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Vacuum High-Temperature Brazing of 3003 Aluminum Alloy

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Abstract. Brazing filler metals based on the Al-Si system are widely used for brazing aluminum alloys. Their melting point is 577 °C (eutectic). It is necessary to conduct comprehensive studies of the technological properties of experimental filler metals and brazed joints to create a brazing filler metal with a reduced melting temperature for vacuum brazing of thin-walled aluminum products made of alloy 3003. The paper presents the research results on high-temperature vacuum brazing of aluminum alloy 3003 with Al-Cu-(Si, Mg) filler metal. It was determined that the amount of magnesium in the filler should be limited due to the risk of porosity formation associated with magnesium vaporization. It was identified that reducing the magnesium content increases the liquidus temperature above 530–550 °C. Therefore, experimental alloys require additional alloying with depressant elements, particularly silicon, to achieve the required melting temperature level. The chemical inhomogeneity of the filler in the initial state (after rapid solidification from the liquid state) and the structure of the brazed joints were investigated using micro-X-ray spectral analysis. Through empirical means, it was determined that a magnesium content of 1.5 % by weight in the filler allows for producing high-quality brazed joints without visible defects. In this case, shear strength is in the range of 0.6–0.7 of the strength of the base material. Tests of brazed joints for three-point bending resulted in an angle close to 180°, which indicates the promising use of experimental brazing filler metal in vacuum brazing of aluminum alloy 3003.

Keywords: vacuum brazing, aluminum alloy, filler metal, magnesium, microstructure.

1 Introduction

In recent decades, aluminum alloys have been widely used in aerospace engineering, the automotive industry, and instrument making due to their undeniable advantages, such as high specific strength and sufficient thermal and electrical conductivity [1–4]. Their application ensures increased weight efficiency and operational lifespan of various products. Aluminum alloys are also used to manufacture air conditioners, refrigerators, and other structures. Traditionally, non-separable joints of aluminum alloys are obtained through welding and brazing methods. It is known [5–9] that aluminum and its alloys are rapidly covered with a thin and dense oxide film of Al_2O_3 in the atmosphere. It protects the surface from further oxidation, providing high corrosion resistance in atmospheric conditions, salt solutions, and certain acids. However, due to its chemical stability and high melting temperature (2072 °C), this dense aluminum oxide layer significantly complicates obtaining non-separable joints. In particular, it hinders the contact between the base metal and the molten filler metal during heating according to the brazing thermal regime [5–9]. Therefore, brazing uses fluxes in a controlled environment or a vacuum [10–14].

2 Literature Review

Fluxes are used in industrial furnace brazing to destroy chemically and thermodynamically stable oxide films. These fluxes consist of alkaline and heavy metal chloride salts enriched with metal fluorides [10, 14]. However, due to their increased corrosive activity, products require thorough and prolonged treatment to remove flux residues after brazing [15].

Furthermore, an alternative to traditional flux-based aluminum brazing is vacuum brazing, which is widely used, particularly for joining chemically active metals and alloys [16].

In general, the mechanism of fluxless vacuum brazing of aluminum alloys is as follows:

– due to a significant difference in the coefficients of linear expansion between aluminum and Al_2O_3, local cracking of the oxide film occurs during heating in a vacuum;

– the molten filler metal can penetrate through these cracks and wet the surface of the base metal, but only if new oxidation of aluminum is prevented in the newly formed cracks. The brazing atmosphere must be completely devoid of oxygen [17].

Most industrial aluminum brazing alloys are based on the Al-(6–12)Si system. According to the Al-Si binary phase diagram, a eutectic phase can also form with aluminum at 577 °C. The oxide film cannot hinder silicon diffusion during the formation of the eutectic phase as a liquid layer between the joining surfaces, which can disrupt the integrity of the surface film Al_2O_3 [18]. Therefore, the presence of silicon in the filler promotes partial disruption of the oxide layer [19]. Increasing the heating temperature 560–590 °C of the 5A06 alloy series enhances the destruction and dispersion of oxide films, but it also increases the degree of oxidation when obtaining joints using induction heating in argon [20].

Additionally, the relatively high melting temperature of the filler metals [7, 8] imposes additional limitations on the use of brazing for alloyed and thermally strengthened aluminum alloys due to the proximity of the brazing temperature to the solidus temperature. Nevertheless, in vacuum brazing, it is necessary to introduce additional elements-activators (e.g., Mg, Bi) into the composition of the filler metals [9].

Eutectic systems such as Al-Ge and Al-Cu can be considered alternative systems for filler metals development, where the minimum eutectic temperature is 420°C and 546°C, respectively [21].

In Al-Cu fillers, the small atomic radius of 1.28Å for copper [22] and, consequently, the high diffusion activity of copper atoms allow them to penetrate the Al_2O_3 film with a few nanometers of thickness. Thus, copper diffusion from the filler through the oxide film can occur, followed by a eutectic reaction with the base metal. Theoretically, with 33 % copper content in the filler, an active eutectic phase capable of disrupting the oxide film will be formed. However, on the other hand, too high a percentage of copper in the filler leads to the formation of a more significant amount of a brittle Cu_xAl_y phase [23], which significantly deteriorates the mechanical characteristics of the brazed joint.

Furthermore, there is evidence that additional silicon alloying in Al-Cu fillers can impede the formation of the $CuAl_2$ phase [24] and increase the fluidity of fillers [25].

Additional magnesium alloying in filler metals can be applied, on the one hand, to reduce the partial pressure of residual oxygen and water vapor because, as known, magnesium reacts with them according to reactions [26]:

$$Mg + \frac{1}{2}O_2 \rightarrow MgO; \quad (1)$$

$$Mg + H_2O \rightarrow MgO + H_2. \quad (2)$$

On the other hand, magnesium atoms can directly react with Al_2O_3 to form $MgAl_2O_4$ and MgO oxides, disrupting the oxide film. It should be noted that Mg reacts with Al_2O_3 to form $MgAl_2O_4$ according to the following equation:

$$3Mg + 4Al_2O_3 \rightarrow 3MgAl_2O_4 + 2Al, \quad (3)$$

when the magnesium content is approximately 0.1–1.0 % by mass [27].

When the magnesium content reaches 2.0–2.5 % by mass, the interaction between magnesium and Al_2O_3 occurs according to the following equation [2]:

$$3Mg + Al_2O_3 \rightarrow 3MgO + 2Al. \quad (4)$$

In the case of the complex Al-Cu-Mg-Si system, there are ¾ eutectic points in the aluminum corner [5]:

1) Al – 27.7 Cu – 2.7 Mg – 5.3 Si; T = 509 °C, L = (Al) + $CuAl_2$ + (Si) + $Al_5Cu_2Mg_2Si_6$;

2) Al – 32.9 Cu – 7.0 Mg – 0.6 Si; T = 502 °C, L = (Al) + Al_3Mg_2 + Mg_2Si + Al_2CuMg;

3) Al – 1.2 Cu – 34.0 Mg – 0.1 Si; T = 448 °C, L = (Al) + Al_3Mg_2 + Mg_2Si + $(AlCu)_{49}Mg_{32}$.

During the solidification of Al-Cu-Si-Mg alloys, the following phases can be formed: $CuAl_2$, $Al_5Cu_2Mg_2Si_6$, Mg_2Si, Al_2CuMg, and $(Al, Cu)_{49}Mg_{32}$. These intermetallic compounds are brittle and can significantly reduce the mechanical properties of the brazed joints. The formation of such intermetallic compounds can be prevented by properly selecting temperature-time parameters in the brazing cycle [9].

The concentration of constituent components in the brazing filler metal is also significant. Therefore, by developing new brazing filler metal and the technological process of brazing, these parameters must be taken into account.

In addition, an important point is the tendency to decrease the melting temperature range of the brazing filler metal. That will avoid the deformation of thin-walled aluminum structures during heating and preserve their geometric parameters.

The article aims to create an experimental brazing filler metal for brazing aluminum alloy 3003, characterized by a reduced melting temperature. The following objectives were formulated to achieve this goal:

– to choose a basic alloying system that provides the necessary melting temperature interval; make brazing filler metal in acceptable condition;

– to determine the temperature of solidus and liquidus; investigate the spread of brazing filler metal on the base metal and the formation of brazed joints; conduct mechanical tests of brazed joints.

This study presents the micro X-ray spectral and mechanical investigations of aluminum alloy 3003 (Al – 1.0–1.5 Mn) joints obtained by high-temperature vacuum brazing with an Al-Cu-Si-1.5Mg experimental filler metal.

3 Research Methodology

3.1 Research objects

Additionally, for conducting the experimental investigations, the alloy 3003 (1.0–1.6 % Mn, no more than 0.7 % Fe, 0.6 % Si, 0.2 % Cu, 0.2 % Ti, 0.1 % Zn, 0.05 % Mg, and balance of Al) was used as the base material in the form of 1.7 mm thick plates. This alloy is manufactured according to the EN 573-3 standard. It is widely used to produce various heat exchange devices, including air conditioners, refrigerators, and other structures.

The eutectic alloy based on the Al-Cu-Si system alloyed with magnesium (1.5–3.1 %) served as the base for producing experimental filler metals.

3.2 Research methods

The experimental brazing filler metals were melted using a laboratory arc melting unit on a cold copper substrate in an argon atmosphere. The solidus and liquidus temperatures were determined by a computational method with the "JMatPro7" software and experimentally using high-temperature differential thermal analysis on the VDTA-8M device. Rapid solidification from the liquid state (Figure 1) with a cooling rate of approximately 105 °C/s was employed to obtain the filler as a microcrystalline ribbon with a 50–100 μm thickness.

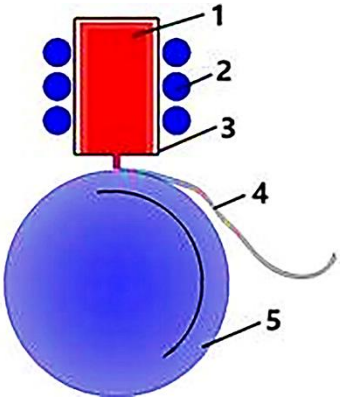


Figure 1 – Simplified scheme of obtaining the filler as rapidly solidified ribbon: 1 – filler melt; 2 – inductor; 3 – crucible; 4 – solidified ribbon; 5 – copper drum

For this, the brazing filler metal ingot 1 was melted using an induction power source 2. After receiving the liquid metal, it was poured onto a copper drum 5, which rotates at high speed and provides ultra-fast cooling with the simultaneous formation of a thin ribbon of brazing filler metal 4.

Before brazing, the aluminum alloy samples were prepared by mechanically removing the oxide film (using diamond disc grinding with a grit size of 120 μm) and chemically cleaning them. The chemical cleaning process involved using aqueous solutions: 15 % NaOH (for degreasing), 20 % vol. HNO_3, and 2 % vol. HF (for etching) with intermediate rinsing steps in distilled water. After cleaning, the samples were used for the flow and brazing experiments in a vacuum furnace with radiant heating. The vacuum chamber pressure during heating was maintained at no less than 6.33 mPa. The brazing of lap joints with the aluminum alloy 3003 was performed using the following parameters: heating temperature of 550 °C with a holding time of 1 minute. The heating rate was set at 15 °C/min.

For metallographic examinations and mechanical testing, samples brazed with microcrystalline ribbon were placed in the gap between the joined samples of the base metal. Plate-shaped lap joint samples were brazed for mechanical testing (Figure 2), with the filler's thickness corresponding to the 1.7 mm base metal thickness.

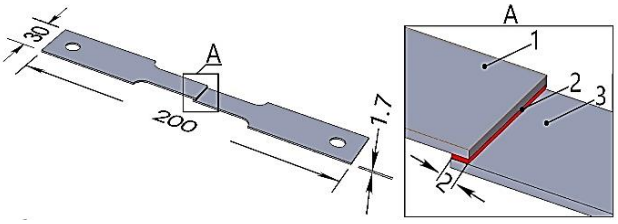


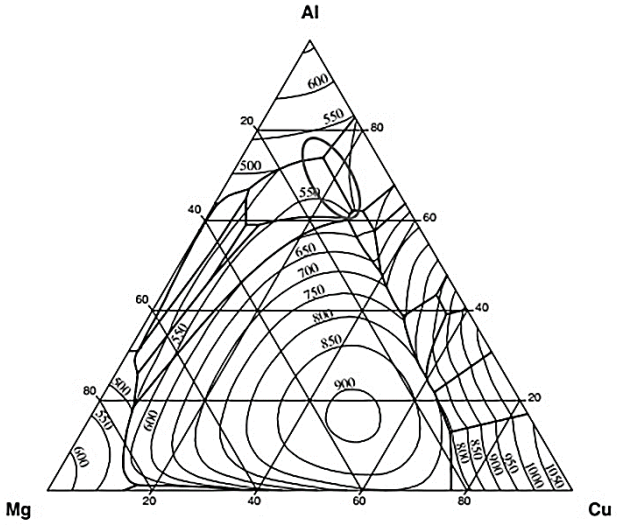
Figure 2 – Schematic representation of the overlapping sample for conducting mechanical tests: 1, 3 – base metal; 2 – brazing filler metal

The microstructure and elemental composition of the brazed joints were studied by the scanning electron microscope (TescanMira 3 LMU), equipped with an Oxford Instruments X-max 80 mm^2 energy-dispersive spectrometer and INCA software. The backscattered electron (BSE) signal was used to visualize the constituent phases by atomic number and study the distribution of elements. The micro X-ray spectroscopic analysis provided high local resolution measurements up to 1 μm.

4 Results

In order to preserve the structure and properties of the base metal (3003 alloys), the temperature range of the high-temperature brazing process needs to be limited and should not exceed 550 °C. Alloys based on aluminum, containing other alloying elements, are within this temperature range. The base system for the filler alloys was Al-Cu-Si. Magnesium was used to disrupt the oxide film [28]. According to the phase diagrams [29] in the ternary Al-Cu-Mg system, several eutectic compositions have temperatures within or close to the required range (Figure 3).

It should be noted that these alloys have an increased amount of magnesium (% mass): Al-18Cu-26Mg (melting temperature 480 °C), Al-32Cu-7.5Mg (melting temperature 503 °C). On the one hand, this magnesium concentration provides the necessary melting temperature range. On the other hand, a high amount of magnesium can negatively affect the brazing process and the mechanical properties of the brazed joints.



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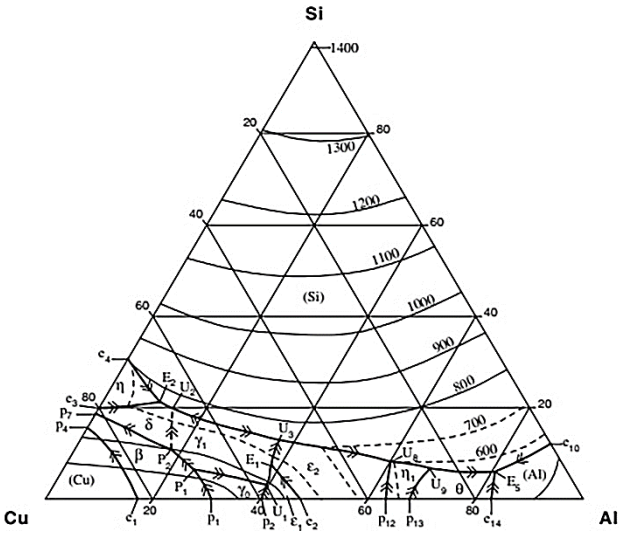


b

Figure 3 – Isotherms of the liquidus (a) on the ternary phase diagram of the Al-Cu-Mg system (% atomic) and the liquidus surface projection of the ternary Al-Cu-Si system (b) [29]

The results of the flow ability investigations indicate that when heating the filler metals with an elevated magnesium content, active dissolution of the base metal in the molten filler occurs, accompanied by intense evaporation of magnesium, leading to the formation of pores, undercuts, and shrinkage cavities (Figure 4).

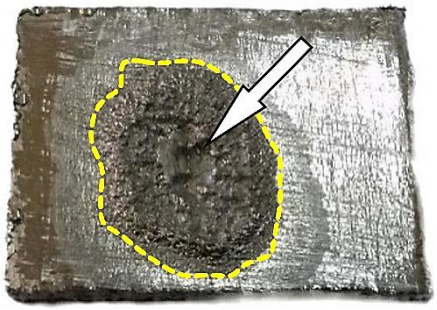
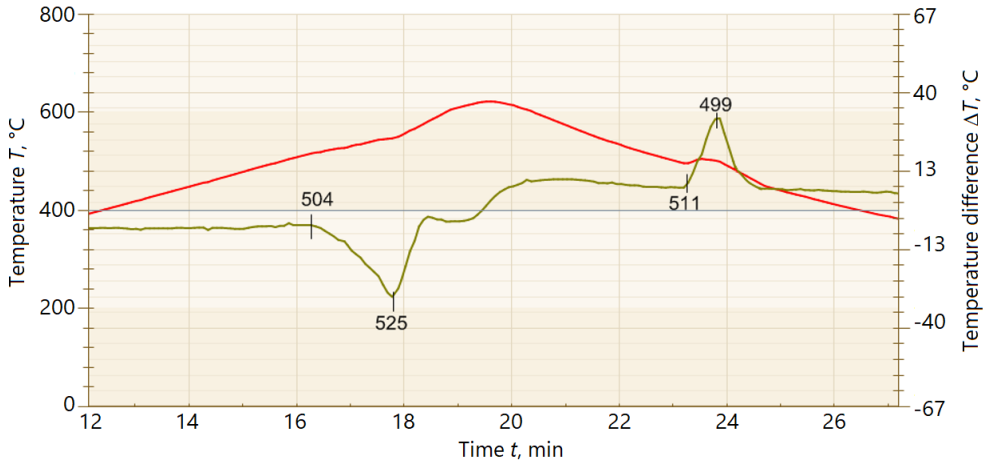


Figure 4 – External view of the Al-Cu-26Mg (% mass) filler droplet after flowing along the 3003 alloys (500 °C, 1 min)

Therefore, aluminum filler metals containing magnesium in the range of 1.5–3.1 % were produced for the experiments. It should be noted that reducing the magnesium content (below 7 % mass) in the ternary alloys increases the liquidus temperature (above 530–550 °C). Therefore, the experimental alloys required additional alloying with depressant elements to achieve the necessary melting temperature level. In this case, silicon alloying was used, which forms a eutectic with aluminum.

Using the example of investigating the melting temperature interval of the experimental Al-Cu-Si-3.1Mg alloy, the thermal curve is shown (Figure 5a), which exhibits endothermic and exothermic thermal effects corresponding to the solidus temperature (504 °C) and the liquidus temperature (525 °C) during heating and cooling, respectively (499 °C and 511 °C).

Reducing the magnesium concentration from 3.1 % to 1.5 % leads to a slight increase in the solidus temperature from 504 °C to 508 °C and the liquidus temperature from 525 °C to 542 °C, as observed on the endothermic curve during heating (Figure 5b).



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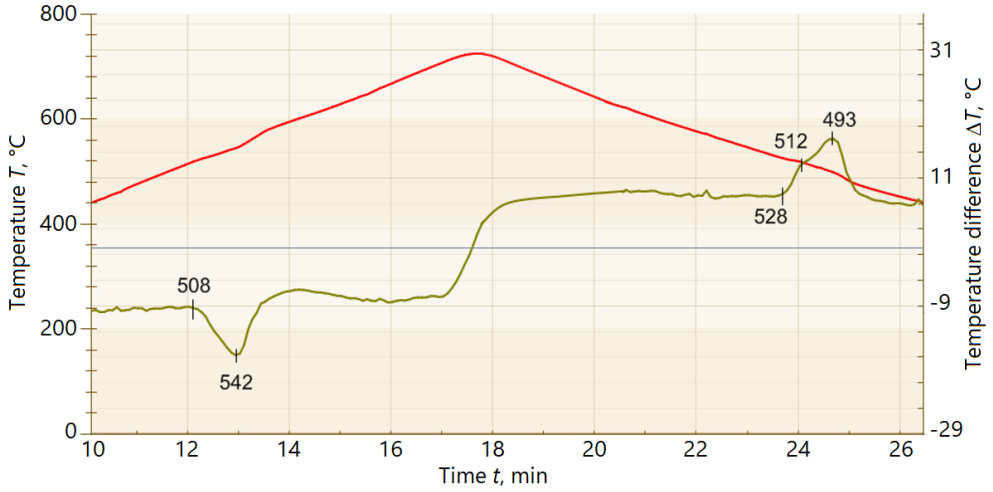


b

Figure 5 – Melting temperature intervals of experimental Al-Cu-Si-Mg alloys containing 3.1 % (a) and 1.5 % (b) magnesium

Notably, the calculated solidus and liquidus temperatures (510 °C and 540 °C, respectively) align well with the experimental data obtained using high-temperature differential thermal analysis (508 °C and 542 °C, respectively) for the Al-Cu-Si-1.5Mg filler metal (Figures 5b, 6).

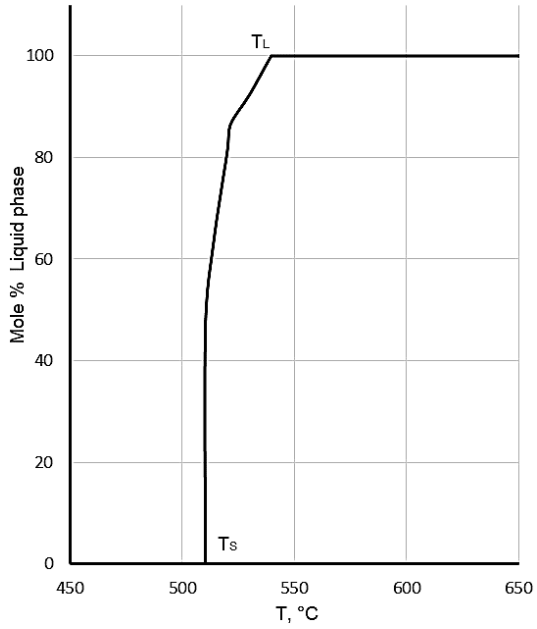
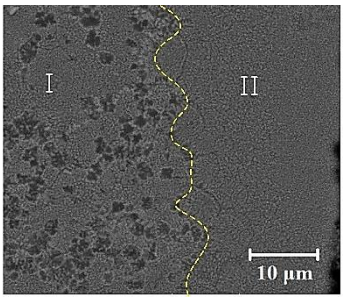


Figure 6 – Calculated solidus and liquidus temperatures of the Al-Cu-Si-1.5Mg alloy

The experimental Al-Cu-Si-(1.5)Mg filler alloy in microcrystalline foil was used for further investigations. It is characterized by a heterogeneous structure containing two zones (Figure 7a).



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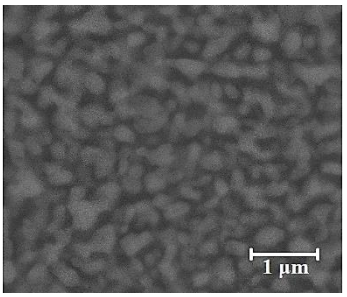

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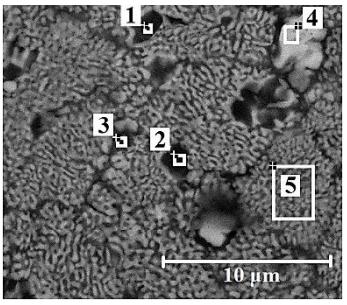


c

Figure 7 – Microstructure of the Al-28Cu-4Si-1.5Mg filler metal ribbon: a – after rapid solidification; b – zone No. 2; c – phases within zone No. 1, where the chemical composition was determined

Zone No. 1 is characterized by a eutectic cellular structure formed as separate colonies, with individual phases crystallizing along their boundaries, with a size of 0.23–0.35 μm (Figure 7b). The copper content in phases No. 1–3 ranges from 7.53 % to 11.4 % by mass (Table 1).

Table 1 – Chemical composition of individual phases in the rapid solidification filler metal

Spectrum No.	% wt.			
	Mg	Al	Si	Cu
1	0.48	77.01	1.62	20.89
2	0.89	79.94	3.08	16.09
3	1.23	71.24	4.22	23.31
4	0.69	54.02	2.42	42.87
5	0.48	67.30	1.95	30.26

In phase No. 4, it increases to 42.9 %. It should be noted that although these phases form the eutectic, they are significantly smaller, and the eutectic's copper content does not exceed 30.3 %.

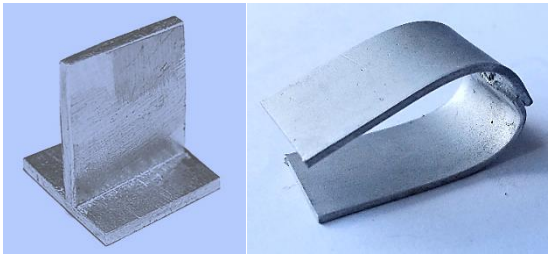
An ultrafine homogeneous structure characterizes the metal in zone No. 2, but determining the local chemical composition of each phase is not possible due to the resolution of the electron microscope and the size of individual particles (their size does not exceed 0.5 μm). These morphological features are due to the extremely high cooling rate (105 °C/s) inherent in the rapid solidification process from the liquid state.

Additionally, the metal in zone No. 2 crystallizes upon contact with the copper disc, which contributes to a faster cooling rate than the cooling rate in zone No. 1, resulting in an ultrafine structure. However, this does not affect the chemical composition of these zones, as confirmed by the results of local micro-X-ray spectroscopic analysis, which show that the chemical composition of both zones contains approximately the same concentration of the alloying elements.

Using microcrystalline ribbon Al-Cu-Si-(1.5)Mg filler metal with a fine-dispersed structure and homogeneous chemical composition positively affects brazing. T-joint samples of aluminum alloy 3003 obtained by vacuum brazing at a temperature of $T = 550$ °C (1 min, microcrystalline ribbon filler metal in the gap) using this experimental filler demonstrate good wetting of the base metal and the formation of high-quality brazed joints without erosion (Figure 8a).

It is known that in the presence of silicon and iron in aluminum and its alloys, triple compounds of these metals, α-(Al-Fe-Si) and β-(Al-Fe-Si) with a plate-like or needle-like morphology, are formed. The formula Fe_2SiAl_8 describes the composition of the α-phase, and the β-phase is described by $FeSiAl_5$ [30].

The results of micro-X-ray spectroscopic studies are shown: microstructure of the 3003 alloys after plastic deformation and annealing consists of a solid solution in which elongated particles of a light phase enriched with iron and manganese are present (Figure 9, Table 2).



a b

Figure 8 – The external appearance of the T-joint sample after brazing (a) and plate specimen after 3-point bending tests (b)

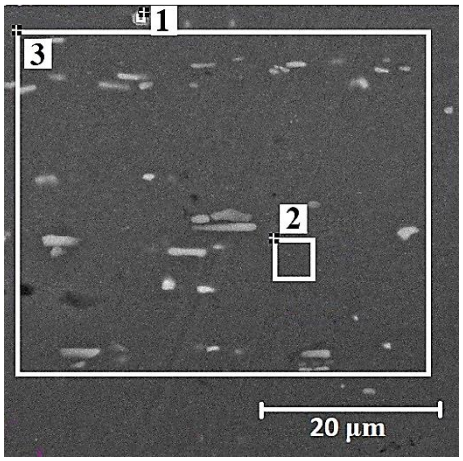


Figure 9 – Microstructure of the base metal

Table 2 – Chemical composition of individual phases in the rapid solidification filler metal

Spectrum No.	% wt.				
	Al	Si	Mn	Fe	Cu
1	83.21	2.71	6.28	7.80	0.00
2	99.04	–	0.78	0.00	0.18
3	98.59	0.00	1.13	0.28	0.00

They have a shape close to polyhedral and are oriented in the rolling direction. According to literature sources, these particles can be classified as complex intermetallic phases $Al_6(Mn, Fe, Si)$, which are inherent to the base metal [30].

The solid solution based on aluminum (spectrum No. 2, Table 2) contains a small concentration of manganese and copper, which correlates well with the phase diagrams indicating the limited solubility of these elements in aluminum at room temperature [21].

After brazing, the base metal retains the texture formed during its manufacturing (through deformation). In the solid solution, a dispersed needle-like structure is observed (Figure 10), which is poorly visualized.

These morphological features are characteristic of alloys in this series after partial decomposition of the α-solid solution, leading to dispersed intermetallic phases like $(Fe, Mn)Al_6$ [30].

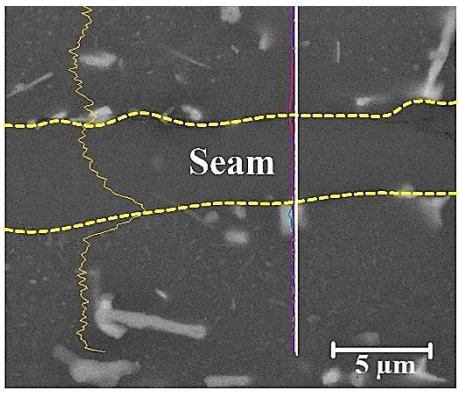
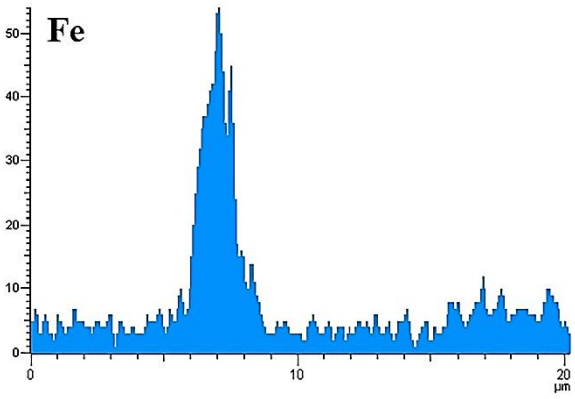


Figure 10 – Electron image of the microstructure of the brazed joint

The light phase in the base metal, contrasting with the aluminum solid solution, contains elevated concentrations of iron, manganese, copper, and silicon (Figure 11), which are typical for the Al_6(Fe, Mn, Si, Me) phase [27].

Micro-X-ray spectroscopic studies confirmed the presence of a homogeneous solid solution structure based on aluminum in the brazed joint through scanning with an electron beam (Figure 10).

Individual inclusions of a phase with an elevated copper concentration are observed in some areas of the joint. Local micro-X-ray spectroscopic analysis revealed that a homogeneous solid solution structure based on aluminum, containing 2.7 % copper, is formed in narrow gaps with a width of 3–5 µm.



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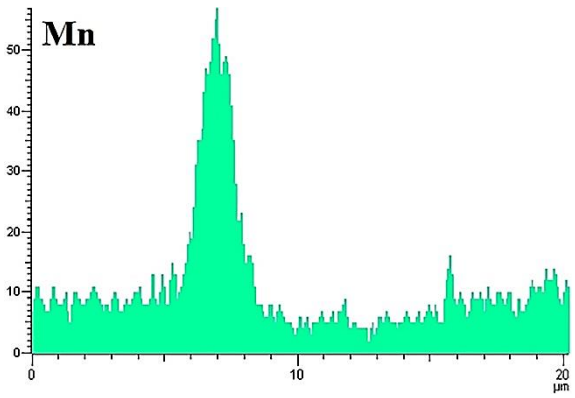

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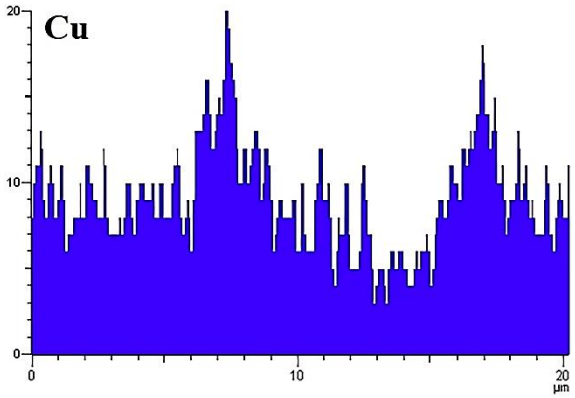

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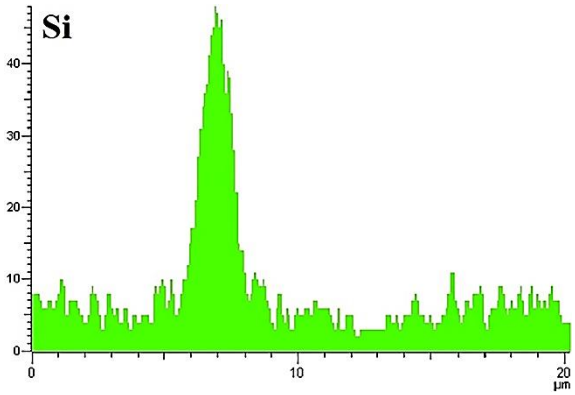

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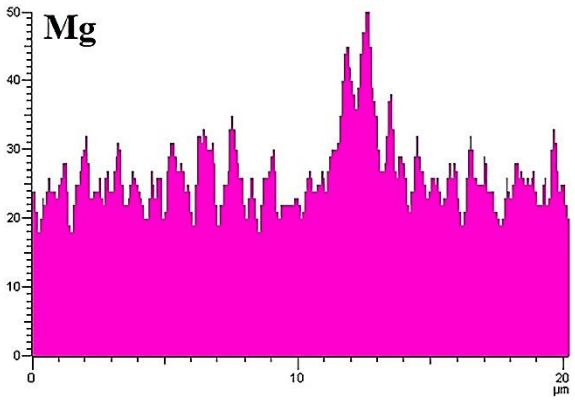

e

Figure 11 – Qualitative distribution of Fe (a), Mn (b), Cu (c), Si (d) and Mg (e) in the brazed joint during scanning with an electron beam

In wider gaps (over 15 µm), closer to the fillet regions, a phase enriched with copper crystallizes within the background of the solid solution (Figure 12).

The presence of this phase is characteristic of the initial state of the brazing alloy. It may correspond to the intermetallic phase $AlCu_2$ identified in the rapidly solidified ribbon of the alloy at the initial state (Figure 13).

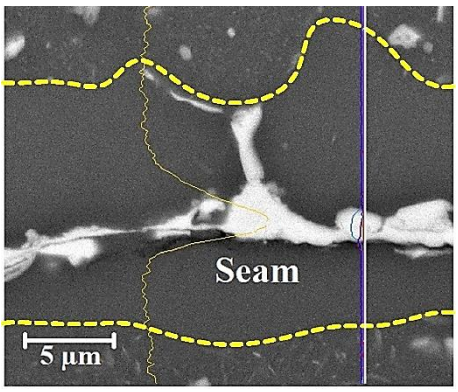

Figure 12 – Electron image of the microstructure of the brazed joint

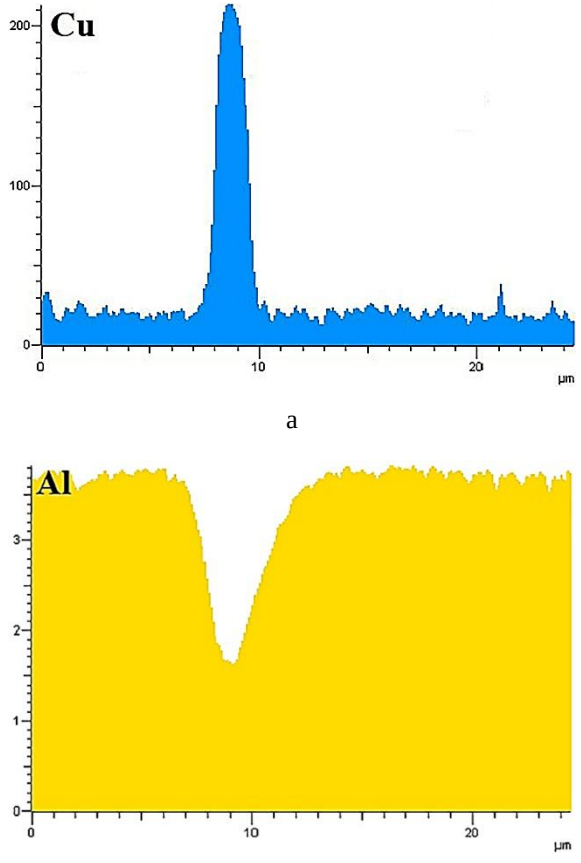

Figure 13 – Qualitative distribution of Cu (a) and Al (b) in the brazed joint

The geometric and temperature-time parameters of the vacuum brazing process determine these features in the formation of brazed joints. Upon heating, the brazing alloy melts wets the solid base metal, and interacts with it.

5 Discussion

The obtained results promote mutual diffusion processes and lead to the complete decomposition of the structural components [31], which are inherent in the initial state of the brazing filler metal (resulting from rapid liquid-state solidification). Upon further cooling, the brazing alloy crystallizes under non-equilibrium conditions and in the presence of concentration gradients (of copper and other alloying elements of the brazing alloy and base metal).

As a result of diffusion processes [32], a microstructure forms in the brazed joint, which differs in morphological structure and chemical composition from the initial state of the brazing alloy.

The results of mechanical testing of plate specimens of aluminum alloy 3003 obtained using vacuum brazing and the Al-Cu-Si-(1.5)Mg filler metal showed that the shear strength of the brazed joints is at the level of 0.6–0.7 of the strength of the base material. Fracture of the brazed specimens occurs at the brazed joint.

When testing the brazed joints under three-point bending, an angle close to 180° was obtained (Figure 9 b).

These test results indicate the high quality and reliability of the brazed joints obtained by vacuum high-temperature brazing of aluminum alloy 3003 and indicate the promising use of Al-Cu-Si-(1.5)Mg experimental brazing filler metal.

Notably, creating brazing filler metals with a reduced melting temperature is an urgent task today and is particularly important for brazing thin-walled structures from aluminum alloys. Since the high brazing temperature harms the geometric parameters of aluminum products, it leads to stress and deformation of the thin-walled base metal.

6 Conclusions

The formation of joints in aluminum alloy 3003 obtained through vacuum high-temperature brazing by the Al-Cu-Si-Mg filler metal with reduced brazing temperature was studied. Empirically, it has been proven that during the flow of the brazing alloy containing an elevated concentration of magnesium (26 %), active dissolution of the base metal occurs, accompanied by intense evaporation, resulting in the formation of pores, undercuts, and shrinkage cavities.

It has been shown that the solidus and liquidus temperatures of the Al-Cu-Si-1.5Mg brazing filler metal obtained by calculation and experimental methods practically coincide.

Micro-X-ray spectroscopic analysis revealed that during the brazing of aluminum alloy 3003 with the microcrystalline Al-Cu-Si-1.5Mg brazing filler metal, the constituent phases of the brazing alloy dissolved and a crystalized structure of the brazed joint is formed, which significantly differs from the structure of the initial brazing alloy. A solid solution structure based on aluminum is observed in narrow regions of the brazed joint. The morphological structure of the base metal remains unchanged and retains its original structure.

The shear strength of the brazed joints is at the level of 0.6–0.7 of the strength of the base material, and fracture of the brazed specimens occurs at the brazed joint. Testing the brazed joints under three-point bending resulted in an angle close to 180°.

Thus, the research results show the expediency of using the experimental Al-Cu-Si-(1.5)Mg brazing filler metal for vacuum brazing of the 3003 series aluminum alloy.

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