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FORMATION OF THE STRUCTURE AND MECHANICAL PROPERTIES OF JOINTS OF TIAIND INTERMETALLIC ALLOY IN DIFFUSION WELDING

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The impact of technological measures in vacuum diffusion welding on formation of the structure and mechanical properties of joints of TiAlNb intermetallic alloy was studied in the work. It is shown that welding of intermetallic alloy by the method of vacuum diffusion welding at temperature $T_w = 1050$ °C, pressure $P_w = 10$ MPa, for 20 min does not ensure producing sound joints. After welding, the joint line is visible in the butt, along which there is a considerable number of defects in the form of pore lines. Increase of welding parameters up to temperature $T_w = 1200$ °C, pressure $P_{\rm w} = 30$ MPa, welding time of 30 min, as well as application of a ductile interlayer from NbTi alloy 1 mm thick allows improving the conditions of welded joint formation and greatly reducing the number of defects in the butt joint. During welding, common grains and diffusion zone 25-35 µm thick form between the interlayer material and the intermetallic alloy. Application of nanolayered interlayer of Al-Ti system of the total thickness of 25 µm in welding of TiAlNb intermetallic alloy, combined with cyclic loading in the form of 3 cycles of loading-unloading leads to a change of the nature of the structure in the joint zone. In the microstructures of welded joints obtained by optical metallography, the joint line is not visible. Application of electron microscopy allows detecting in the butt joint a diffusion zone 15 to 20 µm thick, close by its chemical composition to that of the intermetallic alloy. Investigation of the compressive strength of welded joints demonstrated that the average strength of joints of TiAlNb intermetallic alloy, produced using an interlayer from NbTi alloy, is equal to 988.2 MPa, and application of a nanolayered interlayer of Al-Ti system in welding allows increasing the average strength of the samples up to 1279.8 MPa. 16 Ref., 2 Tables, 8 Figures.

Keywords: TiAlNb intermetallic alloy, diffusion welding, interlayers, joint microstructure

Titanium aluminides represent an important class of alloys which have a unique set of physico-mechanical properties that makes them highly promising for manufacture of aviation engine elements. The main disadvantage of the above alloys is low ductility at room temperature that makes their technological processing and industrial application more complex.

One of the directions of possible solution of the problem of increasing the ductility and adaptability to fabrication, respectively, is creation of alloys with ortho- or B₂-structure. It is known that niobium addition to γ -TiAl promotes increase of its ductility [1]. With this purpose, titanium aluminides are alloyed by a rather large quantity of niobium (up to 25 at.%) and other β -stabilizers. However, alongside many positive characteristics orthorhombic alloys with up to 25 at.% niobium content, have higher density (6.9 g/cm³). In addition, high niobium content leads to considerable increase of the alloy price.

A modern tendency in development of the technology of heat-resistant TiAl-based intermetallics is creation of a class of alloys, having third β -phase in their composition. Presence of this phase facilitates the technological processing of materials, including also rolling and hot pressing [2-4]. y-TiAl alloys contain 42-46 at.% aluminium and also transient metals in the sum of up to 10 at.% total, as master alloy, which stabilize the primary β -Ti phase (known in the low-temperature ordered state as B₂-phase with BCC lattice) In addition to obligatory alloying by Nb, such β-stabilizers as Mo, Ta, Zr, Cr, W, and V can be used. Their application leads to preservation in the alloy of a small volume fraction of residual B2-phase at solidification, which is ductile both at room, and at high temperatures. Molybdenum having high β -stabilizing activity, can be used for creation of B_2 -phase (thus, TNM abbreviation TiAl-Nb-Mo appeared) [5-7]. Development of TNM type alloys allows solving the problem of low room temperature ductility of intermetallics, as well as increasing the high temperature resistance of the products.

At this moment, researchers are paying attention to creation of intermetallic alloys of Ti–Al–Nb ternary system. The complexity of producing joints by

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diffusion welding method consists primarily, in the presence of an oxide layer on their surface, which prevents formation of a welded joint. Alloys of this group also have insufficient ductility that, in its turn, prevents bonding of the contact surfaces at the stage of physical contact. The works on diffusion welding of titanium aluminides are known.

The authors of [8] studied the possibility of diffusion welding of γ -TiAl alloy. It was found that the highest values of shear strength were obtained in the following mode: $T_w = 1000 \text{ °C}$, $P_w = 10 \text{ MPa}$, t = 300 min (388.4 MPa). Despite the absence of defects in the joints, all the samples failed at shear strength values on the level of 25 % from that of the base metal, which is caused by the presence of brittle α_2 -Ti₃Al phase along the butt joint.

In [9] it was shown that at the same mode of welding TiAl alloy with a high Nb content ($T_w = 1100$ °C, $P_w =$ = 30 MPa, t = 45 min), roughness lowering from 0.261 to 0.062 µm allows increasing the shear strength by 100 MPa (to 383 MPa). Control of sample surface roughness also has a positive effect on joint structure formation with its refinement α_2 -Ti₃Al phase disappears in the butt joint. Heat treatment promotes removal of the joint line, this way somewhat increasing the shear strength, but greatly changing the base material microstructure. It becomes coarse-grained, completely platelike.

With the purpose of intensification of the process of formation of joints of TiAl alloy with a high Nb content in diffusion welding, the authors of [10] use pulsed current as the heat source. It is assumed that plasma, which forms in the gaps between the surfaces, activates and cleans them, removing the oxides and contamination. Optimum welding parameters were as follows: $T_w = 1200$ °C, $P_w = 15$ MPa, t = 60 min. In this mode, the initial lamellar microstructure in the joint zone is transformed into the duplex one, having higher mechanical properties. Pressure increase up to 30 MPa at welding temperature of 1200 °C, promotes intensive grain growth, leading to decrease of the joint rupture strength from 657 to 574 MPa.

The authors of [11] point to the need for running of recrystallization processes in the butt joint, in order to produce a sound joint of TiAl alloy with a high Nb content. Diffusion welding at the temperature higher than 1100 °C and 30 MPa pressure leads to recrystallization on the contact boundary, promoting migration of the interface. Shear strength of the joints rises with temperature and pressure of welding, and reaches the highest value (approximately 400 MPa) at the following mode parameters: $T_w = 1150$ °C, $P_w = 30$ MPa, t = 45 min, $T_w = 1100$ °C, $P_w = 40$ MPa, t = 45 min.

One of the methods for welding surface activation is using interlayers, application of which allows local-

izing plastic deformation directly in the butt joint, and minimizing the requirements to roughness and quality of surface preparation.

Work [12] is a study of the possibility of diffusion welding of γ -TiAl, using a mixture of powders of titanium, aluminium and high-purity carbon as an interlayer. Powders were cold-pressed into cylindrical samples 0.5 mm thick. The thus obtained interlayer was placed between the samples, which were welded and heated up to the aluminium melting temperature (660 °C) under the pressure of 15–55 MPa. The powder mixture entered into a reaction, and formed a TiAl, layer on the boundary with titanium aluminide, and a porous mixture of γ -TiAl and TiC phases in the central section of the joint zone. The highest values of rupture strength (approximately 70 MPa) were obtained at welding pressure of 30 MPa. Its smaller or larger values lead to porosity increase and, as a consequence, to lowering of mechanical property values.

In order to reduce the chemical inhomogeneity in the joint zone, there is a need for application of thinner foil, capable, however, of plastic deformation during welding. Foils produced by the method of electron beam evaporation and condensation in vacuum can be regarded as such materials. As shown by previous studies, during welding they can be transformed into a structure, close in its chemical composition to the material being welded [13].

The objective of this work is investigation of the impact of vacuum diffusion welding of TiAlNb alloy with application of interlayers of Nb–Ti or Al–Ti systems, on formation of the structure and mechanical properties of the joints.

Investigation procedures, materials and sample preparation for welding. TiAlNb intermetallic alloy (Ti-28.80Al-11.27Nb-Cr3.51-3.1Zr, wt.%) was used for investigations. The alloy was developed at PWI and produced by the method of electron beam melting with application of lighter β -stabilizers, namely Cr and Zr at reduction of Nb concentration and Al content reduced to 28.80 wt.%.

The developed alloy has the following advantages. First, Nb increases the creep resistance as a result

of lowering of diffusion mobility of the elements, strengthens γ and α_2 - phases, as well as improves TiAl oxidation resistance.

Secondly, Zr and Cr, similar to molybdenum, stabilize β -phase. Zr and Cr are lighter than molybdenum, so that the developed alloy has lower density that is a weighty argument for the aerospace industry. Density of the produced alloy is 4.11 g/cm³, that is almost 1.7 times smaller than for TNM alloys (6.9 g/cm³). In addition, Cr, particularly in microquantities, improves the corrosion resistance.

The initial ingot produced by the method of electron beam melting, had a nonuniform coarse microstruc-



Figure 1. Microstructure of intermetallic alloy after ICZM in the initial condition (white arrow shows the direction of the alloy crystallization in melting)

ture, inhomogeneous distribution of elements through the ingot field, as well as many of pores and cracks. All these disadvantages exactly determined its low mechanical properties at room temperature. It is known that before application of the cast intermetallic material, it should be subjected to gas-static isothermal pressing, many hour heat treatment or rolling [14].

Ingot treatment was performed by the method of induction crucibleless zone melting (ICZM) [15]. Sample microstructure (Figure 1) after zone remelting consists of grains of 31.5 µm average size, elongated in one direction, which also have internal lamellar structure that consists of $\gamma + \alpha_2$ lamellar colonies, along which precipitates of light-coloured layers of β -phase and acicular precipitates of α -hase appear. In the sample center the intergranular boundaries are thin and have the thickness of 2 µm [16].

Metal cutting up into samples for welding was conducted in an EDM machine. Samples of $10 \times 10 \times 5$ mm size were cut out for welding. Surfaces to be joined were ground on a diamond ring and degreased in alcohol.

Welding of intermetallic alloy was performed in U-394M unit. The uniformity of sample heating was ensured due to application of electron beam heater of a circular shape, which was mounted on the butt level. The welding process parameters were as follows: welding temperature $T_w = 1050-1200$ °C, welding pressure $P_w = 10-30$ MPa, welding time t = 20-30 min, vacuum in the working chamber was maintained on the level of $1.33 \cdot 10^{-3}$ Pa. Sample welding was performed with application of static and cyclic loading. Figure 2 gives the cyclograms of welding process.

After the rarefaction on the level of $1.33 \cdot 10^{-3}$ Pa has been achieved in the vacuum chamber, sample heating was performed. After reaching the required temperature and soaking at the mode for several minutes, welding pressure is applied, to equalize the temperature field (duration is determined by sample size).

The total welding time at application of static loading was equal to 20 min, and at application of cyclic loading it was 30 min. At cyclic loading three cycles of sample loading-unloading were applied.

Sample welding was conducted both without application of interlayers, and with interlayers in the form of foil. Niobium-titanium alloy or nanolayered foil of Al–Ti system were used as interlayers.

The interlayer from NbTi alloy was produced by the technology of cold-hearth electron beam melting of intermetallic alloy of TiAl system and niobium alloy 5VMTs. Niobium in the alloy promotes an increase of heat resistance, ductility and oxidation resistance. The composition of the produced NbTi alloy was as follows: Nb–43.49Ti–3.06W–2.35Al, wt.%.

As shown by investigations, metal hardness is uniform and equal to HV-1810–1930 MPa over the ingot cross-section. The interlayer from NbTi alloy was cut out in the EDM machine, which was followed by grinding its surfaces. The interlayer thickness was 1 mm.

Nanolayered foil based on Al–Ti system was produced by electron beam evaporation and condensation in vacuum. The deposition process consists in layer-by-layer condensation of elements on a horizon-



Figure 2. Cyclogram of VDW process: a — VDW with static loading; b — VDW with cyclic application of pressure; T_w — welding temperature; P_w — force of pressing the samples together; t_1 — duration of heating to T_w ; t_2 — duration of soaking at T_w ; t_3 — welding time; t_4 — cooling time

tal substrate, which rotates and which is fixed on the shaft of UE204 unit.

Nanolayered foil (Table 1) for application as an interlayer was selected proceeding from the composition of materials, which were welded, so that the interlayer components acted as base material alloying elements. The foil is characterized by uniform distribution of elements across the thickness.

Investigations of the structure and phase composition of the produced joints were conducted using the methods of optical microscopy in Neophot-32 microscope and scanning electron microscopy (SEM) in CAMSCAN 4 microscope, fitted with energy-dispersive analysis system Oxford Inca Energy 200. In order to determine the chemical composition of elements in the joint zone, investigations were conducted on flat samples, which were prepared by the standard procedure, using grinding-polishing equipment of Struers Company. This procedure was used to prepare transverse macrosections of both the foil and the welded joints.

In order to reveal the sample microstructure by optical metallography method, etching was performed in a reactive consisting of a mixture of hydrofluoric and nitric acid, in the following proportion: 1 part of hydrofluoric acid (HF) and 3 parts of nitric acid (HNO₃). The photos of the joint microstructure were taken by a digital camera C-3000 of OLYMPUS Company.

Sample microhardness was measured in hardness meter M-400 of LECO Company with a diamond pyramid. The load was 25 g.

Samples for mechanical testing were cut out of welded joints by EDM machine. The sample size was $4 \times 4 \times 8$ mm. Metallographic studies of the structure and chemical composition were conducted on one of the samples obtained after welding, and the others were used for evaluation of mechanical properties of the welded joints.

Welding of intermetallic alloy TiAlNb without application of interlayers. Welding of intermetallic TiAlNb alloy was conducted at temperature $T_w =$ = 1050 °C, pressure $P_w =$ 10 MPa, for 20 min. As shown by metallographic investigations of the sam-

Table 1. General characteristics of Al/Ti foil

Foil	Total foil thickness, μm	Layer thickness, nm		Foil composi- tion, at.%		Foil composi- tion, wt.%	
		Al	Ti	Al	Ti	Al	Ti
Al/Ti	25	30	25	47.14	52.86	33.45	66.55

ples, the joint line is visible in the butt. A considerable number of defects are located in the form of a pore sequence along this line, that is readily revealed at chemical etching of the joint (Figure 3, a).

Increase of welding mode parameters to $T_w = 1200 \text{ °C}$, $P_w = 30 \text{ MPa}$ at soaking for 30 min, allows greatly reducing the number of defects in the butt joint (Figure 3, *b*). Material microhardness directly in the joint zone is equal to *HV* 4800 MPa, at 20 μ m distance from the butt it is *HV* 4730, and at 50 μ m distance it is *HV* 4180 MPa, respectively.

Welding of intermetallic alloy TiAlNb using an interlayer based on NbTi alloy. Application of an interlayer of a softer material than the intermetallic alloy in welding allows improving the conditions of welded joint formation (Figure 4). In welding in the following mode: $T_w = 1050 \text{ °C}$; $P_w = 15 \text{ MPa}$ and t = 30 min, a diffusion zone is observed in the butt joint, where the metal structure differs from that of the intermetallic TiAlNb alloy (Figure 4, *a*). This zone is separated from two sides (relative to the interlayer) from the intermetallic alloy by clearcut lines, along which clustering of defects is observed.

At increase of welding parameters up to temperature $T_w = 1200$ °C and pressure value $P_w = 30$ MPa and preservation of soaking time t = 30 min, the number of defects in the butt joint becomes much smaller (Figure 4, b).

Application of electron microscopy allows revealing significant diffusion of chemical elements in the butt joint (Figure 5). Figure 5, a, shows half of the welded joint.

As one can see from Figure 4, *b* and Figure 5, *a*, common grains form in the butt joint between the interlayer material and the intermetallic alloy during welding.



Figure 3. Microstructure (×25) of TiAlNb alloy joint zone in welding in the following mode: $a - T_w = 1050$ °C, $P_w = 10$ MPa, t = 20 min; $b - T_w = 1200$ °C, $P_w = 30$ MPa, t = 30 min

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Figure 4. Microstructure of TiAlNb alloy joint zone in welding with application of an interlayer from NbTi alloy in the following mode: $a - T_w = 1050$ °C, $P_w = 15$ MPa, t = 30 min (×25); $b - T_w = 1200$ °C, $P_w = 30$ MPa, t = 30 min (×200)

A diffusion zone 25–35 μ m thick is observed along the interlayer — TiAlNb alloy interface. Chemical composition of this zone corresponds to the following element content: 47.04Ti–29.31Nb–20.28Al–1.97Cr–1.4W, wt.%. In the middle part the chemical composition of the interlayer (66.34Nb–28.83Ti–3.48W–1.35Al, wt.%) is preserved due to its considerable thickness.

As shown by investigation results, an increased level of microhardness values up to HV 5090 MPa is observed in the near-contact zone of the interlayer — intermetallic alloy. Microhardness in the intermetallic alloy proper at 0.1 mm distance from the butt joint, is equal to HV 3670 MPa, at 2 mm it is HV 4120 MPa, and in the interlayer central part it is HV 2440 MPa, respectively.

Welding of intermetallic alloy TiAlNb using nanolayered interlayer of Al-Ti system. Welding of intermetallic alloy TiAlNb, using nanolayered interlayer of Al–Ti system of total thickness of 25 μ m was conducted at the following parameters: temperature $T_w = 1200$ °C, pressure $P_w = 30$ MPa, soaking time t = 30 min. Analysis of joint microstructure shows that during welding a diffusion zone approximately 20–25 μ m thick forms in the place of location of the nanolayered interlayer (Figure 6, *a*). Finely-dispersed precipitates are observed in the middle of the diffusion zone. The lines of interface with the intermetallic alloy pass on both sides from this zone. No cracks or pores were found in the butt joint.

Application of cyclic loading in welding (3 loading and unloading cycles) leads to a change of the nature of the structure in the joint zone. No contact line is found in welded joint microstructure, obtained by optical metallography. The line of contact of the nanolayered interlayer with the intermetallic alloy as an element of the microstructure is absent (Figure 6, b).

Electron microscopy allows revealing in the butt joint a diffusion zone approximately $15-20 \mu m$ thick (Figure 7, *a*), close in its composition to the intermetallic alloy.

Chemical composition of elements in the joint zone is equal to: 67.12Ti-31.31Al-1.57Cr, wt.%. That is after welding of the intermetallic alloy, using a nanolayered



Speetrum	Chemical composition, wt.%						
Spectrum	Al	Ti Cr		Nb	W		
2	1.35	38.83	-	66.34	3.48		
3	20.28	47.04	1.97	29.31	1.4		
4	31.38	56.56	2.56	9.49	-		

Figure 5. Microstructure of the zone of TiAlNb alloy joint, produced with application of an interlayer from NbTi alloy in the following mode: $T_w = 1200$ °C, $P_w = 30$ MPa, t = 30 min (*a*); distribution of chemical elements in the butt joint (*b*); content of chemical elements in individual regions in the joint zone (*c*)



Figure 6. Microstructure of TiAlNb alloy joint zone in welding using a nanolayered interlayer of Al–Ti system in the following mode: $a - T_w = 1200 \text{ °C}, P_w = 30 \text{ MPa}, (\times 500); b - T_w = 1200 \text{ °C}; 3 \text{ cycles of pressure } P_w = 30 \text{ MPa}; t = 30 \text{ min} (\times 50)$



Figure 7. Microstructure of zone of TiAlNi alloy joint, produced using a nanolayered interlayer of Al–Ti system in the following mode: $T_w = 1200$ °C, 3 cycles of pressure $P_w = 30$ MPa, t = 30 min (*a*); distribution of chemical elements in the butt joint (*b*) and chemical element content in individual regions of the joint zone

interlayer, a diffusion zone forms in the butt joint, in which an increased content of titanium (67.12 %), aluminium (31.31 %), and a small content of chromium on the level of 1.57 wt.% (Figure 7, b) are found.

As shown by investigation results, an increase of microhardness values up to HV-5160–5400 MPa is observed in the diffusion zone. Microhardness values in the intermetallic alloy are equal to HV 4370 MPa.

Investigations of mechanical properties of welded joints. Investigations of compressive strength

of welded joints were conducted according to ASTM D695 standard. Mechanical properties of the alloys at room temperature were studied by uniaxial compression, using INSTRON 8802 testing machine and extensometer 2620-601. Strain rate was $2 \cdot 10^{-4} \cdot s^{-1}$. Application of the compression method is the most effective, when samples dimensions are small. A diagram in σ - ϵ coordinates is realized. Table 2 gives the parameters of samples and results of compressive testing of the samples.

Table 2. Parameters of samples and res	ults of compressive testing of samples
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Sample number	Joint type	Sample area <i>F</i> , mm	Tensile strength, σ_t	Yield limit $\sigma_{0.2}$	Relative reduction in area at maximum force $\delta^{(\sigma)}$, %	Modulus of elasticity <i>E</i> _t , MPa
1-1		15.52	1021.2	637.0	10.5	71893.0
1-2	Welding with interlayer of NbTi alloy	16.10	1044.4	608.0	11.8	87872.0
1-3		15.54	898.8	600.0	9.7	84306.0
2-1		16.28	1153.1	605.0	12.3	90327.0
2-2	Welding with nanolayered interlayer	16.34	1435.3	628.0	19.1	84784.0
2-3		16.92	1250.9	615.0	13.6	95480.0



Figure 8. Diagram of testing samples from TiAlNb alloy, produced in welding with an interlayer of NbTi alloy (*1*) and in welding with nanolayered interlayer (*2*)

Proceeding from test results, a series of diagrams was derived in σ - ϵ coordinates, which are given in Figure 8.

Mechanical testing of the joints for compression revealed that the average strength of the joints from TiAlNb intermetallic alloy, produced using an interlayer from NbTi alloy, is equal to 988.2 MPa, and average strength of samples, produced with a nanolayered interlayer of Al–Ti system is 1279.8 MPa.

Conclusions

1. Welding of intermetallic alloy TiAlNb by vacuum diffusion process at temperature $T_w = 1050$ °C, pressure $P_w = 10$ MPa, for 20 min, does not ensure producing sound joints. After welding, a contact line is observed in the butt joint, along which a large number of defects are located in the form of linear porosity.

2. Increase of welding mode parameters up to temperature $T_w = 1200$ °C, pressure $P_w = 30$ MPa, soaking time t = 30 min, and use of a ductile interlayer from NbTi alloy 1 mm thick allows improving the conditions of welded joint formation and greatly reducing the number of defects in the butt joint. Common grains and a diffusion zone 25–35 µm thick form along the interlayer intermetallic alloy boundary between the interlayer material and the intermetallic alloy during welding.

3. Application of nanolayered interlayers of Al– Ti system of the total thickness of 25 μ m and cyclic loading in the form of 3 cycles of loading-unloading in welding the intermetallic alloy TiAlNb leads to a change of the nature of the structure in the joint zone. No joint line is visible in the microstructure of welded joints, produced by optical metallography. Application of electron microscopy allows revealing in the butt joint a diffusion zone 15 to 20 μ m thick, close in its composition to that of the intermetallic alloy.

4. Investigations of compressive strength of welded joints showed that the average strength of joints from intermetallic alloy TiAlNb, produced using an interlayer from NbTi alloy, is equal to 988.2 MPa, and application of nanolayered interlayer of Al–Ti system in welding allows increasing the average strength of the samples up to 1279.8 MPa.

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