient less than a certain value ∇T_{\max} (∇T , ∇T_{\max} are temperature gradients, °C).

2. Under the condition $\nabla T > \nabla T_{\max}$, the alloy microstructure consists of a large number of flat or fibrous eutectoid grains, for which their orientation relative to the sample axis is practically arbitrary.

To evaluate the mechanism of welded seam formation during electron beam welding of the Ti-TiB microcomposite alloy, taking into account the above results, three possible options can be assumed:

1. After the weld pool formation by the electron beam, rapid crystallization occurs through eutectic transformation with the formation of secondary TiB fibers, preferably oriented in the direction of the temperature gradient (from the welded seam axis to the samples being welded).

2. After the weld pool formation by the electron beam, rapid crystallization occurs with fixation of metastable state of a boron solid solution in titanium, followed by eutectoid decomposition in the solid state with formation of an alloy microstructure oriented in the direction of heat removal (at $\nabla T < \nabla T_{\max}$) or misoriented (at $\nabla T > \nabla T_{\max}$).

3. Upon the first option implementation, the partial formation of metastable phases occurs, followed by eutectoid decomposition at elevated temperatures due to residual heating or final annealing of the welded joint.

Determination of predominant mechanism for the welded seam formation during electron beam welding of the Ti-TiB microcomposite alloy is possible on the basis of analysis of structural features and phase composition of the weld seal material.

Fig. 3 shows the microstructure of the welded seam material in the plane of section, which approximately coincides with the plane of joint of the welded elements near their edge.

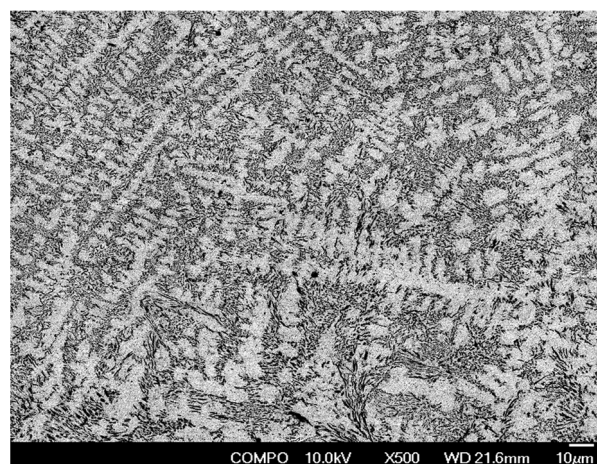


Fig. 3. The microstructure of material in extreme region of the welded seam of the Ti-TiB alloy in longitudinal section ($U_e = 60$ kV, $I_e = 90$ mA, $v_e = 10$ mm·s⁻¹)

The structural features of the material in extreme region of welded seam of the Ti-TiB alloy (see Fig. 3) in comparison with microstructure of welded seam in other regions (see Fig. 2) are due to the presence of a free surface in such extreme region, which provides additional heat removal. Taking into account low thermal conductivity of titanium alloys, this can be an influential factor accelerating the welded seam cooling in its extreme zones. The size of TiB inclusions in extreme area of welded seam is much smaller and their direction in direction perpendicular to the welded seam is more clearly defined. At that, the general distribution of TiB microfibrils has a dendritic character. In

central regions of welded seam the microstructure of the Ti-TiB alloy has a transitional form: from cellular to dendritic. Formation of such structure is practically impossible during eutectoid decomposition in solid state, which excludes this mechanism as the only one and typical for welded seam formation during electron beam welding of the Ti-TiB microcomposite alloy.

To analyze the probability of the 1st or 3rd option from the above described mechanisms of welded seam material structure formation during electron beam welding of the Ti-TiB microcomposite alloy, a spectral analysis of the TiB fibers was carried out to determine the boron content. 60 fibers were used in the sample. The results of analysis of composition deviation for containing boron phases are shown in Fig. 4.

The results obtained indicate that a greater number of fibers in the welded seam zone correspond to TiB_n with $n \sim 1$, which is consistent with the results of X-ray phase analysis. At the same time, up to 13% of containing boron fibers have deviations from this composition (50 at.%Ti, 50 at.%B), $\Delta c/c > 1\%$.

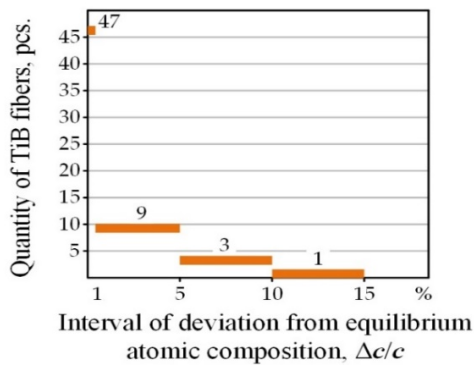


Fig. 4. The results of analysis of TiB microfibers statistical distribution according to the deviation from the equilibrium composition ($n = 1$) based on the Auger spectral analysis of the Ti-TiB microcomposite alloy in the welded seam

This indicates that significant part of reinforcing fibers in the welded seam is in a metastable state and an increase in temperature to a level ensuring the diffuse processes occurrence will lead to relaxation to the thermodynamically stable state (TiB_n with $n \sim 1$). In such a state, when boron is deficient near the microfiber, diffusion of extra Ti atoms from its volume to the surface to the titanium matrix should be expected. Intensification of such type diffusion processes is a positive factor influencing the mechanical properties of the alloy as a whole, since it helps to reduce mechanical stresses at the interface between titanium matrix and TiB microfiber.

Microstructure of material near the heat-affected zone of the Ti-TiB alloy in the longitudinal section of the welded seam ($U_e = 60$ kV, $I_e = 90$ mA, $v_e = 10$ mm·s⁻¹) is shown in Figure 5. It should be noted that in this area there is a greater directivity of TiB microfibers in the direction

from the heat supply zone to the colder base metal. This is evidenced mainly by the cross sections of the TiB fibers in the plane parallel to the joint of the welded elements (Fig. 5).

The results of analysis of TiB_n microfibers statistical distribution by deviation from the equilibrium composition ($n = 1$) based on the Auger spectral analysis of the Ti-TiB microcomposite alloy with respect to the boron containing composition are shown in Fig. 6. Comparison of TiB_n microfibers distribution by deviation from the equilibrium composition of the welded seam (see Fig. 4) and the heat-affected zone (see Fig. 6) allows us to state that there are much more microfibers, containing boron and being in a non-equilibrium state with respect to the boron concentration, in the welded seam material near the heat-affected zone than in the central region of the seam welded material. This may indicate a higher crystallization rate of the melt in contact with the Ti-TiB base material, which remained in the solid state.

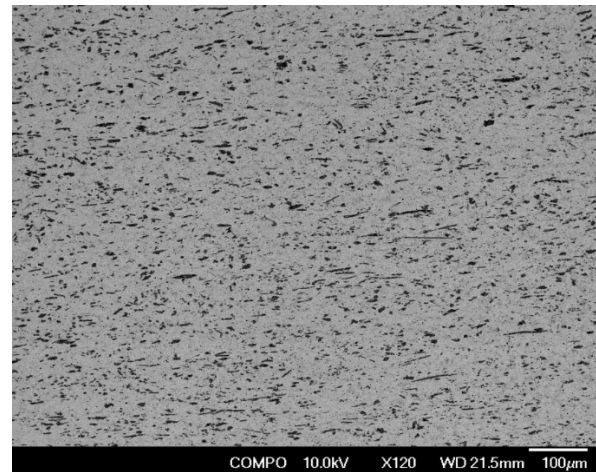


Fig. 5. Microstructure of the welded seam material near the heat-affected zone of welded joint of the Ti-TiB alloy in the longitudinal section ($U_e = 60$ kV, $I_e = 90$ mA, $v_e = 10$ mm·s⁻¹)

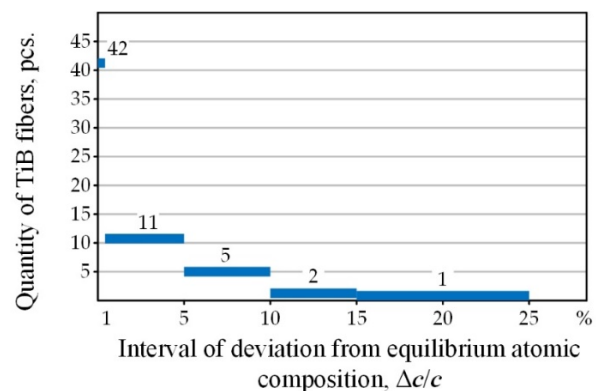


Fig. 6. The results of analysis of statistical distribution of TiB_n microfibers according to the deviation from the equilibrium composition ($n = 1$) based on the Auger spectral analysis of the Ti-TiB microcomposite alloy at the "welded seam" – "base material" boundary

The fact of existence of non-equilibrium phase formations of the TiB_n type with $n < 1$ in the material of the welded joint and at the boundary with the heat-affected zone gives grounds to believe that after the weld pool formation by the electron beam, rapid crystallization occurs through eutectic transformation with the formation of secondary TiB fibers, preferably oriented in the direction of the temperature gradient (from the welded seam axis to the welded samples) with partial formation of metastable phases.

4. Conclusions

1. The structure of the Ti-TiB alloy in longitudinal section of the welded seam, formed under the conditions of electron-beam welding, is characterized by distribution of TiB microfibrils in the form of a dendritic type clusters

throughout the volume of (α -Ti) titanium matrix, decrease in their size by 7-10 times, and the ratio of thicknesses with their length is from 1:6 to 1:7.

2. For boride microfibrils, formed in the welded seam material under the conditions of electron-beam welding, a deviation from the thermodynamically stable composition of TiB_n ($n = 1$) in microfibrils up to 30% of their total amount is typical.

3. Formation of microstructure of the Ti-TiB alloy during electron-beam welding occurs as a result of rapid crystallization through eutectic transformation with TiB microfibrils formation. A high crystallization rate probably leads to partial fixation of the metastable state of boron solid solution in titanium, followed by eutectoid decomposition in the solid state at elevated temperatures due to residual heating.

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Особливості кристалізації матеріалу зварного шва у сплаві Ti-TiB в умовах електронно-променевого зварювання

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Анотація. Природні металеві композиційні матеріали є перспективним класом сучасних конструкційних матеріалів, які необхідно зварювати. Такі матеріали можна зварювати плавленням, що було встановлено на прикладі сплаву Ti-TiB. Підвищені

експлуатаційні властивості таких матеріалів визначає їхня мікроструктура, що характеризується наявністю мікрОВОЛОКОН боридів, карбідів або силіцидів у металевій матриці. Для збереження механічних властивостей матеріалів у зварному з'єднанні необхідно забезпечити формування армуючих мікрОВОЛОКОН у матеріалі зварного шва. Визначення механізму формування боридних мікрОВОЛОКОН, що утворюються в матеріалі зварного шва, стане основою для оптимізації режимів зварювання плавленням, зокрема електронно-променевим зварюванням.

Метою цього дослідження було визначення механізму формування боридних мікрОВОЛОКОН, що утворюються в матеріалі зварного шва. Аналізуються два найбільш вірогідні варіанти механізму формування, які передбачають евтектичний розпад під час кристалізації з рідкого розплаву або евтектоїдний розпад із метастабільного закристалізованого сплаву. Третьою версією був змішаний варіант двох вищевказаних механізмів.

У роботі наведені результати металографічного аналізу особливостей розподілу боридної фази та аналізу елементного складу боридних волокон на основі локальної Оже-електронної спектроскопії. Об'єктом дослідження було з'єднання зі сплаву Ti-TiB, отримане електронно-променевим зварюванням. Факторами аналізу були особливості розмірів, орієнтації та характеру розподілу мікрОВОЛОКОН боридної фази у різних його областях зварного шва. Також досліджувався характерний елементний склад боридних мікрОВОЛОКОН, який характеризував відповідність рівноважним фазам.

Визначено ступінь відхилення співвідношення бору та титану у такій фазі від термодинамічно рівноважного у різних шарах матеріалу зварного шва, сформованого електронним променем у вакуумі. Встановлено залежність розподілу боридної фази у різних умовах теплового обміну матеріалу зварного шва на бокових поверхнях та у центральних областях. Показано, що для частини боридних мікрОВОЛОКОН, що утворюються в матеріалі зварного шва, характерне відхилення від термодинамічно стабільного складу TiB_n ($n = 1$) до TiB_n ($n = 0,85$). Дендритоподібний характер розподілу боридних мікрОВОЛОКОН та наявність метастабільних фазових утворень на основі Ti та B надав підстави для пропозиції щодо переважаючого механізму формування структури матеріалу зварного шва у сплаві Ti-TiB у процесі кристалізації.

Аналіз гіпотетичних варіантів механізму формування боридних мікрОВОЛОКОН, які утворюються в матеріалі зварного шва, показав, що утворення дендритного виду структури характерно для росту кристалів нової фази у рідкій фазі. Для такого росту характерно формування рівноважних фаз. Наявність у зварному шві значної кількості нерівноважної боридної фази свідчить про залишок у титановій матриці нерівноважного бору та подовження росту боридних волокон в закристалізованому зварному шві. Визначеним механізмом формування боридних мікрОВОЛОКОН, які утворюються в матеріалі зварного шва, є евтектичний розпад під час кристалізації з рідкого розплаву із формуванням мікрОВОЛОКНА TiB та подальший рост таких кристалів за рахунок евтектоїдного розпаду із метастабільного закристалізованого сплаву Ti-TiB. Отримані результати дають можливість розуміти механізм формування зварного шва в зварюваних природно-композиційних металевих матеріалах, що дозволяє розробляти рекомендації щодо оптимізації технології зварювання таких матеріалів.

Ключові слова: титанові метали; борид титану; мікроструктура; механічні властивості; зварне з'єднання; електронно-променеве зварювання; термічне оброблення.