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INFLUENCE OF TEMPERATURE OF HEATING IN VACUUM ON BEHAVIOUR OF OXIDE FILM ON THE SURFACE OF γ-TiAl INTERMETALLIC ALLOY

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The paper deals with the influence of temperature of heating in vacuum and soaking time on oxide film recovery on the surface of γ-TiAl intermetallic alloy. Analysis of publications on diffusion welding of titanium aluminides showed a considerable scatter of parameters in welding modes recommended by the authors (temperature, pressure and process time). Influence of concentration of oxygen contained in oxide layer on contact surfaces of samples, on their adhesion during heating in vacuum was analyzed. It is experimentally established that with increase of the temperature of heating pre-oxidized samples of titanium aluminide from 900 up to 1200 °C, the nature of surface relief in the contact zone changes from flat to voluminous one. It is found that heating of oxidized samples of γ-TiAl intermetallic alloy under vacuum at 1200 °C for 20 min at 5 MPa pressure allows reducing oxygen content in the butt joint 5 times from 40.99 to 6.12–7.74 wt.%. 18 Ref., 2 Tables, 7 Figures.

Keywords: *γ-TiAl intermetallic alloy, oxide, vacuum, heating temperature*

γ-TiAl-based intermetallic alloys are regarded as promising materials for manufacturing components of aviation equipment, operating at elevated temperatures. The advantage of such alloys over other alloys is due mainly to low specific density and high heat resistance.

References [1, 2] showed the possibility to produce γ-TiAl welded joints with application of multilayer foils of Al–Ti system. Here optimum welding modes were determined: heating temperature T = 1200 °C, compression pressure P = 20 MPa, time of soaking at pressure t = 20 min.

Works of other authors [3–7] show considerable differences as to the applied welding modes (Table 1). So, [3] recommends welding temperature T = 1350 °C at pressure P = 10 MPa and process time t = 45 min. In References [4, 5] the following welding mode is believed to be optimum: T = 1150–1200 °C at pressure P = 15–20 MPa and process time t = 60 min. The authors of [6] recommend substantial increase of welding process time up to t = 180 min at temperature T = 1100 °C and pressure P = 20 MPa. In Reference [7] satisfactory results were obtained at application of increased values of pressure P = 300 MPa and interlayer from titanium alloy VT1.

It is known that weldability of titanium alloys is determined by their gas absorption, thickness of oxide film on contact surfaces, as well as structural transformations in the metal [8]. In vacuum diffusion welding of titanium-based alloys cleaning of the surfaces being welded from oxide films is associated with their dissolution in the metal, as well as development of tensile stresses in the butt at pressure application, leading to cracking of harder and more brittle oxide films, and with recrystallization processes.

At heating in vacuum complex physical-chemical processes proceed on contact surface of the abutted parts, associated with autovacuuming of their internal volume, heating of gas inside it, sorption and desorption and oxide film destruction. So, Reference [9] shows the principal possibility of metal autovacuum-

Table 1. Recommended modes of welding γ-TiAl intermetalic alloys

Alloy, at.%	Welding mode			Source
	T, °C	P, MPa	t, min	
Ti–48Al–2Mn–2Nb	1350	10	45	[3]
Ti–38Al	1200	20	60	[4]
Ti–47Al–4 (Cr, Nb, Mn, B)	1150	15	60	[5]
Ti–47Al–4 (Cr, Nb, Mn,B)	1100	20	180	[6]
Ti–44.7Al–4.65Nb–2.73Mn–0.31B	850	300	20	[7]

ing at heating and self-cleaning of contact surfaces from oxide films.

The authors of [10], based on investigation of titanium alloys VT1, OT4, VT6 come to the conclusion that the process of oxide film dissolution in the alloy metal matrix is controlled by oxygen diffusion in it. Analysis of kinetic curves of oxide dissolution in VT1 alloy is indicative of decaying nature of the process of oxide dissolution. Unlike VT1 alloy, in aluminium-containing alloys OT4 (3.5Al, 1.5Mn) and VT6 (6Al, 4.5V) the nature of oxide film dissolution is more complicated. Oxidation in air of titanium alloys, doped with aluminium, is accompanied by formation of a layer with higher aluminium content on their surface.

Research performed in [8] showed that annealing at heating temperature T = 700 °C for 12–15 min under the conditions of autovacuuming of samples from OT4-1 alloy (1.5Al, 1.0Mn) with initial oxide film thickness of about 54 nm (light-blue colour) ensures its discoloration that corresponds to oxide film thickness of about 20 nm.

By the data of [11] with increase of heating temperature and development of physical contact between the surfaces being welded, conditions are in place for «non-oxidizing» annealing that leads to oxygen dissolution in titanium, and chemical compounds of aluminides remain in the butt joint zone in the form of films. Welded joint formation will be controlled by diffusion process of lowering aluminium concentration to the level, when existence of its chemical compounds is impossible.

Thus, it can be assumed that to produce joints from intermetallic alloys based on titanium aluminides, having increased aluminium content, the question of oxide film recovery on contact surfaces of the samples, is also important.

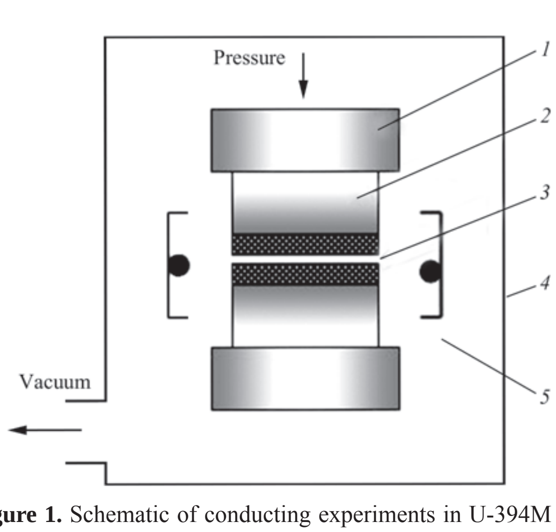

Figure 1. Schematic of conducting experiments in U-394M unit (for description of *1*–*5* see the text)

Known are works devoted to investigation of oxidation resistance of both intermetallic alloys based on γ-TiAl [12–14], and of coatings on these alloys [15, 16].

For γ-TiAl intermetallic alloys, however, the influence of technological parameters of diffusion welding on physical-chemical state of the contact surface has not been studied. In this connection the objective of the work consisted in studying the influence of heating temperature on dissolution of oxide film on γ-TiAl surface.

Materials and methods of investigation. Investigations were conducted on samples of γ-TiAl intermetallics (Ti–33.36Al–4.97Nb–2.68Cr, wt.%). Sample size was 10×8×3 mm. Sample surfaces were polished on P400 sandpaper. Sample heating was performed in U-394M unit (Figure 1). Samples were heated at the rate v = 150 °C/min up to 800 °C temperature and soaked for 5 min, which was followed by their oxidation in air.

Furtheron samples of intermetallics (*2*) with a layer of oxide on contact surfaces (*3*) were placed in the abutted condition between the dies (*1*) and heated in the unit vacuum chamber (*4*) by slot-type electron beam heater (*5*) up to temperature: T = 900, 1050 and 1200 °C for t = 20 min in vacuum $B = 1.33 \cdot 10^{-3}$ Pa. Sample preloading force was P = 5 MPa. After cooling to 20 °C the samples were taken out of vacuum chamber and separated. Sample surfaces were studied by the methods of optical and electron microscopy. Micromechanical characteristics of surface metal layer were evaluated using PMT-3 instrument with 2 N load.

It should be noted that basic methods for studying the kinetics of interaction of titanium alloys with oxygen, conducted in 1970–1980, were as follows: interference indication, ellipsometry and gravimetry. In our case, investigation of the structure and determination of its elemental composition were conducted by the method of X-Ray microanalysis (XRMA) in an analytical complex, consisting of scanning electron microscope JSM-35 CF of JEOL, and X-ray spectrometer with dispersion by X–ray quantum energy (model INCA Energy-350 of Oxford Instruments Company). As noted in a number of works [17, 18] this method has been applied with success to study the kinetics of metal oxidation.

Experimental results and discussion. After oxidation the sample surface had dense oxide film of dark-gray colour (Figure 2). Microhardness of γ-TiAl intermetallic alloy in the initial state is equal to 4.6 GPa. A significant increase in microhardness up to 13 GPa is found after oxidation.

According to the data of [6], oxide film of dark-gray colour on γ-TiAl surface consists of TiO_2 (rutile) and α-Al_2O_3.

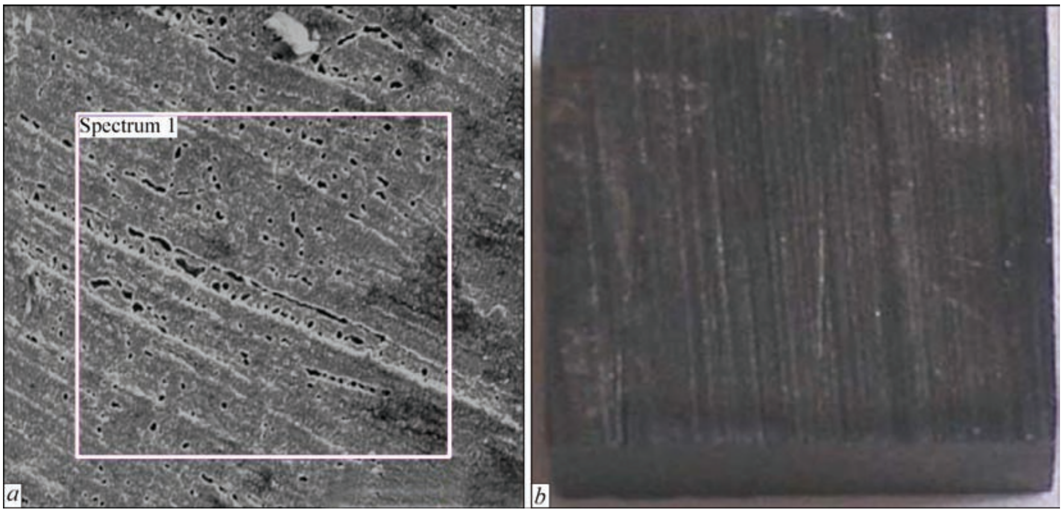

Figure 2. Appearance of the surface of γ-TiAl sample after heating in vacuum and oxidation in air: *a* — electron (×250); *b* — optical (×25) microscopy

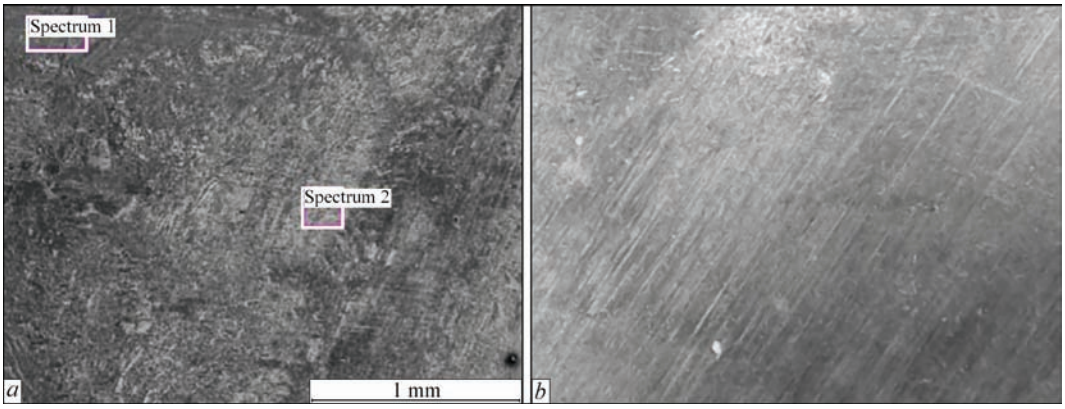

Figure 3. Appearance of γ-TiAl sample after heating in vacuum at 900 °C: *a* — electron (x30); *b* — optical (×50) microscopy

After oxidation, oxygen content on the sample surface is equal to 40.99 wt.% (Table 2).

As shown by our studies, autovacuuming of samples (Figure 1) at their heating in vacuum up to 900 °C and soaking for 20 min, allows lowering oxygen content on the contact surface from 40.99 to 28.62–29.32 wt.%. Individual areas of adhesion are found on sample surface. Their total number, however, is small (Figure 3). It should be also noted that autovacuuming of samples also results in the change of colour of their contact surfaces from dark-gray to gray. Such a change of surface colour, in our opinion, can be associated with oxygen dissolution in the sample metal that is in agreement with the data of works [8, 10].

Table 2. Element content on the surface of γ-TiAl sample

Treatment mode	Point number	Element content, wt.%				
		[O]	Ti	Al	Cr	Nb
After oxidation	1	40.99	36.48	17.83	1.38	3.31
Heating at 900 °C	1	28.62	41.80	22.50	2.44	4.64
	2	29.32	44.71	21.32	1.79	3.03
Heating at 1050 °C	1	13.01	53.22	27.47	2.29	4.01
	2	13.89	53.84	25.46	2.20	4.62
Heating at 1200 °C	1	6.12	77.56	11.58	0.00	4.74
	2	7.74	67.06	16.70	1.26	7.25

Increase of the temperature of sample soaking in vacuum to 1050 °C leads to appearance of longer regions of adhesion with oxygen content of 13.01–13.89 wt.% (Figure 4). In addition to adhesion regions having the colour of unoxidized metal, individual regions of light-grey colour are also observed on contact surface of the samples.

At further increase of heating temperature up to 1200 °C, the nature of surface relief changes significantly (Figure 5), transforming from flat into a voluminous one. Separation ridges are observed in adhesion portions, presence of which is indicative of bonding of these parts of contact surfaces.

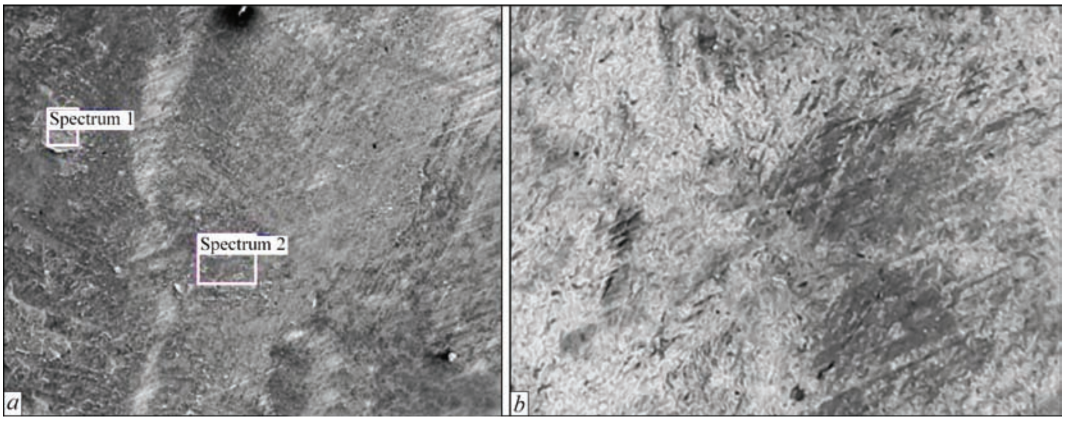

Figure 4. Appearance of γ-TiAl sample after heating in vacuum at 1050 °C: *a* — electron (×30); *b* — optical (×50) microscopy

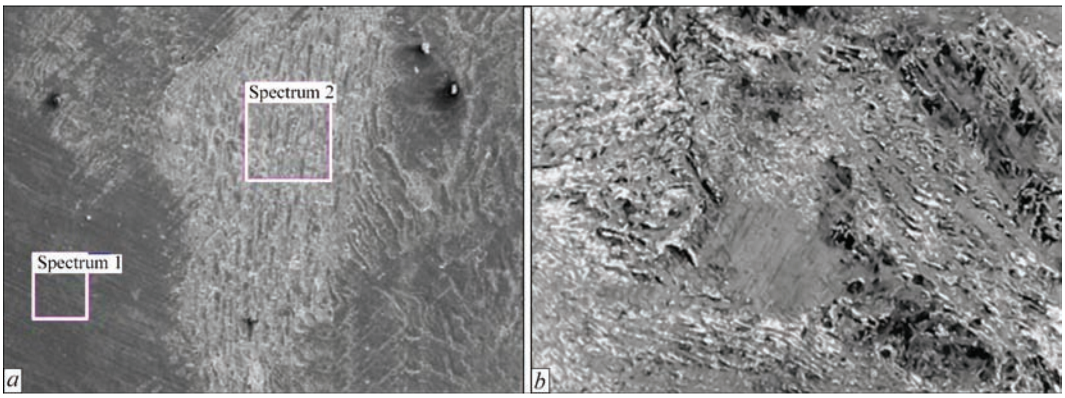

Figure 5. Appearance of γ-TiAl sample after heating in vacuum at 1200 °C: *a* — electron (×30); *b* — optical (×50) microscopy

At heating temperature of 1200 °C adhesion areas take up 90 % of sample surface. Soaking at this temperature ensures complete discoloration of the contact surface that is similar to the mechanism of self-cleaning of doped titanium alloys from oxides [8]. Oxygen content on the surface of γ-TiAl samples is equal to 6.12–7.74 wt.%. Evaluation of micromechanical characteristics on the sample surface after their auto-vacuuming at T = 1200 °C shows that microhardness decreases from 13 (after oxidation) to 6.5 GPa, as a result of oxygen dissolution in the matrix of intermetallics.

Figure 6 is the graph of heating temperature influence on oxygen content on contact surface of γ-TiAl samples.

As is seen from the graph, titanium aluminide heating at 1200 °C for 20 min in vacuum allows oxygen content in the butt to be reduced 5 times: from 40.99 after oxidation to 6.12–7.74 wt.%.

Experimental results agree with those of [6], in which the influence of diffusion welding parameters on formation of physical contact in the butt joint was studied on γ-TiAl alloy. The authors of the work come to the conclusion that optimum welding temperature

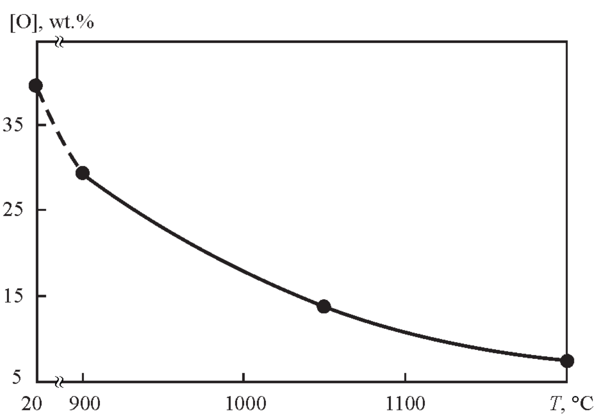

Figure 6. Influence of heating temperature on oxygen content on contact surface of γ-TiAl samples

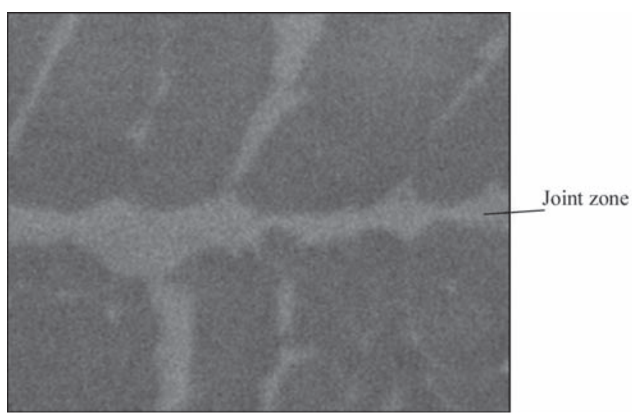

Figure 7. Microstructure (×3000) of the zone of γ-TiAl joint produced at diffusion welding in the following mode: T = 1200 °C, t = 20 min, P = 7 MPa

is T = 1200 °C at soaking time t = 60 min, and pressure P = 10 MPa that in the authors' opinion allows producing welded joints without residue of oxide film in the butt weld.

As shown by our studies, vacuum diffusion welding of γ-TiAl intermetallic alloy in the following mode: T = 1200 °C, t = 20 min, P = 70 MPa, also provides joints (Figure 7), which have no defects in the form of micropores or residue of oxide inclusions.

Thus, it is shown in the case of investigation of oxygen content on γ-TiAl surfaces after oxidation in air and heating in vacuum, that sample heating up to 1200 °C at soaking for 20 min promotes reduction of oxygen cotent on contact surfaces by more than 5 times from 40.99 in as-oxidized state up to 6.12–7.74 wt.%. Analysis of fracture surface of samples after their autovacuuming shows that dissolution of oxide film in intermetallic alloy matrix in the contact zone results in complete discoloration of the mated surfaces that is similar to the mechanism of self-cleaning of doped titanium alloys from oxides. Vacuum diffusion welding of γ-TiAl (Ti–33.36Al–4.79Nb–2.68Cr, wt.%) in the following mode: temperature T = 1200 °C, pressure P = 70 MPa, process time t = 20 min provides formation of defectfree joints.

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