Р а з д е л III

ПАЙКА. АДГЕЗИОННЫЕ ПОКРЫТИЯ. АДГЕЗИОННЫЕ ЯВЛЕНИЯ В ТЕХНОЛОГИЧЕСКИХ ПРОЦЕССАХ ПОЛУЧЕНИЯ МАТЕРИАЛОВ

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BRAZING OF ALUMINA CERAMIC AND GRAPHITE TO TITANIUM BY AMORPHOUS FOIL Ti—20Zr—20Cu—20Ni AS THE FILLER METAL

Brazed joints of alumina ceramic and graphite with titanium were tested for shear strength. This was followed by investigation of their microstructure and phase compositions using scanning electron microscopy and EDS analysis. Active diffusion of alloy and ceramic components during brazing resulted in the formation of sophisticated microstructures characterized by a non-uniform distribution of elements. No intermetallic layers were found at the titanium interface, both in graphite-titanium and ceramic-titanium brazed joints. This new effect, and post-braze diffusion heat treatment, may improve the strength and ductility of brazed joints of dissimilar base materials.

Keywords: *brazing, alumina ceramic, graphite, Ti—Zr—Cu—Ni amorphous foil, microstructure.*

Introduction

Alumina ceramic brazed to metals is widely used for applications in optical instruments, energy converters, Tokamak windows, electronic and aerospace devices, rocket nozzles, and abrasive tools. This is due to its satisfactory combination of hardness, mechanical strength with low thermal and electrical conductivities [1, 2].

Bulk graphite tiles are brazed as armor to cooled metal substrates in modern fusion reactors. Due to such physical characteristics as high thermal conductivity and resistance to thermal shock, high sublimation temperature, and

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light weight, graphite is considered as the ideal material for limiters, diverters, and various shields used inside plasma chambers [3, 4].

Also, graphite brazed to metals is suitable in thermal management systems that are being developed for various electronics and space exploration applications [5, 6]. Typical substrates in these structures are stainless steel or refractory metals. Therefore, titanium is an attractive light-weight and corrosion-resistant option to substitute in the place of heavy metals in many brazed graphite-to-metal structures.

Most ceramic- or graphite-to-titanium joints are now brazed by active filler metals based on the Ag—Cu eutectic activated by 1,25—4,5% (mass) of titanium. Interaction and interfacial reactions of the active filler metals with ceramics and titanium are well investigated. They are mainly characterized by the formation of intermetallic layers at the interface of the base materials and intermetallic phases precipitated within the joint metal [7—11].

These filler metals provide good wetting of ceramics — but they do not flow on ceramic surfaces. Therefore, preforms of active filler metals should be placed between ceramic and titanium parts to cover the full surface of the projected brazed joint.

Reactions and joint formation of ceramics or graphite with high-titanium filler metals are not studied, yet. Particularly, the application of active filler metals with a high content of titanium can prevent the scavenging of Ti at the ceramic interface [12] to improve the density and strength of brazed joints. This work is aimed to prove the feasibility of joining ceramic and graphite to titanium using the new near- eutectic alloy Ti—20Zr—20Cu—20Ni (% (mass)), which can be considered as an ”ultra-active“ brazing filler metal due to its total higher content of titanium and zirconium, as much as 60% (mass). The new ”ultra-active“ filler metals of the Ti—Zr—Cu—Ni family [13] have the same range of brazing temperatures as active braze alloys but a lower coefficient of thermal expansion (CTE), which is much closer to titanium or ceramics than that of such traditional active alloys as CusilABA® or Ticusil®.

This circumstance may result in a significant decrease of residual thermal stresses in ceramic—to—titanium brazed joints. So this paper presents experimental results of vacuum brazing, mechanical testing, and study of microstructure of titanium-to-alumina and titanium-to-graphite brazed joints made by an ”ultra-active“ filler metal, TiBraze200, applied in the form of amorphous foil 50 microns thick.

Experimental procedure

Titanium Grade 2 (CP Titanium), Titanium Grade 5 (Ti—6Al—4V alloy), sintered alumina ceramic, and isomorphic graphite bars (supplied by McMaster-Carr Corp.) were used as base materials. Alumina bars 6x6 mm and graphite bars 12,5x6 mm were brazed as ”bridges“ to titanium of the standard double-lap specimens (fig. 1), for testing according to AWS C3.2M/C3.2:2008, Standard Methods for Evaluating the Strength of Brazed Joints. The base metal thickness was 3,175 mm and the width was 12,5 mm. Five specimens were fabricated for every combination of base materials, and the brazed specimens were subjected to tensile testing to determine shear strengths. Shims of amorphous filler metal TiBraze200 (Ti—20Zr—20Cu—20Ni (% (mass)) foil 50 microns thick were pre-

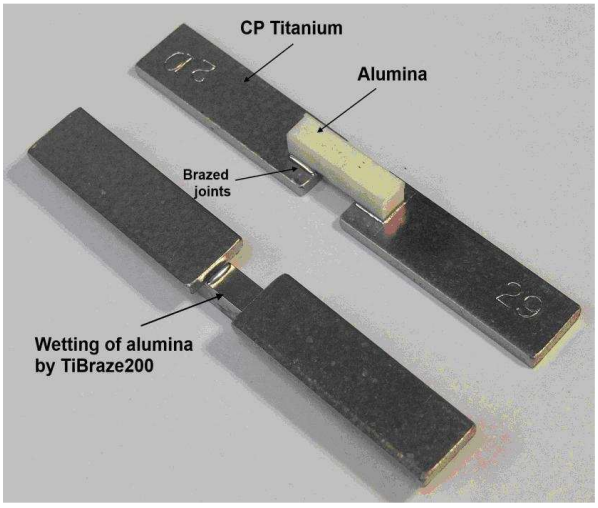

Fig. 1. Alumina—titanium double-lap specimens brazed in vacuum at 920 °C, 20 min

Рис. 1. Зразки оксидо-алюмінієва кераміка—титан, що паяні в вакуумі при 920 °С, 20 хв

placed between base material parts that were brazed in a vacuum furnace at 10^{-2}—10^{-3} Pa. Specimens were loaded by dead weights during heating and cooling. The dead weights provided compression ~250 g/cm^2. Process parameters are: 920 °C, 20 min for alumina to titanium joints, and 970 °C, 40 min for graphite to titanium joints.

The same brazed specimens were cut, mounted in KonductoMet® and polished for studying the microstructure of resulting the brazed joints. Polished cross-sections of brazed joint were etched for 1—2 min with a water solution containing hydrofluoric acid (3—4 mL) and nitric acid (3—4 mL). Finally, the samples were washed with ethanol.

Using an optical microscope, micrographs of each sample were studied in order to determine the microstructure and joint quality. Images included macro-sections, fillets, interfaces, joint metals, and defects. SEM with EDS analysis was done in a Jeol JSM-5900LV scanning electron microscope for studying the fine structure and phase compositions of the joint metal and diffusion zones.

Results and discussion

Shear strength of graphite—to—titanium brazed joints wasn't measured, because all the samples failed in the graphite body (fig. 2), even with overlaps as small as one thickness of the graphite bar itself. Shear strength of ceramic-to-titanium brazed joints also was tested using “bridge-like” specimens (see fig. 1). Some alumina—titanium joints also failed along the ceramic body (see fig. 2), which confirms that the brittleness of a ceramic body played a crucial role in testing: this means that the geometrical design of the specimens successfully used for metal—to—metal joints testing should be adapted for metal—to—ceramic specimens. However, these test results at least demonstrated that the adhesion of the braze alloy TiBraze200 to ceramics and graphite is sufficiently strong to resist significant shear loads, and titanium—to—ceramics or

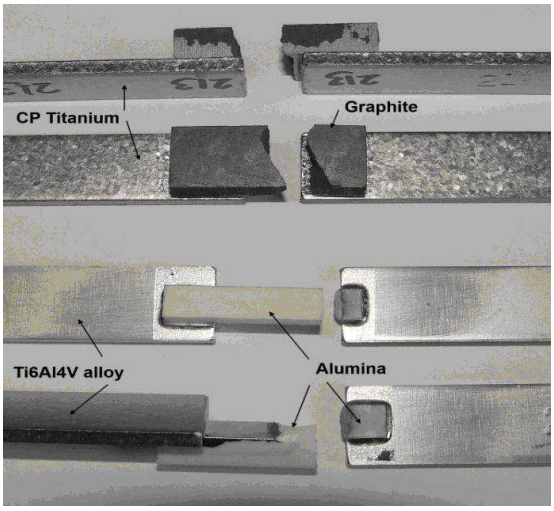

Fig. 2. Brazed joints failed in ceramic or graphite bodies during mechanical testing

Рис. 2. Паяні зразки, зруйновані по тілу кераміки або графіту під час механічного випробовування

titanium—to—graphite brazed joints can respond to the requirements of reliability at larger overlaps. Three alumina—titanium joints, which failed in the joint metal, exhibited shear strength in the range of 93—112 MPa. That is 12% higher than the tensile strength of traditional alumina—Kovar joints manufactured by Ag—Cu active filler metals containing 1,25—1,75% Ti [14]. Suggesting that tensile strength of dissimilar brazed joints usually is higher than shear strength, due to the smaller effect of stress concentration at the edges, we can expect significant gain in the tensile strength of Al_2O_3—Ti joints brazed by TiBraze200.

TiBraze200 as a titanium—rich filler metal in the form of amorphous foil exhibited good wetting of titanium, ceramics, and graphite base materials, and spreading along ceramic or graphite surfaces (fig. 1, 2), which is not typical for traditional active filler metals [15]. Overheating above the regular brazing temperature of TiBraze200 and longer holding time when brazing ceramic and graphite (in comparison with metal—to—metal joining) gave better results, both in the quality and strength consideration for ceramic and graphite brazed joints. Recommended parameters for use in brazing processes are presented in table 1. The titanium- and zirconium-rich filler metal TiBraze200 forms dense brazed joints with alumina ceramic. However, titanium—graphite joints have a number of micro-voids at the titanium interface (fig. 3). A hypothesis regarding this is: these micro-voids appear due to the insufficient amount of liquid filler metal that is predominantly infiltrated into graphite pores and that reacts with graphite forming (Ti,Zr)C carbides. A similar ”scavenging effect“ was explored in the ceramic—to—metal joints brazed by titanium-containing active filler metals [12]. Infiltration of the braze liquid into the graphite body also resulted in a lack of shaped fillets in the titanium—graphite joints.

T a b l e 1. **Brazing process parameters**

T а б л и ц я 1. **Параметри процесу паяння**

Brazing Filler Metal TiBraze200	Metal—to—metal: CP Ti, Ti—6Al—4V, Ti—3Al—2,5V alloys	Metal—to—ceramic: Alumina ceramic and graphite
Brazing temperature	890—900°C	920—970°C
Holding time	10—12 min	20—40 min

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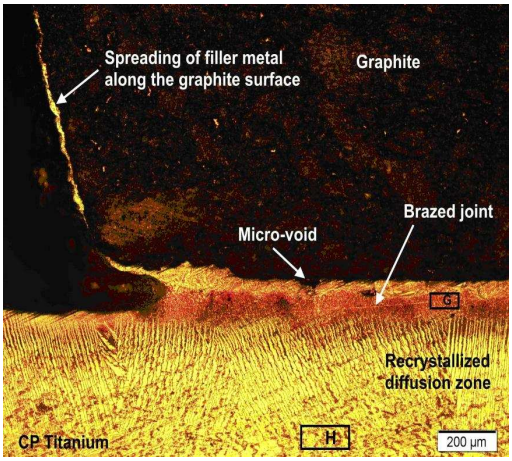
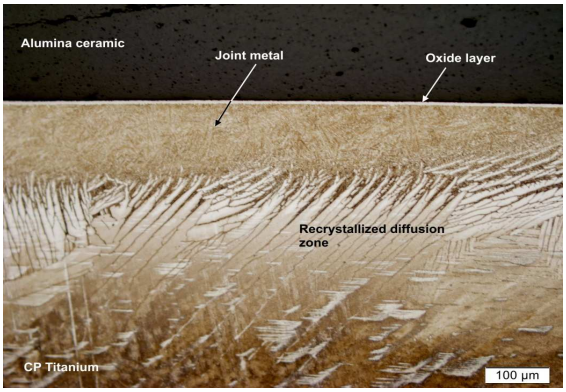


Fig. 3. Fillet area of a titanium—graphite joint brazed by TiBraze200. Box G is inside the joint metal and box H is in titanium, outside the diffusion zone, x100

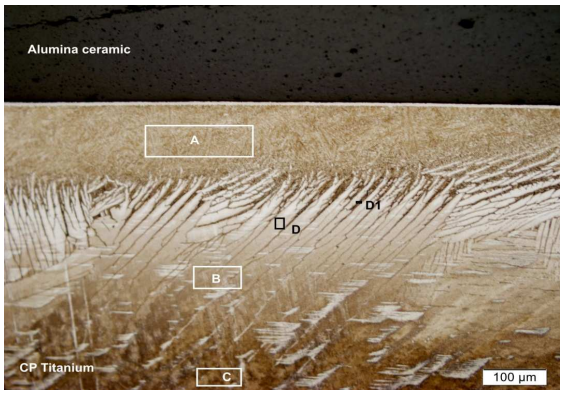
Рис. 3. Зона припою, з'єднання титан—графіт, що було впаяно припоєм TiBraze200. Позначки G — припайний метал, H — титан поза дифузійної зони, x100

The alumina—titanium joints are characterized by smooth fillets and a very thin oxide layer at the interface. The necessity of overheating testifies that a reaction of molten filler metals with ceramics is inactive or slow if compare to brazing metal—to—metal joints. The limited thickness of the oxide layer did result in a sufficient contact strength of joint metal with ceramic or graphite. This is confirmed by fracture in the ceramic bodies, instead of in the joint metals (see fig. 2).

The microstructure of alumina joints with titanium comprises a solid solution zone on the ceramic side and a recrystallized diffusion zone on the base metal side, which has typical pattern for the interaction of Ti—Zr-based filler metals with titanium (fig. 4, *a*). The diffusion zone is wider and more developed



a



b

Fig. 4. Microstructure (*a*) and the map of EDS-measured phase compositions (*b*) in the Al_2O_3—titanium joint brazed by TiBraze200 amorphous foil, x200

Рис. 4. Мікроструктура (*a*) та мапа ЕДС-виміру фазового складу (*b*) паяного з'єднання Al_2O_3—титан, що було впаяно припоєм TiBraze200 в вигляді аморфної фольги

than that in metal—to—metal joints due to a higher brazing temperature and longer exposure time in the temperature range above the liquidus temperature of the filler metals. Fig. 4, *b* shows a map of measured compositions of different zones and phases in the microstructure of brazed joint. The XRD spectra pattern from the overall joint metal is presented in fig. 5, while all compositions marked in the microstructure map fig. 4, *b* are presented in table 2. Very important is a fact that no intermetallic layers were formed at the titanium interface despite the TiBraze200 filler metal containing a significantly large amount of copper and nickel, 20% (mass) of each. The absence of intermetallic layers on the metal side appears to be a significant positive distinctive feature of "ultra-active" brazing filler metals.

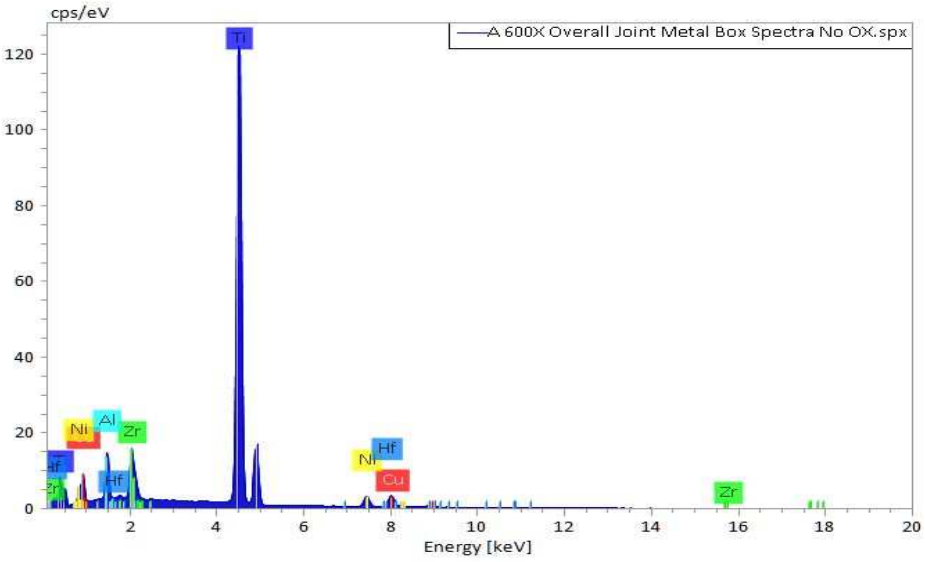


Fig. 5. EDS-spectra pattern from the joint metal: Box A in fig. 4, *b*

Рис. 5. ЕДС спектр паяного металу: помітка А на рис. 4, *b*

T a b l e 2. **EDS analysis of the areas indicated in Fig. 4, *b* (% (mass))**

T а б л и ц я 2. **ЕДС аналіз зони, що вказана на рис. 4, *b* (% (мас.)**

Measured area in fig. 4, b	Al	Ti	Ni	Cu	Zr
A Joint metal	3,4	76,6	5,1	7,4	7,6
B Titanium under diffusion zone	2,8	96,5	0,5	0,1	0,02
C Titanium body	1,0	98,8	0	0,04	0,07
D Large crystals in the diffusion zone	1,4	96,7	0,8	0,8	0,3
D1 Between large crystals in the diffusion zone	2,1	87,9	6,2	3,2	0,6

The significant growth of titanium content in the joint metal (area A in fig. 4, *b*) has engaged our attention, as the initial composition of TiBraze200 contained only 40% (wt.) of titanium. This means that dissolution of titanium from the base metal into the liquid braze was sufficiently intense to substitute zirconium bound by oxygen into the oxide layers E and F (fig. 6). In their turn,

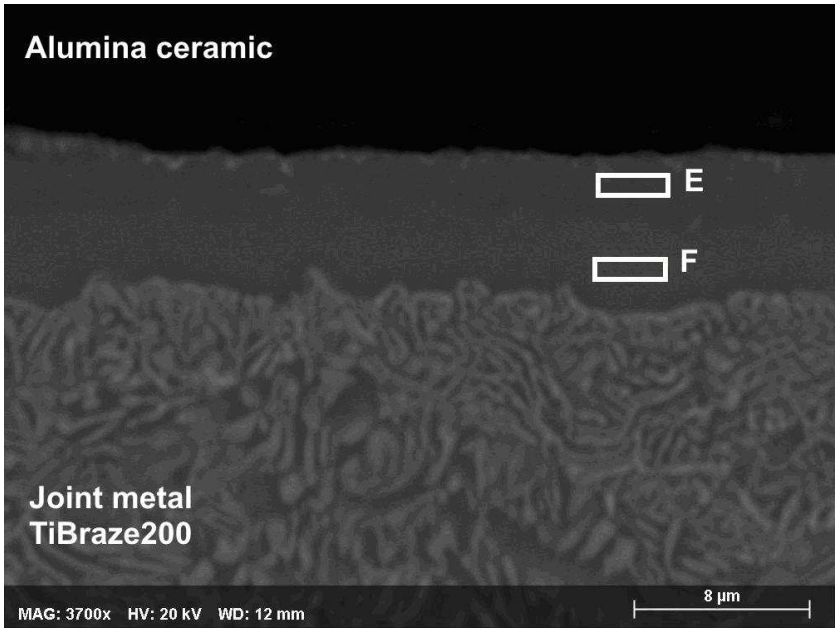


Fig. 6. Double—layer of complex oxides at the ceramic interface in the alumina—titanium joint brazed by TiBraze200, x3700

Рис. 6. Подвійний шар складних оксидів біля міжфазної границі з'єднання Al_2O_3 кераміка—титан паяного припоєм TiBraze200, x3700

copper and nickel diffused into the base metal, especially along the titanium grain boundaries. If we compare the contents of Cu and Ni in titanium grains (the box D in fig. 4, *b* and table 2) with the contents of these elements between titanium crystals (box D1), we can see that the space between titanium crystals is saturated by copper and nickel, while solid titanium grains contain an insignificant amount of these elements. As well, aluminum also rather saturates spaces between titanium crystals, along the titanium grain boundaries (table 3). Such a non-uniform re-distribution of alloying components in the base metal resulted in the full recrystallization of titanium and the formation of a sort of "Widmanstädtten-like" structure of base titanium near the brazed joint. The same type of titanium structure was found also in [13, 16].

Noteworthy also is the diffusion of aluminum deeply into the titanium body, especially if compared with copper or nickel. These were not transported further than the diffusion zone, while aluminum was found in titanium body at the distance about 400 microns from the joint. This point is still not explicable.

A study of the oxide layer at the ceramic interface by and EDS analysis and SEM at high magnification showed a double-layer structure (see fig. 6) with the following compositions of each layer presented in table 3.

Table 3. **EDS analysis of the interface layers indicated in fig. 6 (% (mass))**

Таблиця 3. **ЕДС аналіз міжфазних шарів, що вказані на рис. 6 (% (мас.))**

Measured areas	Al	Ti	Ni	Cu	Zr	O
E	12,1	61,8	0,8	1,5	4,8	19,0
F	6,7	68,6	0,6	1,1	4,6	18,5

Both oxide layers are presented by complex oxides (Al, Ti, Zr)O_x that differ mainly by content of aluminum and titanium (table 3). The separation into two layers can be explained thus: (a) The first layer E (see fig. 6) was formed immediately after contact of the liquid filler metal with alumina, and the stochastic-equilibrium oxide $(Al_2TiZr)O_5$ was formed at the surface of ceramic; (*b*) This dense oxide layer plays a role as a diffusion barrier for aluminum transport into the braze, as well as for titanium transport from the braze to the ceramic surface. Therefore, the layer F has non-equilibrium composition with understandable prevailing of titanium over aluminum in the composition.

Similar double-layered structure of the interface reaction products was found in [8, 17]. There was discussed the brazing of alumina by silver-copper eutectic alloyed with 2,9% (mass) of titanium. The Ti_3Cu_3O compound was identified in the oxide layer of the ceramic surface. According to data of table 3, this compound was not formed in our case. It is interesting to note that the oxygen content in the layer E (see fig. 6), and the appropriate layer I [8], are close to each other: 39% (at.) and 34% (at.), while the aluminum content is quite different: 14% (at.) in our case against 1,4% (at.) in the layer I.

A thin $Cu_2(Ti,Al))_4O$ layer adjacent to alumina was also found in [11], where was considered the brazing of alumina with Ti—6Al—4V alloy by Cu—40Ag—5Ti filler metal. The authors also found highly dispersed Ti_2Cu and $(Ti, Al)_3Cu$ phases that were not identified in our work.

Microstructures of a titanium-graphite joint brazed by TiBraze200 amorphous foil are shown in fig. 3 and 7, as well as graphite base material infiltrated by the filler metal. The depth of penetrating the graphite body by liquid braze is up to 500 microns. This effect can improve the strength of graphite brazed joints made using amorphous foil filler metals. There were no intermetallic layers found at the titanium interface and that is atypical for titanium—to—carbon joints brazed with low-titanium filler metals such as Ticusil® [6].

As expected, the composition of joint metal (box G in fig. 3) is close to that of the alumina—titanium brazed parts (tables 2 and 4), while distribution of elements in the diffusion zone is quite different. In contrast with alumina-titanium brazed joints, the distribution of copper, nickel, and zirconium is relatively uniform in the graphite-titanium joints (table 4). These elements penetrated solid titanium at the distance at least 400 microns together with carbon that was not perceived before by other researchers. So active diffusion in solid titanium could be ignited by overheating almost to α-β transition

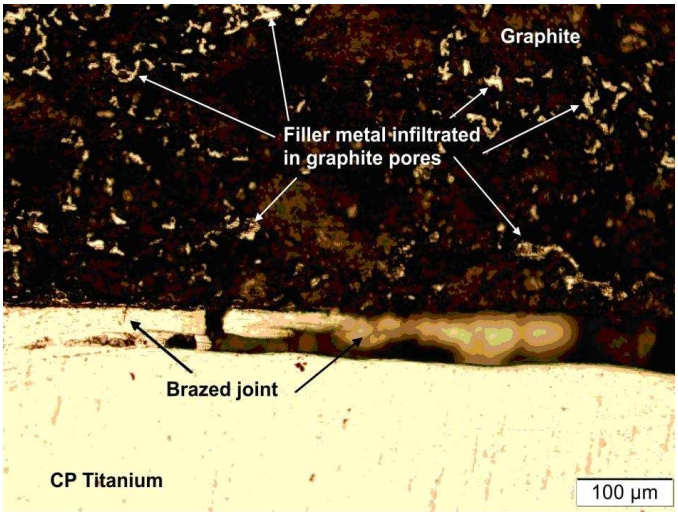


Fig. 7. Filler metal infiltrated in the graphite pores when brazed by TiBraze200, x200

Рис. 7. Металевий припій TiBraze200, що пропитує пори в графіті при паянні, x200

T a b l e 4**. EDS analysis of the areas indicated in fig. 3 and 8 (% (mass))**

T а б л и ц я 4**. ЕДС аналіз зон, вказаних на рис. 3 та 8 (% (мас.))**

Measured areas	C	Ti	Ni	Cu	Zr
G Joint metal	3,9	73,5	6,2	7,6	8,8
H Titanium body	3,6	80,6	4,2	5,9	5,7
K Intermetallic layer	37,7	52,5	1,5	2,1	6,0

temperature and holding for a longer than usual time during brazing. The ”Widmanstädtten-like“ structure of base titanium metal in the diffusion zone is similar to the structure described above in the alumina—titanium brazed joint, and one hypothesis is that it was formed in the same way.

A high concentration of carbon in the joint metal was also noticed (see table 4), but surprisingly, no carbide phases were found in the joint metal even at the high magnification of x3300 (fig. 8). The intermetallic layer at the graphite interface (fig. 8, 9) is definitely developed up as (Ti, Zr)C carbide phase (see table 4). The irregular shape of this layer (see fig. 8) has engaged our attention because usually intermetallic layers look like a solid strip of the thickness equal along the interface of the base material. Such a specific shape of the carbide layer characterized by irregular thickness can be assigned to porosity of the graphite surface and/or infiltration of graphite pores by liquid

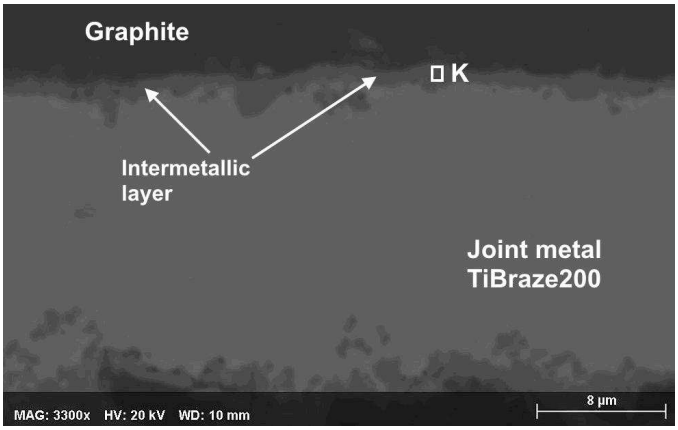


Fig. 8. Intermetallic layer at the graphite interface in the joint brazed by TiBraze200, x3300

Рис. 8. Інтерметалічний шар на міжфазній границі біля графіту з'єднання, паяного припоєм TiBraze200, x3300

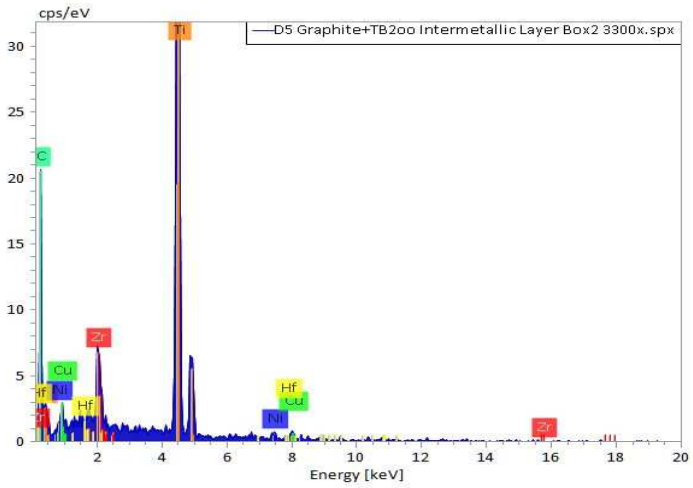


Fig. 9. EDS-spectra pattern from the intermetallic layer: Box K in fig. 8

Рис. 9. ЕДС спектр інтерметалічного шару: помітка К на рис. 8

filler metal. The filler metal in open pores changes a local profile of the graphite surface contacted with the liquid metal bath and affects formation of continuous flat intermetallic layer.

Conclusions

The “ultra-active”, amorphous filler metal Ti—20Zr—20Cu—20Ni (% (mass) is suitable for brazing alumina ceramic or graphite to titanium in vacuum by overheating 30—70 °C compared to regular brazing temperature in order to intensify interfacial reactions and provide formation of dense and strong joints. The liquid filler metal exhibited flowing along the ceramic or graphite surfaces in contrast with traditional active, low-titanium braze alloys.

A double oxide layer is formed at the alumina ceramic interface, whereby two sub-layers have different aluminum contents but same oxygen contents.

Both alumina—titanium and graphite-titanium brazed joints do not have reaction intermetallic layers at the titanium interfaces. This is a new effect of brazing with ”ultra-active“ filler metals, which is promising in view of increasing ductility of brazed joints of dissimilar base materials.

Aluminum (in the ceramic—titanium joint) or carbon (in the graphite—titanium joint) diffuse deeply, at least by 400 microns, in the titanium body. Diffusion of copper and nickel from the joint metal is limited by the diffusion zone, where these elements are distributed not uniformly: they saturate the space between titanium grains, which contain only an insignificant amount of Cu and Ni. The recrystallized diffusion zone of the base metal gives a possibility to improve the microstructure with a more uniform distribution of alloy components by additional, post-braze heat treatment that may increase the strength of brazed joints.

РЕЗЮМЕ. Була випробувана міцність при зсуві паяних з’єднань оксидно-алюмінієвої кераміки та графіту з титаном. Досліджено їх мікроструктури та фазові склади за допомогою скануючої електронної мікроскопії та ЕДС аналізу. Активна дифузія сплаву та керамічних компонентів у процесі паяння спричиняє утворення складних мікроструктур, які характеризуються нерівномірним розподілом елементів. У паяних сполуках на поверхні поділу з титаном, як для графіту, так і для оксидної кераміки, шари інтерметалідів не були знайдені. Цей новий ефект, а також дифузійна термообробка після паяння можуть сприяти підвищенню міцності та пластичності паяних сполук на основі різнорідних матеріалів.

Ключові слова: *паяння, оксидно-алюмінієва кераміка, графіт, аморфна фольга Ti—Zr—Cu—Ni, мікроструктура.*

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Пайка оксидно-алюминиевой керамики и графита с титаном с использованием аморфной фольги Ti—20Zr—20Cu—20Ni в качестве припоя

Испытана прочность при сдвиге паяных соединений оксидно-алюминиевой керамики и графита с титаном. Исследованы их микроструктуры и фазовые составы с помощью сканирующей электронной микроскопии и ЭДС анализа. Активная диффузия сплава и керамических компонентов в процессе пайки приводит к образованию сложных микроструктур, и которые характеризуются неравномерным распределением элементов. В паяных соединениях на границе раздела с титаном, как для графита, так и для оксидной керамики, слои интерметаллидов не были обнаружены. Этот новый эффект и диффузионная термообработка после пайки могут повысить прочность и вязкость паяных соединений на основе разнородных материалов.

Ключевые слова: *пайка, оксидно-алюминиевая керамика, графит, аморфная фольга Ti—Zr—Cu—Ni, микроструктура.*