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M. HEBDA*#, P. KACZOR**, K. MIERNIK*

VACUUM BRAZING OF STAINLESS STEEL DEPENDING ON THE SURFACE PREPARATION METHOD AND TEMPERATURE OF THE PROCESS

This paper discusses issues related to optimising the technological parameters of the process of brazing gold in a vacuum furnace. An investigation of the brazing process was carried out for materials used in constructing components for aircraft engine fuel systems. The vacuum brazed material was AMS 5510 stainless steel (in the form of plates and pipes). AMS 4787 (BAu-4) was used as the brazing filler. In particular, the influence of the method of preparing the surface on solder spreading and the thickness of the diffusion zone were analysed. The best spreading of solder was obtained for nickel plated surfaces. When the sample surface was more rough or scratched, the effect of the spreading of solder was limited and the diffusion process of the solder into the base material became dominant. Moreover, the influence of the brazing temperature on microstructure changes and on interdiffusion of the AMS 5510 stainless steel/BAu-4 solder system was determined. It was observed that an increase in the brazing temperature modifies the morphology of the formed joint by forming a massive and rounded phase. Furthermore, an increase in the brazing temperature enhances the exchange of components.

Keywords: Vacuum brazing; Stainless steel; Surface preparation; Brazing temperature

1. Introduction

The development of industry and its branches, such as aviation and astronautics, has resulted in a demand for new materials possessing specific properties. The result, in turn, of introducing new, previously unknown materials has been the need to develop methods of joints which will allow to make connections that have the required quality and properties. One of the methods that has in recent years played an important role in the production of various elements is soldering. Soldering is a process in which two or more metal items are joined together by melting and flowing a filler metal (solder) into the joint. The filler metal has a lower melting point than the adjoining metal and is distributed between the fitting parts by capillary action. The brazing process takes place if the melting point of the filler metal exceeds 450 °C. Brazing is one of the most popular joining methods, particularly in case of thin-walled elements. This method, known since ancient times, currently allows to generate a highly complex design and combination of materials with differing properties and chemical composition. For instance, Zaharinie et al. [1] investigated brazing vacuum process of sapphire to Inconel 600 for gas pressure sensor application. Prakash et al. [2] studied the ceramic-metal brazed joints (SiC- to stainless steels and to Inconel 718). Srinivasan et al. [3] optimised process conditions for high-temperature vacuum brazing of Inconel 600 sleeves to 316L stainless steel cable sheath. Wojarski and Tillmann [4] analysed dissimilar aluminium-steel joints brazed in a vacuum. Furthermore, thus brazing is possible to obtained also high purity alumina joints suitable for application in rapid cycle proton synchrotron [5]. As a result, the brazing process is widely used in various industries. Through a selection of appropriate methods of soldering, process parameters and the geometry of solder joints, it is possible to make permanent and reliable connections between bonding machine parts. The most popular method used in industry is brazing in vacuum furnaces. Other possibilities of soldering are for example a laser soldering process investigated by Nishikawa and Iwata [6], laser brazing technique analysed by Saida [7] or an ultrasonic soldering studied by Wei et al. [8]. Brazing has many advantages over other metal-joining techniques, such as welding, e.g. it (i) receive extremely clean joints without the need for secondary finishing, (ii) is superior and (iii) produces less thermal distortion than welding due to uniform heating of the brazed piece. Moreover, brazing is simple to perform (easily adaptable and automated for mass production) and cost effective, which in times of economic crisis is of great importance. Furthermore, a properly conducted process of brazing guarantees high strength, tightness and corrosion-resistant connections. Thus, this technique is used for the production of aircraft engine components, such as turbines, fuel systems or hydraulic lines as well as in the construction of car bodies or structural components operating at high temperatures. The research of Wu et al. [9] showed that vacuum brazing of Ni-NiCr

^{*} CRACOW UNIVERSITY OF TECHNOLOGY, INSTITUTE OF MATERIALS ENGINEERING, 24 WARSZAWSKA STR., 31-155 KRAKÓW, POLAND ** PRATT & WHITNEY TUBES SP. Z O.O., 4 GRABSKA STR., 32-005 NIEPOŁOMICE, POLAND

[#] Corresponding author: mhebda@pk.edu.pl

laminated composite to Cr18-Ni8 steel is an attractive candidate for the modern aero-engines material to improve the thrust to weight ratio. Honggang [10] studied the vacuum brazing of TiAl alloy to 40Cr steel for application in manufacturing automotive engine (to reduce the weight and production costs of vehicles). Soltani Tashi et al. [11] investigated diffusion brazing of titanium alloy and stainless steel as a material used in the aerospace and shipbuilding industries. Nelson [12] analysed braze process for the repair of damaged turbine components made of nickel-based superalloys.

Surface preparation for the soldering process is an important procedure in order to achieve joints with suitable properties. The choice of surface purification methods depends not only on the shapes and sizes of the components, but also on the type of material from which they were made. For steel and cast iron a technique that is often used is grinding and sandblasting or shot blasting. However, these processes cause micro-inequalities to form, and these have a significant impact on the spreading of solder, hence on the quality and strength of the connections that are made. For instance, Sanchez et al. [13] studied the most suitable parameters (inter alia surface preparation, thickness of Ni plating and joint clearance) for carrying out high quality Cu/Cu and Cu/SS joints performed by vacuum brazing. Nowacki et al. [14] investigated the influence of brazing parameters (roughness of the surfaces to be joined and the distance between them as well as duration of the brazing) on microstructure and properties of vacuum brazed 14-5PH steel. Mirski and Piwowarczyk [15] discussed mechanical and chemical methods of preparing a hardmetal surface for brazing.

The results presented in this paper focus on optimising the technological parameters of the process of brazing gold in a vacuum furnace. In particular, the influence of the roughness of the surfaces on solder spreading and the thickness of the diffusion zone were analysed. Moreover, the influence of the brazing temperature on microstructure changes and on interdiffusion of the AMS 5510 steel/BAu-4 solder system was determined.

2. Materials and Methods

An investigation of the brazing process was carried out for materials used in constructing components for aircraft engine fuel systems. The vacuum brazed material was AMS 5510 stainless steel (in the form of plates and pipes) with a chemical composition as shown in Table 1. AMS 4787 (BAu-4) was used as the brazing filler with a chemical composition as shown in Table 2.

Chemical composition of AMS 5510 stainless steel

TABLE 1

Desig-	Chemical composition wt. [%]									
nation	C	Si	Mn	Р	S	Cr	Ni	Ti	Ν	Fe
AMS	0.08	0.25-	2.0	0.04	0.02	17.0-	9.0-	0.7	0.1	Dal
5510	max	1.00	max	0.04	0.05	19.0	12.0	max	max	Dai

Chemical composition of brazing filler AMS 4787 (BAu-4)

Designation	Chemical composition wt. [%]					
Designation	Au	Ni	Total impurities			
AMS 4787	82 ± 0.5	Bal.	0.15 max			

The method of surface preparation of the connected elements has a significant impact on the quality of the brazed joint. Therefore, in order to optimise the technological parameters of the brazing process, studies were conducted on the process of the spreading of braze and on the thickness of the diffusion layer obtained according to the method of preparing the surface of the material (AMS 5510).

This investigation was carried out on six samples in the form of sheets with dimensions of 7×12.5 cm and a thickness of 3 mm. The surfaces of the samples were subjected to various surface treatments as described in Table 3. An equal amount of solder (34.50 ±0.15 mg) for each experiment, was applied on the sheet surface prepared as in Table 3. A braze filler in the form of wire was combined with the stainless steel sheets using a spot welding Telwin Digital Spotter 5500. Next the samples were brazed in a vacuum furnace with a graphite chamber (Seco Wariwck) at 1046°C. The temperature profile of the process is presented in Figure 1.

TABLE 3

Designation of samples depending on type of surface treatments performed

Designation of samples	Type of surface treatments performed				
S1	surface after the nickel plating process, then cleaned with acetone				
S2	surface after grinding with abrasive paper with gradation of 120, then cleaned with acetone				
S3	surface after grinding with abrasive paper with gradation of 240, then cleaned with acetone				
S4	surface after grinding with abrasive paper with gradation of 400, then cleaned with acetone				
S5	surface scratched with a sharp tool, then cleaned with acetone				
S6	surface in the state of delivery (no cleaning or polishing procedures)				

Next the samples were prepared according to Figure 2 in order to perform the metallographic observation. Samples were etched using Kalling's reagent (2g CuCl₂, 40mL HCl, 40-80mL ethanol (95%)). Microscopic observation was performed using a Nikon optical microscope, model Eclipse LV150. Measurements of the diffusion layer's thickness were analysed using a Buehler OmniMet Modular imaging system. Roughness measurement was performed using the Surftest SJ-301 device.

For the optimised procedure for surface preparation of materials to be joined (nickel plated surfaces) we also studied the effect of the soldering temperature that was applied (1002°C, 1046°C and 1066°C) on the quality of the diffusion connection



Fig. 1. Temperature profile used to investigate the process of spreading and the thickness of diffusion of solder depending on the method of surface preparation of the joined materials. Stage I – Preparation of the vacuum (2×10^{-3} Tr) inside the furnace chamber. Stage II – Heating at 17°C/min. Stage III – Annealing at 770°C for 40 min (for oxide reduction from the surface of the samples). Stage IV – Heating at 17°C/min. Stage V – Annealing at 1046°C (proper soldering process). Stage VI – Cooling to ambient temperature



Fig. 2. Scheme of sample preparation for the metallographic observation (investigating the spreading of braze and the diffusion layer's thickness): a) perspective view, b) cross-section



Fig. 3 View of individual elements undergoing the process of assembly: a) tube whose tip is coated with a layer of nickel, b) brazing filler ring, c) element together with the inserted brazing filler ring, d) sample after assembly

of the braze-base material. Measurements were carried out for samples in the form of tubes having a diameter of 25.4 mm. The method of connecting elements for brazing is presented in Fig. 3. The solder in the form of a wire ring (Fig. 3b) with equal amount (68.10 \pm 0.15 mg) for each experiment, was placed in the detail (Fig. 3c) to be connected to the tube (Fig. 3a). Fig. 3d shows the sample after assembly. The test specimens were collected after the brazing process according to the scheme presented in Fig. 4. Evaluation after brazing consisted of SEM observation as well as EDS analysis of the braze cross-section (using a Jeol JSM-5510LV Scanning Electron Microscope (SEM) with an energy dispersive spectrometer (EDS) IXRF Systems Model 500 Digital Processing).



Fig. 4 Scheme of sample preparation to study the effect of temperature on the quality of the diffusion connection of braze-base material: a) perspective view, b) cross-section

3. Results and discussion

3.1. Measurement of the spreading of braze filler and the diffusion layer's thickness depending on the method of surface preparation

Before proceeding to the study of the spreading of braze filler, surface roughness measurement was performed (depending on the type of surface treatments that were done as described in Table 3). The obtained results are presented in Table 4. The area of spreading, was adopted as a measure of spreading. Fig. 5 presents a view of the area of spreading of braze filler on the stainless steel surface depending on the surface preparation method (described in Table 3). The results of braze filler

TABLE 4

Surface roughness depending on the type of surface treatment as described in Table 3

Designation of samples	Surface roughness Ra [µm]
S1	0.43
S2	0.39
S3	0.31
S4	0.24
S5	0.68
S6	0.43

Braze filler spreading measured in perpendicular directions (designated as X and Y) as well as the area covered by the brazed filler depending
on the type of surface treatment described in the Table 3

Designation of samples	Braze filler spreading measured in x-direction [mm]	Braze filler spreading measured in y-direction [mm]	Surface area covered by braze filler [mm ²]
S1	14.42 ± 0.61	13.91 ±0.27	157.56 ±6.71
S2	11.67 ±0.27	10.98 ±0.04	100.79 ±3.47
S3	12.06 ±0.13	11.45 ±0.17	108.66 ± 3.35
S4	12.43 ±0.18	11.40 ±0.13	111.77 ±3.67
S5	12.39 ±0.23	11.44 ±0.19	111.74 ±4.83
S6	12.67 ±0.26	11.51 ±0.05	115.12 ±3.81



Fig. 5. View of the spreading of braze filler on the stainless steel surface depending on the surface preparation method: 1) surface after the nickel plating process, 2) surface after grinding with abrasive paper with gradation of 120, 3) surface after grinding with abrasive paper with gradation of 240, 4) surface after grinding with abrasive paper with gradation of 400, 5) surface scratched with a sharp tool, 6) surface in the state of delivery

spreading measured in perpendicular directions (designated as X and Y) as well as the area covered by the brazed filler are summarised and presented in Table 5. It was found, based on the obtained results, that the greatest braze filler spreading was recorded for the sample marked S1, whose surface before the brazing process was coated with nickel. The smallest of the surface areas covered by braze filler was measured for sample S2 (after grinding with abrasive paper with gradation of 120). It was about 33% lower than for sample S1. Furthermore, it was observed that there were no pits on the brazing filler only for the sample whose surface was nickel plated before measurement (S1). For all samples the thickness was measured of the diffusion layer of the braze filler into the base material. The obtained results are shown in Figure 6. The largest thickness of the diffusion layer was obtained for sample S2 (after grinding



Fig. 6. Results of the thickness of the diffusion layer of the braze filler into the base material depending on the method of surface preparation (sample designation in accordance with Table 3)

with abrasive paper with gradation of 120). The thickness of the diffusion layer was almost ten times higher than for sample S1 after the nickel plated process.

On the basis of all the obtained results it was found that the method of preparing the surface of the connected elements had a huge impact on the spreading of braze filler. The ability of braze filler to spread on the surface of the samples that were scratched or ground with abrasive paper is lower than for the surface after the nickel plated process. This effect is mainly the result of the micro-roughness occurring in the surface layer of the material, which hinders the spread of the liquid solder. Moreover, for surfaces of samples polished with variable gradation of abrasive paper it was observed that the spreading of braze filler increased with a decreasing surface roughness. Furthermore, it was observed that covering the surface of the nickel layer helped the spreading of the braze filler by reducing surface tension at the interface between the bonded parts. It can be also assumed, that a better wettability occur because this phenomenon is related to spreading as well as to surface properties. This effect is highly beneficial since it guarantees filling in the braze filler throughout the soldering gap and thereby obtaining a permanent and tight connection. In addition, there were no negative effects (burnings, pitting, etc.) occurring on the surface of the braze filler only for samples with a nickel plated surface (S1). These results are consistent with studies obtained by [16,17] who report the best spreading, and thus the wettability of the surface after nickel-plating operation. Moreover, it is also important that nickel layer protects the stainless steel surface from oxidation [17].

The process of preparing the surface of the joined materials significantly affects the diffusion of braze filler into the base material. All scratches and roughness formed on the surfaces of the samples are special places of penetration for the molten solder. They intensify the diffusion process of the braze filler components into the deeper layers of the base material. The sample, after grinding its surface with abrasive paper with gradation of 120, was characterised by the smallest spreading of braze filler and simultaneously by the furthest diffusion of braze filler into the base material.

Skipping the technological procedures, such as cleaning, grinding or nickel plating, in order to properly prepare the surface for brazing does not preclude carrying out the brazing process. However, not performing these procedures limits the spreading of braze filler and thereby lowers the properties of the obtained connection.

3.2. Characteristics of the brazing joint depending on the brazing temperature

During brazing several processes take place: interdiffusion between the base material and solder, Au–Ni solid solution decomposition and the precipitation process during ageing. These processes affect the distribution of elements, structure and properties of the joint [14]. The thickness changes of the diffusion layer of the brazing filler into the base material depending on the temperature of the soldering process are shown in Table 6.

TABLE 6

Thickness of the diffusion layer of the brazing filler into the base material depending on the temperature of the soldering process

Designation of samples	1	2	3
Temperature [°C]	1002	1046	1066
Thickness of the diffusion layer of the brazing filler into the base material [µm]	45 ±8	62 ±12	77 ±9

From the results obtained it was found that a higher brazing temperature increases the thickness of the diffusion layer of the solder into the base material. This effect is a natural consequence of the increased mobility of atoms and thereby quickens the diffusion process as the temperature increases.

The microstructure evaluation with the EDS line analysis of the cross-section joints for three different brazing temperatures observed using SEM are presented in Figures 7-9. Measurements of chemical composition microanalysis of Fe, Cr, Ni and Au carried out in the base material and solder (light and dark area) are shown in Table 7.



Fig. 7. SEM microstructure with the EDS analysis (from left, changes of the intensity of Fe, Cr, Ni and Au) of the cross-section brazed joints for a sample brazed at 1002°C

The microanalysis of the chemical composition (Figs 7-9, Table 7) that was performed revealed diffusion of the solder elements (gold) into the base material as well as Fe and Ni from the base material to the solder. Regardless of the brazing temperature, significantly increased levels of Fe, Cr and Ni were found in the darker phase than in the lighter phase of the solder.

Furthermore, it was observed that the brazing temperature change influences a change in the morphology of the formed joint. At a temperature of 1002°C the diffusion processes occurred more slowly than at 1066°C, therefore a phase rich in Fe, Cr and Ni (dark area in Figures. 7-9) was formed on the border of



Fig. 8. SEM microstructure with the EDS analysis (from left, changes of the intensity of Fe, Cr, Ni and Au) of the cross-section brazed joints for a sample brazed at $1046^{\circ}C$



Fig. 9. SEM microstructure with the EDS analysis (from left, changes of the intensity of Fe, Cr, Ni and Au) of the cross-section brazed joints for a sample brazed at 1066°C

the braze filler- base material. It had the shape of small, irregular precipitates that were locally interconnected. The center of the joint was free from the darker phase. This effect depends on the temperature used for brazing. With an increase in the brazing temperature the darker phase occurring in the joint adopted an increasingly massive and rounded character. It became the dominant phase in the joint. At a temperature of 1066°C an area can be observed in the joint in which both sides of the base material are connected by a formed darker phase of the solder.

Based on Figs. 7-9 and EDS analysis results (Table 7) as well as on the binary alloy phase diagram [18] it can be concluded that as a result of the Au–Ni solid solution decomposition the brazed joint is composed of two phases. The bright region is the Au[Ni] solid solution rich in gold, while the dark phase is Ni[Au] solid solution rich in nickel. According to EDS results (Table 7), the chemical compositions of formed gold–nickel solid solutions depends on the applied brazing temperature. Moreover, the diffusion of Fe and Cr from the base material to the Ni[Au] phase has been observed. The obtained results are in full agreement with the analysis reported by Nowacki [19], Liaw [20] and Sun [21].

4. Conclusions

The method of preparing the surface for the brazing process has a significant impact on the spreading of braze filler. The best spreading of solder was obtained for nickel plated surfaces. Moreover, there were no negative effects (burnings, pitting, etc.) occurring on the surface of the braze filler only for samples with a nickel plated surface. When the sample surface was more rough or scratched, the effect of the spreading of solder was limited and the diffusion process of the solder into the base material became dominant.

An increase in the brazing temperature enhances the exchange of components, i.e. the solder and the base material, which increases the thickness of the diffusion layer in the joints.

TABLE 7

Temperature [°C]	Element [wt. %]	Base materials (main elements)	Solder – light area	Solder – dark area
1002	Fe	70.24 ±7.12	12.25 ± 3.03	22.90 ±4.20
	Cr	18.46 ±4.58	3.70 ± 1.83	4.73 ±2.15
1002	Ni	9.01 ±2.11	16.25 ± 3.03	41.15 ±4.66
	Au	_	67.80 ±2.49	31.22 ±1.60
	Fe	71.26 ±7.25	10.75 ±2.76	41.18 ±5.56
1046	Cr	18.68 ±4.67	5.76 ±2.22	10.74 ± 3.34
1046	Ni	8.96 ±2.12	6.80 ± 1.93	27.89 ±3.81
	Au		Solder – light area 12.25 ± 3.03 3.70 ± 1.83 16.25 ± 3.03 67.80 ± 2.49 10.75 ± 2.76 5.76 ± 2.22 6.80 ± 1.93 76.70 ± 2.63 7.52 ± 2.34 4.57 ± 1.99 5.07 ± 1.70 82.85 ± 2.80	20.19 ± 1.29
	Fe	70.61 ±7.15	7.52 ±2.34	38.61 ±5.30
1066	Cr	19.03 ±4.66	4.57 ± 1.99	7.87 ± 2.80
1000	Ni	8.57 ±2.05	5.07 ±1.70	32.69 ±4.04
	Au	_	82.85 ± 2.80	20.83 ±1.28

Analysis of the chemical composition of Fe, Cr, Ni and Au carried out in the base material and solder (light and dark area)

Furthermore, it was observed that an increase in the brazing temperature modifies the morphology of the formed joint by forming a massive and rounded Fe, Ni, Cr-rich phase.

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