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REACTIVE-FLUX BRAZING OF ALUMINIUM TO TITANIUM

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ABSTRACT

At brazing dissimilar joints of AD1 aluminium to VT1-0 titanium at the temperature of 605–610 °C by Al–12Si brazing filler metal in argon application of reactive flux of $KAlF_4$–$10K_2SiF_6$ system with additives of CoF_2, K_2ZrF_6 compounds, promotes production of a sound joint due to formation of a low-melting alloy of Al-Si system on the contact surface. The low-melting alloy of Al–Si system newly-formed at reactive-flux brazing can independently fulfill the function of brazing filler metal at formation of a dissimilar metal joint. Cobalt reduced from the flux has little influence on weld structure and joint strength. At application of Al–12Si brazing filler metal and reactive flux of $KAlF_4$–$10K_2SiF_6$–$5K_2ZrF_6$ system, which contains potassium-zirconium fluoride (K_2ZrF_6), a certain refinement of the structure (dendrites of aluminium-based solid solution) is observed from the aluminium side that promotes an improvement of shear strength of aluminium-titanium brazed joints.

KEYWORDS: aluminium, titanium, reactive-flux brazing, Al–Si brazing filler metal, reactive flux of KF–AlF_3–K_2SiF_6 system, brazed joint

INTRODUCTION

Aluminium and titanium structures are optimal by strength/weight ratio, have high corrosion resistance and strength and are characterized by a wide spectrum of potential application in automotive and aerospace industry.

At present methods of welding and brazing of aluminium to titanium are actively developed. Their mechanically loaded joints are used in structures of various products. Formation of a sound joint of aluminium and titanium, as well as of their alloys is a complex problem, because of a considerable difference of physico-chemical properties of the metals (melting temperature. thermal expansion coefficient, heat conductivity, corrosion resistance), active interaction with gases (O_2, N_2, H_2), presence of a dense film from refractory oxides on the surface and ability to form brittle intermetallic compounds.

Effective application of electromagnetic radiation, filler material, joint configuration at laser braze-welding [1–6]; improvement of the geometrical shape of the rotating tool and optimization of its movement modes in friction stir welding [7–9] — this is an incomplete range of methods aimed at breaking up the highly stable oxide film, formation of a favourable weld structure and strong joint. At spot stir braze-welding (close to melting temperature of Zn–Al eutectic) of A2014 aluminium alloy and Ti6Al4V alloy application of a double coating (Al and Zn) on titanium increases (by 110 %) the shear strength of the joint [10], compared to the traditional technology.

High-temperature brazing in vacuum (purged by argon) by aluminium brazing filler metal below the critical temperature of titanium ($T < 800$ °C), aluminium($T < 630$ °C), and their alloys is the best choice as to the cost and preservation of mechanical properties of a joint of dissimilar metals [11–16]. Temperature limitation is due to undesirable changes of the microstructure and properties of both the thin-walled base metal and the joint: i.e. below α↔β-phase transformation in Ti [17, 18] and considerable loss of aluminium alloy strength at heating [19]. At titanium interaction with liquid aluminium and brazing filler metals (Al–Me systems (Me = Ag, Cu, Si), Al–Si–Cu, Al–Si–Mg) brittle intermetallic compounds form, which are close to $TiAl_3$ [20–22], Al_2Ti [23], and $Al_xSi_xTi_z$ [12,14, 24, 25] by their stoichiometric composition, and whose interlayer has different influence on brazed joint strength. For instance, increase of soaking time ($t \leq 25$ min) at brazing temperature of 620 °C promotes an increase of the strength of Al/Ti joint brazed with Al–12Si–1Mg brazing filler metal [12]. At 620 °C temperature silicon diffusion from the brazing filler metal into Al results in isothermal crystallization of the solid solution, and a double layer from $Al_5Si_{12}Ti_7$, $Al_{12}Si_3Ti_5$ intermetallic compounds forms on titanium.

Prior application of an interlayer from (67Ag–33Al) alloy or (50Zn–50Al) coating on titanium at brazing by Al–Si brazing filler metal by immersion into the flux melt [26] and in vacuum [27] does not lead to any significant increase of shear strength ($\tau_{sh} \leq 40$ MPa) of Al/Ti joint.

Based on investigation results it was established that application of reactive flux of KF–AlF_3–K_2SiF_6 salt system improves wetting and formation of the joint between the parts at brazing of aluminium alloys with low (≤ 0.7 wt.%) magnesium and aluminium content with steel [28–30]. At reactive-flux braz-

Table 1. Chemical composition of base materials and brazing filler metal, wt.%

Metal	Si	Fe	Cu	Mn	Mg	Cr	Ti	Al	O	N	H	C
AD1	0.15	0.30	0.30	0.05	0.02	0.05	0.10	99.3	–	–	–	–
VT1-0	0.1	0.15	–	–	–	–		–	0.2	0.04	0.01	0.07
AK12	10–13	<1.5	<0.6	<0.5	<0.1	<0.1Zn	<0.1	–84.3	–	–	–	–

ing active cleaning of the aluminium contact surface takes place, silicon is reduced from K_2SiF_6 compound in a short time, its content in the weld changes as a result of diffusion in the liquid state and properties of the joint of similar and dissimilar metals change accordingly.

Improvement of aluminium and titanium wetting by brazing filler metal at flux brazing, when their surface is covered by a strong refractory oxide film, and possibility of weld alloying by elements reduced from the reactive fluoride flux, are a factor in the formation of quality joints of components from dissimilar materials.

This work presents the results of studying the structure and strength of titanium-aluminium joint, formed with application of Al–12Si brazing filler metal and powder flux of $KF–AlF_3–K_2SiF_6$ salt system with CoF_2, K_2ZrF_6 additives in temperature-time modes that were established for high-temperature brazing of aluminium in argon.

MATERIALS AND EXPERIMENTAL PROCEDURE

Experiments were performed using tee-samples (40×40×1 mm substrate, 40×5×1 mm strip, assembled with a narrow gap of less than 0.1 mm) from AD1 aluminium alloy and VT1-0 titanium, AK12 brazing filler metal of Al–Si system (Table 1) and non-hygroscopic reactive fluxes: $KA1F_4–10K_2SiF_6$, $KA1F_4–10K_2SiF_6–CoF_2$, $KA1F_4–10K_2SiF_6–5K_2ZrF_6$.

Preparative synthesis method with application of reagents (hydrofluoric acid HF, $Al(OH)_3$ aluminium hydroxides and KOH potassium hydroxides and SiO_2 silicon oxide) were used to obtain $KA1F_4–10K_2SiF_6$ reactive flux. Reactive flux was mixed with addition of ready CoF_2 (ch.cl), K_2ZrF_6 (ch.cl) chemical compounds to produce the required compositions of homogeneous dispersed powder mixture. Before brazing metal samples were cleaned in water solutions of: 15 % NaOH and degreased, 20 vol.% HNO_3, 2 vol.% HF and etched, and washed in distilled water between the operations. Tee-samples were assembled by placing a strip from aluminium alloy (titanium) on base metal substrate. Powder flux of ~ 0.06 g weight (in the sample upper zone) and a sample (0.17 g) of Al–12 Si aluminium brazing filler metal with flux (in the sample lower zone) were applied along the line of contact of the strip with base metal substrate. Flux brazing was performed at the temperature of 600–620 ± 2 °C in pure argon atmosphere (vol.%): 99.987Ar, $0.002O_2$, $0.01N_2$, $0.001H_2O$ at dew point temperature $T = –58$ °C. Sample image was obtained with Panasonic FZ-30 digital camera. Brazed joint microstructure was studied using optical (Neophot-32) and scanning electron microscope (JSM840). Brazed joint strength was determined by tensile testing of overlap samples (two assembled plates of the following dimensions: 55 mm length, 15 mm width of the working part, 1.0 mm thickness) in R-5 tensile testing machine with maximum force of 50 kN. Mathematical processing methods with application of HSC 6.0 program were used for calculation of Gibbs energy.

INVESTIGATION RESULTS AND DISCUSSION

Based on calculations (using HSC 6.0 program) of the change of Gibbs free energy (ΔG) the nature of running of chemical reactions (1), (2) at aluminium interaction with chemical compounds (K_2SiF_6, CoF_2) under the high-temperature conditions was determined as follows:

$$4Al + 3K_2SiF_6 = K_3AlF_6 + 3KAlF_4(g) + 3Si\ (D\,G_{600\,^{\circ}C} = -942\ \text{kJ}). \qquad (1)$$

aluminium interaction with potassium hexafluorosilicate (K_2SiF_6) by reaction (1) can result in silicon reduction in the composition of double fluoride is possible.

Conducted research of interaction of reactive flux of $KF–AlF_3–K_2SiF_6$ salt system on aluminium substrate (that is above the temperature of formation of Al–Si double eutectic) in high-purity argon atmosphere [31] showed that two processes proceed on aluminium surface: silicon reduction from the composition of potassium hexafluoride and contact-reactive melting of silicon with aluminium. Such an interaction results in formation of a metallic layer of Al–Si system, which improves the wetting and capillary properties of the brazing filler metal and can independently perform the function of brazing filler metal at narrow gap filling.

Proceeding from calculation results, under the brazing conditions cobalt can be reduced by aluminium from CoF_2 fluoride by the following reaction (2):

$$2Al + 3CoF_2 = 2AlF_3 + 3Co\ (\Delta G_{600\,^{\circ}C} = -926.123\ \text{kJ}). \qquad (2)$$

Balance of processes (1) and (2) is shifted completely towards the interaction products.

The sequence of chemical reactions (3) and (4) of potassium fluorozirconate (K_2ZrF_6) with aluminium occurring at high temperatures was studied in work [32]:

$$4Al + 3K_2ZrF_6 \rightarrow K_3AlF_6 + 3KAlF_4 + 3Zr, \quad (3)$$

$$9Al + 3Zr \rightarrow 3Al_3Zr. \quad (4)$$

X-ray structural analysis confirms the appearance of $KAlF_4$, K_3AlF_6, and Al_3Zr compounds in the products of reactions (3), (4). When brazing filler metal of Al–Si system is used, silicon interact with K_2ZrF_6. Based on the results of X-ray diffraction analysis of the products obtained after vacuum heating of K_2ZrF_6 + (Si) or K_2ZrF_6 + (Al–7Si) mixtures at 700 °C, showed that the reaction between K_2ZrF_6 and silicon does not take place [32]. At temperature modes of brazing, reduction (processes 1–3) of metals such as Si (conditionally referred to metals), Co, Zr by aluminium from K_2SiF_6, CoF_2, K_2ZrF_6 chemical compounds is possible.

Interaction in the heterogeneous system of "salt melt (KF–AlF_3–K_2SiF_6 flux) – metal alloy (Al–Si brazing filler metal) – solid metal (Al, Ti)" determines the nature of wetting and formation of a strong permanent joint by brazing filler metal at brazing.

Formation of brazed joint of AD1 alloy was studied using tee samples with AD1 alloy strip and Al–12Si brazing filler metal with flux placed in its lower zone, and just the flux applied in its upper zone (Figure 1, *a*). At heating of such a sample up to 605 °C temperature, filling of the gap takes place in the lower zone, its length reaching 40 mm (Figure 1, *b*), and in the upper zone (at application of just the flux) the gap is filled to length L = 16 mm (by newly-formed low-melting Al–Si alloy). Increase of soaking time to 16 s at heating of this sample leads to completion of the joint formation by newly-formed low-melting alloy of Al–Si system (Figure 1, *c*). Temperature rise from the moment of brazing filler metal melting is the main factor for improvement of wetting and capillary properties of the brazing filler metal at the joint formation. High mutual solubility of molten brazing filler metal and newly-formed low-melting alloy, which belong to the same Al–Si metal system, has a positive influence on the kinetics of narrow gap filling.

Thus, proceeding from the conducted studies, the following sequence of running of the process of formation of aluminium brazed joint in argon was established: melt of flux of KF–AlF_3–K_2SiF_6 salt system wets and cleans the metal surface; new (cleaned) state of the surface activates the process of silicon reduction from the flux (by aluminium), further interaction of silicon with aluminium as a result of contact melting promotes formation of low-melting Al–Si alloy in the form of a continuous layer, which improves base metal wetting by the brazing filler metal. In the case of brazing filler metal absence the newly-formed alloy (Al–Si) under the impact of capillary forces fills the gap and its solidification takes place at cooling (Figure 1). In both the cases we obtain a sound brazed joint that differs only by the brazed seam width.

At aluminium brazing with titanium without using the brazing filler metal, heating up to the temperature of 585 °C also leads to silicon reduction from the flux on aluminium contact surface and formation of a low-melting alloy of Al–Si system, which improves wetting of both the base metals, and independently fulfills the function of brazing filler metal during brazing. Obtained sample of aluminium substrate with titanium strip demonstrates good filling of the capillary gap (Figure 2).

The difference of wetting of titanium substrate by aluminium filler of Al–Si system consists in that an interlayer of intermetallic compounds forms on the contact surface [20, 23].

Results of the conducted experiments showed that in order to achieve filling of the gap by brazing filler metal at brazing of titanium with aluminium and application of titanium as substrate (with just the flux), it is necessary

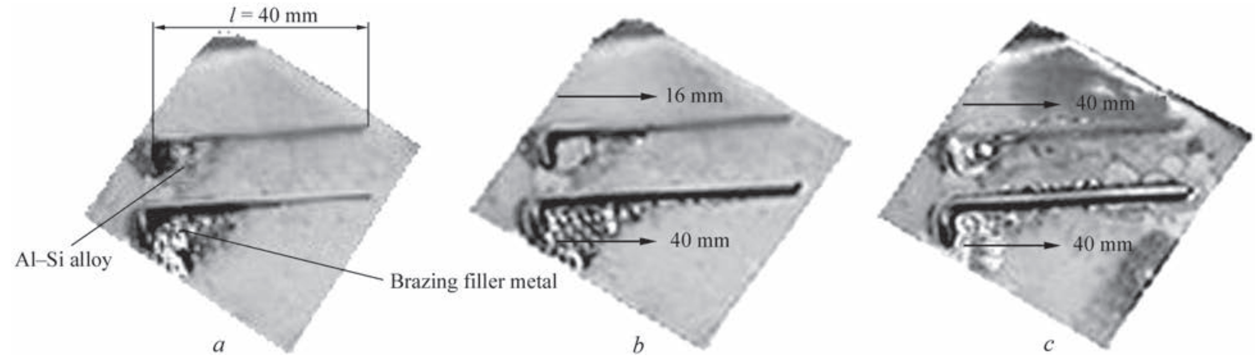

Figure 1. Appearance of a tee-sample with molten Al–12Si brazing filler metal and $KAlF_4$–$10K_2SiF_6$ flux after heating of an aluminium joint (AD1) up to the following temperature: *a* — 585 °C, t_0; *b* — 605 ± 2 °C, t = 8 s; *c* – 605 ± 2 °C, t = 16 s

to raise the temperature and increase the soaking time by approximately 1.5–2.5 times (Figure 3).

Further increase of brazing temperature (by 5–7 °C) promotes formation of a joint of titanium substrate with aluminium strip, both using Al–12Si brazing filler metal, and without the filler, with application of $KAlF_4$–$10K_2SiF_6$ reactive flux.

Based on the conducted experiments, the temperature range ($T = 610 \pm 2$ °C) was determined for formation of a sound Al/Ti brazed joint (on Al and Ti substrate) in high-purity argon with 100 % filling of a narrow ($a \leq 0.1$ mm) gap at application of reactive $KAlF_4$–$10K_2SiF_6$ flux and Al–12Si brazing filler metal and at application of just the flux without using the brazing filler metal. Base metal brazing is conducted at temperature close to 0.95 of aluminium alloy solidus temperature, and further temperature rise can lead to complete softening, for instance, of thin-walled structure elements. Active running of the chemical reaction between the flux salt melt and base metal contact surface limits the time of soaking at brazing temperature.

Detailed study of microstructure of joints produced by reactive-flux brazing with brazing filler metal application demonstrated good weld formation (Figure 4). Samples were brazed in the same temperature-time modes: temperature of 610 ± 2 °C, soaking time $t = 30$ s. Based on the results of metallographic investigations it was found that at application of the brazing filler metal (Al–Si) and $KAlF_4$–$10K_2SiF_6$ flux the brazed seam structure consists of dendrites of aluminium-based solid solution (α-Al), Al–Si eutectic (*e*), precipitating in interdendritic spaces and a continuous intermetallic layer (IML) on titanium of thickness $\delta = 8$–10 μm (Figure 4, *a*).

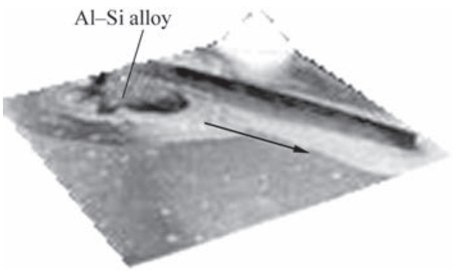

Figure 2. Appearance of a tee sample of AD1 (substrate)/VT1-0 (strip) with $KAlF_4$–$10K_2SiF_6$ flux without application of brazing filler metal after heating up to temperature of 605 ± 2 °C, $t = 18$ s in argon

Local X-ray microprobe analysis revealed that silicon content in α-Al solid solution is equal to (0.54 %), and in the eutectic component it is (~12.8 %) at application of Al-Si brazing filler metal and $KAlF_4$–$10K_2SiF_6$ reactive flux. Intermetallic layer on titanium is formed by $Ti_{37.24}Al_{61.5}Si_{1.26}$ compound, which by its stoichiometric composition [20] is close to $TiAl_3$ phase (37.2 % Ti), containing a small silicon concentration.

In joints formed with reactive flux application without the brazing filler metal (Figure 4, *b*) a much smaller quantity of the eutectic component forms in thin interdendritic α-Al spaces and a thinning of the intermetallic layer to $\delta \leq 3$ μm is observed.

At application of Al–12Si brazing filler metal and $KAlF_4$–$10K_2SiF_6$–$2CoF_2$ reactive flux silicon content in the solid solution (α-Al) and in the eutectic component almost does not change (Figure 5, *a*, Table 2).

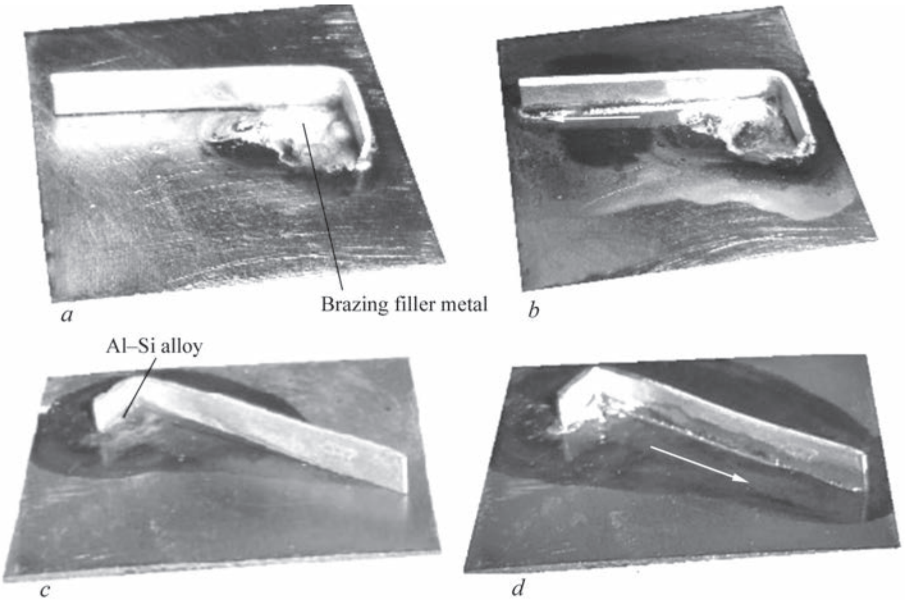

Figure 3. Appearance of a tee sample of VT1-0 (substrate)/AD1(strip) with Al–12Si brazing filler metal and $KAlF_4$–$10K_2SiF_6$ flux after heating up to the temperature of 585 °C, t_0 (*a*); 610 ± 2 °C, $t = 26$ s (*b*) and without brazing filler metal application (585 °C, t_0) (*c*); after joint solidification (610 ± 2 °C, $t = 44$ s) (*d*) in argon

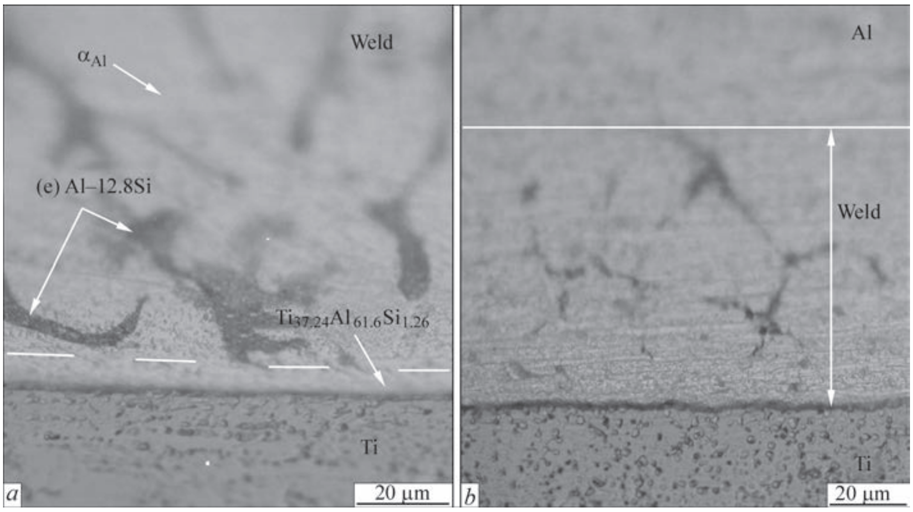

Figure 4. Microstructure of Al/Ti joint brazed with Al–12Si filler and $KAlF_4$–$10K_2SiF_6$ flux (*a*); without filler application, just with $KAlF_4$–$10K_2SiF_6$ flux (*b*)

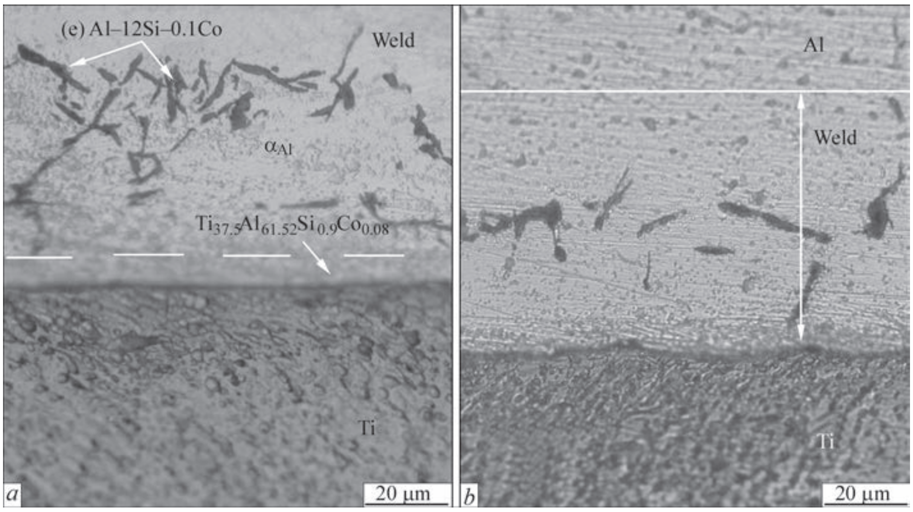

Figure 5. Microstructure of Al/Ti joint brazed by Al–12Si filler and $KAlF_4$–$10K_2SiF_6$–$2CoF_2$ flux (*a*), $KAlF_4$–$10K_2SiF_6$–$2CoF_2$ without filler application (*b*)

Cobalt content (0.10 wt.%) in the eutectic does not influence the dimensions of aluminium-based solid solution.

At application of Al–12Si brazing filler metal and $KAlF_4$–$10K_2SiF_6$–$5K_2ZrF_6$ reactive flux containing K_2ZrF_6 potassium-zirconium fluoride, silicon concentration in α-Al sol.s remains on the same level, and in the eutectic component it decreases to 10.63 wt.% (Figure 6, *a*, Table 3).

Table 2. Chemical element content in Al/Ti brazed joint, wt.%

Component	Al	Si	Co	Ti
α-Al sol.s	99.46	0.53	0.01	0
(e)	87.9	12.0	0.10	0
IML (Ti)	61.52	0.9	0.08	37.5

Table 3. Chemical element content in Al/Ti brazed joint, wt.%

Component	Al	Si	Zr	Ti
α-Al sol.s	98.84	0.56	0.16	0
(e)	89.18	10.63	0.19	0
IML (Ti)	62.83	0.8	0.17	36.2

Results of X-ray microprobe analysis showed that at application of $KAlF_4$–$10K_2SiF_6$–$5K_2ZrF_6$ flux, containing potassium-zirconium fluoride (IV) (K_2ZrF_6) there is 0.19 wt.% Zr in the eutectic component (Table 3). Moreover, certain refinement of dendrites of aluminium-based solid solution is observed (Figure 7).

The same effect of microstructure refinement is observed at alloying of eutectic alloy of Al–12.4Si system by zirconium (0–0.5 wt.%) [33]. Increase of the eutectic phase content up to 12 vol.% at zirconium alloying of the cast aluminium alloy and decrease of α-Al volume, respectively is shown in the case of Al–12.4Si–0.2Zr cast alloy, that promotes increase of tensile strength up to $\sigma_t = 100$ MPa [33].

Based on mechanical testing results it was found that at the temperature of 20 °C the maximum strength of overlap Al/Ti sample is equal to $\tau_{sh} = 61$ MPa at

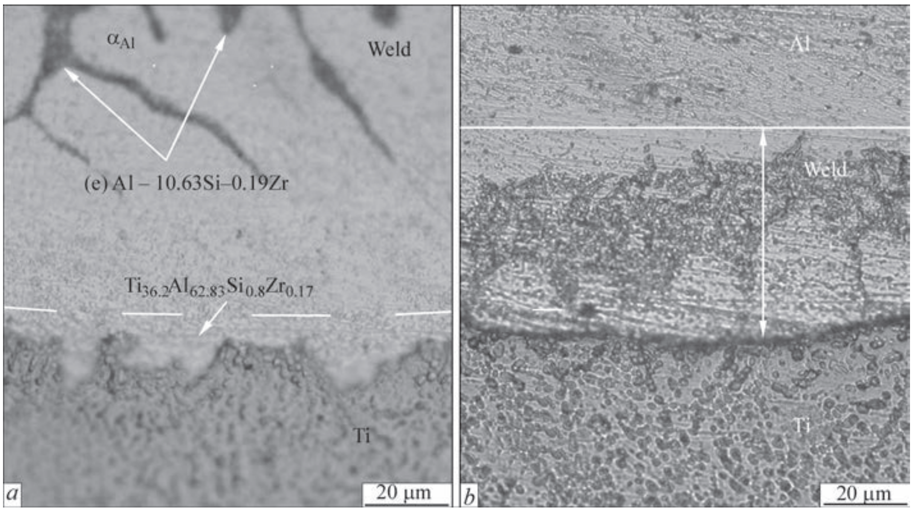

Figure 6. Microstructure of Al/Ti joint, brazed by Al–12Si filler and $KAlF_4$–$10K_2SiF_6$–$5K_2ZrF_6$ flux (*a*), $KAlF_4$–$10K_2SiF_6$–$5K_2ZrF_6$ flux without filler application (*b*)

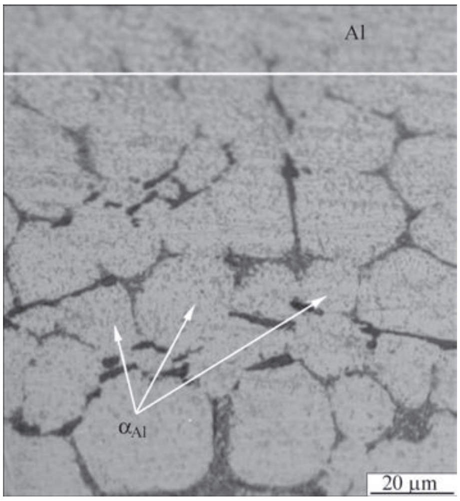

Figure 7. Microstructure of an area the weld of Al/Ti joint brazed with Al–12Si filler and $KAlF_4$–$10K_2SiF_6$–$5K_2ZrF_6$ flux

application of Al–12 % Si brazing filler metal and $KAlF_4$–$10K_2Si$–$5K_2ZrF_6$ reactive flux (Figure 8).

CONCLUSIONS

1. At brazing different joints of AD1 aluminium with VT1-0 titanium by Al–12Si brazing filler metal, application of KF–AlF_3–$10K_2SiF_6$ reactive flux promotes cleaning of base metal surface and formation of a sound joint (at the temperature of 605–610 °C in argon).

2. At reactive-flux (KF–AlF_3–$10K_2SiF_6$) brazing of dissimilar aluminium-titanium joints without application of brazing filler metal, a low-melting alloy of Al–Si system forms on the aluminium contact surface at the temperature of 585–610 °C as a result of silicon reduction by aluminium from the flux. This alloy independently fulfills the function of brazing filler metal.

3. At application of Al–12Si brazing filler metal and $KAlF_4$–$10K_2SiF_6$–$5K_2ZrF_6$ reactive flux, which contains potassium-zirconium fluoride (IV) (K_2ZrF_6), a certain refinement of the structure (dendrites of aluminium-based solid solution) of the brazed seam is observed from the aluminium side that improves an increase of shear strength of aluminium-titanium brazed joints.

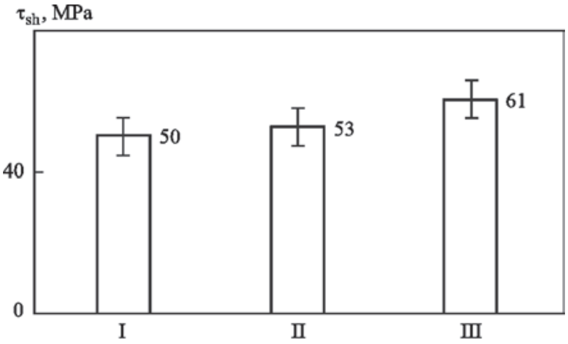

Figure 8. Strength of Al/Ti overlap joint brazed by Al–12Si filler using the following reactive fluxes: $KAlF_4$–$10K_2Si_6$ (I), $KA1F_4$–$10K_2SiF_6$–$2CoF_2$ (II), $KAlF_4$–$10K_2Si_6$–$5K_2ZrF_6$ (III)

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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