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Original Research Article

Brazeability of Grade 2 titanium and AZ31B magnesium alloy under flux cover

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ABSTRACT

The article presents the basic issues related to brazing of poorly brazeable materials such as titanium and magnesium alloys. The text contains exemplary chemical compositions of fluxes used in brazing of poorly brazeable materials and presents the results of composition and technological process-related tests concerning fluxes used for brazing of Grade 2 titanium and AZ31B magnesium alloy. The article also presents the brazing properties of fluxes and test results concerning the mechanical properties of brazed joints as well as the results of metallographic examination utilising light and electron microscopy.

1. Introduction

Titanium and its alloys are becoming increasingly popular in highly modern industries and sectors of economy as well as in the production of medical equipment and implants, sports equipment, jewellery and metal accessories. This trend is related to exceptional physicochemical properties of this metal, such as high temperature and corrosion resistance, low density and high mechanical strength. Titanium is rated among lightweight structural materials characterised by high relative strength (strength-to-density ratio) within a wide temperature range. The relative strength of titanium alloys is approximately 1.5 times higher than that of high-strength alloy steels. The relative strength of other light metal alloys, e.g. Al or Mg is also lower than that of Ti alloys especially at higher operating temperatures [1,3,5].

Magnesium alloys also find applications in automotive industry, aviation as well as in the production of electro-technological and electronic equipment. These alloys belong to the cheapest light metal alloys and are characterised by advantageous properties including relatively high mechanical strength, very low density, good vibration damping ability and high corrosion resistance. In the automotive industry Mg alloys are used in the production of seat and sunroof frames, engine elements, radiator housing, gearbox elements and pedal bearers. Other applications include telephone casings, aerials and videophones [1,5].

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1.1. *Titanium brazeability*

Titanium is a chemically active metal characterised by high chemical affinity for all chemical elements, and particularly for gases present in the air such as oxygen, nitrogen and hydrogen. In the air titanium oxidises relatively slowly. However, a stable TiO_2 oxide formed during heating impedes brazing processes. The intensity of TiO_2 formation is particularly high at 650–700 °C [2–4].

Presently, brazing of highly critical elements made of titanium and its alloys is performed exclusively in vacuum or pure, chemically neutral and controlled atmospheres [7,8,16]. Fig. 1 presents a TWC INVAC-manufactured high-vacuum furnace with removable bottom used for titanium brazing [6].

However, it sometimes becomes necessary to braze big-sized titanium elements or elements permanently fixed to larger structures. In such situations it is difficult or impossible to perform brazing in vacuum. Brazing of elements in the air atmosphere requires the use of highly active specialist brazing flux ensuring the proper course of brazing phenomena. Such flux should reduce the surface tension of brazing metals ensuring required wettability and spreadability of brazing metals as well as ensuring the filling of capillary brazing gaps [2,4]. Flux action also includes chemical reaction and dissolving of oxide layers present on workpiece and brazing metal surfaces as well as the prevention of their re-oxidation during brazing.

Most of available fluxes recommended for brazing of various metals, even those of the most stable oxides, are unsuitable for brazing of titanium as they fail to provide the proper protection of reactive titanium against oxidation at brazing temperatures and do not create appropriate conditions for wetting workpiece surfaces with a brazing metal. Fig. 2 presents the test of Ag 245 silver brazing metal spreadability (45% Ag) on the Grade 2 titanium base under the cover of high-fluoride F47A flux. The flux is produced at Institute of Welding and is used for brazing of stainless (chromium–nickel) steels and brasses. As can be seen, the brazing metal entirely failed to wet titanium.

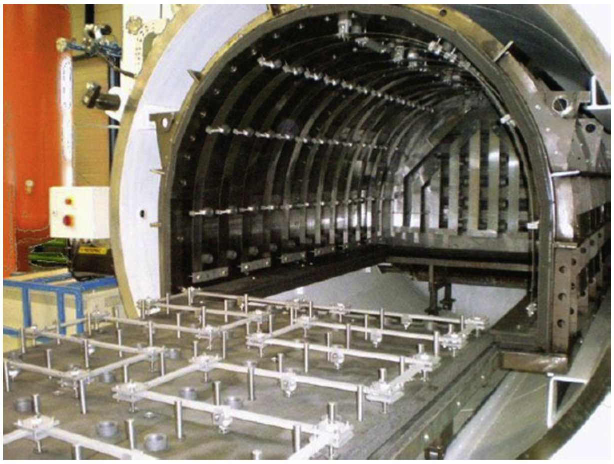


Fig. 1 – TWC INVAC-made high-vacuum furnace with removable bottom used for titanium brazing [6].

Fig. 2 – Lack of spreadability of the Ag 245 silver brazing metal on the Grade 2 titanium base, in the air, under the F47A flux cover.

Specialist reference publications concerned with brazing seldom provide information on the composition of fluxes for flame or induction brazing of titanium in the air. The examples of compositions found (% by weight) are presented below [4,9–11]:

- 45% NaCl, 36% KCl, 10% AgCl, 9% LiF,
- 50% KF, 46% LiF, 4% AgCl,
- 50% LiCl, 25% KF, 25% $MgCl_2$,
- 48% NaCl, 30% $MgCl_2$, 22% LiCl.

However, practical brazing tests revealed that these fluxes fail to provide good brazeability of titanium using silver brazing metals (low brazing metal spreadability) and are not durable after their manufacture [9–11]. The spreadability of first two fluxes on titanium surface was limited and reached 100–120 mm^2. Another two fluxes do not allow wetting of titanium surface using silver brazing metal. They do not contain transition metal chlorides which could react with the titanium and be reduced on the surface.

This has led to the conclusion that it is necessary to develop chloride–fluoride flux for brazing of titanium in the air and meeting the criteria of good brazeability and higher chemical stability.

1.2. *Brazeability of magnesium alloys*

Recent years have seen significant interest in brazing of Mg–Al–Zn–Mn (AZ31B) magnesium alloys. However, brazing of such alloys causes a serious problem as the film of MgO and Al_2O_3 oxides present on the surface is very durable and difficult to remove during brazing. Making brazed joints in the air atmosphere requires the use of highly active flux containing halides of alkaline metals such as Ca, Na and Li [4,11,13].

Similarly as in the case of fluxes used for brazing of titanium, fluxes used for brazing of magnesium alloys should provide good brazeability and removal of oxide layers from the surface of workpieces and brazing metals [4,11,13].

Reference publications [11,13] provide the following compositions of fluxes for brazing of magnesium alloys (% by weight):

- 45% KCl, 25% NaCl, 24% LiCl, 6% NaF,
- 54% KCl, 30% $CaCl_2$, 12% NaCl, 4% NaF,
- 51.5% LiCl, 40% $CaCl_2$, 8.5% KCl,
- 55.5% $CaCl_2$, 29.8% LiCl, 14.7% NaCl,
- 35% KCl, 30% LiCl, 15% $CdCl_2$, 10% $ZnCl_2$, 10% ZnF_2,
- 49.5% LiCl, 31.5% KCl, 9% NaCl, 8% NaF, 2% Na_3AlF_6,
- 74,7% LiCl, 25.3% KCl.

The selection of appropriate flux for brazing of magnesium alloys also depends on the type of filler metal used. As initial brazing tests revealed the fluxes failed to provide satisfactory brazeability of magnesium alloys.

2. Experimental tests results of flux for brazing titanium Grade 2

2.1. Base and filler metals

The tests involved the use of base metal in the form of a 2.5 mm thick Grade 2 titanium sheet according to ASTM B265 and Ag 245 silver brazing metal (B-Ag45CuZn-665/745) according to PN-EN ISO 17672:2010, in the form of a wire with a diameter of 2.0 mm. The Ag 245 brazing metal was singled out for brazeability tests due to the required melting point and versatility. This filler metal is recommended for brazing of titanium [9,10,14]. Fluxes were made using the mixtures of fluorides and chlorides of intermetals and alkali metals.

2.2. Testing the brazing properties and the measurements of wetting angle

The brazing properties of test fluxes were carried out using the classical spreadability tests. A plate with dimensions of 40 mm × 40 mm × 2.5 mm was used as the base. The brazeability tests were preceded by degreasing with acetone and chemical etching in the mixture of acids (30 cm^3 HF + 55 cm^3 HNO_3 + 15 cm^3 H_2O). On each titanium base test piece, first silver brazing metal (0.2 g) and, next, flux (0.2 g) was applied. The test piece underwent heating from underneath using a neutral oxy-acetylene flame with an attachment having an output of 160 dm^3 of C_2H_2/h. Heating was interrupted 3 s after melting of the brazing metal. Afterwards, the test piece was cooled in the air. Fig. 3 presents the scheme of the brazing metal spreadability tests.

Eighty two flux samples were developed and made in the laboratory, changing their chemical composition on the basis of melting observations, brazing properties, amount of flux slag and ease of its removal from brazed surfaces. On the basis of the spreadability tests conducted it was determined that the most advantageous flux was that designated as F60T containing such compounds as $KF{\cdot}2H_2O$; KHF_2; LiCl and $ZnCl_2$ [17,18]. Evaluation of the spreadability Ag 245 under the flux F60T cover on the titanium surface was based on measurement of surface dissolution of the braze (which reached 274 mm^2 – the average value of three measurements) and on measurement of wetting angle. During the spreadability test of the Ag 245 brazing metal under the F60T flux cover it was possible to determine a wetting angle θ amounting to 9° (Fig. 4). The chemical stability of the newly developed flux amounts to approximately 12 months.

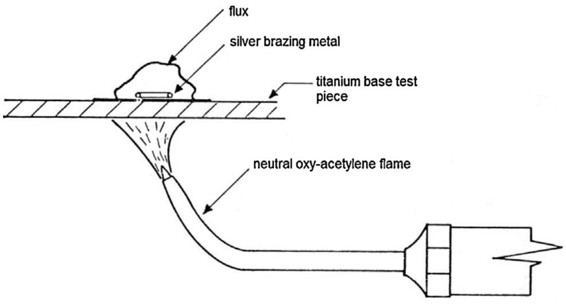


Fig. 3 – Scheme of the Ag 245 brazing metal spreadability tests on titanium surface.

2.3. Making test brazed joints

2.3.1. Flame brazing

The surface of titanium test pieces in the form of plates (40 mm × 40 mm × 2.5 mm) (test of brazing metal penetration into the overlap joint gap, visual testing and metallographic examination) and strips (100 mm × 10 mm × 2.5 mm) (static shearing test) was prepared in a similar manner as in the case of the spreadability tests. Heating was carried out using a neutral oxy-acetylene flame. The overlap joint (plates were placed horizontally, freely without pressure, flux placed between plates) was made dosing the flux manually at the overlap edge. In these conditions the brazing gap width was formed spontaneously during the inflow and solidification of the brazing metal. The test enabled the assessment of brazing metal capillarity in the presence of F60T flux. After brazing metal solidification and cooling of the surface in the air the test pieces were immersed in cold water in order to remove (post) flux slag entirely.

2.3.2. Induction brazing

Induction brazing was carried out using an NG-40 medium frequency inverter device. The heating parameters were the following:

- frequency: 22.5 kHz,
- type of inductor: 2-coil, water-cooled copper inductor,
- brazing time: 10 s.

Fig. 5 presents brazed test pieces and the inductor shape.

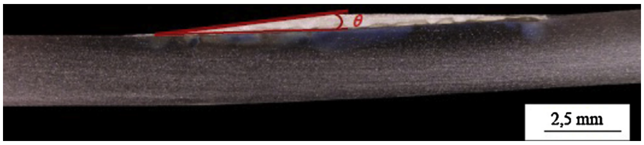


Fig. 4 – Wetting the titanium base with molten Ag 245 brazing metal, wetting angle 9°.

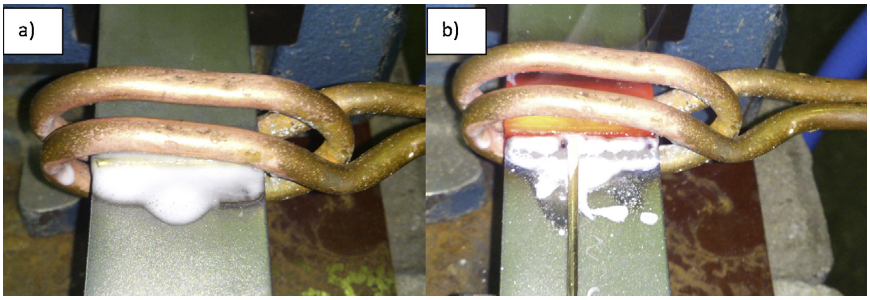


Fig. 5 – Position of the test pieces and inductor shape during induction brazing of titanium using the F60T flux and the Ag 245 silver brazing metal: before heating (a), 7 s after heating start (b).

2.4. *Static shearing test of brazed joints*

In order to determine the mechanical properties of the Grade 2 titanium overlap joints it was necessary to carry out static shearing tests. The static shearing tests of the brazed joints were carried out on five test pieces prepared using a neutral oxy-acetylene flame with an attachment having an output of 160 dm^3 of acetylene/h. The length of the overlap in the brazed joints of Grade 2 titanium amounted to 3.0 mm. For each test piece the shearing area amounted to 30 mm^2. In order to ensure the axiality of load eliminating the bending of the joint it was necessary to use elements which corrected fixing of the test pieces in the holders.

The average shear strength of 5 test pieces amounted to 163.0 MPa, with a standard deviation of 10.0 MPa and the brazing gap amounting to, on average, 0.13 mm. The braze contained scrap of cohesive character. The obtained shear strength of the joints brazed using the Ag 245 silver brazing metal was restricted within a range 160–200 MPa [4].

2.5. *Metallographic examination*

Fig. 6a and b presents the macrostructure of the joint made using flame heating and induction heating respectively. The metallographic examination conducted revealed the overlap joint of good quality, with the gap filled along the whole length of the joint.

The F60T flux enables obtaining, both by flame heating and induction heating of Grade 2 titanium in the air atmosphere, the flat face of the braze, free from surface imperfections. In the brazed joints it was possible to observe the concave shape of the braze face.

The microstructure of the brazed joints (Fig. 7) was revealed by etching the titanium test pieces in Buehler's reagent of the following chemical composition (% by volume): 2% HF + 3% HNO_3 + 95% H_2O (distilled). The central area of the flame heated brazed joint was subjected to observations.

On the boundary of titanium with the Ag 245 braze it was possible to observe the formation of columnar crystal precipitates. The work [14] presents data concerning the possibility of the formation of solid solutions on the basis of Cu_3Ti_2, Cu_4Ti_3, $CuTi_2$ intermetallic phases while brazing titanium with Ag–Cu types of eutectic filler metals. For this reason it is possible to assume that the precipitates formed are the mixture of Cu_3Ti_2, Cu_4Ti_3, $CuTi_2$ phases. In turn, the central light area of the braze contains a structure being a eutectic mixture.

The EDS analysis of the brazed joint required the use of a FESEM FEI Nova NanoSEM 230 scanning electron microscope. The analysis of the EDS chemical analysis was conducted at 6 points, starting in the base metal and moving towards the braze. The results of the measurements and the identification of phases formed (including the columnar crystals, point 2 and 3) on the border of Ti–Ag 245 shown in Fig. 8.

On the basis of the measurements conducted it was possible to observe the slight diffusion of the silver brazing metal components to the base metal at point 1 (Ti content at this point amounts to 88.6% by weight.). In the braze area adjacent to titanium, on the surface of round precipitates (point 2) it was possible to observe a significant titanium content (54% by weight), which, if combined with the

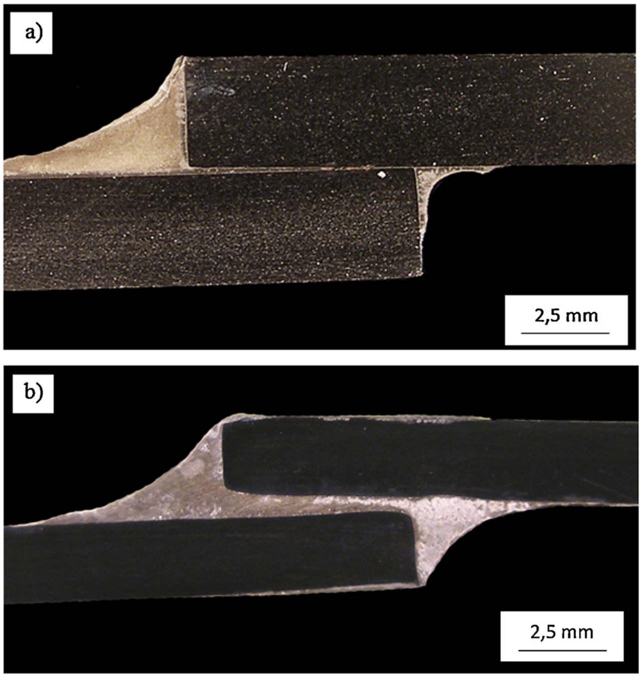


Fig. 6 – Macrostructure of the joint of Grade 2 titanium made using the Ag 245 silver brazing metal under the F60T flux cover, flame brazing-gap width 0.13–0.15 mm (a) and induction brazing-gap width 0.23–0.31 mm (b), chemical etching using Adler's reagent.

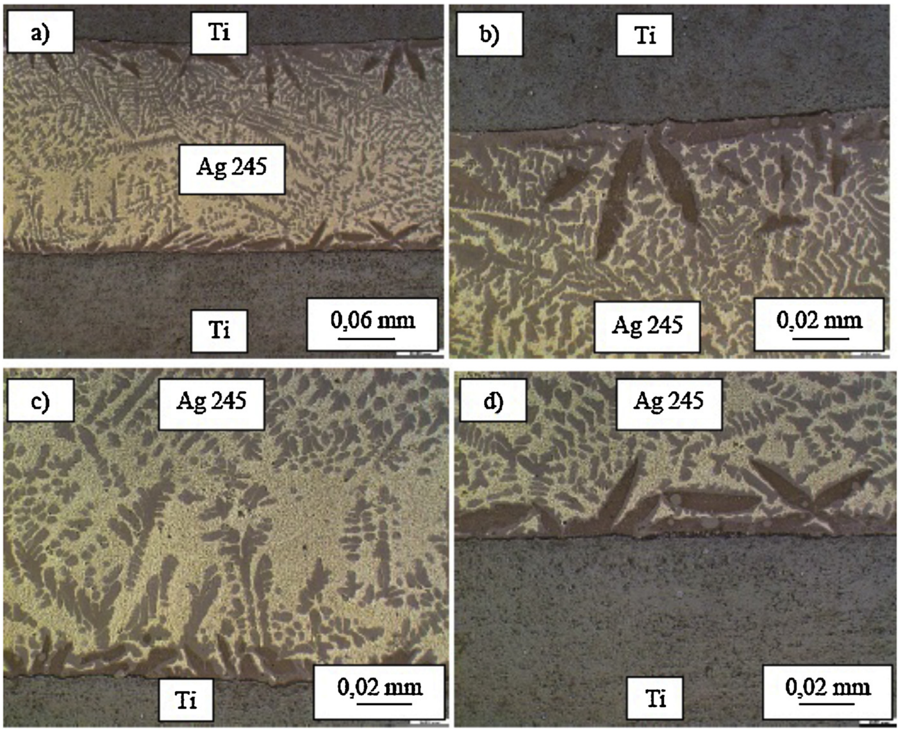


Fig. 7 – Microstructure of the joint of Grade 2 titanium made using the Ag 245 silver brazing metal and the F60T flux, chemical etching using Buehler's reagent: Ti–Ag 245–Ti (a), Ti–Ag 245 top (b), braze (c), Ag 245–Ti bottom (d).

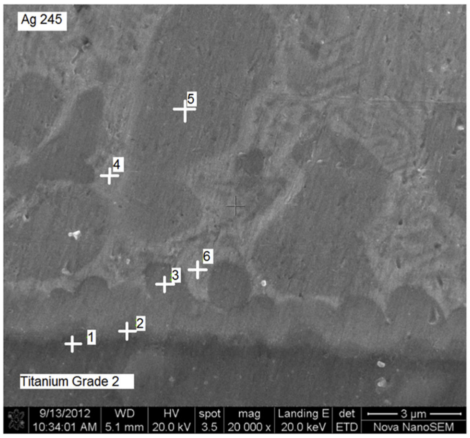
significant amount of present copper (30.5% by weight) favoured the formation of intermetallic phases from the Cu–Ti system, type Cu_3Ti_2, Cu_4Ti_3, $CuTi_2$. In turn, at point 3, on the braze surface, approximately 2 μm away from the titanium surface it was also possible to observe titanium, yet its amount was significantly lower (19.4% by weight).

2.6. *Derivatographic measurements*

The melting point of the F60T flux was determined using the method of differential scanning calorimetry (DSC). To this end it was necessary to use a DSC Mettler Toledo 821e scanning micro-calorimeter connected with a Thermo Haake compression refrigerator.

On the DSC diagram for the F60T flux heated in the air atmosphere (Fig. 9) it is possible to observe four endothermic effects. The first effect can be observed at 46 °C and is characterised by a low enthalpy $\Delta H = +31.2$ J/g, the second endothermic effect observed at 133.7 °C is the highest of all the effects as it is characterised by a significant enthalpy $\Delta H = +150.4$ J/g; this effect is connected with the evaporation of water bounded in the flux. The third effect is present at 446.8 °C and can be ascribed to the evaporation of some flux components.

On the basis of the curve analysis (Fig. 9) the temperature of 576 °C can be adopted as the final value of the flux melting point. The entire melting of the flux at this temperature is proper as regards brazing processes utilising silver brazing



Point	Content , % by weight			
	AgL	TiK	CuK	ZnK
1	1.2	88.6	6.7	3.5
2	2.9	54.0	30.5	12.6
3	6.1	19.3	49.6	25.0
4	60.8	0.9	16.9	21.4
5	18.3	0.8	52.7	28.2
6	58.8	3.0	16.6	21.6

Fig. 8 – Microstructure of the Ti–Ag 245 joint with EDS measurements in 6 points.

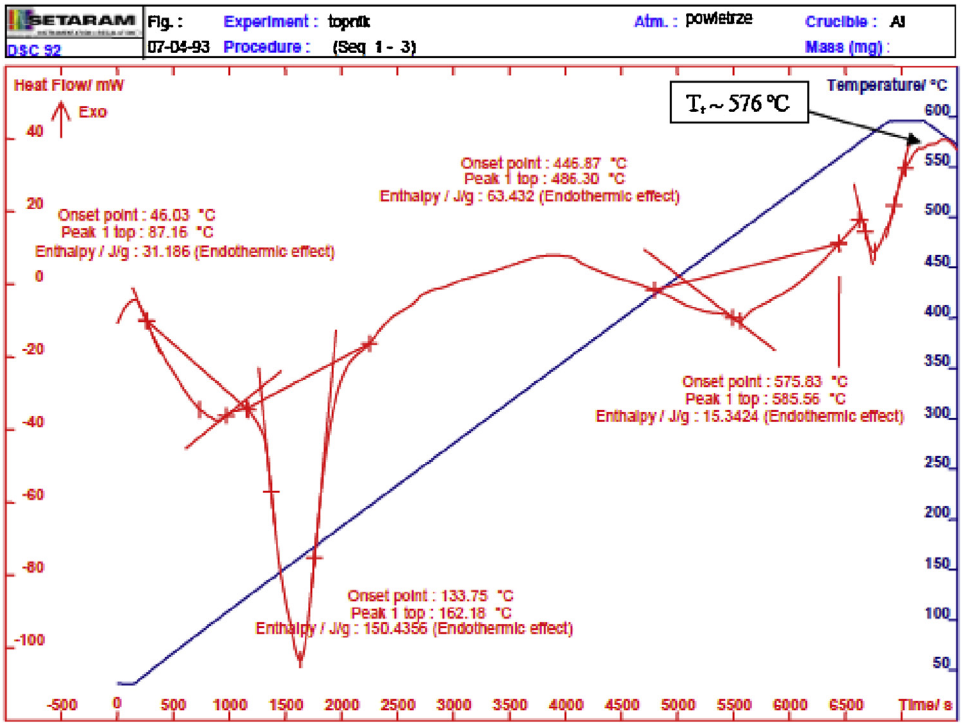


Fig. 9 – DSC diagram of F60T flux heating in the air atmosphere.

metals, e.g. Ag 245. In accordance with the brazing process assumptions the first to melt is the flux, whose effect prepares the titanium surface for wetting and spreading of the brazing metal.

3. Experimental tests results of flux brazing of AZ31B magnesium alloy

3.1. *Base and filler metals*

The tests involved the use of base metal in the form of a 2.3 mm thick Mg–Al–Zn–Mn (AZ31B) magnesium alloy sheet. The filler metal which underwent spreadability and wettability tests on the magnesium base was the MgAl9Zn3Mn1 grade magnesium brazing metal (M001 according to PN-EN ISO 17672, BMg-1 according to AWS A 5.8, having a melting point of 443–599 °C) in the form of a rod with a diameter of 2.0 mm. Brazing consumables used in the tests were the mixtures of fluorides and chlorides of intermetals and alkali metals [12].

3.2. *Testing the brazing properties and the measurements of wetting angle*

The tests involved the use of 2.3 mm thick round plates with a diameter of 50 mm as the base. The brazeability tests were preceded by degreasing with acetone and etching in 10% aqueous solution of HNO_3. On each Plate 0.2 g of the brazing metal and 0.2 g of the flux were applied. The heating was performed using a propane-air flame (Fig. 10).

The heating of the test pieces was finished 5 s after the melting of the brazing metal. Following the solidification of the brazing metal the test pieces were left in the air atmosphere for cooling. Next, after cooling the test pieces were immersed in cold water in order to remove flux slag. The tests, in addition to evaluating the basic criterion of brazeability assessed on the basis of the area on which the brazing metal had been spread, also involved the analysis of the flux behaviour, i.e. its softening, melting and spreading, the chemical cleaning of the base surface, the efficiency of covering and protecting the molten brazing metal against oxidation as well as the type and easy removing of flux slag.

During the tests on flux for brazing of the Mg–Al–Zn–Mn (AZ31B) magnesium alloy 15 flux test compositions were developed, being changing on the basis of the verification of melting point, activity and brazing properties. For each test flux version 3 spreadability tests were performed.

On the basis of spreadability tests conducted it was determined that the most advantageous flux was that designated as FMAG containing such compounds as KCl, LiCl

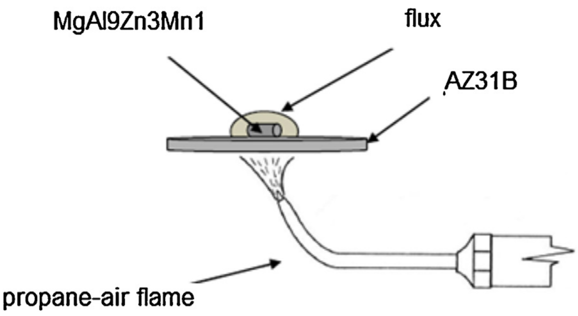


Fig. 10 – Scheme of brazing metal spreadability test on magnesium base.

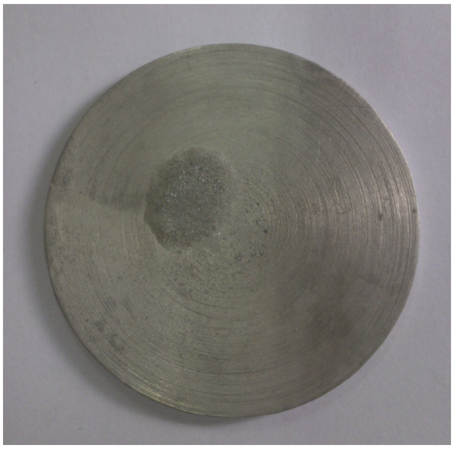


Fig. 11 – Spreadability of the MgAl9Zn3Mn1 grade magnesium brazing metal on the AZ31B magnesium alloy base using the FMAG flux.

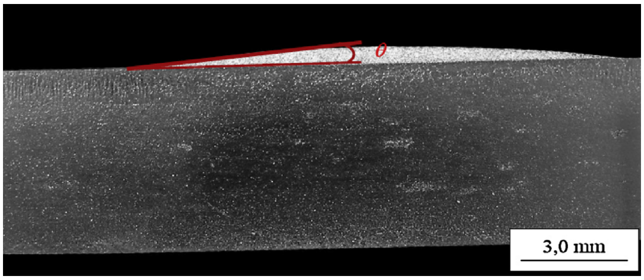


Fig. 12 – Wetting of the AZ31B magnesium base with the molten MgAl9Zn3Mn1 brazing metal, wetting angle 16°.

H_2O, $CaCl_2$ and NaF [19]. The spreadability of the MgAl9Zn3Mn1 braze on magnesium surface reached 94 mm^2 – average value of three measurements.

Fig. 11 presents the spreadability of the MgAl9Zn3Mn1 grade magnesium brazing metal on the AZ31B magnesium alloy base using the FMAG flux.

The spreadability test was followed by the graphic determination of a wetting angle. The wetting angle determined amounted to 16° (Fig. 12), which indicates very good wettability of the AZ31B magnesium base with the MgAl9Zn3Mn1 brazing metal.

3.3. *Making test joints*

The base metal used in the tests was in the form of plates (30 mm × 30 mm × 2.3 mm) made of the Mg–Al–Zn–Mn (AZ31B) magnesium alloy. The filler metal used in the tests was the MgAl9Zn3Mn1 grade magnesium brazing metal. The heat source was a propane–air flame.

The overlap joints (plates were placed horizontally, freely without pressure, overlap length of approximately 5 mm, flux in the brazing gap and at the overlap outlet) were made dosing the flux manually at the overlap end. In these conditions the brazing gap width was formed spontaneously during the inflow of the brazing metal. After brazing and solidification the brazes were immersed in cold water in order to remove flux slag.

3.4. *Mechanical properties – static shearing test*

The static shearing test of the brazed joints made of the AZ31B magnesium alloy using the MgAl9Zn3Mn1 magnesium filler metal and the FMAG flux involved five test pieces. The shearing area amounted to 26 mm^2. In order to ensure the axiality of load eliminating the bending of the joint it was necessary to use elements which corrected fixing of the test pieces in the holders. The average shear strength of 5 test pieces amounted to 47.6 MPa, with a standard deviation of 2.4 MPa and the brazing gap amounting to, on average, 0.11 mm.

The shear strength value obtained is relatively low, yet the analysis of reference publications [15] revealed that such joints made with magnesium filler metals are characterised by a shear strength of approximately 50–60 MPa. In all cases the test pieces underwent separation in the brazed joint, on the boundary between the braze and the material being brazed.

3.5. *Macroscopic metallographic examination of overlap joints*

The metallographic joint revealed that while using the FMAG flux it is possible to obtain an overlap joint characterised by good quality and the joint gap filled entirely along the whole length of the overlap amounting to 5 mm. The width of the brazing gap amounted to 0.11 mm. The joint did not contain any brazing imperfections. The use of the FMAG flux enables obtaining the smooth and proper shape of the braze face. Fig. 13 presents the macrostructure of the overlap joint made of the Mg–Al–Zn–Mn (AZ31B) magnesium alloy using the MgAl9Zn3Mn1 grade magnesium brazing metal and FMAG flux.

3.6. *Metallographic examination of overlap joints*

The overlap joints underwent metallographic examination using a Leica-manufactured MeF 4M light microscope. Fig. 14 presents the microstructure of the flame brazed joint made of the Mg–Al–Zn–Mn (AZ31B) magnesium alloy using the MgAl9Zn3Mn1 grade magnesium brazing metal and the FMAG flux.

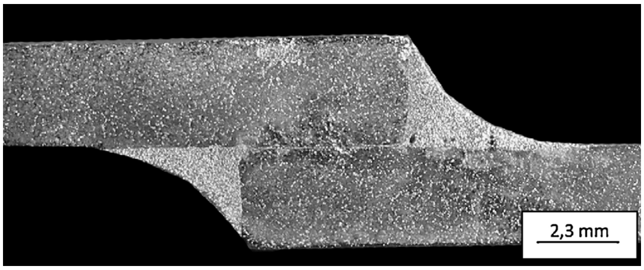


Fig. 13 – Macrostructure of overlap joint made of Mg–Al–Zn–Mn (AZ31B) alloy using the MgAl9Zn3Mn1 magnesium brazing metal and the FMAG flux, gap width 0.11 mm, chemical etching with 10% solution of acetic acid.

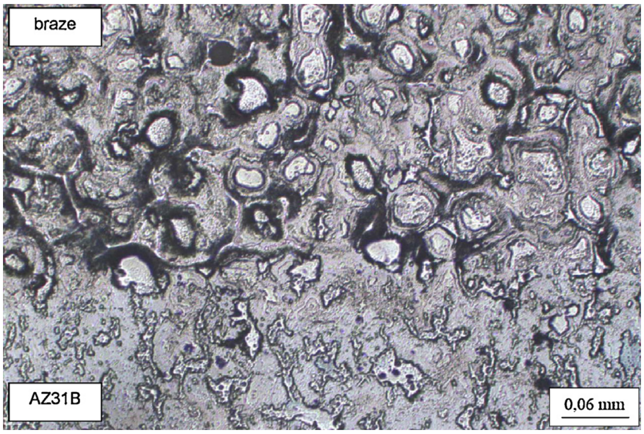


Fig. 14 – Microstructure of the transition zone of the joint made of the AZ31B (MgAl3Zn1Mn0.2) alloy using the MgAl9Zn3Mn1 filler metal (braze at the top), etching with Nital.

The metallographic examination of the joint revealed its good quality – very few brazing imperfections such as gas cavities in the joining area were observed. The joint did not contain any elaborate diffusion zones either. The structure of the braze itself was relatively complex. Reference publications state [16,19] that it is possible to observe the presence of the precipitates of the β – $Mg_{17}Al_{12}$ intermetallic phase in the matrix of the solid solution α – Mg (Mg–Al–Zn).

4. Discussions

All of the test results revealed that it is possible to carry out brazing titanium Grade 2 and magnesium alloy AZ31B in the atmospheric air. In order to obtain good quality joints, it is necessary to use the special fluxes: for titanium Grade 2 F60T flux, and for the magnesium alloy AZ31B flux FMAG. The overlap joints obtained in this way are characterised by good quality, shear strength and did not contain any brazing imperfections. After brazing the joints contained relatively small amounts of flux slag.

The recipe of both fluxes due to their innovative chemical composition have been reported and patented in Polish Patent Office [17–19].

5. Conclusions

1. The test results concerning the spreadability of the Ag 245 brazing filler metal on the surface of titanium, using the F60T flux, revealed that it is possible to develop flux not containing expensive and light-sensitive silver chloride (AgCl). This compound can be replaced by, e.g. zinc chloride ($ZnCl_2$), which does not undergo light-induced decomposition.
2. The most advantageous test results concerning the brazing properties of fluxes as well as the quality and strength of brazed joints made of Grade 2 titanium using fluxes and the Ag 245 brazing metal were obtained for the F60T flux containing such compounds as $KF{\cdot}2H_2O$, KHF_2, LiCl and $ZnCl_2$. In turn, in the case of the joints made of the AZ31B alloy brazed with the MgAl9Zn3Mn1 magnesium filler metal the best results concerning brazing properties were obtained for the flux designated as FMAG, containing such compounds as KCl, LiCl H_2O, $CaCl_2$ and NaF.
3. The activity of the developed flux F60T is sufficient within the temperature range 550–800 °C, whereas the activity of the FMAG flux is sufficient within the temperature range 450–600 °C.
4. The F60T flux enables obtaining proper brazed joints both by C_2H_2–O_2 flame heating and by induction heating. In turn, the FMAG flux is recommended for flame brazing of the AZ31B magnesium alloy using the MgAl9Zn3Mn1 magnesium filler metal.
5. The brazed overlap joints made of Grade 2 titanium using the Ag 245 brazing metal and the F60T flux were characterised by good quality and relatively high shear strength amounting to 163 MPa. In turn, the overlap joints made of the AZ31B magnesium alloy using the MgAl9Zn3Mn1 magnesium filler metal and FMAG flux were characterised by good quality and shear strength amounting to 47.6 MPa. After brazing the joints contained relatively small amounts of flux slag.

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