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MECHANICAL AND STRUCTURAL PROPERTIES OF JOINTS OF STAINLESS STEEL AND TITANIUM BRAZED WITH SILVER FILLER METALS CONTAINING TIN

WŁAŚCIWOŚCI MECHANICZNE I STRUKTURALNE ZŁĄCZY STALI NIERDZEWNEJ I TYTANU LUTOWANYCH SPOIWAMI SREBRNYMI ZAWIERAJĄCYMI CYNĘ

Joining of titanium and its alloys with stainless steel by means of welding methods and obtaining joints characterised by good operation properties constitutes today a significant problem in relation to research and technology. Apart from specialised welding technologies, brazing is one of the basic methods applied for joining these having diversified physical and chemical properties material combinations. Brazing is especially recommendable in the production of systems and heat exchangers for chemical industry as well as subassemblies of nuclear reactors and aircraft engines and accessories. Similarly as in case of welded joints of stainless steel and titanium, the mechanical properties of brazed joints of the aforesaid materials are connected with the occurrence of hard and brittle intermetallic phases appearing in the form of continuous layers on braze boundaries.

This work reports testing of strength properties and investigation of structures of vacuum-brazed joints of stainless chromium-nickel steel (X6CrNiTi18-10) and titanium (Grade 2) at 820÷900°C for 5÷20 min by means of silver brazing filler metals with tin grade B-Ag68CuSn-730/755 (Ag68Cu28Sn4) i B-Ag65CuSnNi-740/767 (Ag65Cu28Sn5Ni2). These filler metals assure better wettability of stainless steel hard to wettable in vacuum brazing.

The structural tests were conducted taking advantage of optical microscopy; by means of a scanning electron microscope (SEM) and energy-dispersion spectrometer (EDS).

The test results allowed to specify the most convenient brazing parameters of the tested material system from the mechanical properties point of view and to determine of qualitative and geometrical changes in joint structures depending on temperature and brazing times.

Keywords: brazing, vacuum brazing, stainless steel, titanium, intermetalics phases, mechanical properties of brazed joints, brazing parameters

Łączenie metodami spawalniczymi tytanu i jego stopów ze stalą nierdzewną i uzyskanie w efekcie połączeń o wymaganej dobrej jakości oraz korzystnych własnościach eksploatacyjnych stanowi wciąż ważny i aktualny problem badawczy oraz technologiczny. Lutowanie twarde obok specjalistycznych metod spawania i zgrzewania jest jedną z podstawowych metod łączenia tego układu materiałowego o zróżnicowanych własnościach fizycznych i chemicznych. Metoda ta jest szczególnie zalecana w produkcji instalacji oraz wymienników ciepła dla przemysłu chemicznego, a także podzespołów reaktorów nuklearnych oraz osprzętu i silników lotniczych. Własności mechaniczne połączeń lutowanych tytan – stal nierdzewna, podobnie jak połączeń spawanych i zgrzewanych tych materiałów, są związane z występowaniem twardych i kruchych faz międzymetalicznych wydzielających się w postaci ciągłych warstw na granicach lutowin.

W niniejszej pracy przedstawiono wyniki badań własności wytrzymałościowych oraz struktur połączeń stali nierdzewnej chromowo-niklowej X6CrNiTi18-10 z tytanem Grade 2 lutowanych próżniowo w temperaturach 820÷900°C i czasach 5÷20 min lutami srebrnymi, zawierającymi niewielki dodatek cyny, w gat. B-Ag68CuSn-730/755 (Ag68Cu28Sn4) i B-Ag65CuSnNi-740/767 (Ag65Cu28Sn5Ni2). Luty te zapewniają korzystniejszą zwilżalność trudno zwilżalnej w warunkach lutowania próżniowego stali nierdzewnej.

Badania strukturalne połączeń prowadzono z wykorzystaniem mikroskopii świetlnej, mikroskopu elektronowego - skaningowego (SEM) oraz spektrometru dyspersji energii (EDS).

Przeprowadzone badania pozwoliły na ustalenie najkorzystniejszych ze względu na własności mechaniczne parametrów lutowania badanego układu materiałowego a także na określenie zmian strukturalnych w połączeniach, wykonywanych w różnych warunkach temperaturowo – czasowych procesu.

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1. Introduction

Apart from specialised welding technologies, brazing is one of the basic methods applied for joining titanium and its alloys with stainless steel in the production of systems and heat exchangers for chemical industry as well as subassemblies of nuclear reactors and aircraft engines and accessories [1÷6]. Quite often in case of titanium constructions, technical specifications allow for the replacement of some titanium elements with those made of considerably cheaper stainless steel. This, however, is conditioned by good operation properties (especially mechanical) of joints. Brazing is applicable also in the aforesaid area.

Similarly as in case of welded joints of titanium and stainless steel, the mechanical properties of brazed joints of the aforesaid materials are connected with the occurrence of hard and brittle intermetallic phases [7÷9]. Titanium, being a reactive metal, forms intermetallic phases with the basic components of most brazing filler metals. Particularly negative on the mechanical properties of brazed joints is the impact of intermetallic phases formed in a peritectic reaction as continuous brittle and hard layers on braze boundaries. Such layers, poorly connected to a parent metal and braze, being significantly different from them in relation to thermal expansion coefficient, facilitate cracking as early as at the stage of braze solidification. In the event of the occurrence of tensile or shearing stress, the layers are also responsible for significant weakening of the joint [11÷16].

Research on brazing technology of titanium (Grade 2) with stainless steel (grade X6CrNiTi18-10) using silver brazing alloys were conducted in previous years in Institute of Welding in Gliwice [17, 18].

This article presents the results of tests related to time and temperature conditions of vacuum brazing of stainless chromium-nickel steel with titanium by means of B-Ag68CuSn-730/755 (Ag68Cu28Sn4) i B-Ag65CuSnNi-740/767 (Ag65Cu28Sn5Ni2) silver brazing alloys and their influence on the quality and mechanical properties as well as the structure of joints. These filler metals assure better wettability of stainless steel hard to wettable in vacuum brazing [10].

The purpose of the above investigation was to determine the optimum technological brazing conditions ensuring the best possible quality and mechanical properties of joints.

2. Experimental and research results

2.1. Parent and brazing filler metals

The following parent metals were used during the investigation:

- stainless steel – a rod of a diameter of 28 mm, grade X6CrNiTi18-10 according to PN-EN 10088-1, with the chemical composition according to the analysis (% m/m): 0.017% C; 18.09% Cr; 9.64% Ni; 0.78% Si; 1.37% Mn; 0.20% Ti;
- titanium – a 25-mm thick sheet, Grade 2, according to ASTM B 26579 (maximum impurity content: 0.1% C; 0.25% O; 0.03% N; 0.0125% H; 0.03% Fe).

The brazing filler metals used in the tests were silver brazing alloys, grade B-Ag68CuSn-730/755 (Ag68Cu28Sn4) and B-Ag65CuSnNi-740/767 (Ag65Cu28Sn5Ni2) in the form of a band being 0.1 mm thick.

2.2. Preparation of joint samples for strength and structural tests

Shear strength tests and structural investigation of brazed joints of stainless steel (X6CrNiTi18-10) with titanium (Grade 2) involved the application of butt-brazed samples of cylindrical elements having slightly diversified diameters i.e. the stainless steel – Ø 25×15 mm, the titanium – Ø 20×15 mm.

Silver brazing filler metals grade B-Ag68CuSn-730/755 (Ag68Cu28Sn4) and B-Ag65CuSnNi-740/767 (Ag65Cu28Sn5Ni2) in the form of profiles having the dimensions of Ø 20×0.1 mm and Ø 20×0.1 mm was inserted (3 pieces) between elements to be joined distancing in this way brazing gaps between these elements (total thickness of braze layer 0.3 mm).

As it has been proved during tests conducted with limited wettability materials creating brittle phases in joints when used with brazing alloy, this type of a sample (cylindrical sample used for the testing of diffusion-brazed joints) ensures a free course of diffusion processes in a braze and features quite a strong reaction in case of the occurrence of brittle phases impairing the quality of joints [17].

The brazing of steel – titanium samples etched in solutions of appropriate acids was performed in TORVAC-manufactured S-16 furnace in vacuum conditions within the range of $10^{-3} \div 10^{-4}$ mbar applying brazing temperatures of 820, 860 and 900°C measured directly on the samples subject to brazing.

The selection of brazing temperatures in case of steel – titanium joints was conditioned by the brazing properties of the metals to be joined. On the one hand, due to the occurrence of allotropic change ($\alpha \rightarrow \beta$) and excessive growth of titanium grains resulting in the reduction of titanium mechanical properties and creation of brittle intermetallic phases in a braze, the brazing temperature should not exceed 900°C. On the other hand, however, owing to the poor wettability of stainless chromium-nickel steel by silver brazing alloy in vacuum conditions, the brazing temperature should be over 850°C and even exceed 900°C [1÷6, 17].

The hold time of the samples at the brazing temperature was selected within quite a vast range of 5÷20 min (accordingly: 5, 10, 15, 20 min) in order to learn the impact of this parameter on the course of structural changes in the joints. It needs to be emphasized that in case of titanium, the commonly recommended hold time should be possibly short in order to limit the formation of brittle intermetallic phases with brazing alloy components, especially with copper. However, in order to ensure complete wetting of stainless steel, a slightly longer time than that for brazing is required. Preliminary technological tests have proved that the time should not be shorter than 5 min [17].

2.3. Brazed joint strength tests

The brazed joints used in tests were first subject to a qualitative visual inspection which revealed the incomplete fusion of the brazing filler metal and incomplete filling of the gaps in joints produced at 820°C, with the hold times of 5 and 10 minutes respectively. The joints made at 820°C/15÷20 min (hold-time), 860°C/5÷20 min and 900°C/5÷20 min demonstrated comparably good quality and entirely filled-up brazing gaps. On the lateral surface of the element made of titanium (easier wettable and more reactive than stainless steel), the joints were covered with a 3÷5 mm-thick and, near the edge, even 10 mm-thick layer of brazing filler metal; the said thicknesses being dependent on the brazing time and temperature.

Afterwards, the cylindrical test pieces were subject to shearing tests involving the application of special shearing shackles eliminating the effect of crosswise bending during shearing. The tests were carried out using an Instron-manufactured testing machine (model 4210). The test results are presented in Fig. 1 and Fig. 2.

The shearing test results reveal higher values in case of application of B-Ag68CuSn – 730/755 brazing filler metal (Fig. 1). The highest strength values (157÷159 MPa) were obtained for joints brazed at 860°C with the hold time of 10÷15 min as well as for joints brazed at 900°C with the hold time of 5 min (157 MPa). A very similar value (152÷154 MPa), decreasing with an increasing hold time, was obtained for the remaining joints brazed at 900°C as well as those brazed at 860°C with the hold time of 20 min. A relatively lower strength (143÷144 MPa) could be observed in case of the joints made at 820°C with the hold time of 10÷20 min as well as those brazed at 860°C with the hold time of 5 min. It should, however, be emphasized that both for the welding temperature of 820°C and that of 860°C as well as relatively short hold times of 5 and 10 minutes, it was possible to obtain higher shear strength (145 MPa) than for a silver-based B-Ag72Cu-780 brazing filler metal of eutectic composition (84÷129 MPa respectively) [17].

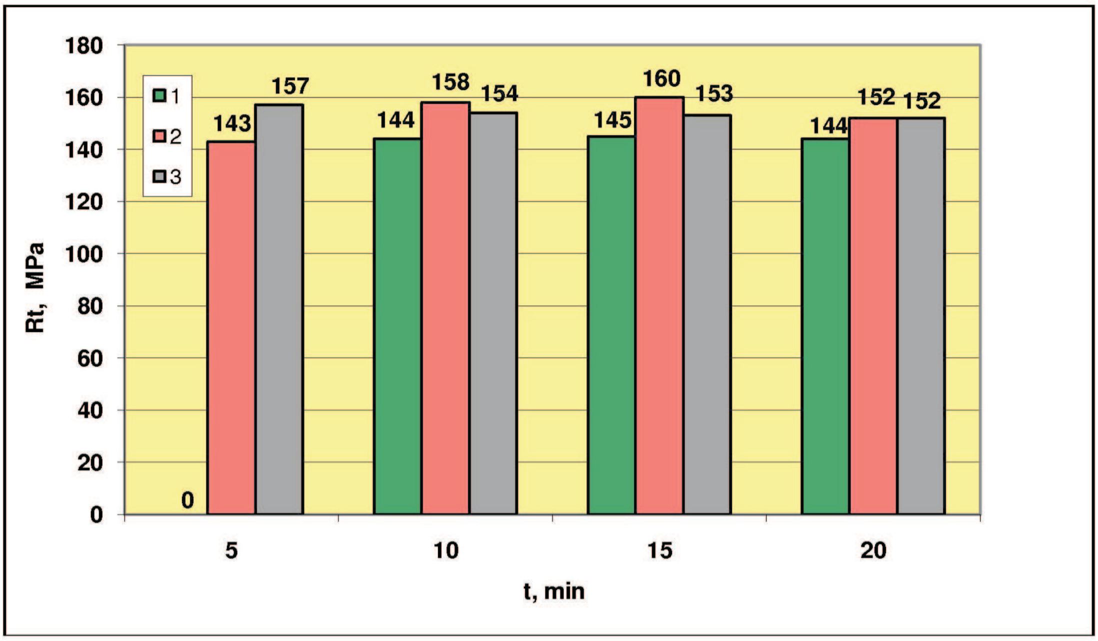

Fig. 1. Shear strength (Rt) for stainless steel – titanium joints brazed with B-Ag68CuSn-730/755 (Ag68Cu28Sn4) silver brazing alloy at temperatures of 820°C (1), 860°C (2), 900°C (3) and time 5÷20 min

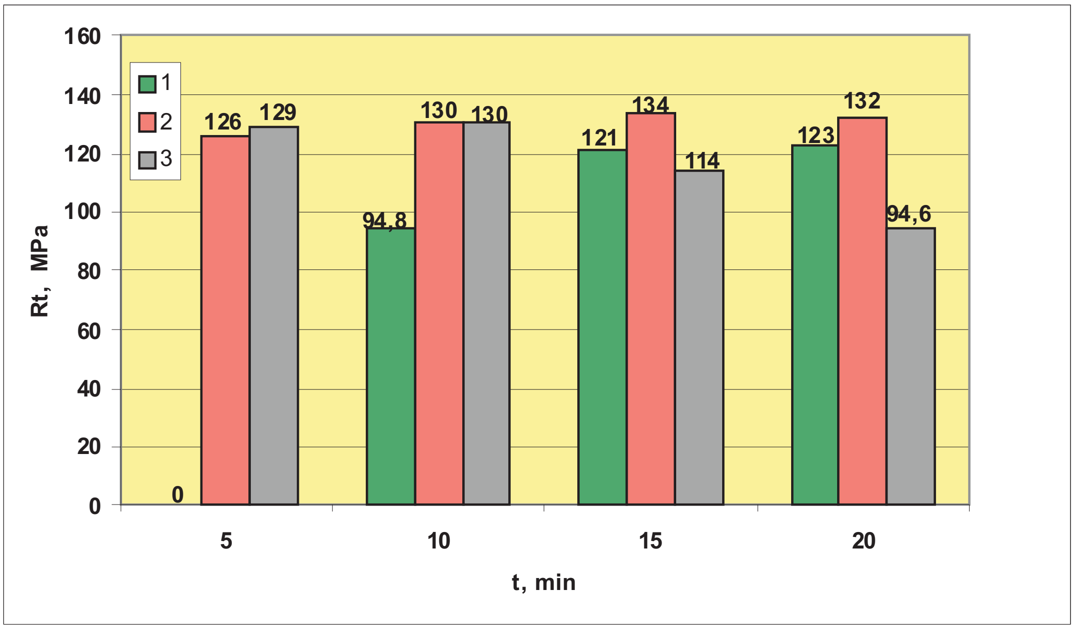

Fig. 2. Shear strength (Rt) for stainless steel – titanium joints brazed with B-Ag65CuSnNi-740/767 (Ag65Cu28Sn5Ni2) silver brazing alloy at temperatures of 820°C (1), 860°C (2), 900°C (3) and time 5÷20 min

The joints made of B-Ag65CuSnNi-740/767 (AgCu28Sn5Ni2) filler metal, brazed in analogous conditions, were characterised by relatively lower shear strength (Fig. 2). The highest strength (129÷134 MPa) was obtained for the joints brazed at 860°C with the hold time of 10÷20 min as well as those brazed at 900°C with the hold time of 5÷10 min.

2.4. Structural examination of joints

The preliminary metallographic examination of the structures of the brazed joints was conducted through the determination of their morphology in the light field using a Leica-manufactured metallographic light microscope MeF4M. Next, representative joints were examined by means of a Hitachi-made SEM VP S-3600N scanning electron microscope (SEM) with variable vacuum, collaborating with an energy-dispersive X-ray spectroscope (EDS) manufactured by THERMO NORAN and equipped with a System Six analyser. The surface of the plane sections of the joints was observed using secondary electron image (SEI) and backscattered electron image (BSE) technologies.

The tests were supplemented with a microanalysis of the chemical composition conducted by means of energy-dispersive X-ray spectroscopy (EDS) at a voltage of 25 kV, using a Thermo Noran-made System Six analyser.

The structures of joints of X6CrNiTi 18-10 stainless steel with titanium (Grade 2), brazed with silver-based B-Ag68CuSn-730/755 (Ag68Cu28Sn4) and B-Ag65CuSnNi-740/767 (Ag65Cu28Sn5Ni2) filler metals at 820, 860, and 900°C with the hold times of 5÷20 min are presented in Fig. 3 and Fig. 4.

The aforesaid structures are characterised by expanded diffusional zones, the width of which increases with the temperature of a joining process and the hold time of joints.

Figures 5÷8 depict the results of the structural analysis of selected joints brazed with the above-mentioned filler metals; the analysis was conducted with a scanning electron microscope (SEM) and an energy dispersive spectroscope (EDS).

The identification of phases in the structures was based on the EDS spectroscopic analysis, available phase equilibrium systems [8, 9, 18] and experience gained in previous tests [17] performed with an energy-dispersive spectroscope (EDS) as well as an electron microanalysis conducted using a transmission electron microscope (TEM), performed for this type of joints brazed with a B-Ag72Cu-780 filler metal of the composition similar to the basis of both of the brazing filler metals.

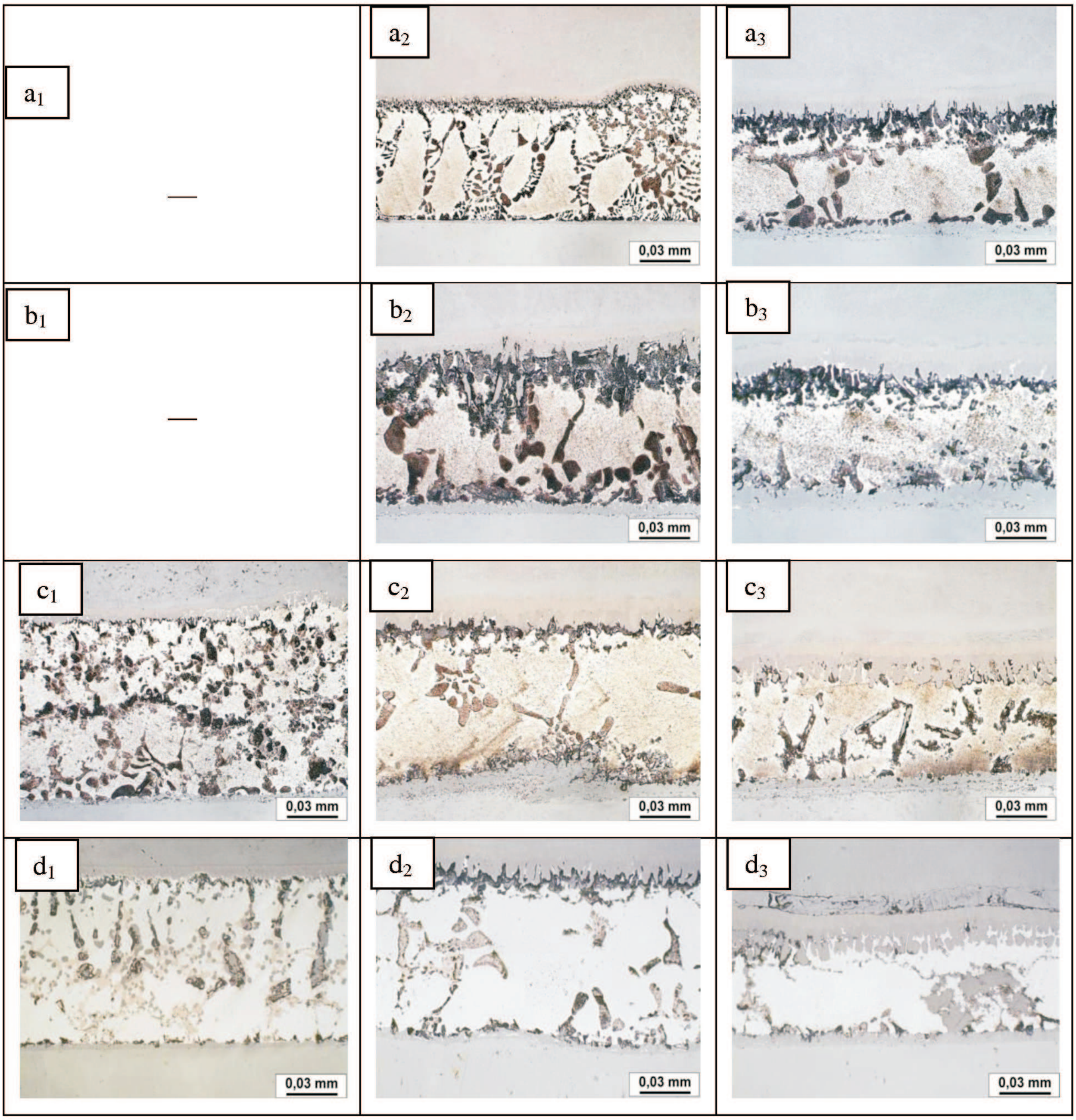

Fig. 3. Microstructure of stainless steel (down) – titanium (up) joints brazed with silver B-Ag68CuSn-730/755 (Ag68Cu28Sn4) brazing alloy at temperatures of 820°C (a_1 - d_1), 860°C (a_2 - d_2), 900°C (a_3 - d_3) and time 5 min (a_1 - a_3), 10 min (b_1 - b_3), 15 min (c_1 - c_3), 20 min (d_1 - d_3). Etch. Buehler

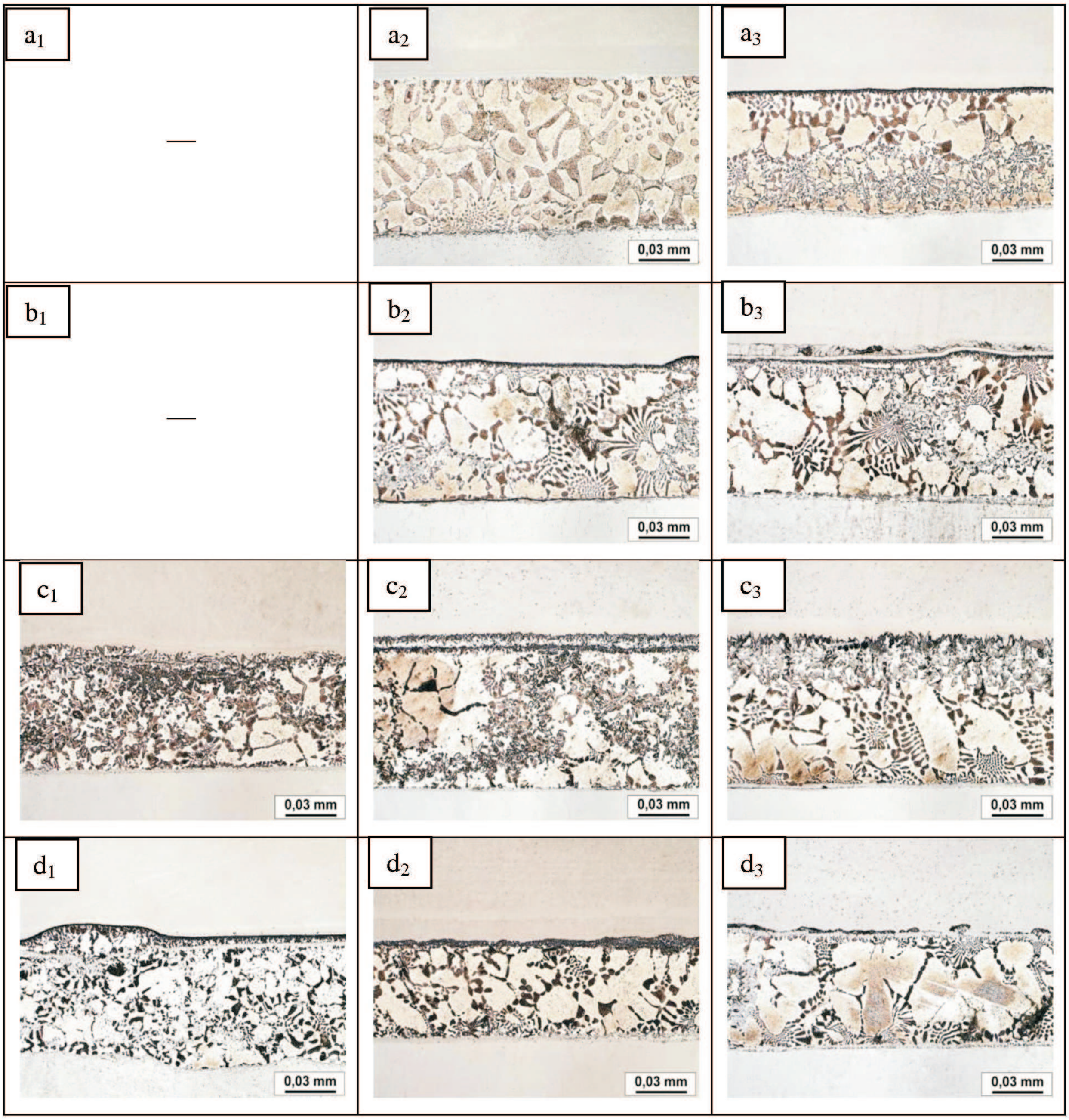

Fig. 4. Microstructure of stainless steel (down) – titanium (up) joints brazed with silver B-Ag65CuSnNi-740/767 (Ag65Cu28Sn5Ni2) brazing alloy at temperatures of 820°C (a_1 - d_1), 860°C (a_2 - d_2), 900°C (a_3 - d_3) and time 5 min (a_1 - a_3), 10 min (b_1 - b_3), 15 min (c_1 - c_3), 20 min (d_1 - d_3). Etch. Buehler

All the values provided below are expressed in "atomic %".

In case of the steel-titanium joints brazed with a B-Ag68CuSn-730/755 (Ag68Cu28Sn4) filler metal at 820÷900°C it was possible to observe the layers of Ti-Cu solid solution (Fig. 5 and Fig. 6) of variable Cu content (within 0.9÷5.9% range) dependent on brazing temperature; the solid solution layers being present on the titanium side. Below the aforesaid layers it was possible to observe another layer of the solution on the basis of a phase corresponding to the stoichiometric formula of the Cu-Ti intermetallic phase (51.6÷52.8% Ti, 44.1÷45.9% Cu, 2.4÷3.0% Ag). Behind this layer, in the joints brazed at 820 and 860°C, against the background of a silver-based Ag-Cu solution (87.6% Ag, 12.4% Cu – Fig. 5), it was possible to observe solid state precipitates on the basis of this element, yet of a slightly different chemical composition (92.3% Ag, 7.7% Cu – Fig.5). In turn, in the joint brazed at 900°C (Fig. 6) it was possible to observe the layers of solid solution precipitates of bases stoichiometrically corresponding to a Cu_3Ti_2 intermetallic phase (58.3% Cu, 39.0% Ti, 2.7% Ag) and a Ti-Cu-Sn phase (46.5% Ti, 16.3% Cu, 26.0% Sn, 11.2% Ag).

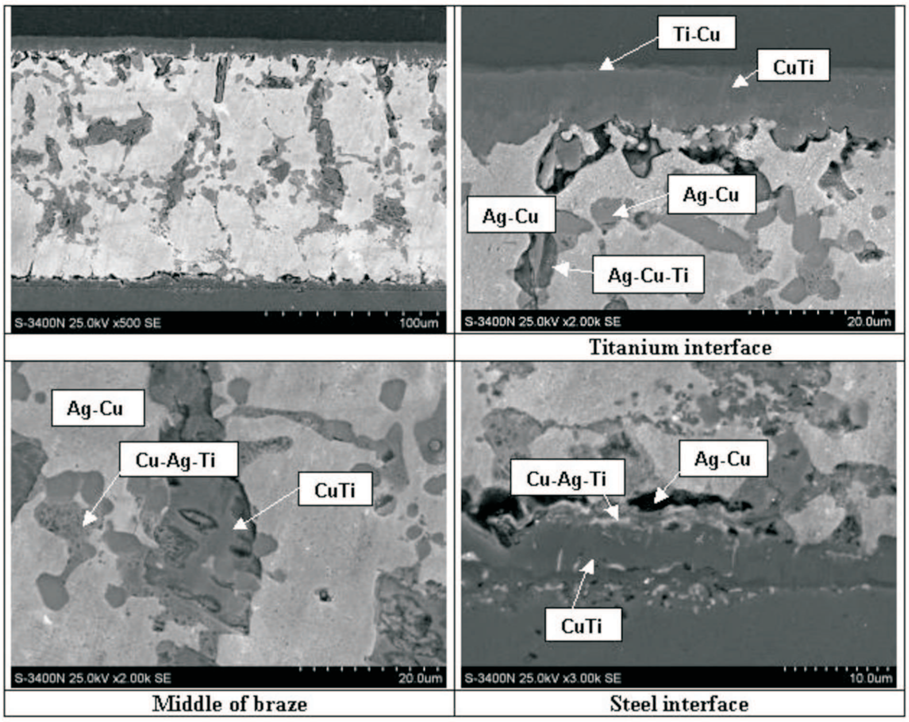

Fig. 5. Microstructure of stainless steel – titanium joint brazed with B-Ag68CuSn-730/755 (Ag68Cu28Sn4) silver brazing alloy at temperature 820°C, SEM

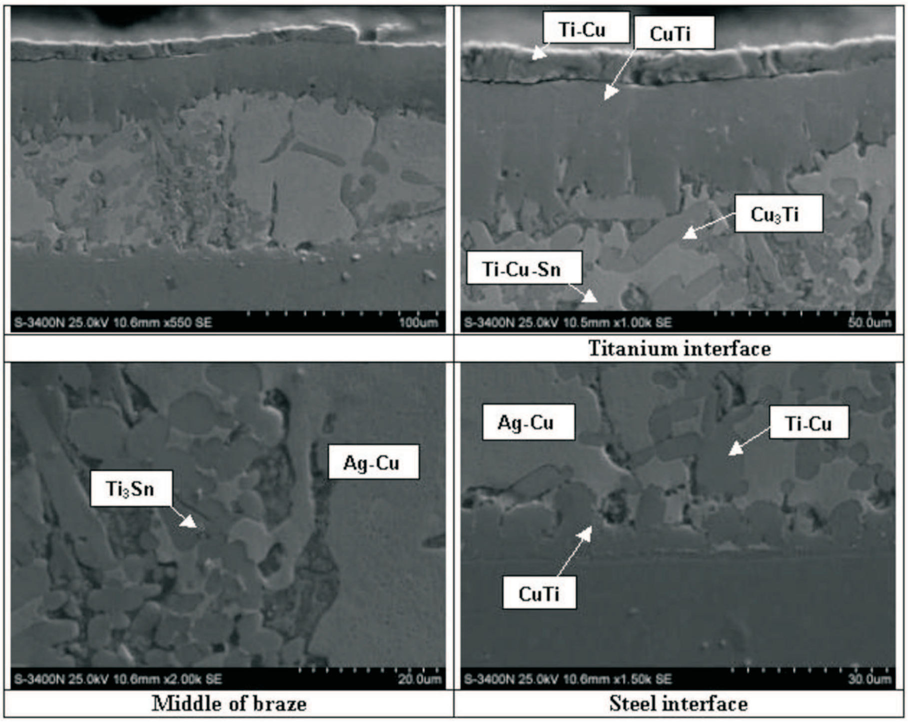

Fig. 6. Microstructure of stainless steel – titanium joint brazed with B-Ag68CuSn-730/755 (Ag68Cu28Sn4) silver brazing alloy at temperature 900°C, SEM

The middle part of the tested brazes in the joints made at 820°C (Fig. 5) was composed of Cu-Ag-Ti solid solution precipitates (92.5% Cu, 4.0% Ag, 3.5% Ti) on the basis of copper and those on the basis of the phase stoichiometrically corresponding to a Cu_4Ti intermetallic phase (77.0% Cu, 21.4% Ti, 1.6% Ag) in the silver-based Ag-Cu solid solution (86.8% Ag, 13.2% Cu). In the joints brazed at 900°C (Fig. 6) the middle part of the braze was composed of solid state solutions of the phase basis corresponding to a Ti_3Sn intermetallic phase (69.6% Ti 22.3% Sn, 6.3% Cu, 1.5% Ag) and the silver-based Ag-Cu solid solution (88.2% Ag, 11.8% Cu).

On the stainless steel side, in all the brazes made at 820 900°C it was possible to observe the layers of solutions with high iron content, of the composition of the basis stoichiometrically corresponding to a Cu-Ti intermetallic phase (32.7÷39.9% Ti, 38.9÷42.9% Cu, 11.5÷15.9% Fe, remainder Cr, Ni,Ag).

In case of the steel-titanium joints brazed with a B-Ag65CuSnNi-740/767 (AgCu28Sn5Ni2) filler metal at 820÷900°C (Fig. 7 and 8), it was possible to observe the layers of Ti-Cu solid solutions (91.7÷98.0% Ti, 2.0÷5.9% Cu, remainder Fe) dependent on brazing temperature; the solid solution layers being present on the titanium side. Below the aforesaid layers, in the joints brazed at 820°C (Fig. 7) and 860°C it was possible to observe further solution layers of the basis corresponding stoichiometrically to a CuTi intermetallic phase (51.6÷52.8% Ti, 44.1÷45.9% Cu, 2.4÷3.0% Ag). In the joints brazed at 900°C it was possible to observe a CuTi2-phase-based solution layer (76.4% Ti, 21.3% Cu, remainder Ag, Sn). Below these layers, in the silver-based Ag-Cu solid solution, it was possible to observe solid solution precipitates based on copper and Ti-Cu-Sn intermetallic phases (65.2% Ti, 12.0% Cu, 21.1% Sn, remainder Ag).

The middle part of the braze in the aforementioned joints made at 820°C (Fig. 7) was composed of precipitates of a solution based on the phase corresponding stoichiometrically to a CuTi intermetallic phase (46.6% Ti, 43.5% Cu, 6.2% Ag, remainder Sn, Fe) present in a silver-based Ag-Cu solid solution (86.1% Ag, 10.8% Cu). In the joints brazed at higher temperatures (Fig. 8), these precipitates corresponded to the copper-based Cu-Ag-Ti solid solution (89.8% Cu, 7.9% Ag, 2.3% Ti).

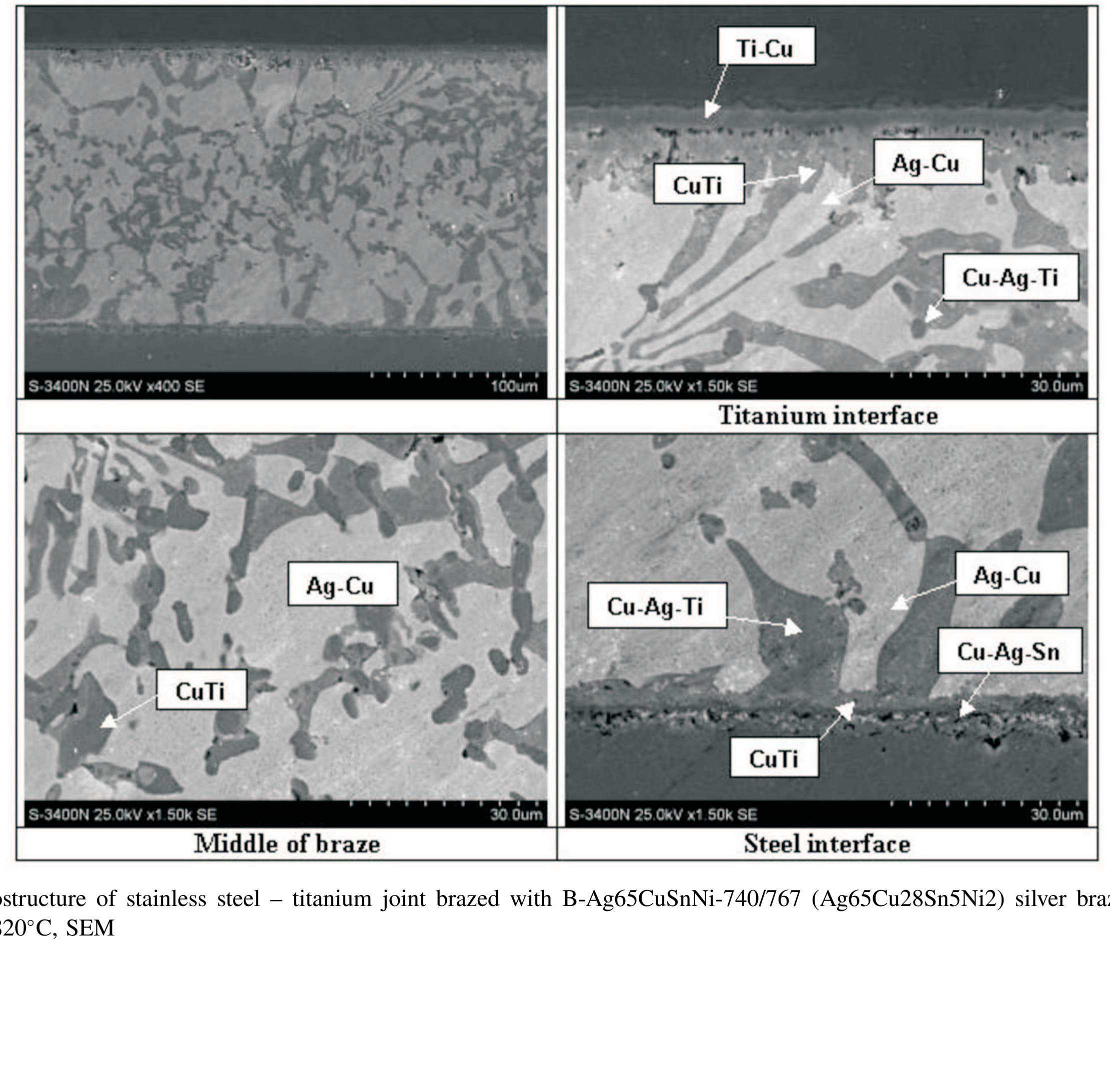

Fig. 7. Microstructure of stainless steel – titanium joint brazed with B-Ag65CuSnNi-740/767 (Ag65Cu28Sn5Ni2) silver brazing alloy at temperature 820°C, SEM

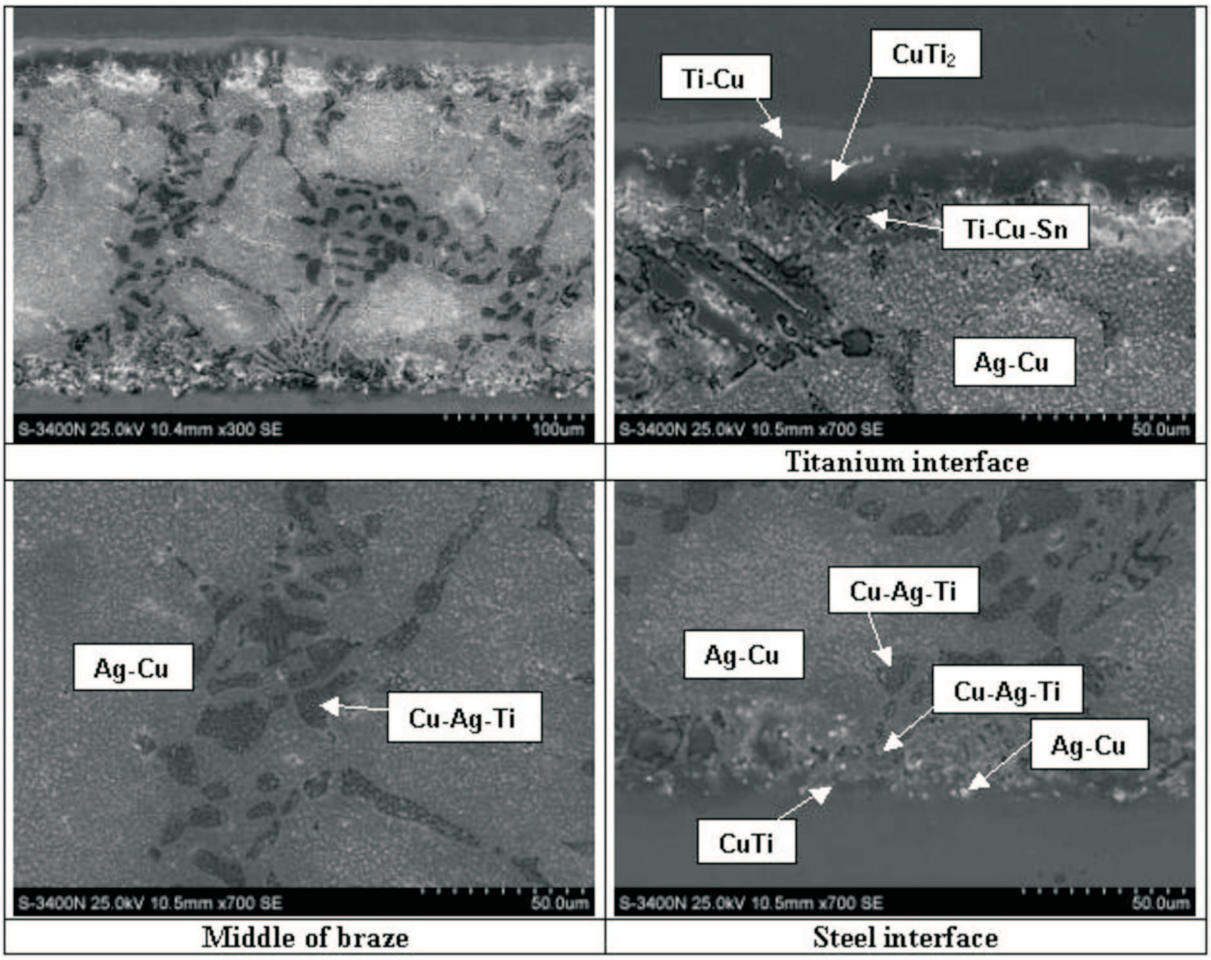

Fig. 8. Microstructure of stainless steel – titanium joint brazed with B-Ag65CuSnNi-740/767 (Ag65Cu28Sn5Ni2) silver brazing alloy at temperature 900°C, SEM

On the stainless steel side of the tested joints (Fig. 7 and Fig. 8), similarly to the joints brazed with the B-Ag68CuSn-730/755 (Ag68Cu28Sn4) filler metal, it was possible to observe the layer of the phase of stoichiometric composition corresponding to the CuTi phase (solid solution based on this phase with high iron content) with the inclusions of the copper-based Cu-Ag-Ti solid solutions (83.4% Cu, 14.6% Ag, 2.2% Ti), copper-based Cu-Ag-Sn solid solutions (92.1% Cu, 4.1% Ag, 2.5% Sn, remainder Fe) and the silver-based Ag-Cu solid solutions (79.4% Ag, 20.6% Cu).

The investigation also involved the observation of joint structures following the rupture (after shearing tests). It was noticed that the joints lost their continuity on the stainless steel side, just behind the brittle layer of the CuTi-phase-based solution with high iron content.

3. Conclusions

1. The tests of the vacuum brazing of steel (X6CrNiTi18-10) and titanium (Grade 2) joints revealed good repeatable quality of the joints made with B-Ag68CuSn-730/755 and B-Ag65CuSnNi-740/767 filler metals, brazed at 820°C, with the hold-time of 15÷20 min and brazed at 820÷900°C, with the hold-time of 5÷20 min respectively.
2. The steel-titanium joints made with B-Ag68CuSn-730/755 and B-Ag65CuSnNi-740/767 filler metals represented the highest shear strength, in 157÷159 MPa and 129÷134 MPa ranges respectively, in case of applying the brazing temperature of 860°C and hold-time of 10÷15 min as well as in case of the temperature of 900°C and the hold-time of 5 min.
3. The structural examination of the steel-titanium joints made with B-Ag68CuSn-730/755 and B-Ag65CuSnNi-740/767 filler metals revealed the

presence of extensively expanded diffusional zones; the test pieces lost their continuity in shear test on the steel-braze border, just behind the CuTi phase with high Fe content.

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