A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 57 2012 Issue 2
DOI: 10.2478/v10172-012-0057-9

A. WINIOWSKI*, D. MAJEWSKI*

IMPACT OF CHEMICAL COMPOSITION OF BRAZING FLUXES ON QUALITY AND MECHANICAL PROPERTIES OF TITANIUM BRAZED JOINTS

WPŁYW SKŁADU CHEMICZNEGO TOPNIKÓW LUTOWNICZYCH NA JAKOŚĆ I WŁAŚCIWOŚCI MECHANICZNE POŁĄCZEŃ LUTOWANYCH TYTANU

Titanium and its alloys are increasingly popular specialist structural materials used in modern technologies. In terms of operational properties, titanium is significantly better than other commonly applied structural materials.

A crucial welding-related issue of today is durable joining of elements made of titanium and its alloys. One of the most popular and recommended joining methods, particularly in case of thin-walled elements of complicated geometry is brazing. Due to high reactivity of titanium, it should be brazed in vacuum or very pure, chemically neutral, controlled atmospheres. Brazing of titanium in air atmosphere (flame or induction brazing) requires highly active, fluoride, specialist brazing fluxes.

Institute of Welding in Gliwice has conducted recipe- and technology-related research on brazing fluxes, which resulted in the development of a new flux characterised by high durability and good brazing properties. The article presents the outcome of the research, including the determination of the impact of the basic chemical components on the brazing properties of fluxes, preparation of recipes of fluxes and assessment of their brazing properties as well as the evaluation of quality and shear strength of brazed joints made with such fluxes.

Keywords: brazing, flame brazing, brazing flux, titanium

Tytan oraz jego stopy stanowią specjalistyczne, coraz szerzej stosowane w nowoczesnej technice materiały konstrukcyjne, przewyższające pod względem właściwości eksploatacyjnych wiele dotychczas stosowanych powszechnie materiałów konstrukcyjnych.

Ważnym i wciąż aktualnym problemem w dziedzinie spawalnictwa jest trwałe łączenie elementów wykonanych z tych materiałów. Jedną z bardziej popularnych i zalecanych metod ich łączenia, zwłaszcza w przypadku wyrobów cienkościennych o skomplikowanej geometrii, jest lutowanie twarde. Z uwagi na silną reaktywność tytanu i jego stopów bardziej odpowiedzialne elementy, lutuje się w próżni lub w bardzo czystych atmosferach kontrolowanych, neutralnych chemicznie. Dla procesu lutowania twardego tytanu w atmosferze powietrza (lutowanie płomieniowe lub indukcyjne) niezbędne są natomiast wysoko aktywne – fluorkowe, specjalistyczne topniki lutownicze.

W Instytucie Spawalnictwa w Gliwicach podjęto badania recepturowo – technologiczne nad topnikami tego typu zakończone opracowaniem nowego, topnika o wysokich właściwościach lutowniczych i trwałości. W prezentowanej poniżej pracy przedstawiono wyniki tych badań. Obejmują one określenie wpływu podstawowych składników chemicznych na właściwości lutownicze topników, tworzenie receptur topników i ocenę ich właściwości lutowniczych a także ocenę jakości oraz wytrzymałości na ścinanie wykonanych przy ich użyciu połączeń lutowanych.

1. Introduction

Owing to relatively low specific gravity, high mechanical strength and good corrosion resistance, titanium is applied in aviation, aerospace and chemical industries as well as in the production of surgical instruments, biomedical implants, jewellery, ironmongery and household appliances [1].

Due to high chemical reactivity of titanium, the brazing of critical machinery components is carried out almost exclusively in furnaces with controlled atmosphere (pure, chemically neutral atmospheres and vacuum) [2÷7].

Quite often it becomes necessary to braze small, thin-walled titanium elements in the air (i.e. ironmongery, lamp housings, spectacle frames, household appliances etc.). There are also cases of stationary brazing

* POLISH WELDING CENTRE OF EXCELLENCE, 44-100 GLIWICE, BL. CZESLAWA 16-18, POLAND

of big-sized titanium elements or elements permanently fixed to a greater structure (elements of plants and chemical equipment). In such cases it is necessary to carry out brazing in the air atmosphere, applying relatively fast flame or induction heating. For this brazing method it is recommended to apply silver filler metal and appropriate high chemically active brazing flux ensuring deoxidation of elements being joined and brazing metal during the whole process [3÷5, 8]. The purpose of the application of the flux is to ensure proper course of capillary actions i.e. required wettability and spreadability of brazing metal on materials being joined and proper filling of capillary brazing gaps of joints.

Most commercially available fluoride fluxes recommended for brazing are useless in brazing of titanium as they do not provide adequate protection of reactive metal against oxidation in brazing temperature and thus do not create proper conditions for the formation of brazed joints representing desired quality. Specialist publications on the subject very seldom refer to the composition of fluxes for flame or induction brazing of titanium in the air [4, 5]. Encountered examples, tested in practice, do not provide sufficient brazing properties for silver filler metals (low spreadability of brazing metals on the surface of titanium); they are also characterised by very low durability. Similarly, the specialist flux for brazing of titanium in the air atmosphere, developed at Institute of Welding in 2007 and designated F70T, due to the content of photosensitive silver chloride (AgCl), demonstrated relatively low durability of only more than ten days, if stored in conditions with limited access of light [8]. The presented results of tests on a new flux for titanium are the consequence of continuing research on flux brazing of titanium, aimed at the development of a flux of higher durability [15].

2. Recipe- and technology-related assumptions – methodology of tests

A flux for brazing of titanium with silver filler metal should meet at least one of the following requirements [2÷5]:

- remove film of titanium oxide and titanium nitride (non-wettable by brazing metals) through the absorption or dissolution of the former, or
- actively affect the surface of metal under the layer of oxides and nitrides; in this way the layer is removed during the flow of brazing metal.

Having met one of the aforesaid requirements, at least one ingredient of the flux should react with titanium, using its ability to reduce metals from molten salts. The surface of titanium is covered then with a layer of reduced metal preventing further oxidation.

Having the foregoing in view one may come to the conclusion that while brazing titanium it is recommended to use molten chlorides of such metals as silver, copper, zinc, tin or manganese. Titanium reacts with these salts in accordance with the following equations [4, 9]:

$$Ti + 4AgCl \rightarrow TiCl_4 \uparrow + 4Ag \downarrow \tag{1}$$

$$Ti + 2Cu_2Cl_2 \rightarrow TiCl_4 \uparrow + 4Cu \downarrow \tag{2}$$

$$Ti + 2SnCl_2 \rightarrow TiCl_4 \uparrow + 2Sn \downarrow \tag{3}$$

$$Ti + 2MnCl_2 \rightarrow TiCl_4 \uparrow + 2Mn \downarrow \tag{4}$$

$$Ti + 2ZnCl_2 \rightarrow TiCl_4 \uparrow + 2Zn \downarrow \tag{5}$$

Titanium tetrachloride ($TiCl_4$) formed in the above reaction escapes as gas from the metal surface, additionally destroying the film of titanium oxide (TiO_2), whereas reduced metal covers pure titanium surface protecting it against oxidation [2, 4].

In order to ensure required range of temperatures for flux activity i.e. 600-800°C, suitable for the temperature of brazing with Ag-Cu-Zn-type silver filler metal containing 45% Ag, the flux should contain an addition of other highly active compounds from the group of fluorides and chlorides such as: $KF{\cdot}H_2O$, KHF_2, $NaHF_2$, LiCl, LiF, NaF, CaF_2 and CsCl, composing mixtures of required melting points [10÷14].

A detailed analysis of publications about the chemical properties of fluoride salts of transition metals and alkali metals such as: AgF, $AgHF_2$, $ZnF_2{\cdot}4H_2O$, CuF_2, MnF_2, CsF and $(NH_4)HF_2$, gave rise to the conclusion that the aforesaid compounds may have a positive effect on the brazing properties of the flux [12÷14]. All of the above fluorides of transition metals should react in the manner similar to that of chlorides i.e. with the formation of titanium-based protective metallic layer.

On the basis of the analysis of publications concerning the properties of the compounds presented [8, 10÷14] and own experiments, the recipe-related assumptions for the flux to be developed were as follows:

- base of the flux will be composed of fluorides and chlorides of lithium, potassium, and sodium or their mixtures of quantitative content selected according to the fusibility criterion (required melting point: approx. 600-650°C);
- components to be used as ingredients increasing flux activity: caesium chloride (CsCl), caesium fluoride (CsF) and ammonium bifluoride ($(NH_4)HF_2$);
- compounds to be used due to their reactivity with metallic titanium, forming a metallic film on the surface of the latter: silver chlorides and fluorides (AgCl, AgF), copper chlorides and fluorides ($CuCl_2$, CuF_2), manganese chlorides and fluorides ($MnCl_2$, MnF_2) and zinc chlorides and fluorides ($ZnCl_2$, ZnF_2).

The tests of brazing properties of test fluxes were conducted in accordance with the methodology developed and verified in Institut's previous works on fluxes, based on the classical spreadability test.

In relation to brazing, currently there are no internationally- or EU-standardised methods for testing of brazing properties. Available methods, if any, are described in often outdated standards of some countries as well as in reference books or research publications [3÷6].

The Institut's tests on the spreadability of the brazing metal involved the use of titanium plate (40×40×2,5 mm) (Grade 2 acc. to ASTM) as the base [16]. The brazing metal, whose spreadability on the aforesaid base in presence of tested fluxes was assessed, was silver filler metal, grade Ag 245 (B-Ag45CuZn-665/745) acc. to PN-EN [17]. Prior to the tests, the titanium sample was degreased with acetone and etched in an appropriate mixture of acids ($HF + HNO_3 + H_2O$). On each sample of the titanium base, approx. 0,2 flux samples were placed. Afterwards, silver brazing metal (also approx. 0,2 g) was laid on the foregoing. The whole of so prepared a sample was then heated from beneath with a stationary oxyacetylene torch with a cap providing output of 160 l of C_2H_2. The heating was interrupted 3 seconds after the melting of the brazing metal.

As the measure of spreadability (wettability) one assumed:

– spreading area of brazing metal (measured by means of graphic processing of a photograph of the sample) and coefficient: $K_p = (P_A - P_o)/P_0$,

where: P_A – arithmetic average of spreading area of brazing metal, mm^2;

P_o – area of the flat projection of spherical filler metal sample of volume V onto the plane base, calculated from the formula: $P_o = [(3/4\ \pi)\ V]^{2/3}$, mm^2;

– height of brazing metal layer after spreading (measured with a micrometer, with accuracy of up to 0.01 mm) and coefficient $K_H = [(D_o - H_A)/D_o]\cdot 100\%$,

where: H_A – arithmetic average of height of brazing metal layer after spreading,

D_o – theoretical diameter of a brazing metal drop of volume V without wetting, calculated from the formula: $D_o = 1{,}2V^{1/3}$, mm.

The spreadability tests were accompanied by the examination of the quality of flux slag and its removability. The spreadability tests for the final versions of fluxes were supplemented with tests on the penetration of brazing metal (in presence of flux being tested) into capillary gaps of overlap joints. The brazing of the aforementioned overlap joints with the test fluxes was conducted in accordance with the intended use of the flux being developed, using an oxyacetylene torch and manually feeding the brazing metal. The tests involved also the determination of shear strength of joints (for the best flux versions) and metallographic quality examination (macro) for the best flux versions.

3. Development and laboratory production of flux samples and determination of their brazing properties

Following the adopted technical assumption, the basic ingredients used in the new fluxes were as follows: $KF\cdot 2H_2O$, KHF_2 and LiCl. Also other, more reactive ingredients such as CsCl, CsF, LiF, NaF, NH_4HF_2 were tested for their usability. Next raw materials selected as flux ingredients were those which come into reduction reaction with titanium. As a result of the reaction, the surface of titanium is covered with a film of reduced metal preventing oxidation. As it was pointed above, the most favourable substances characterised by desired protective properties are molten chlorides of such metals as: zinc ($ZnCl_2$), copper (CuCl, $CuCl_2$), tin ($SnCl_2\cdot 2H_2O$), nickel ($NiCl_2\cdot 6H_2O$), aluminium ($AlCl_3\cdot 6H_2O$), manganese ($MnCl_2\cdot 6H_2O$) and fluorides of such metals as: zinc ($ZnF_2\cdot 4H_2O$), copper ($CuF_2\cdot 2H_2O$), manganese (MnF_2) and silver ($AgF \cdot H_2O$, AgF_2).

The tests aimed at obtaining flux for brazing of titanium in the air atmosphere involved the development and laboratory production of a few dozen flux samples. During the tests the chemical composition of the samples was successively changed as a result of verification of melting point and brazing properties. Table 1 presents selected recipe composition of flux samples, whereas Table 2 presents the brazing properties of the best versions of test fluxes.

According to the analysis of the results presented in Table 2, all the flux samples ensured good brazing metal spreadability expressed in coefficients K_P and K_H. The greatest area of spreading of flux was that provided by fluxes 47T and 48T, yet the surfaces of the layers of molten brazing metal produced using these fluxes were slightly rough – unlike in case of other fluxes, whose application made it possible to obtain smooth layer surfaces.

TABLE 1

Recipe composition of selected fluxes samples

No.	Recipe composition of flux, % (m/m)	Designation of flux								
		5T	26T	27T	40T	47T	48T	62T	63T	79T
1	$KF \cdot 2H_2O$	25	25	25	25	22	24	24	24	24
2	KHF_2	41	39	41	40	52	54	–	–	–
3	LiCl	30	30	30	–	–	–	–	–	–
4	CsCl	–	–	–	27	20	20	36	37	26
5	NH_4HF_2	–	–	–	–	–	–	32	33	32
6	CsF	–	–	–	–	–	–	–	–	10
7	$SnCl_2 \cdot 2H_2O$	4	–	–	–	–	–	–	–	–
8	$ZnCl_2$	–	6	4	8	–	–	8	6	8
9	$ZnF_2 \cdot 4H_2O$	–	–	–	–	6	4	–	–	–

TABLE 2

Spreadability results for silver filler metal Ag 245 (B-Ag45CuZn-665/745) on titanium surface (Grade 2) with use of selected fluxes

No.	No. of flux	Spreadability [1)]					
		P_A, mm^2	S_P, mm^2	$\mathbf{K_P}$	H_A, mm	S_H, mm	$\mathbf{K_H}$, **%**
1	5T	167,33	11,18	**10,45**	0,32	0,01	**90,72**
2	26T	155,95	7,47	**9,67**	0,39	0,02	**88,69**
3	27T	154,49	3,83	**9,57**	0,35	0,02	**89,85**
4	40T	165,43	5,47	**10,32**	0,25	0,02	**92,75**
5	47T	217,73	6,59	**13,89**	0,34	0,03	**90,14**
6	48T	167,91	6,46	**10,48**	0,30	0,05	**91,30**
7	62T	126,09	5,78	**7,62**	0,34	0,04	**90,14**
8	63T	123,02	5,30	**7,41**	0,31	0,03	**91,01**
9	79T	142,75	3,07	**8,76**	0,31	0,02	**91,01**

1) P_A – average value of spreading area (3 measurements); S_p – standard deviation of spreading area of brazing metal; $\mathbf{K_P}$ for P_0 = 14,62 mm^2;
H_A – average value of height of brazing metal layer after spreading (3 measurements),
S_H – standard deviation of height of brazing metal layer after spreading; $\mathbf{K_H}$ for D_0 = 3,45 mm.

The presented compositions of flux samples (Table 1) demonstrated a common feature i.e. relatively high content of fluorine compounds, amounting to 60÷70%. It was not possible to obtain a flux which would properly wet the surface of titanium after decreasing or eliminating one of the fluorine compounds from the recipe and replacing the former with a less toxic substance e.g. chlorides of alkali metals. The preparation of test lots of flux included the verification of application of other fluorides (NaF, LiF) in the composition, in order to eliminate hydrated potassium fluoride. This attempt, however, did not produce desirable results as the application of the aforesaid compounds in the flux composition adversely affected its brazing properties. In turn, the application of 22÷25% $KF \cdot 2H_2O$ (Table 2) in the composition of test versions significantly improved the spreadability and wettability of the brazing metal (Table 2).

It was ascertained that the usefulness of CsF and CsCl in the flux is connected with high activity of these compounds and effective protection of the joining zone, whereas 32% addition of ammonium bifluoride (NH_4HF_2) makes it possible to replace KHF_2 in the flux composition. In addition, the application of NH_4HF_2 instead of KHF_2 in fluxes no. 62T and 63T led to an overall decrease of the amount of harmful fluoride, improving thus health and safety conditions of the brazing process.

The most favourable substances reacting with titanium and utilising its ability to reduce from molten salts are $SnCl_2 \cdot 2H_2O$, $ZnCl_2$ and $ZnF_2 \cdot 4H_2O$ (Table 2). Only these compounds made it possible to wet the surface of titanium with brazing metal. The favourable content of $SnCl_2 \cdot 2H_2O$ in fluxes is approx. 4%, $ZnCl_2$ between 4 and 8%, whereas $ZnF_2 \cdot 4H_2O$ from 7 to 9%. For this reason one may conclude that these salt contents in fluxes are optimum and enable obtaining desired high spreadability and wettability of brazing metal.

In order to determine influence of above fluorides and chlorides of transition metals for quality and amount of protective film of reduced metal, chemical profile analysis were conducted. The chemical profile analysis molten fluxes samples on titanium surface made with an application of Glow Discharge Atomic Emission Spectrometer LECO GDS850A. The result of chemical profile analysis is presented in Figure 1 (sample 26T with $ZnCl_2$, Table 1). The amount of protective zinc coating came to 70% (depth 0,7 μm).

In Figure 2 was presented titanium surface after chemical profile analysis. The round point indicate the place where was carried out the chemical profile analysis. The dark colours places indicate flux slag. This flux slug was not remove, in order not to lead to remove zinc coating film after rinse the sample in hot water.

More detailed tests on this issue made it possible to select fluxes (presented in Table 3) for further tests of mechanical and metallographic properties.

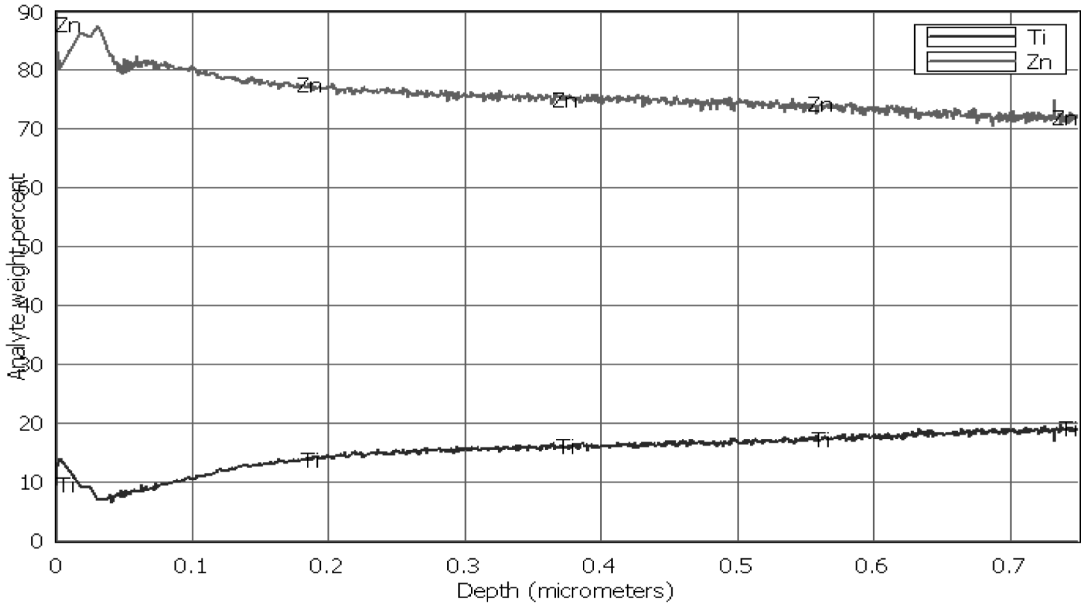

Fig. 1. Chemical profile analysis of molten flux 26T on titanium surface

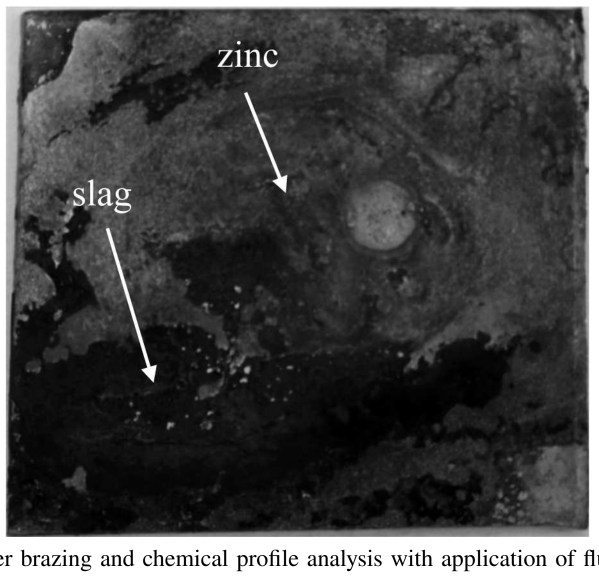

Fig. 2. The titanium sample surface after brazing and chemical profile analysis with application of flux 26T

TABLE 3

Recipe composition of selected flux samples for tests of mechanical and metallographic properties

No.	Recipe composition of flux % (m/m)	Designation of flux								
		5T	26T5	26T6	40T	47T	48T	62T6	62T7	79T5
1	$KF{\cdot}2H_2O$	25	25	25	25	22	24	24	24	24
2	KHF_2	41	38	37	40	52	54	–	–	–
3	LiCl	30	30	30	–	–	–	–	–	–
4	CsCl	–	–	–	27	20	20	36	36	27
5	NH_4HF_2	–	–	–	–	–	–	32	31	32
6	CsF	–	–	–	–	–	–	–	–	10
7	$SnCl_2{\cdot}2H_2O$	4	–	–	–	–	–	–	–	–
8	$ZnCl_2$	–	7	8	8	–	–	8	9	7
9	$ZnF_2{\cdot}4H_2O$	–	–	–	–	6	4	–	–	–

4. Quality tests (visual inspection and metallographic examination) and properties of mechanical joints made with the use of newly developed fluxes

The effect of the fluxes from Table 3 on the quality and mechanical properties of brazed joints was assessed by means of visual inspection, macroscopic metallographic examination and strength tests of test overlap joints made with these fluxes. The samples (40x40x2.5 mm) were made of titanium plate, grade 2; the filler metal being silver brazing metal, grade Ag 245 (B-Ag45CuZn-665/745) [16,17].

The samples were heated with a typical oxyacetylene torch with a cap providing output of 160 l/h of acetylene.

The brazing of the overlap joint (plates were put horizontally, freely, without pressure) was conducted with feeding brazing metal manually, at the overlap outlet. In such conditions, the size of the brazing gap settled on its own, during the inflow of brazing metal. The above test enables quite strict assessment of the capillary properties of the brazing metal in presence of the flux under investigation.

Immediately after brazing, in order to remove (post) flux slag entirely, the samples with solidified brazing metal were immersed in cold water (thermal shock).

It proved impossible to produce overlap joints using the fluxes designated 40T, 47T, 48T, 62T7 and 79T5 – as they failed to ensure proper wettability of the overlap joint gap with the brazing metal. It was, however, possible, using fluxes 5T, 26T5, 26T6 and 62T6, to produce joints for metallographic examination and those for shear strength tests.

While analysing the strength test results presented in Table 4 one may notice that the highest shear strength was demonstrated by the overlap joints made with the flux designated 26T5, whereas the lowest shear strength characterised the overlap joints made with the fluxes designated 26T6 and 62T6, which, in addition, demonstrated significant scatter of results (the highest standard deviation). Also the very process of brazing of the overlap joints with the use of these three fluxes was rather problematic as the brazing metal proved reluctant to penetrate the capillary gap of the joint.

TABLE 4

The shear strength test results lap joints brazed with the use of newly developed fluxes

No.	Designation of flux	Shear strength R_t, MPa	
		Average value [1)]	Standard deviation
1	5T	136,00	5,84
2	26T5	163,04	10,04
3	26T6	109,48	18,51
4	62T6	108,49	19,44

[1)] Average value of 5 measurements

The macrostructures of the joints brazed with the use of test fluxes are presented in Figures 3÷6.

The macroscopic examination revealed that the best quality of the titanium overlap joint with the filling of the joint gap along the entire length of the overlap can be obtained applying the flux designated 26T5. In addition, the flux enables obtaining the smooth face of a braze, free from surface imperfections.

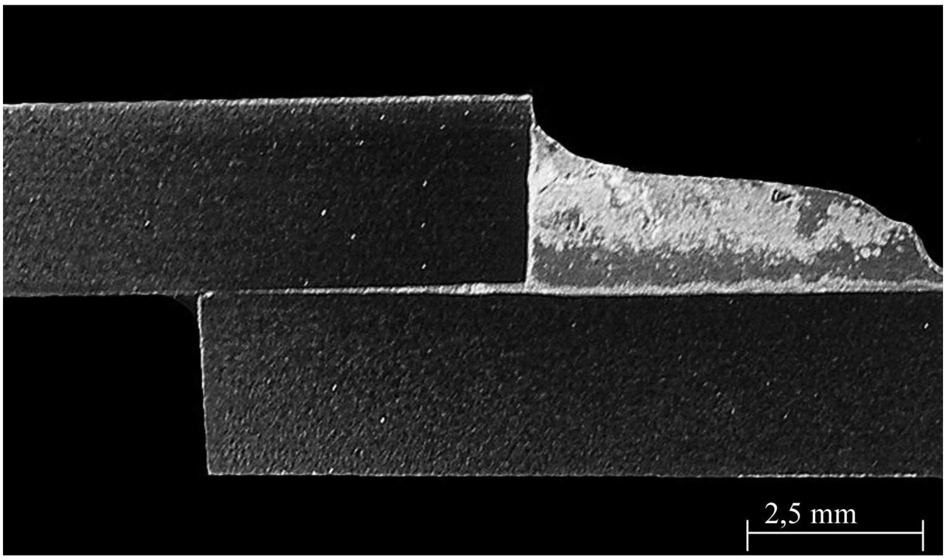

Fig. 3. Macrostructure of brazed joints of titanium Grade 2 made with silver braze metal type Ag 245 and flux 5T (Table 3) (etchant Adler)

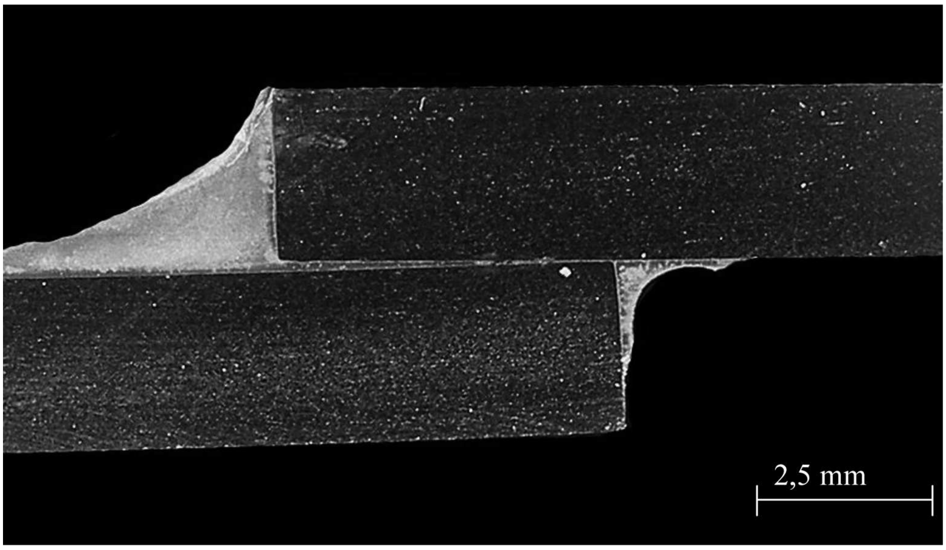

Fig. 4. Macrostructure of brazed joints of titanium Grade 2 made with silver braze metal type Ag 245 and flux 26T5 (Table 3) (etchant Adler)

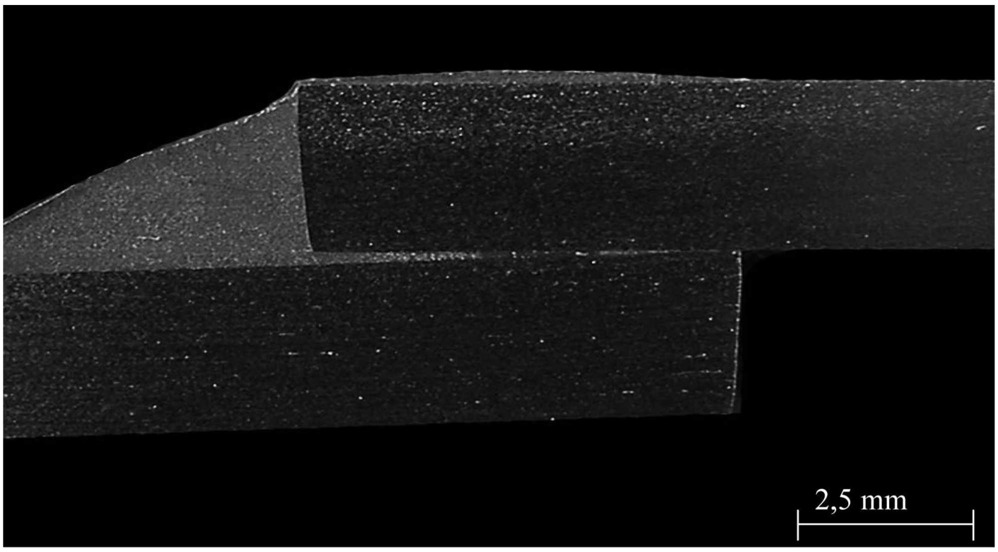

Fig. 5. Macrostructure of brazed joints of titanium Grade 2 made with silver braze metal type Ag 245 and flux 26T6 (Table 3) (etchant Adler)

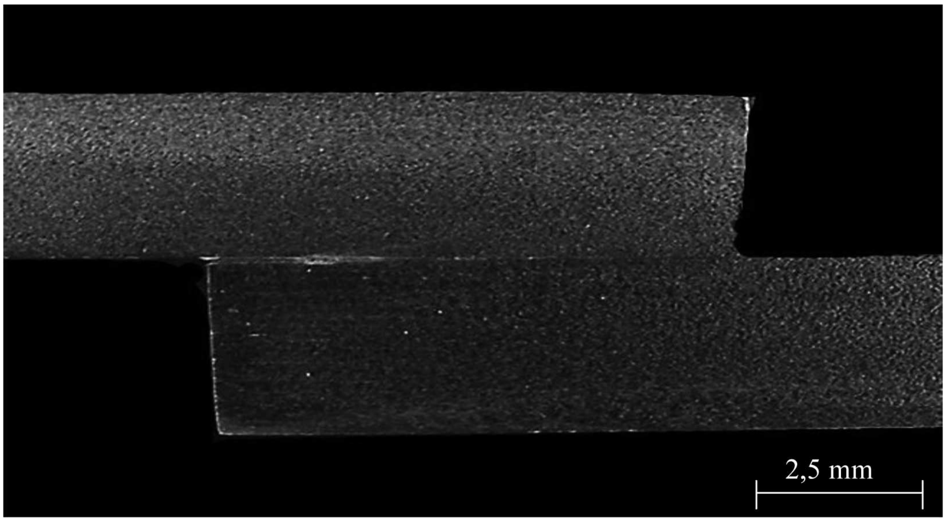

Fig. 6. Macrostructure of brazed joints of titanium Grade 2 made with silver braze metal type Ag 245 and flux 62T6 (Table 3) (etchant Adler)

The recipe- and technology-related tests revealed that, technically, the best flux was that designated 26T5. The flux is characterised by good brazing properties as well as provides good quality and relatively high shear strength of a joint made with silver brazing metal, grade Ag 245, which, in addition, does not contain any photosensitive substances and can be stored for approximately 6 months.

5. Conclusions

1. The total content of fluorine compounds making up the composition of the flux for brazing of titanium with silver brazing metal, grade Ag245, in the air atmosphere should amount to 60÷70%. Otherwise, flux does not provide sufficient wettability and spreadability of the brazing metal on titanium base.
2. The results of the recipe- and technology-related tests proved that it is possible to develop a flux for brazing of titanium with silver brazing metal, grade Ag245, not containing expensive and photosensitive silver chloride (AgCl), which can be replaced by zinc chloride $ZnCl_2$, not decomposing if exposed to light.
3. The most favourable results of tests of brazing properties, quality and strength of the joints of titanium, brazed with silver brazing metal, grade Ag245, are obtained with a flux containing 7% addition of $ZnCl_2$ and compounds such as $KF{\cdot}2H_2O$, KHF_2 and LiCl.

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