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THE DIFFUSIVE BARRIERS IN COPPER WITH AUSTENITIC STEEL JOINTS BRAZED WITH Cu-P-Ag FILLER METAL

BARIERY DYFUZYJNE W LUTOWANIU TWARDYM MIEDZI ZE STALĄ AUSTENITYCZNĄ PRZY UŻYCIU SPOIWA TYPU Cu-P-Ag

This work presents problems resulting from brazing of copper with austenitic steel type 18/9 (18/10). Both metals show considerable differences in physical-chemical and mechanical properties, which demonstrate essential influence on the properties of brazed joints, particularly with the use of filler metals based on Cu-P (+Ag).

Brazed joints of copper with austenitic steel with the use of L-Ag15P (CP 102 according to PN-EN 1044) silver filler metal with phosphorus have been made. Brazing of different kinds of steel as well as nickel and its alloys with this filler metal is forbidden because of creation on fragile reactionary phases at the boundary of brazed joint on steel side (nickel). However, considering cases of application of this group of filler metals on Cu-P (+Ag, Sn) matrix for steel brazing, a diffusive barrier on Cr-Ni (18/10) steel surface should be created, preventing formation of the fragile intermetallic Fe_2P and Ni_2P phases. It has been achieved by putting on a galvanized coat of copper of up to 50 μm thickness. Considering the Cu coat adhesion to steel surface, its surface was developed by chemical etching. Basing on surface roughness tests' results, etching parameters have been selected, suitable for correct joint of copper galvanized layer with steel, from the point of view of mechanical adhesion.

Research results have been presented basing on microscopic observations with the use of light microscopy, electron microscopy and the analysis of chemical elements with EDX method, micro-hardness measurements as well as static shear test of the obtained brazed joints.

Keywords: heteronymous metals, brazing, copper – phosphorous filler metals, diffusive barrier (Ni, Cu), metallographic investigations, micro-hardness, mechanical strength

W pracy przedstawiono problemy wynikające z lutowania twardego miedzi ze stalą austenityczną typu 18/9 (18/10). Obydwa metale wykazują znaczne różnice we właściwościach fizykochemicznych i mechanicznych, które mają istotny wpływ na właściwości złączy lutowanych wykonanych szczególnie przy użyciu spoiw na bazie Cu-P (+Ag).

Wykonano połączenia lutowane miedzi ze stalą austenityczną z użyciem lutu srebrnego z fosforem gat. L-Ag15P (CP 102 wg PN-EN 1044). Lutowanie tym lutem różnorodnych stali oraz niklu i jego stopów jest zabronione z uwagi na tworzenie się kruchych faz reakcyjnych na granicy połączenia lutowanego od strony stali (niklu). Z uwagi jednak na przypadki stosowania tej grupy lutów na osnowie Cu-P (+Ag, Sn) do lutowania stali należy stworzyć barierę dyfuzyjną na powierzchni stali Cr-Ni (18/10), uniemożliwiającą tworzenie się kruchych faz międzymetalicznych typu Fe_2P i Ni_2P. Zrealizowano to przez nałożenie powłoki galwanicznej z miedzi o grubości dochodzącej do 50 μm. Mając na uwadze przyczepność powłoki Cu do powierzchni stali rozwinięto jej powierzchnię przez trawienie chemiczne. Na podstawie wyników chropowatości powierzchni, wybrano parametry trawienia, właściwe dla poprawnego połączenia warstwy galwanicznej miedzi ze stalą z punktu widzenia adhezji mechanicznej.

Wyniki badań przedstawiono na podstawie obserwacji mikroskopowych za pomocą mikroskopii świetlnej, mikroskopii elektronowej i analizy pierwiastków metodą EDX, pomiarów mikrotwardości oraz statycznej próby ścinania wykonanych połączeń lutowanych.

1. Introduction

The joining of copper with Cr-Ni 18/9 (18/10) with austenitic structure are applied e.g. in coolers and cooling devices construction as well as in brazing of stator windings of high power output generators [8,11,13]. The stator winding consists of a packet of current-generating conductors made of oxygen-free copper, between which there are tubes of 1H18N9T acid-resistant steel with flowing distilled water for copper winding cooling. The

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brazed joints of copper conductors' packet with tubes of 18/9 steel in copper ferrule is carried out by brazing with the use of L-Ag15P filler metals without fluxing agent. This filler metal allows for brazing of the whole packet without fluxing agent, with no need to apply the additional flux and thus, no necessity of ex-fluxing slag removal. However, Cr-Ni steel is also joined in copper brazing process, which is very undesirable due to fragility of Cu -18/9 connection.

A great difference of thermal conductivity as well as oxides of various melting temperatures and resistance present on their surfaces are of essential meaning in brazing of pair Cu-18/10 metals. However, the similarity in properties can be seen in linear expansibility coefficients of both metals, and this fact results in reduction of the risk of significant strains and cracks formation during cooling down of the joints [4].

2. Properties of the joined metals

Basic physical-chemical and mechanical properties of copper and austenitic steel of X6CrNiTi18-10 symbol (number 1.4541) according to PN-EN 10088-1:1988 standard, being equivalent to 1H18N10T steel, according to PN-71/H-86020 standard, have been presented in Table 1.

TABLE 1

Basic physicochemical and mechanical properties of copper and 1H18N10T austenitic steel [5-10]

Properties	Copper	1H18N10T steel
Melting point, T_t, °C	1083	1450
Boiling point, T_w, °C	2595	–
Density ρ, kg/m^3	$8.9 \cdot 10^3$	$7.9 \cdot 10^3$
Linear expansion α coefficient at the temperature of 20°C, 1/K	$17.0 \cdot 10^{-6}$	$18.0 \cdot 10^{-6}$
Specific thermal conductance *lambda* at the temperature of 0°C, W/m·K	397	14.6
Yield strength $R_{0.2}$, MPa	60	205
Tensile strength R_m, MPa	240	500-750
Shear strength R_t, MPa	160	–
Relative elongation A_{10}, %	45	40
Hardness, *HB*	50	130-190
Longitudinal elasticity module at the temperature of 20°C, GPa	119	200

3. Brazing of copper with 1H18N10T austenitic steel

It is possible to carry out the brazing process of copper with austenitic steels using Ag-Cu-Zn silver filler metals, under the fluxing agent protection. Filler metals with addition of tin, gallium or cadmium, decreasing their melting temperature, might also be used [11]. The greatest influence of cadmium consists in decrease of melting temperature of silver filler metals, however it is more and more often withdrawn from their production process due to poisonous and carcinogenic activity. The example of silver, non-cadmium filler metals are filler metals of AG 102 symbol, according to PN-EN 1044 (L-Ag55Sn, according to DIN 8513) and AG 104 (L-Ag45Sn, according to DIN 8513) [2,11-13]. Chemical composition of the most frequently used silver filler metals has been presented in Table 2.

TABLE 2

Chemical composition and the melting temperature of silver filler metals to joining of copper with austenitic steel [2,3,11-13]

No	Symbol of filler metal according to PN-EN 1044	Symbol of filler metal according to DIN 8513 (PN)	Chemical composition, percentage by weight %				Melting point, °C
			Ag	Cu	Zn	Sn	
1.	AG 203	L-Ag44 (LS45)	44	30	26	–	675 – 735
2.	AG 102	L-Ag55Sn	56	22	17	5 Sn	620 – 655
3.	AG 104	L-Ag45Sn	45	27	25.5	2.5 Sn	640 – 680
4.	AG 105	L-Ag40Sn	40	30	28	2 Sn	650 – 710
5.	AG 106	L-Ag34Sn	34	36	27.5	2.5 Sn	630 – 730

In case of brazing of Cr-Ni acid-resistant steels with silver filler metals including zinc, the danger of knife-line attack exists [1]. Contrary to filler metals including tin, the filler metal of AG 203 (LS45) symbol is particularly suitable for brazing of elements operating under difficult conditions, with dynamic loads [12].

Brazed joint of copper with 18/9 austenitic steel made with AG 102 (L-Ag55Sn) filler metal in fluxing covering, has been showed in Figure 1. The structure of braze consists of eutectic mixture and the originally crystallized crystals of dendritic structure [2,3].

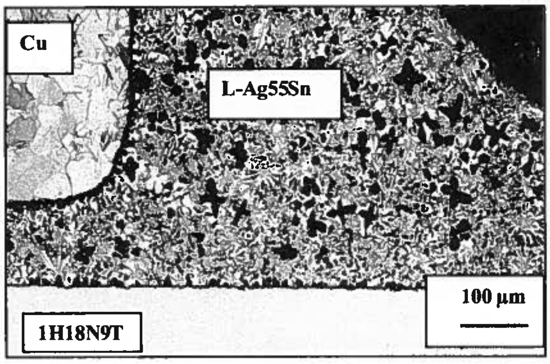


Fig. 1. Cu-1H18N9T brazed joint, carried out with AG 102 filler metal in fluxed cleading, chemically etched by Mi17Cu agent

The use of Cu-P filler metals for Cr-Ni steels containing iron and nickel is absolutely undesirable. Even a small content of phosphorus (within 0,05-0,3 percentage by weight range) in L-Ag40Cd silver filler metal causes formation of 2 µm-width reaction zone. It should be stressed, that the reaction zone containing iron phosphides Fe_2P does not appear directly on steel boundary, but at the distance of several um from St 60 steel, in the braze (Fig.2) [14]. Fragile intermetallic phases of Fe_2P and Ni_2P demonstrate limited solubility in copper [1-3,15-16]. Maximum solubility of iron phosphide in copper is about 1.5 percentage by weight [1,17].

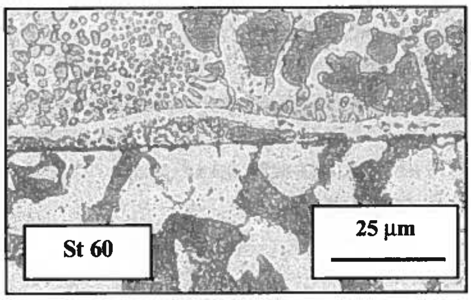


Fig. 2. Reaction zone with iron phosphides in L-Ag40Cd + 0,3% P brazed joint [14]

Brazed joints with steel using filler metal containing phosphorus are very fragile. Even a light impact or small deformation cause decohesion of the joint.

The L-Ag15P (according to DIN 8513) filler metal widely applied in industrial practice is presently known as CP 102 filler metal, according to valid PN-EN 1044 standard, including 5% P, 15% Ag and the rest Cu (percentage by weight). Its melting point is 645-800°C. It allows for obtaining the largest plasticity and fatigue strength in Cu-Cu brazed joints [18]. The possibility of moistening and brazing of copper without the use of fluxing agent constitutes one of basic properties of this filler metal. This is possible due the following chemical reaction:

$$12Cu_2O + P_4 = 2Cu(PO_3)_2 + 22Cu \quad (1)$$

Copper phosphates form as a result of reduction of copper oxides, forming a grey coat on the surface of braze. Due to lack of corrosive threat, there is no necessity to remove this thin coat from the joint surface [3,15].

The results of reaction of L-Ag15P filler metal with 1H18N9T steel have been shown in figures 3a and 3b. The reaction zone between melted filler metal and austenitic steel as well as lack of wettability of filler metal are very distinct (Fig.3a) [1-3].

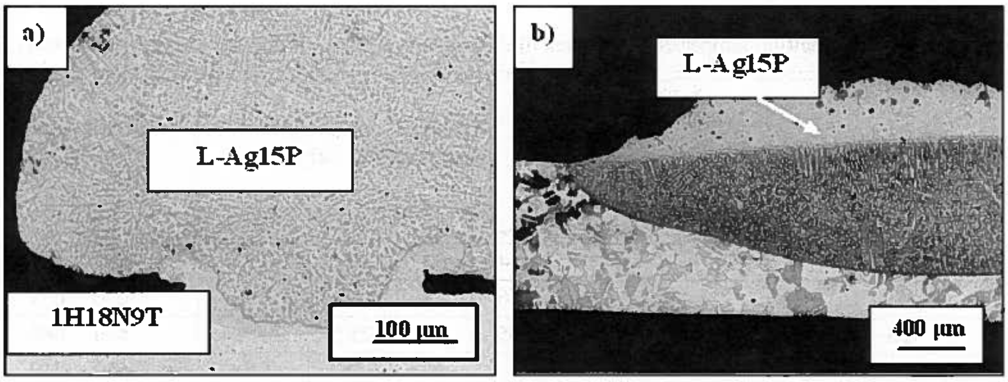


Fig. 3. The layer of melted L-Ag15P filler metal on a tube from 1H18N9T steel: a – in treated state, b – chemically etched by V2A agent

The coats protecting against formation of the fragile intermetallic phases are recommended in case of necessity of brazing copper with austenitic steel using filler metals with addition of phosphorus. Coating of steel surface with the suitable thickness layer not containing iron and nickel, will ensure creation of good brazed joint, not leading to its fragility [3].

4. Research

The research consisting in application of diffusive barriers as galvanized coats spread on steel 18/10 surface has been carried out at Welding Technology Division of the Institute of Production Engineering and Automation of Wroclaw University of Technology. A necessary thickness of galvanized coats, spread in order to avoid formation of fragile intermetallic phases in brazed joints of cuprum with austenitic steel 1H18N10T using copper-phosphorous filler metal L-Ag 15P, has been estimated.

4.1. Preparation of 1H18N10T steel surface for spreading of galvanized coats

Metal plates of 2 mm thickness of Cu 99,9E (M1E) (according to PN-77/H-82120) cathode melted copper as well as X6CrNiTi18-10 (according to PN-EN 10088-1:1998) or 1H18N10T (according to PN-71/H-86020) chromium-nickel steel have been taken to make the joints. L-Ag15P filler metal of 0.2 mm thickness in the form of strip has been used.

The tests of Cu – steel 18/10 joints have been carried out for various methods of surface preparation, distance elements application and thickness of the applied galvanized coats of nickel and copper.

The preparation of steel surface in its as-cast condition consisted in cleaning with abrasive paper (160), degreasing and then washing in ultrasonic bath. However, this way of preparation of the surface has not ensured good joint between galvanized layer and steel surface. The authors decided to increase the roughness of steel surface by chemical etching in order to improve adhesion of galvanized layers. Steel etching has been carried out in the water solution of hydrochloric and nitric acids of the following composition: 40 ml of HCl, 40 ml of HNO_3 and 80 ml of distilled water. The samples have been prepared in the ULTRON ultrasonic bath for 5 minutes after the etching process.

The results of measurements of roughness have been shown in Table 3. The measurements have been carried out with the use of profilograph manufactured by Taylor Hobson.

TABLE 3

Values of Ra, Rz and Rt roughness parameters for different ways of 1H18N10T steel surface preparation

Method of surface preparation	Ra*, μm	Rz**, μm	Rt***, μm
Raw state (without etching)	0.1827	2.0517	3.1020
Ground with abrasive paper (160)	0.5451	4.5564	7.6861
Etching 1.5 min	0.2776	2.1773	5.0319
Etching 3 min	0.5073	3.3269	6.4781
Etching 5 min	0.7198	5.8195	7.9596
Etching 7.5 min	1.0670	8.3488	11.6028
Etching 10 min	4.5951	25.3389	31.0936
Etching 12.5 min	4.7805	30.8680	39.7372
* Ra – mean arithmetical deviation of profile from mean line			
** Rz – height of roughness according to ten points of profile			
*** Rt – maximum roughness height			
ATTENTION ! new symbols according to PN-EN ISO 4287:1999			

It has been established while analyzing measurements of steel sheet surface roughness, that the best values of roughness have been obtained after 10 minutes of etching. The protective coats on the steel surface were spread following etching, ultrasonic bath washing and drying of the samples.

The authors decided to apply copper as the layer constituting the diffusive barrier on the surface of steel. The steel was covered with a layer of nickel in order to activate stainless steel surface, which is easily becomes passive under the influence of air [19].

During nickel plating and the copper plating processes, platinum wire of 0.5 mm diameter, coiled in spring and connected to positive pole of current feeder constituted anode. A sample of 1H18N10T steel of 2 mm thickness and 20×20 mm active surface constituted cathode. The distance between anode and cathode was about 50 mm. The electrolyte has been mixed with magnetic stirrer. Galvanized layers have been put on at room temperature.

Nickel-plating of 1H18N10T steel surface has been carried out in Watts bath [20], whose chemical composition and operating parameters of have been presented in Table 4. The nickel coat has been of up to of 5 μm thickness.

TABLE 4

Chemical composition and parameters of work of Watts nickel-plating bath, surface of nickel-plating – 400 mm^2

Bath composition for nickel plating	Amount	Operating bath parameters			
		Current strength, A	Voltage, V	Time of coating, min	Mixing
Distilled water H_2O, ml	500				
Nickel sulfate $NiSO_4 \cdot 7H_2O$, g	125				
Nickel chloride $NiCl_2 cdot 6H_2O$, g	10	0.3	4.0	12	no
Boric acid H_3BO_3, g	12.5				

Sulfate bath has been used for application of mat copper coat [20] and its chemical composition as well as operating parameters have been presented in Table 5. Initially, the copper coat was of 30 μm thickness. Later, because of the copper coat dilution during brazing process, its thickness was increased to 50 μm. Table 6 presents the times of copper coats spreading.

TABLE 5

Chemical composition and parameters of sulphate bath to spreading mat copper coats, surface of coating with copper – 400 mm^2

Bath composition for copper plating	Amount	Bath work parameters		
		Current strength, A	Voltage, V	Mixing
Distilled water H_2O, ml Copper sulfate (II) $CuSO_4 \cdot 5H_2O$, g Sulfuric acid (VI) H_2SO_4, ml	500 125 15	0.7-0.9	2.8-3.0	yes

TABLE 6

Time of coating of copper coat (in min)

Coat thickness, μm	Current strength	
	0.7 A	0.9 A
30	25	21
50	32	26

4.2. C_2H_2 – O_2 flame bazig

The Cu-1H18N10T samples with the layer of Cu (+ Ni) and lap width of 12.5 mm have been prepared for brazing process. The oxy-acetylene blowpipe with 3A tip, without an additional flux has been used.

We have also carried out the Cu-1H18N10T joints without nickel-copper layer covering of steel surface for comparison purposes. In case of this brazing method of copper with Cr-Ni steel, we have used AG 500 flux based on hydroxyfluorineborate lithium-potassium compound from FH10 group, according to PN-EN 1045, active within 550-800°C temperature range [13].

4.3. Metallographic research

The metallographic research has been carried out with the use of Olympus CA25 light-microscope, coupled with CAMEDIA C 3030 digital camera and the system of image storing. Evaluation of the galvanized layers on steel surface has been conducted with Phillips SEM 515 scanning electron microscope as well as type 9800, X-ray microanalyzer (Edax, USA).

It appears from the conducted observations that in case of Cu-1H18N10T steel brazed joint without the galvanized coat, the filler metal has not wet the steel surface sufficiently and, as a result, no correct joint has been obtained (Fig. 4).

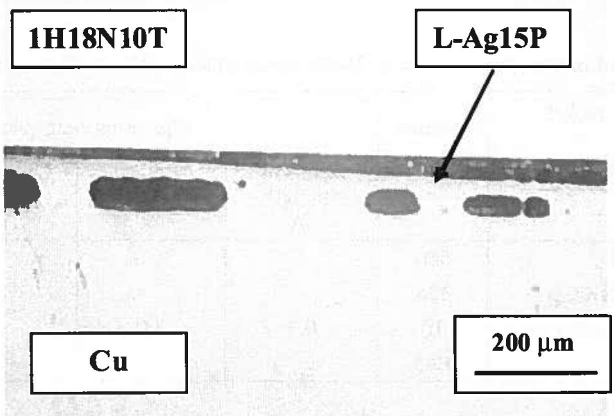


Fig. 4. Cu-1H18N10T brazed connection with copper coat (thickness 30 μm), carried out with L-Ag15P silver filler metal, non-treated state

Vanadium distance elements of 0.1 mm thickness have been applied in brazing tests in order to ensure a proper brazing gap.

The microstructure of Cu (M1E)-steel (1H18N10T) brazed joint made of L-Ag15P filler metal, with visible nickel layer of 5 μm thickness and copper layer of 25-30 μm thickness has been shown in Figure 5a.

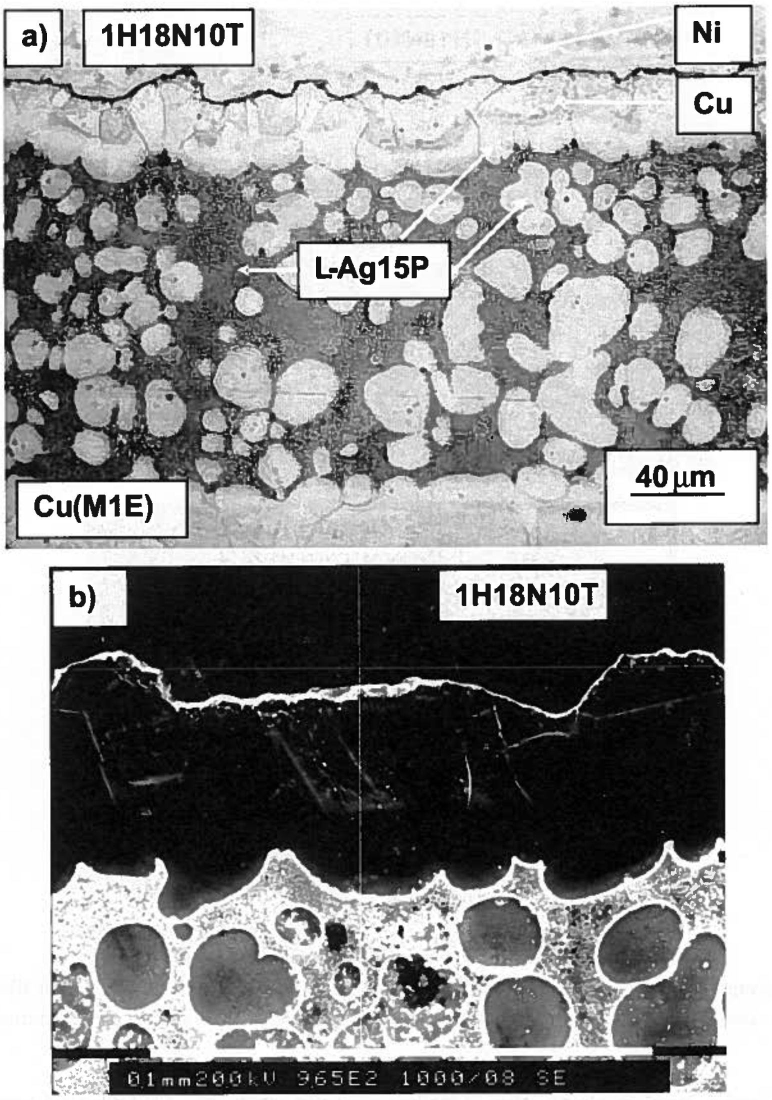


Fig. 5. Cu -1H18N10T brazed connection with nickel and copper coat, carried out with L-Ag15P silver filler metal, gap 0,12 mm, electrolytically etched in 10% chromic acid and then chemically etched by Mi18Cu agent, light microscopy (a) and electron microscopy SEM (b)

Nickel layer is poorly visible in figure 5b. The EDX analysis, conducted at the indicated point, demonstrated, apart from nickel, small quantities of chemical elements diffusing from the steel side (percentage by weight): 0.74 % Cr, 2.05 % Fe, and 6.33 % Cu from the copper layer side. A narrow transient zone, resulting from diffusion process during brazing, has formed at the boundary of nickel and copper layer. This zone contains: 22.15 % Ni and 77. 01 % Cu as well as traces of Fe and Cr. A layer of copper with clearly coarse grained structure is visible next from top. The EDX analysis has shown the presence of 99 % Cu, 0.54 % Ni as well as traces of Cr and Fe, but without phosphorus. L-Ag15P braze adheres to the layer of copper. Round dark crystals visible in braze contain 12.86 % P, 20.56 % Ag while the rest is Cu. Bright eutectic, analyzed on 5×8 mm surface shows 13.85 % P, 16.45 % Ag and the rest of Cu.

The layer of copper spread on Cr-Ni steel surface has been shown in Figures 6 a and 6 b. The L-Ag15P filler metal perfectly moistened the layer of copper. A bright narrow transient zone, containing 4.44 % Ni, 10.30 % Cr, 39.32 % Fe as well as 45.78 % Cu and traces of Mn, is visible at the boundary of copper and steel layer (Fig. 6b). A dark zone of L-Ag15P braze, adhering to the copper galvanized layer, demonstrates the presence of 1.49 % P, 5.72 % Ag and over 92 % of Cu.

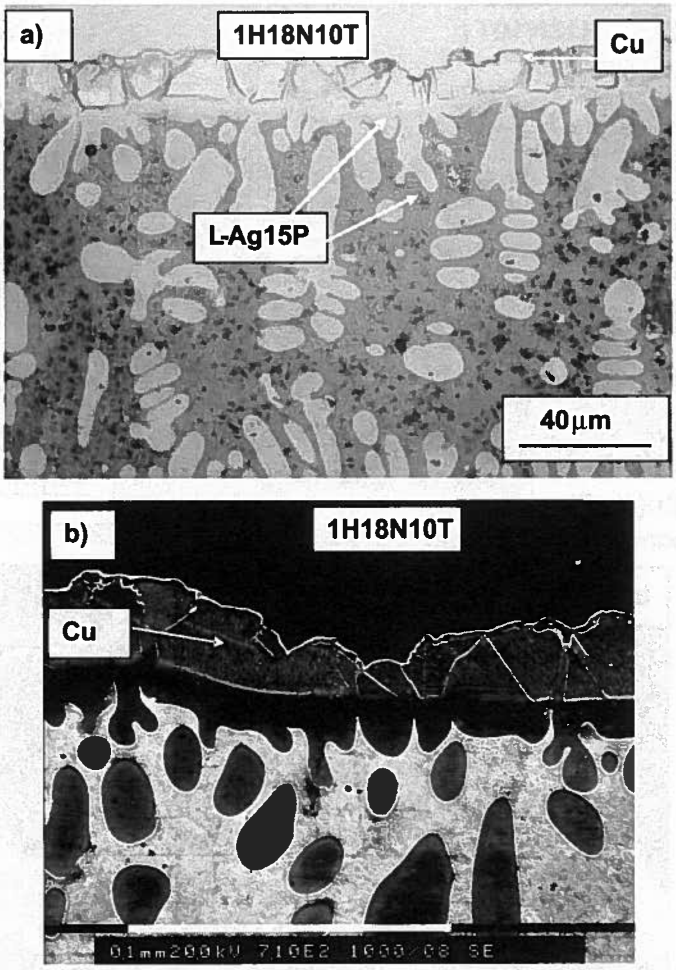


Fig. 6. Cu-1H18N10T brazed connection with copper coat from steel side, carried out with L-Ag15P silver filler metal, electrolytically etched in 10% chromic acid and then chemically etched by Mi18Cu agent, light microscopy (a) and electron microscopy SEM (b)

Because of the developed steel surface, the copper coat demonstrates good joint with steel base. For this reason we have resigned from the primary nickel layer while carrying out subsequent brazed joints for strength tests.

Measurements of micro-hardness (Fig.7) have shown, that the 1H18N10T steel demonstrates the highest hardness of 150 HV 0.050 in the joint, while copper – 43.3 HV 0.050. Average L-Ag15P braze hardness is 110 HV 0.050. The layer of copper spread on steel surface is soft, and its hardness is 56.3 HV 0.050 on the average.

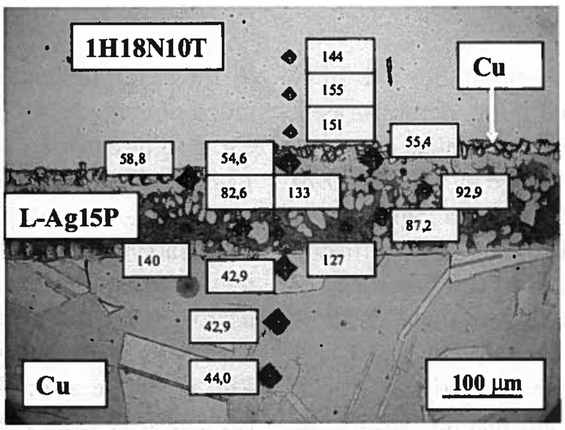


Fig. 7. Microhardness of Cu -(+Cu) 1H18N10T brazed joint by use L-Ag15P filler metal

4.4. Strength tests

The dimensions of samples for static shear test have been presented in Figure 8.

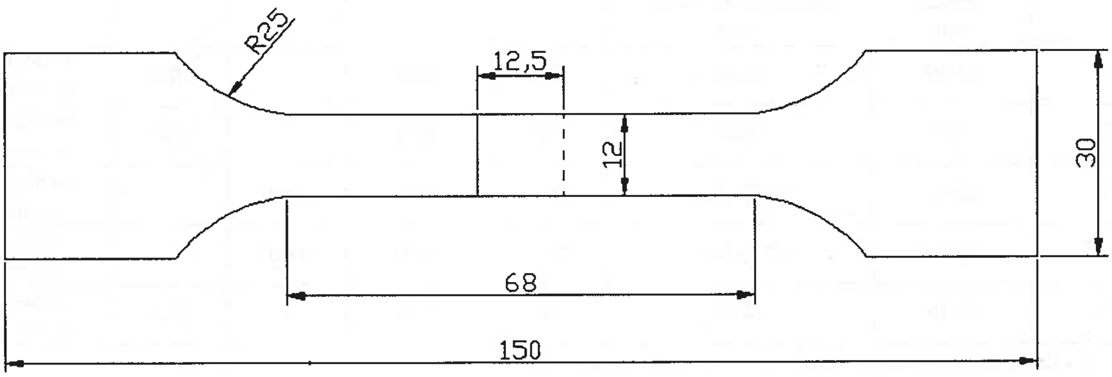


Fig. 8. Shape and dimensions of brazed joint to static shear test

Brazed joints have been made without and with copper protective layer of about 50 μm thickness. In case of copper and austenitic steel joints without the copper layer, easy destruction of the samples has been observed during clamping for mechanical processing (Fig. 9). The L-Ag15P filler metal layer exists on copper surface.

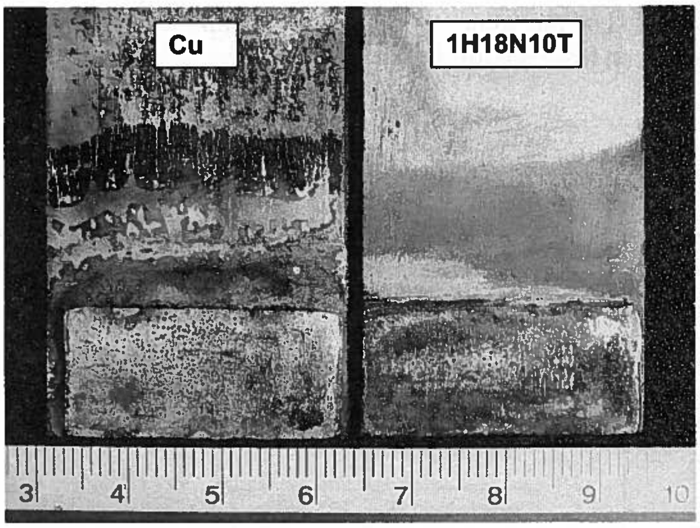


Fig. 9. Wasted Cu-1H18N10T brazed joint without copper coat

Static shear test of brazed overlap joints, by their expansion, has been carried with a universal strength maschine INSTRON 3369, according to PN-EN10002-1:2004 [21] standard. Speed of transverse beam moving was 0.2 cm/min. The range of machine load was 100 kN. Spacing inserts have been applied in order to ensure alignment eliminating bending of the joints.

The results of static shear test of Cu-1H18N10T brazed overlap joints have been presented in Table 7.

In three cases the fracture has been at the joint, where shear strength fluctuated within 26.0 – 30.6 MPa. In two another cases the fracture has been behind the overlap, in heat influence zone, at copper side, and tensile strength of the joint has been 205 – 209 MPa.

Figure 10 presents tensile (shear) graphs for two cases of brazed joints decohesion. In case of No.1 sample,

TABLE 7

Results of static shear test of Cu-1H18N10T overlapped joints

Sample No.	Overlap dimensions		A, mm²	F_t (F_m), N	R_m, MPa	R_t, MPa	Comments on fracture
	Width of joint, mm	Width of overlap (thickness of sheet), mm					
1.	11.90	13.30	158	4840	–	30.6	truncation in overlap
2.	12.05	14.30	172	4480	–	26.0	truncation in overlap
3.	12.15	13.20 (2.0)	24.3	4990	205.0	–	rupture in copper
4.	12.10	13.80 (2.0)	24.2	5050	209.0	–	rupture in copper
5.	12.15	14.80	180	4730	–	26.3	truncation in overlap

F_t (F_m) – the greatest cutting strength (max strength of tension)
R_m – tensile strength
R_t – shear strength

the fracture has been in the overlap, so shear strength R_t of the joint has been calculated. For No.3 sample, the destruction process has occurred in native material – copper, so for this case, tensile strength R_m has been calculated. It should be stressed, that the joints which broke down in the overlap, have been loaded with slightly smaller destructive force than in the case of broken down joints in copper. Therefore, it is sufficient to inctease the width of overlap to at least 15 mm in order to achieve the destruction process in native material (Cu), beyond the overlap.

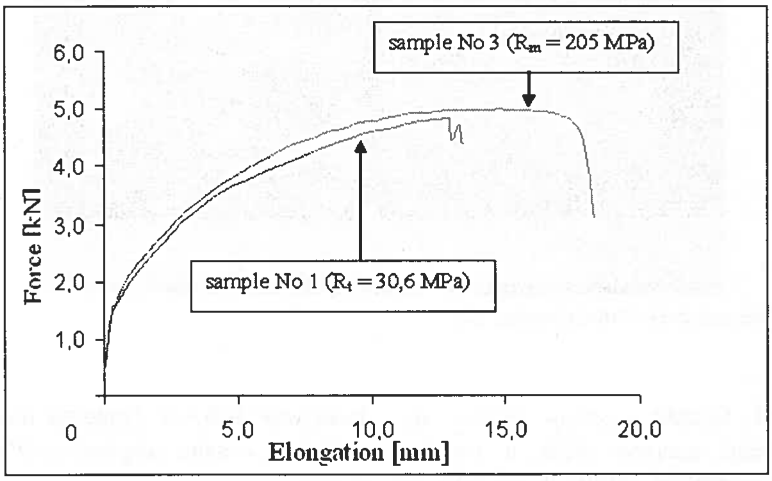
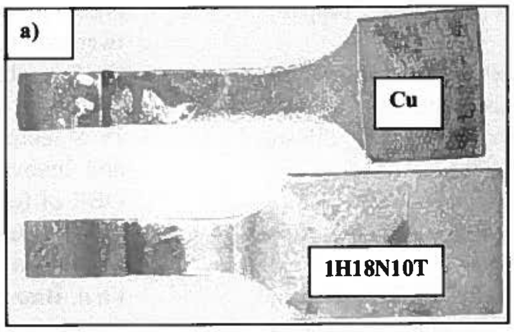


Fig. 10. Diagram of force – elongation dependence for two cases of destruction of Cu – (+Cu) 1H18N10T brazed joints

Figures 11a and 11b present characteristic fractures of the samples. Truncation in the overlap has the form of mixed fracture. Traces of copper and filler metal, proving good joint between galvanized layer and 1H18N10T steel, exist on its surface on the steel side (Fig. 11a).



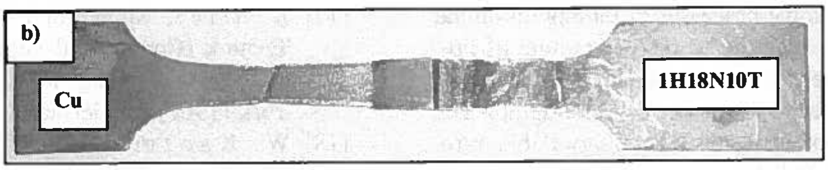


Fig. 11. Exemplary fractures in Cu -1H18N10T brazed joints, a – shearing in overlap (sample No. 1), b – break in copper (sample No. 3)

5. Summary

In spite of significant differences in physical-chemical properties of copper and 18/9 (18/10) Cr-Ni steel, it is possible to obtain satisfactory, brazed joints using filler metals on Cu-P (+Ag) matrix. The following conclusions might be formulated basing on the conducted research:

- filler metals based on Cu-P (+ Ag, Sn) applied for copper brazing cannot be used directly for brazing of any kind of steel, because of possibility of formation of the fragile intermetallic phases of Fe_2P (Ni_2P) and insufficient filler metal moistening on steel surface,
- in case of copper – phosphorous filler metals application to obtain Cu-steel Cr – Ni joints, diffusive barriers constituting protective copper coats spread on steel surface with galvanic method are recommended in order to prevent formation of intermetallic phases contributing to fragility of brazed connections. The lack of copper coat on the surface of steel makes impossible obtaining correct Cu-1H18N10T brazed joint,
- spreading of galvanized coats on 1H18N10T steel surface requires its considerable development with chemical etching (Ra = 4.6 μm, Rt = 31 μm),
- copper coats of 50 μm thickness spread on 1H18N10T steel surfaces constitute an effective diffusive barrier, preventing formation of the fragile intermetallic phases during C_2H_2-O_2 gas brazing process of Cu-1H18N10T sheets of 2 mm thickness and brazing surface of 375 mm^2,
- static shear test of brazed joints proved to be an effective test of galvanized copper coat adhesion evaluation to Cr-Ni steel surface, as the diffusive barrier in brazed joints of copper with 1H18N10T austenitic steel.

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