Structure and Properties of Graphite-Molybdenum Brazed Joints

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Abstract: The paper presents the results of X-ray microspectral studies of dissimilar brazed joints of molybdenum with graphite. It is shown that during active brazing of graphite with molybdenum, mutual diffusion processes occur, and the adhesion-active brazing filler metals penetrates into graphite, and interacts with it, which leads to the formation of carbide phases. When using the Ti-Cr-V and Cu-Ti-Ni systems brazing filler metals, titanium carbides are formed. The zirconium carbides are formed, when using the brazing filler metals based on the Zr-Pd(Mo) systems and the CxMey(Mo, Cr) carbides are formed using the brazing filler metals of the Pd-Ni-Cr-Ge system. The results of tests for three-point bending showed that the using of Pd-Ni-Cr-Ge brazing filler metals provides stable strength at the level of 34-37 MPa, destruction occurs along graphite.

Keywords: graphite; molybdenum; brazed joints; brazing filler metal; structure; properties.

1. Introduction

Production of permanent joints of high-temperature materials molybdenum and graphite by brazing is significant for many branches of industry related structure operation at high temperatures. The low density of graphite, good thermal conduction close to metal thermal conduction, high thermal resistance, and, in particular, resistance to thermal shocks, significantly exceeding the resistance of most ceramic materials, have stipulated its application in different branches of engineering [1-5]. On the other hand, the application of graphite is limited by its most important disadvantage, i.e., low mechanical strength.

This limiting factor can be eliminated by developing structure containing joints of graphite with metallic materials. However, the essential step for developing an effective structure is the safe joining of the dissimilar materials. At that, brazing is the most practically feasible and economical method of joining graphite materials between each other as well as with metallic materials [3-8].

The problems, typical for many pairs of dissimilar materials [7; 9; 10], also appear in molybdenum-graphite brazed joints. The difficulty of joining graphite materials with molybdenum is caused by significant differences in chemical composition, thermal-physical and physical-mechanical characteristics, namely, thermal conduction, coefficient of elasticity, and heat expansion.

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Molybdenum differs by low oxidation resistance and high sensitivity to detrimental impurities. Sublimation of MoO3 oxide starts at a temperature of more than 500 °C. It becomes significant at 600 °C, and with a further increase of temperature (above 800 °C), the oxide is melted, resulting in superactive oxidation of molybdenum in the air atmosphere [11]. Therefore, brazing shall be carried out in a vacuum. Other brazing problems are related to its wetting and filling of a brazing gap with a brazing filler metal. Brazing of graphite can be performed by preliminary metalizing of the surface being brazed (plasma or electrolytic methods) with further application of the ductile inactive brazing filler metals [6] as well as without coating, but using adhesion-active brazing filler metals. Currently, the commonly used filler metals include Ti-(Zr)-based [12-14], Cu-and Cu-Ag-based [15-18] brazing filler metals, etc.

Coatings are often used by brazing [15; 16]. The wetting behaviour of AgCu non-active eutectic filler metal on the surface of Cr-deposited graphite was investigated by Z.B. Chen and athers [18]. The graphite and graphite were successfully joined with AgCu nonactive filler metal by vacuum brazing after surface modification. But the brazing filler metal based on the copper-silver system does not provide brazed joints with performance characteristics at high temperatures.

An important task is a correct selection of the chemical composition of brazing filler metal, its solidus and liquidus temperature. The brazing filler metal should readily wet the materials being brazed, it should be sufficiently strong and at the same time, ductile, should readily deform, promoting relaxation of stresses, arising during brazing and cooling to room temperature. Selection of the brazing filler metal requires using carbide-forming elements, having a chemical interaction with carbon, and forming carbide compounds at the interface during active brazing. The latter contains elements (Ti, Zr, Cr, Ta, Nb, V, Hf) having a high chemical affinity with carbon and forming with them strong adhesive bonds [12-18].

It should be noted that the content of the active constituent requires control. The rise of its concentration results in an increase of brazing filler metal brittleness, an increase in the amount of carbide interlayers and a decrease of the brazed joint strength [8; 19; 20].

The use of active brazing filler metal ensures good wetting of the graphite. The authors [21] presented the investigations on brazing Graphite with molybdenum (TZM) alloy at a high temperature of 1300 to 1700 °C. They used the following brazing filler materials Ti-56Ni, Ti-8.5Si, Ti-33Cr and Ti-30V-3Mo. The results show that the microstructure of TZM/graphite braze metal was composed of NiTi and Ni3Ti compounds when using Ti-56Ni brazing filler, and the discontinuous TiC compounds were found at the Ti-56Ni/graphite side. When using the Ti-8.5Si, Ti-33Cr and Ti-30V-Mo brazing fillers, the metallurgical combination was obtained due to the formation of the Ti-Mo solid solution at the TZM/brazing filler side and the TiC reaction layer at the graphite/brazing filler side. The fracture was occurred at the interface of TiC/graphite during shear test, which indicated the poorer bonding strength between the graphite and TiC. The shear strength range of the joint was 9–12 MPa. TiC compounds were formed at the interface between Ti-56Ni, Ti-8.5Si, Ti-33Cr, and Ti-30V-3Mo brazing filler metals and graphite, and the brazing joint was fractured at the junction of TiC compounds and graphite. Molybdenum (TZM alloy: Mo-0.06Zr-0.4-0.55Ti-0.01-0.04C) is characterized by a higher recrystallization temperature (1400 °C) and mechanical properties compared to ordinary molybdenum (MCh).

Brazing of molybdenum grade MCh at such a high temperature will lead to significant grain growth and recrystallization, which will adversely affect the mechanical properties of the brazed joints. Therefore, brazing Graphite (MPG-6) with molybdenum (MCh) is carried out at a lower temperature. The application of surface treatment of the base materials prior to brazing of graphite (MPG-6) with molybdenum (MCh) (with brazing filler metal Ti–40Zr–8.5Nb–1.5Be) increases the connection of area of brazing filler metal and base metal [12]. The application of pressure (5 kg) during brazing (T =1400 °C, holding time of 20 min) contributes to the partial extrusion of the brazing filler metal from the gap and provides shear strength at

the level of 28 MPa. It is known that using beryllium brazing filler metal requires special technological methods for the protection of service personnel and the creation of specific laboratories.

Thus, in the current work, it is proposed further decrease of the temperature by brazing dissimilar materials graphite/molybdenum with adhesive-active brazing filler metals. This will save the original properties of molybdenum, and avoid the processes of its recrystallization. It is show X-ray microspectral investigations and the mechanical properties of brazed joints graphite-molybdenum by vacuum brazing with filler metals: Ti-Cr-V, Cu-Ti-Ni, Zr-Pd, Zr-Pd-Mo.

2. Experimental

Polycrystalline adhesion-active brazing filler metals (Ti-Cr-V, Cu-Ti-Ni, Zr-Pd, Zr-Pd-Mo) were used in the investigations in cast form (Table 1). Relatively low melting temperatures characterize them.

Table 1. *Brazing filler metal and brazing mode.*

No	Brazing filler metal	$T_{brazing}$, °C	Exposure, min	Heating rate °C/min	Cooling rate °C/min
1	Ti-V-25Cr	1500	5		
2	Cu-Ni-20Ti	1160	1		
3	Zr-25Pd	1100	5	15	5,5
4	Zr-Pd-5Mo	1150	3		
5	Pd-Ni-Ge-19Cr	1235	3		

The ductile palladium-based brazing filler metals Pd-Ni-Cr-(Me) were also used. They can be produced by traditional methods of metallurgical treatment in the form of a thin (50 µm) ribbons. It is used to manufacture embedded elements of any geometry that simplifies the technological process of brazing.

The experimental adhesion-active brazing filler metals were melted by arc method on a cold substrate with an argon atmosphere. For all alloys the solidus and liquidus temperatures were determined using high-temperature differential thermal analysis in a high-purity helium atmosphere at a 40 °C/min heating rate.

2.1 Brazing procedure

The molybdenum of MCh grade and graphite MPG-6 grade as base materials were used for investigations. Samples sized $40 \times 5 \times 5$ mm were prepared for brazing. The capillary brazing of lap samples was carried out in a furnace with radiation heating in a vacuum of about 1.33·10-4 Pa. Experimental procedure of brazing is shown in Figure 1.
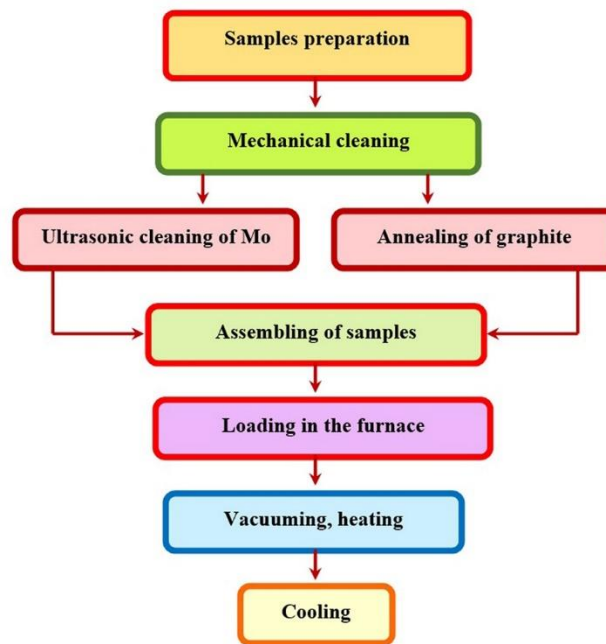
Figure. 1. *Flow chart of experimental procedure of brazing.*

Prior to brazing, the samples of Mo were cleaned mechanically using a diamond disc with a grain size of 100 μm, then ultrasonic cleaning was carried out in ethanol and isopropyl alcohol. Graphite samples were annealed at temperature 1200 °C for 1 hour before brazing. Graphite samples were annealed at the temperature of 1200 °C for 1 hour to remove gases before brazing. During the lap joint assembly, the filler metal was placed in the gap and fixed by spot capacitor welding. The brazing temperature for each filler metal as well as heating and cooling rates are shown in Table 1. A low cooling rate (5.5 °C/min) was chosen to prevent the destruction of the brazed joints due to differences in the coefficients of linear expansion of the brazed materials.

2.2 Metallographic and mechanical examinations

Metallographic examinations were carried out on the brazed lap samples, from which standard technologies produced microsections. The microstructure of the samples and element composition were examined using a scanning electron microscope TescanMira3 LMU equipped with energy-dispersive spectrometer Oxford Instruments X-max 80 mm2 controlled by INCA software. The locality of the measurements made up to 1 mm. Examination of the distribution of chemical elements and microstructure filming (without chemical etching) were carried out in backscattered electron (BSI) mode, providing phase contrast and allowing to differentiate structural constituents due to atomic weight differences.

To determine the mechanical properties, the butt samples were brazed and used a servo-hydraulic machine MTS 810 for three-point bending tests at room temperature.

3. Results and discussion

Micro-X-ray spectral examination of the brazed joints, produced using high-frequency heating and adhesion-active brazing filler metal based on Ti-Cr-V system, showed good wetting of both brazed materials and formation of brazed seams without cracks (Figure. 1a).

A seam structure consists of a matrix, carbides, and eutectic. The seam matrix is a solid solution based on molybdenum (its content makes up to 49 wt. %) with eutectic inclusions.

There is the formation of titanium carbides in a carbon–brazing filler metal interface (from the carbon side) in the form of a grey band of 3-5 mm width (Figure. 2a, b) as well as separate single inclusions, located in the matrix from the graphite side.
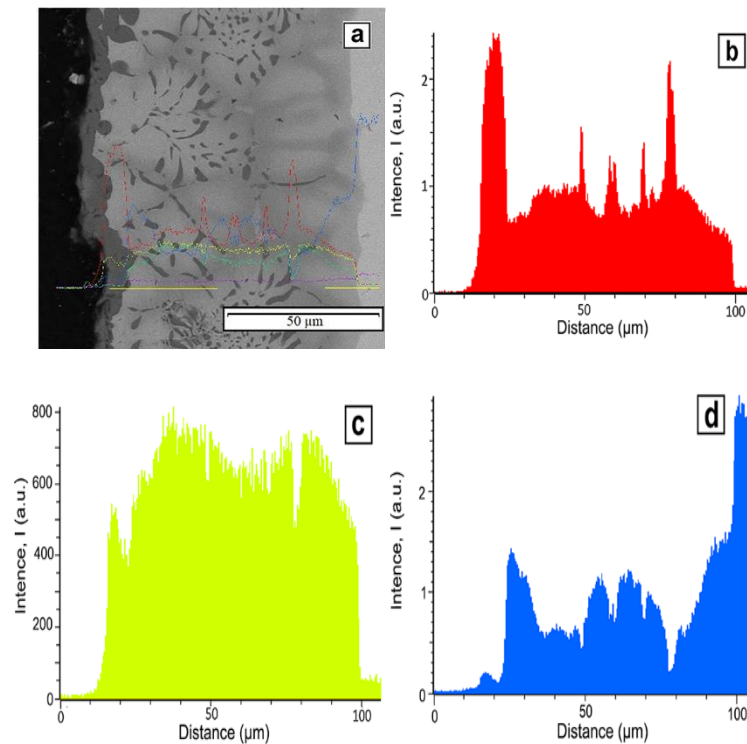

Figure. 2. *The microstructure (a) and distribution of titanium (b), vanadium (c), and molybdenum (d) in graphite – molybdenum brazed seam produced using Ti-Cr-V system brazing filler metal.*

It is proved by micro-X-ray spectral examination when studying the quality distribution of the elements along the scanning line (normal to the brazed seam). The same areas demonstrate an insignificant increase in vanadium and molybdenum concentration (see Figure. 2c, d) and a decrease of chromium concentration.

Obtained data correlate well with the results of local micro-X-ray spectral analysis, which demonstrate the discrete concentration of the elements in the carbide layer (wt. %), i.e., titanium – 69.07%, molybdenum – 4.94%, vanadium – 3.52%, chromium – 1.11%. Carbon concentration makes 21.36%.

Penetration of the brazing filler metal in porous graphite at 200-400 mm distance occurs in the brazing. That result in pore filling and carbide phase formation in the separate areas of the zone boundary to the seam (Figure. 3).
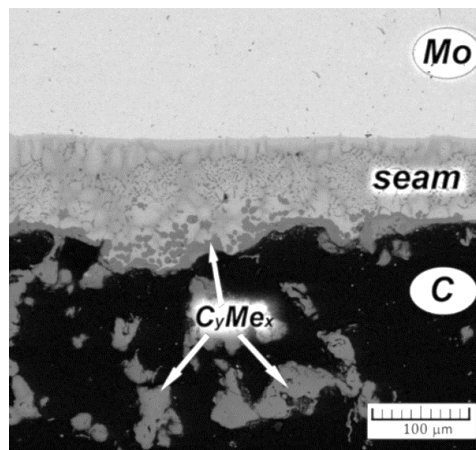
Figure. 3. *The microstructure of graphite-molybdenum brazed joint in brazing with Ti-Cr-V system based brazing filler metal.*

The concentration of titanium in such precipitations reduces to 37.57%, and the amount of other elements insignificantly rises, namely, vanadium to 15.94 %, chromium to 9.68%, molybdenum to 8.67%, carbon to 28.14%. The disadvantage of this brazing filler metal is the presence of pores in the brazed seam.

Among the perspective brazing filler metals for graphite brazing are the alloys based on inert to carbon metals (copper, silver, gold etc.) doped with chemically active elements: titanium, chromium, zirconium etc. Cooper in pure form does not wet graphite, but the presence of titanium in the Cooper alloy reduces the contact angle of wetting at 10 wt. % concentration of titanium, the contact angle falls to zero, resulting in the complete spread of copper-titanium alloy [22]. We have used Cu-Ti-Ni system alloy as the brazing filler metal. It is characterized by a wide melting temperature interval (Figure. 4).
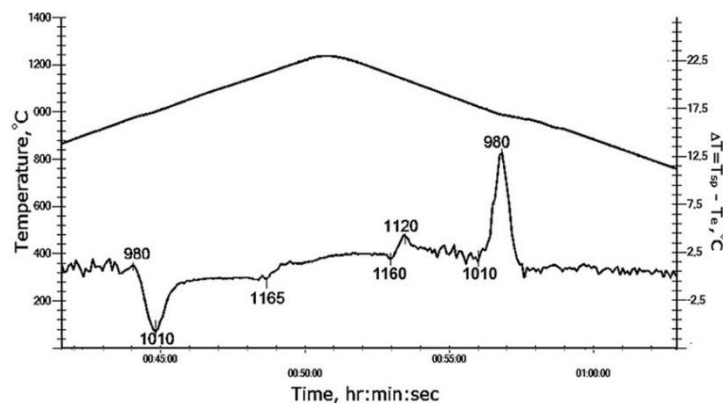

Figure. 4. *The temperatures of solidus and liquidus of Cu-Ti-Ni system alloy.*

Brazing of dissimilar graphite-molybdenum joints was carried out at liquidus temperature of Cu-Ti-Ni system brazing filler metal. Cast brazing filler metal located close to the gap melts on heating and penetrates the brazing gap.

The samples have a good appearance; the brazing filler metal wets molybdenum, graphite, and complete fillets are formed. However, a more detailed examination of the brazed joints using electron microscopy detects single defects in the form of cracks in some samples in the brazed seam zone. Received results of metallographic examination show the absence of stability in the brazed seam quality.

Zirconium-palladium system alloys are of particular interest as brazing filler metals. Zirconium is referred to as a carbide-forming element, which provides good wetting of graphite

[23], but has a high melting temperature. Zirconium melting temperature can be reduced by palladium doping. In accordance with the binary constitution diagram [24] for metallic systems, the concentration of palladium in the amount of 27.5 wt. % provokes the eutectic reaction $L \leftrightarrow (\beta Zr) + PdZr_2$ providing the minimum melting temperature 1030 °C (Figure. 5).
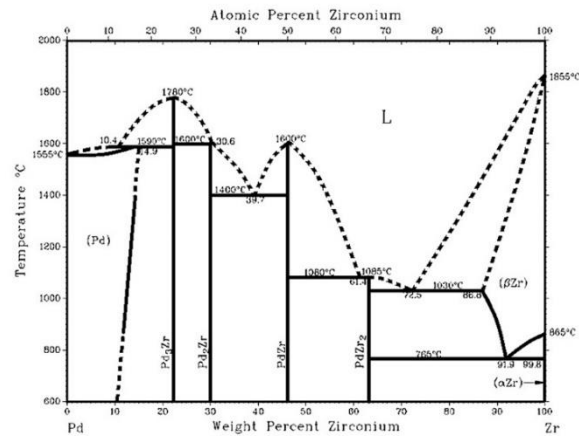

Figure. 5. *The constitution diagram of the binary metallic system Pd-Zr [24].*

Eutectic brazing filler metals differ from the brazing filler metals in the structure of solid solutions because melting occurs at a specific temperature; no melting temperature interval is present. Besides, the eutectic brazing filler metals (except for silver) are characterized by increased brittleness.

The hipper-eutectic alloy Zr-Pd with liquidus temperature 1075 °C was used for the experiments. A qualitative investigation of the distribution of the chemical elements in the graphite-molybdenum brazed joint, produced using Zr-Pd system alloy (Tm = 1100 °C, t = 1 min) showed that palladium and zirconium have a uniform distribution in the seam (Figure. 6a, b, d).
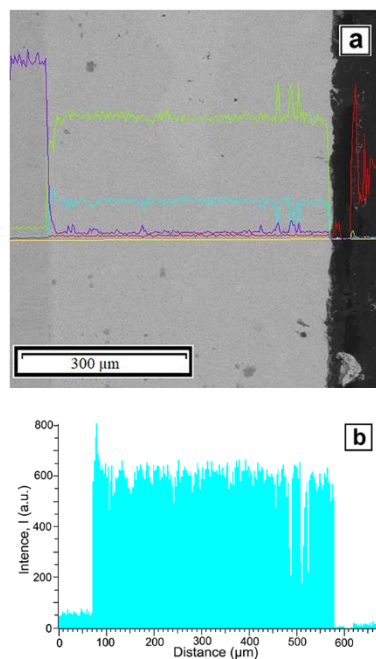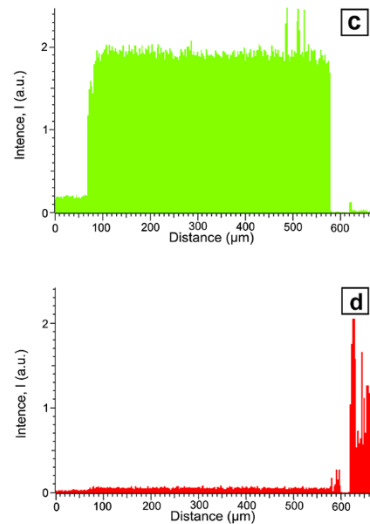
Figure. 6. *The microstructure of graphite – molybdenum joint produced using the pilot Zr-Pd (a) system brazing filler metal and distribution of elements, i.e, palladium (b), zirconium (c);carbon (d).*

Only in some areas of the brazed seam from the side of the brazing filler metal-carbon interface, there are peaks with the maximum concentration of zirconium and insignificant synchronous increase of carbon concentration (Figure. 6c) that indicate the formation of the carbide phase.

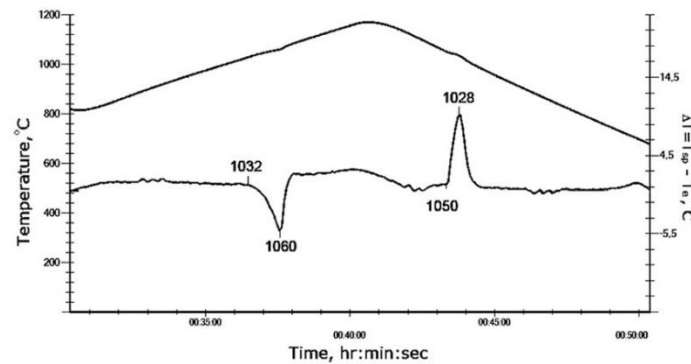A ternary alloy with a lower liquidus temperature was also used for research (Figure. 7).

Figure. 7. *The thermal curve of Zr-25Pd-5Mo alloy received using DTA.*

Formation of carbides at the brazing filler metal-carbon interface verifies the results of local micro-X-ray spectral examination of the microstructure produced by the spread of Zr-Pd-5Mo brazing filler metal over graphite (Figure. 8, Table 2, spectrum 5).
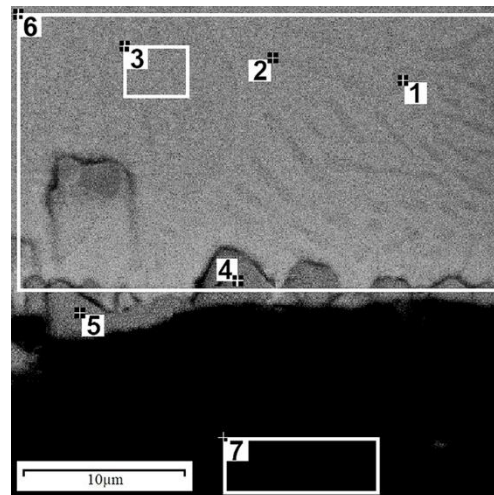
Figure. 8. *The microstructure and examined areas of brazing filler metal – carbon interface.*

Table 2. *Composition of separate phases of brazing filler metal – carbon interface.*

Spectrum No.	Chemical elements, wt.%			
	C*	Zr	Mo	Pd
1	5.97	77.82	6.74	9.47
2	9.97	58.37	0.00	31.67
3	0.00	79.52	7.77	12.71
4	0.00	99.69	0.00	0.31
5	35.90	64.10	0.00	0.00
6	0.00	72.65	4.27	23.08
7	99.35	0.54	0.11	0.00

* Carbon concentration was qualitatively determined

Zirconium carbides are nucleated on the surface of the graphite plate and solidify not in the form of a solid band, but in the form of discrete, separate cut particles of Zr2C (Figure. 8).

One of the methods of reducing stress in the brazing of dissimilar materials is the application of ductile brazing filler metals, which are characterized by acceptable melting temperature and the presence of a solid solution structure that provides the formation of ductile brazed seams in the brazed joints. The excellent representatives of such brazing filler metals are Pd-Cr-Ni-Ge system brazing filler metals, which are used for brazing very critical and loaded parts [25]. It can be used to manufacture the embedded elements of any conFigureuration that raises the feasibility of the brazing process. Application of this brazing filler metal provides good wetting of graphite and molybdenum (at Tm = 1235 °C, τ = 3 min), formation of dense quality seams without defects (Figure. 9a, b). Zonal solidification of the brazed seam is observed. A diffusion layer of around 60 µm width is formed at the molybdenum side. It contains an insignificant amount of brazing filler metal constituent elements (Figure. 9a, b, Table 3, Spectrum 4).
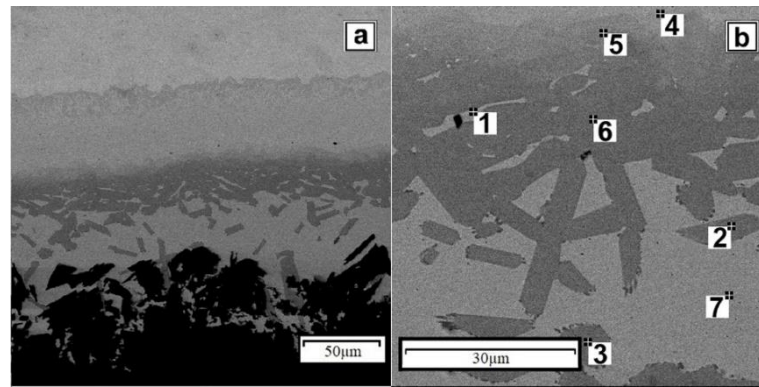
Figure. 9. *The microstructure (a) and examined areas (b) of Mo-C joint brazed with Pd-Cr-Ni-Ge system brazing filler metal.*

Table 3. *Element composition of brazed joints using Pd-Cr-Ni-Ge system brazing filler metal.*

Spectrum No.	Chemical elements, wt.%						
	C^*	Si	Cr	Ni	Ge	Mo	Pd
1	-	0.35	8.43	30.78	1.58	3.57	55.30
2	26.43	0.06	21.47	1.46	0.00	49.71	0.87
3	30.62	0.08	20.28	1.24	0.00	47.77	-
4	-	0.11	3.35	2.72	0.00	89.00	4.82
5	30.84	0.04	11.12	0.43	0.00	57.57	-
6	30.16	0.08	19.76	0.52	0.00	49.48	-
7	8.93	0.26	8.30	30.97	1.09	2.79	47.65

* Carbon concentration was qualitatively determined

In the next zone, there is a phase in the form of regular-shaped particles based on molybdenum, containing about 30 % carbon and having an increased concentration of chromium (Table 3) is formed. This is confirmation of the formation of carbide. They are also formed when using an adhesive-active layer of zirconium [26].

Based on the data of micro-X-ray spectral analysis, the matrix of the brazed seam is the palladium-nickel solid solution (see Figure 9b, Table 3, spectra 1, 7) containing other elements in insignificant amounts. There is also penetration of the brazing filler metal in graphite at a small distance (up to 60 µm).

The formation of permanent joints in brazing has its own peculiarities related with the presence of concentration gradient at the interface, narrow brazing gap, solidification of brazed seams in non-equilibrium conditions. This leads to the mutual diffusion of the liquid filler brazing metal - a solid substrate at the base - metal interface. As a result, there is a possibility for variation of the composition of the brazing filler metal compared to the initial state, formation of new phases, namely, intermetallic, carbides etc. Interaction of liquid brazing filler metal with (base material) graphite is observed. The brazing filler metal penetrates into the graphite, ruins the bonds, and promotes the transfer of separate grains in the brazed seam. As a result, we observe dispersed particles of graphite in the matrix (solid solution) of the brazed seam metal (Figure. 9a).

Considered brazing filler metals were used for brazing of graphite-molybdenum butt samples for mechanical three-point bending tests (Figure. 10a, b).
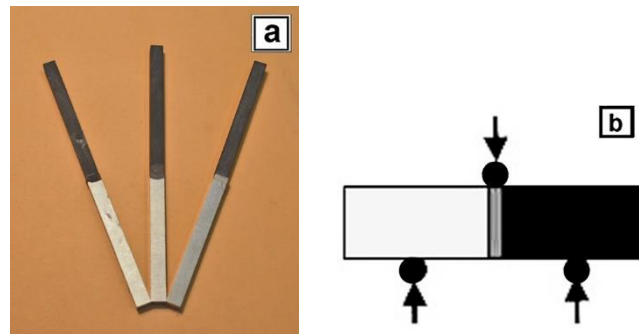
Figure. 10. *The butt samples C+Mo for mechanical test (a), testing scheme (b).*

The mechanical tests showed that the strength of the brazed joints produced by brazing filler metals of Cu-Ti-Ni and Zr-Pd-Mo systems is characterized by significant scattering of data (Figure. 11).

The joints produced using the brazing filler metals of the Zr-Pd and Pd-Cr-Ni-Ge systems differ in stable strength values.
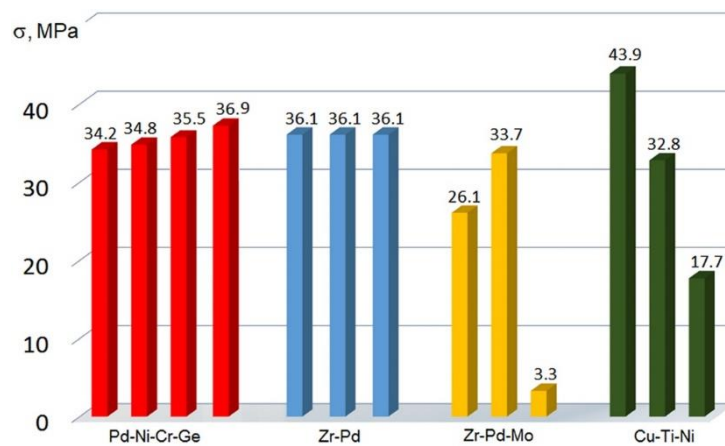

Figure. 11. *The bending strength of C-Mo joints.*

Higher strength values were received by brazing with the brazing filler metal of the Pd-Cr-Ni-Ge system. In all cases, failure of the samples occurs along the graphite (Figure.12).
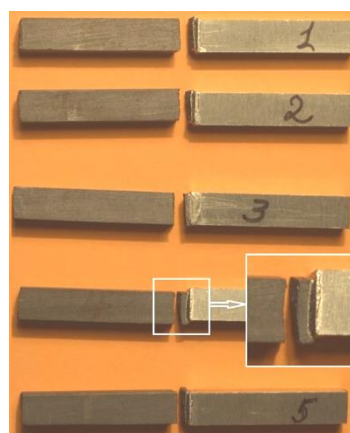

Figure. 12. *The appearance of graphite-molybdenum joints produced with Pd-Ni-Cr-Ge system brazing filler metal after mechanical tests.*

The study of the surface of fractures of C + Mo butt specimens after mechanical tests showed that when using brazing filler metals Cu-15Ni-20Ti and Zr-25Pd-5Mo   a large spread of strength values is observed and an inhomogeneous fracture surface is revealed on the fractures: partly along the brazing filler metals and partly along the graphite (Figure. 13, a).
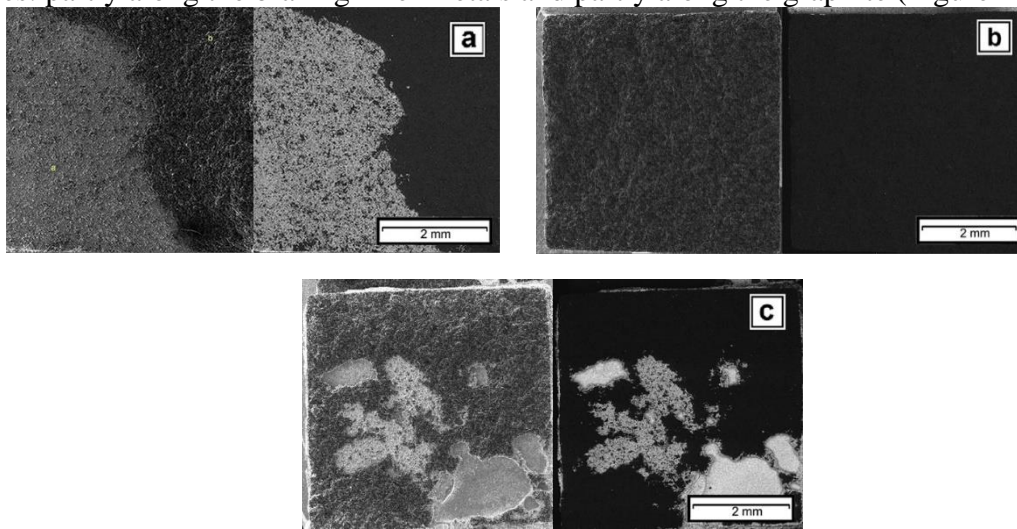

Figure. 13. *Fracture surface of C + Mo brazed joints obtained using brazing filler metal: Cu-15Ni-20Ti (a); Zr-25Pd-5Mo (b); Zr-25Pd (c).*

Brazing filler metals   Zr-Pd   и Pd-Ni-Cr-Ge provided stable strength results for brazed joints, and only carbon was detected on the fracture surface (Figure. 13, b). In practice, we obtained the strength of graphite after the thermal heating mode during brazing. When brazing with brazing filler metal Zr-25Pd (T= 1100 ºC/15 min), areas with different chemical compositions were found on the fracture surface: the constituent elements of the brazing filler metal and pure carbon, Mo, but the strength is at the level 36 MPa.

4. Conclusion

Obtained data of micro-X-ray spectral examination of graphite-molybdenum joints indicate the active interaction of the liquid brazing filler metal and the base material during brazing. This results in the formation of carbon-saturated compounds in the brazed seams. In some areas of graphite (close to seam), the brazing filler metal penetration into graphite also results in the formation of phases saturated with carbon (carbide).

Carbides of different metals are, respectively, formed in the brazed seams using different doping systems. Application of Ti-Cr-V and Cu-Ti-Ni system brazing filler metals provoke the formation of titanium carbides, Zr-Pd(Mo) – zirconium carbides, Pd-Ni-Cr-Ge system - CxMey(Mo, Cr) carbides.

Peculiarities of the formation of the microstructure of brazed joints are in good correspondence with the mechanical test data. Analysis of the results of three-point bending tests of the brazed joints showed the perspective of applying the Pd-Ni-Cr-Ge and Zr-Pd system brazing filler metal for vacuum brazing of dissimilar graphite-molybdenum joints, which provide full-strength (36 MPa) of the joints as well as stability of their production. It is reached due to the solidification of the solid solution based on nickel-palladium system (Mo) in the brazed seam and inclusions of complex carbide CxMey(Mo, Cr) precipitating in the plastic matrix (solid solution). The failure in mechanical tests occurs on the base metal (graphite).

Scientists are developing new types of carbon (composite) and graphite functional materials with improved performance for future high-temperature applications. Their use requires the development of methods for obtaining permanent joints in creating critical

combined structures from dissimilar materials. This is the reason for the further development of technological processes for high-temperature brazing of specific dissimilar materials and the development of appropriate brazing filler metals.

5. Acknowledgment

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