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BRAZING A GRAPHITE COMPOSITE TO MOLYBDENUM ALLOY TZM USING ACTIVE COPPER-BASED FILLER METALS WITH CHROMIUM ADDITIVE

LUTOWANIE TWARDE KOMPOZYTU GRAFITOWEGO ZE STOPEM MOLIBDENOWYM TZM AKTYWNYMI SPOIWAMI MIEDZIANYMI Z DODATKIEM CHROMU

The paper presents issues of brazing the graphite composite CFC 222 with the molybdenum alloy TZM. Both materials demonstrate significant differences in physicochemical and mechanical properties that significantly affect brazing conditions and properties of the brazed joints. The performed brazing operation was preceded by a wettability test that decided selection of the filler metal.

From among various copper-based filler metals, the best appeared a copper brazing filler metal with some addition of active chromium. Presented is a model of the wedge test, helpful at optimising the brazing process of two materials with different properties. Width of the brazing gap was selected on the ground of metallurgical examinations after the wedge test and transferred to the joint with a parallel gap. Applied were various forms of copper-based filler metals in that chromium was present as an alloying component, a component of the brazing paste, powder between copper covers and as a galvanic coating of a pure-copper strip.

Evaluation of brazed joints of the composite CFC 222 with the TZM alloy is presented on the grounds of metallographic examinations by means of light microscopy and microhardness measurements, electron microscopy, EDX analysis of elements and XRD analysis of phase composition of the reactive zone.

Keywords: graphite composite CFC 222, molybdenum alloy TZM, vacuum brazing, copper-based brazing filler metal Cu-Cr, wettability, wedge test, brazing gap, metallographic examinations, EDX and XRD analyses

W pracy przedstawiono problematykę lutowania twardego kompozytu grafitowego CFC 222 ze stopem molibdenowym TZM. Obydwa materiały wykazują znaczne różnice we właściwościach fizykochemicznych i mechanicznych, które mają istotny wpływ na warunki lutowania i właściwości uzyskanych połączeń lutowanych. Wykonanie połączeń poprzedziła próba zwilżalności, która zdecydowała o wyborze lutu.

Spośród różnych lutów na osnowie miedzi najlepszy okazał się lut miedziany z dodatkiem aktywnego chromu. Przedstawiono model próby klinowej, pomocny w optymalizacji procesu lutowania obydwu materiałów o zróżnicowanych właściwościach. Na podstawie próby klinowej, w wyniku badań metalograficznych, dobrano szerokość szczeliny lutowniczej i przeniesiono ją na złącze ze szczeliną równoległą. Stosowano różne postacie lutów miedzianych, w których chrom występował jako składnik stopowy, składnik pasty lutowniczej, proszek między okładkami miedzianymi i jako pokrycie galwaniczne taśmy z czystej miedzi.

Ocenę połączeń lutowanych kompozytu CFC 222 ze stopem molibdenowym TZM przedstawiono na podstawie badań metalograficznych za pomocą mikroskopii świetlnej i pomiarów mikrotwardości, mikroskopii elektronowej, analizy pierwiastków metodą EDX oraz analizy składu fazowego strefy reakcyjnej metodą dyfrakcji rentgenowskiej XRD.

1. Introduction

Graphite materials and graphite-based composites are classified to the group of advanced materials the most bonding-resistant. Their bonding is most often carried-out by adhesive bonding and vacuum brazing. At brazing, it is required to activate interfaces of materials [1-3] and to use active filler metals . Graphite materials demonstrate low density but their thermal conductivity is within the range of conductivity of metals (between unalloyed steel and aluminium). With temperature rise, mechanical strength of graphite increases and reaches the highest strength of all known materials in the range between 2000 and 2500°C [4]. Graphite-based composite materials demonstrate clearly higher mechanical properties than polycrystalline graphite [5]. In the modern

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technique, a high demand exists for joining graphite with metals in the case of large and complexly shaped parts. Graphite and its composites are widely applied in power industry, electrical engineering, machine building, metallurgy and aerospace industry, as well as in biomedical engineering. Figures 1 and 2 show exemplary applications of a graphite-based composite material in joints exposed to high-energy plasma in nuclear power engineering [6] and in rotary anodes of X-ray tubes [7].

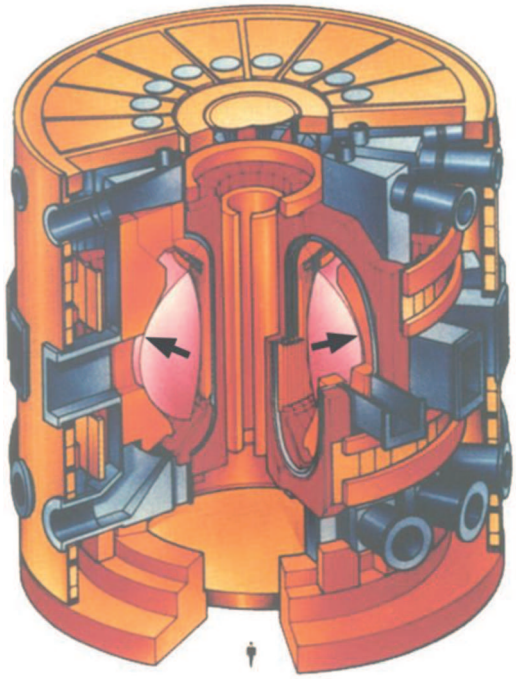

Fig. 1. Lay-out of a nuclear power plant. Arrows indicate the areas to be lined with CFC graphite [6]

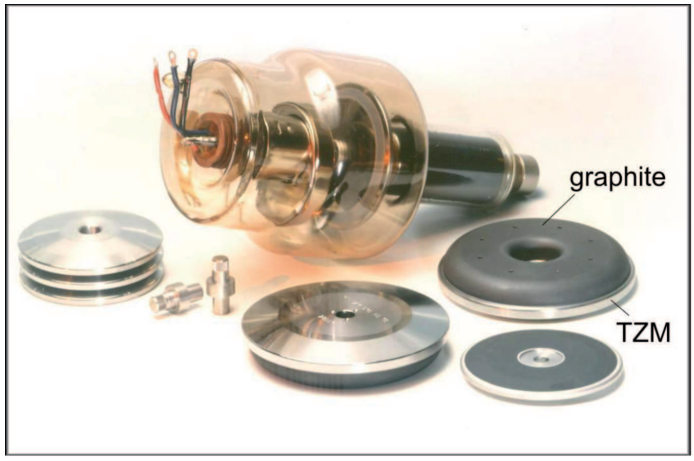

Fig. 2. Rotary anodes X-ray tubes made as joints of graphite-based composite and TZM alloy [7]

2. Brazed materials

Basic physicochemical and mechanical properties of the graphite composite CFC 222 and the molybdenum alloy TZM are shown in Table 1.

The composite CFC 222 is often described as "graphite reinforced with graphite" since both its matrix and reinforcing fibres are made of graphite. Content of fibres in CFC 222 is 55 to 65 vol.%. In the composite structure, present are numerous gaps and pores between individual bunches of graphite fibres, see Fig. 3.

TABLE 1

Basic physicochemical and mechanical properties of graphite composite CFC 222 and alloy TZM [8,9]

Material designation	Density ρ kg/m^3	Melting point T_t °C	Linear expansion coefficient α 1/K	Thermal conductivity λ W/m·K	Young's modulus E GPa	Unit elongation A %	Hardness HV
CFC 222	$1{,}6 \cdot 10^3$	3800 (decomposition)	$1{,}13 \cdot 10^{-6}$	40-130*	70-90*	0.2-0.4	221 HV 0.015
TZM	$10.15 \cdot 10^3$	2620	$5.3 \cdot 10^{-6}$	126	320	2-16.5**	200-340 HV 10**

* In parallel with graphite fibres
** Depending on thickness and kind of the semi-product

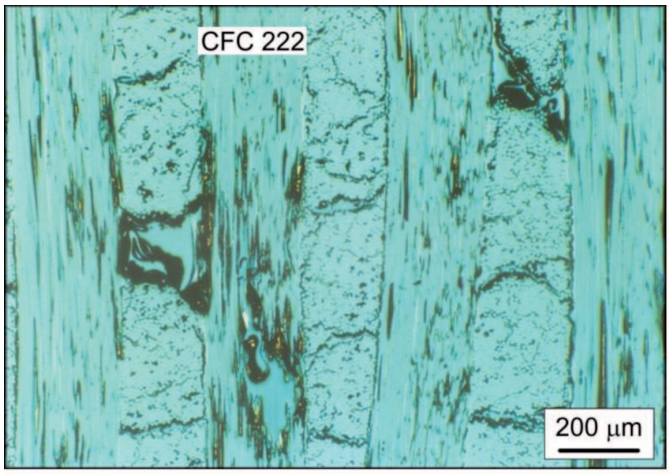

Fig. 3. Composite structure of the graphite CFC 222

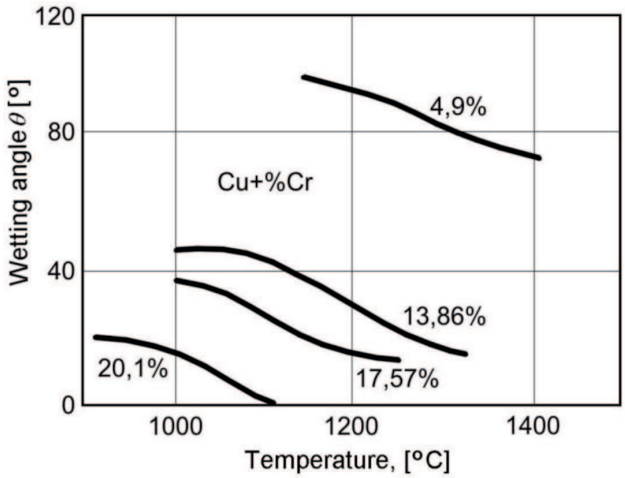

Fig. 4. Influence of active chromium in copper on wettability of graphite [12]

Porosity of the graphite composite reaches 10 vol.%. This heterogeneity and inconsistency of structure is favourable for elastic deformation of the composite during cooling-down a brazed joint. However, deformability of the composite CFC 222 is low, with unit elongation from 0.2 to 0.4% only [8]. Its properties are diverse and depend on the arrangement direction of graphite fibres. Thermal conductivity of this material reaches 130 W/m·K in parallel direction to the fibre arrangement direction and ranges between 5 and 30 W/m·K in perpendicular direction.

The TZM alloy is a high-temperature creep-resisting molybdenum-based alloy that includes also 0.5 wt.% Ti, 0.08 wt.% Zr and 0.01 to 0.04 wt.% C [9]. It is characterised by high recrystallisation temperature (1480°C), high resistance to thermal shocks and good weldability. Thanks to these advantages, the TZM alloy is used for parts working at high temperatures, like guiding nozzles for copper drawing, electrodes for resistance welding or for bolts and nuts in heat-treating furnaces.

3. Brazability of graphite materials

Like ceramic materials [10,11], graphite materials demonstrate susceptibility to brazing, first of all by means of active filler metals. In the case of graphite, these brazing alloys should contain carbide-forming additions of chromium, titanium, silicon and zirconium. Activity of these metals, necessary for wetting graphite with a liquid brazing filler metals, decreases in the given sequence. Chromium shows the strongest influence as an alloying addition to copper in percentages above 20 wt.%, see Fig. 4.

Selection of active components should also consider their influence on deformability of the filler metal. The mentioned additives lead to strengthening the solid solution within the solubility limits and after exceeding a limit to precipitating intermetallic phases [4]. This increases hardness and decreases deformability of the alloy. Application of pure copper for brazing does not guarantee wetting of graphite, because in any case:

- no carbide phase is formed on the interface graphite-braze; this concerns metals of IV, V and VI group of the periodic table plus boron,
- carbon does not dissolve in pure copper; this concerns metals of VIII group and some metals of VII group of the periodic table,
- copper atoms do not diffuse into the graphite lattice; this concerns alkali metals and boron.

4. Tests of wetting graphite with active brazing filler metals

For graphite wettability testing, selected were copper-based filler metals with active additives: chromium, titanium and zirconium. The brazing filler metals were designated as CuCr1, CuCr1Ti CuCr1Zr and CuCr1ZrTi. Content of chromium as an alloying component in the brazing alloys was ca. 1 wt.% and content of zirconium was 0.08 wt.%. The filler metals CuCr1 and CuCr1Zr were usually applied in form of a strip 2×0.1 mm thick. The filler metals CuCr1Ti and CuCr1ZrTi were applied in form of a set of two strips CuCr1 and CuCr1Zr with a 0.015 mm thick titanium foil placed between them.

Tests of graphite wettability with copper brazing filler metals were carried-out in a vacuum furnace made by Balzers. The applied vacuum was not lower than 10^{-4} mbar.

The best results were obtained for the brazing alloy CuCr1 with its melting point below that of pure copper, close to the eutectic temperature of 1077°C (eutectic mixture Cu-Cr containing 1.56 at.% or 1.28 wt.% Cr [13]). Microstructure of the brazing alloy CuCr1 in cast condition is shown in Fig. 5.

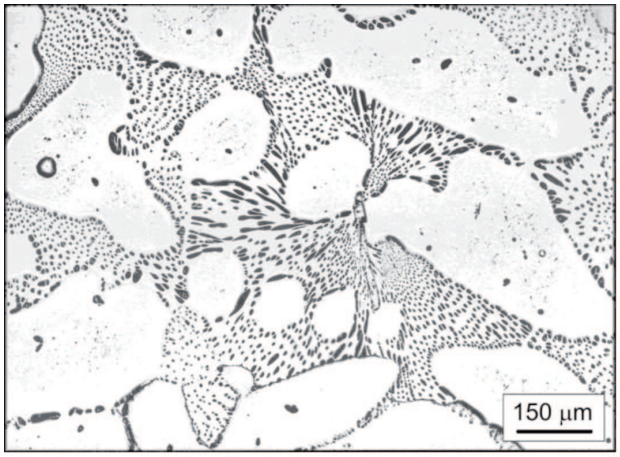

Fig. 5. Microstructure of the brazing alloy CuCr1 in cast condition

The clearly visible dark particles are precipitates of chromium in the eutectic mixture. Copper and chromium do not create any intermetallic phases. Solubility limit of chromium in copper at the eutectic temperature is 0.65 at.% [14]. It declines with temperature and at 400°C is less than 0.03 at.%, so chromium can precipitate from supersaturated solid solution.

The copper matrix of the brazing alloy is soft, with its hardness of 71 HV 0.015, so it demonstrates high plastic deformability.

Wetting angle between graphite and the brazing filler metal CuCr1 used as a strip was 20° at 1150°C for brazing time of 5 min. The angle for the TZM substrate in the same conditions was 25°. Wetting a substrate with a filler metal is considered good when the angle is less than 30° [10,11,15,16]. The brazing alloy CuCr1 showed good adherence to graphite, in contrast to the other brazing filler metals that demonstrated similar wetting angles but no adherence to the graphite substrate.

Figure 6 shows two drops of CuCr1 marked *A* and *B* on the graphite substrate, mounted in resin.

Microscopic observations were carried-out using a light microscope Polyvar-Met made by Reichert-Jung Optische Werke AG. To reveal structural components of the brazed joints, they were vacuum deposited with a thin layer of ZnSe. The small drop of the brazing alloy, marked *A* in Fig. 6, probably splashed from liquid brazing alloy at final phase of the wetting test. The wetting angle is here ca. 97°, so wetting the graphite is unsatisfactory. The small brazing alloy drop does not show any reactive zone on the interface with graphite that would permit indirect wetting with liquid brazing alloy. Chemical composition of the reactive zone was determined by EDX analysis using an X-ray microanalyser coupled with a scanning microscope Can Scan Sydu 86 made by Elektronen-Optik. Chromium content revealed in the matrix of the small brazing alloy drop was below 0.5 wt.% (0.31 and 0.4 wt.% depending on the location). This concentration of active chromium is probably too low for correct wetting. In the larger solder drop *B*, attention should be paid to the clear reactive zone ca. 1 to 2 μm wide, on the boundary with graphite. In this zone, chromium diffusing from liquid brazing alloy connects with carbon from the graphite composite, creating a carbide phase. This zone is hard (1631 HV 0.015), so it can not be too wide, because it would be then susceptible to brittle cracking. The EDX analysis revealed in this zone over 90 wt.% Cr. The XRD analysis of phase composition in this zone was performed using an X-ray diffractometer URD-6 made by Carl Zeiss Jena and radiation of a copper X-ray tube with 0.15405 nm wavelength, filtered by nickel foil. The XRD analysis revealed in this zone first of all chromium carbides Cr_3C_2, and next the carbides Cr_7C_3, $Cr_{23}C_6$ and Cr_2C.

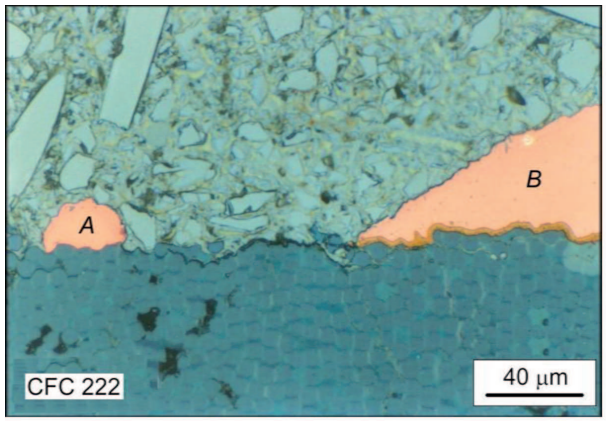

Fig. 6. Substrate of graphite composite CFC 222 wetted with two drops of CuCr1 brazing alloy

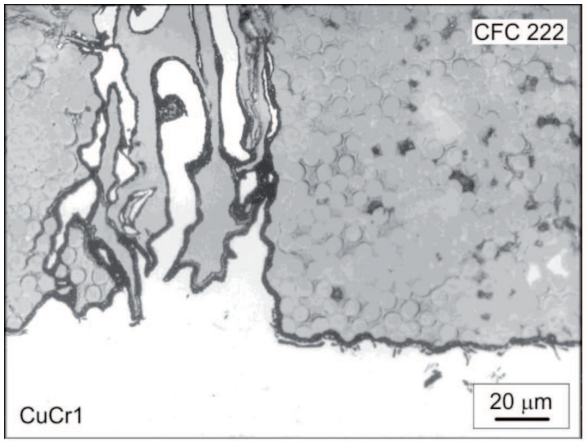

Fig. 7. Capillary action of the brazing alloy CuCr1 in CFC 222 structure

The brazing filler metal CuCr1 is capillarly drawn into narrow gaps in the graphite structure, climbing between the fibres to the height reaching 0.5 mm, see Fig. 7. This leads to higher mechanical load capacity of the brazed joint.

5. Selection of brazing gap in the wedge test

The brazing gap was selected on the grounds of the wedge test model for flat specimens, as shown in Fig. 8.

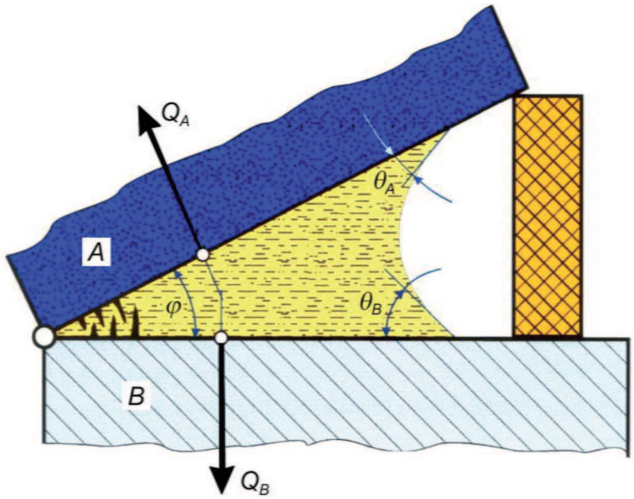

Fig. 8. Wedge test model at brazing dissimilar materials A and B

The wedge test plays a special role in examining physicochemical phenomena occurring during brazing dissimilar materials A and B [16,17]. On its grounds, it is possible not only to evaluate wettability (angles θ_A and θ_B) and capillarity of the brazing filler metal, but also to observe results of the crystallisation process of hard phases present in the brazed joint. Application of the wedge test is important mainly at brazing dissimilar materials with significantly different physicochemical properties.

The heat fluxes Q_A and Q_B are carried-away from liquid brazing alloy included in the gap with divergence angle φ perpendicularly to the surface of the materials creating the wedge gap. This is accompanied by crystallisation of the joint material. Columnar crystals can originate in the direction parallel to that of heat abstraction, especially on the side of the material with higher thermal conductivity. More intensive heat abstraction and also earlier solidification of liquid brazing alloy takes place in the area with smaller volume of the joint, at the gap top. In a small volume of the brazing alloy, crystal nuclei originate almost simultaneously, usually in the places with lattice defects. Flux density of the diffusing atoms is higher in a narrower than in a wider gap , which can be conductive to creating hard intermetallic phases with participation of active brazing alloy components. Different shrinkage of the brazed materials can easily lead to cracks of the joint in the area of narrow brazing gap. Tensile shrinkage stresses usually lead to cracks and delaminations in the material with lower deformability and lower thermal expansion.

Wider brazing gap results in longer diffusion length for the atoms migrating between both brazed materials and does not ensure high overcooling necessary for disequilibrium crystallisation. Creation of hard and brittle intermetallic phases in the joint can be then difficult. If the phases originate yet, they can be isolated in the soft matrix of the braze. Larger volume of plastic braze material permits greater stress compensation by shrinkage of the joint.

Evaluation of crystalline structure of a joint after the wedge test permits also selecting a filler metal and a suitable method of activating surfaces of the materials to be brazed.

The method and conditions of brazing significantly affect results of the wedge test. Diversified heating and cooling conditions of a brazed joint significantly influence its crystalline structure. In evaluation of a braze structure, a great role play metallographic examinations using light and electron microscopy, as well as microhardness measurements. They facilitate analysis of reactive zones and intermetallic precipitates in the joint structure.

The brazed materials, i.e. graphite composite CFC 222 and molybdenum alloy TZM, are characterised by over 4-fold difference in linear expansion coefficients. This requires using a solder with high plastic deformability in a properly wide brazing gap.

For vacuum brazing, the specimens of CFC 222 were preliminarily ground on abrasive papers (up to No. 400) and degreased in alcohol bath, in an ultrasonic washer.

The TZM alloy was pickled in the reagent containing 95 ml HCl and 5 ml HNO_3 for 0.5 min at ambient temperature. The brazed materials were loaded with 360-g Al_2O_3 plates (ca. 0.1 MPa). Strips of the filler metal CuCr1 were placed between the composite and the TZM alloy. Conditions of the wedge test for the examined joint were analogous as in the case of the wettability test.

A brazed joint with a wedge-shaped slit was prepared, of that cut-out were specimens with brazing gaps 22, 70, 100 and 160 μm wide, shown in Fig. 9.

Microstructures of individual brazed joints with specified brazing gaps are shown in Fig. 10.

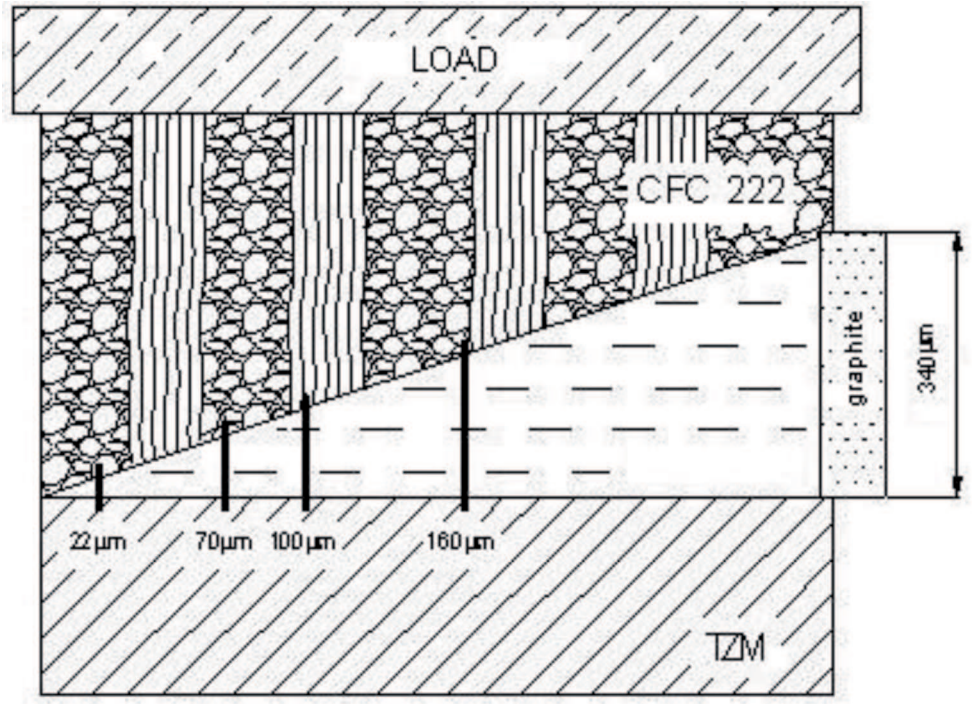

Fig. 9. Locations of cutting-out specimens from the brazed joint in the wedge test

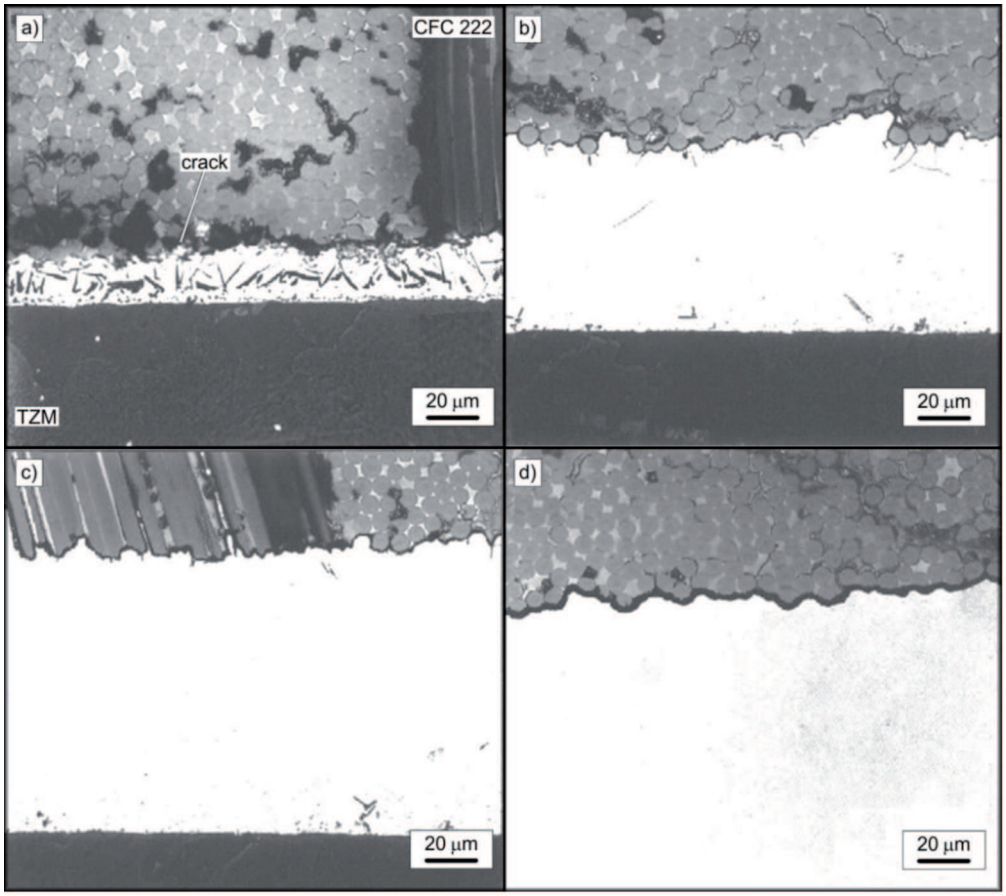

Fig. 10. Microstructures of brazed joints of composite CFC 222 with TZM alloy with various widths of brazing gaps: 22 μm (a), 70 μm (b), 100 μm (c) and 160 μm (d)

The narrow, 22 μm wide gap is conductive to creating cracks and delaminations in graphite, i.e. in the material with lower plasticity than that of the TZM alloy. The reactive zone here is narrow and amounts to ca. 0.5 μm. Acicular precipitates appear in the braze, containing ca. 90 wt.% of molybdenum. The acicular precipitates hardening the joint gradually disappear at wider brazing gap and at a 70 μm wide gap are no longer present, see Fig. 9b. No cracks were observed in the graphite zone adjacent to the joint. With increasing width, more and more visible became the reactive zone on the interface braze-graphite. At the slit 100 μm wide, width of this zone is approximately 2 μm. The greater the braze thickness, the wider is the reactive zone. It is well visible at 160 μm wide slit (Fig. 9d) and rises with increasing width. At the slit 320 μm wide in the wedge test, its width reaches ca. 5 μm. Because of its significant hardness (1631 HV 0.015) and resulting brittleness, a wide brazing gap is unfavourable and undesirable.

6. Metallographic examinations of brazed joints

The performed wedge test permits, on the ground of the joint structure analysis, selecting the gap width within 100 to 130 μm. Width of the gap was transferred to the parallel joint. Brazing surface of the specimens 12 mm thick was 12×25 mm. Bodies of the TZM alloy were shaped as rectangular prisms 12×12×25 mm or tubes dia. 40 mm wall thickness 10 mm with the surfaces faced for brazing, see Fig. 11.

A controlled width of the brazing gap was obtained by means of 100 μm thick spacers made of vanadium, niobium or graphite.

The copper-based filler metals with chromium were applied in the following form:

- CuCr1 strip, with total thickness of 0.2 mm,
- Cu strip 0.2 mm thick with electroplated chromium layer,
- Brazing paste being a mixture of Cu and Cr powders joined by a bonding agent, with Cr content 1, 2 and 5 wt.%,
- Cr powder placed between covers of pure copper, each 0.1 mm thick.

Figure 12 shows a brazed joint of the composite CFC 222 with the molybdenum alloy TZM, made with use of a CuCr1 strip 0.2 mm thick and niobium spacers 0.1 mm thick.

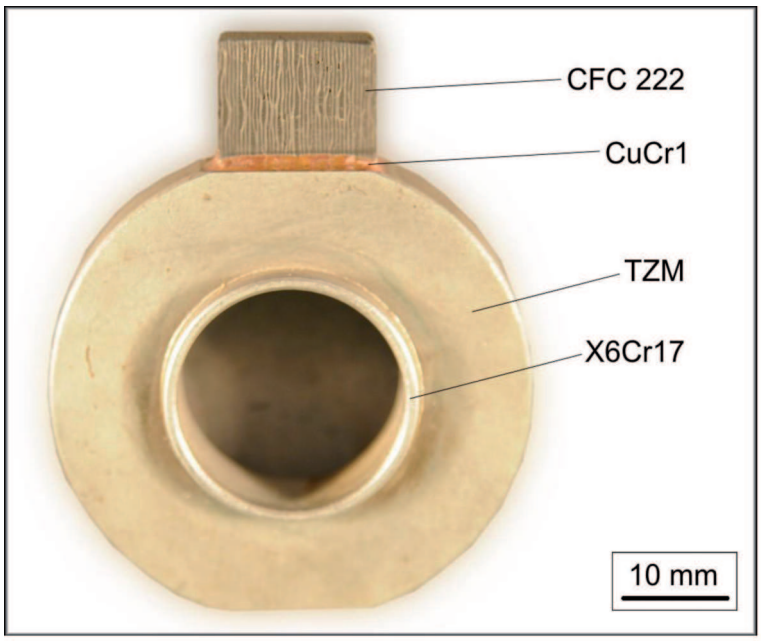

Fig. 11. View of a brazed joint CFC 222-TZM-stainless steel X6Cr17

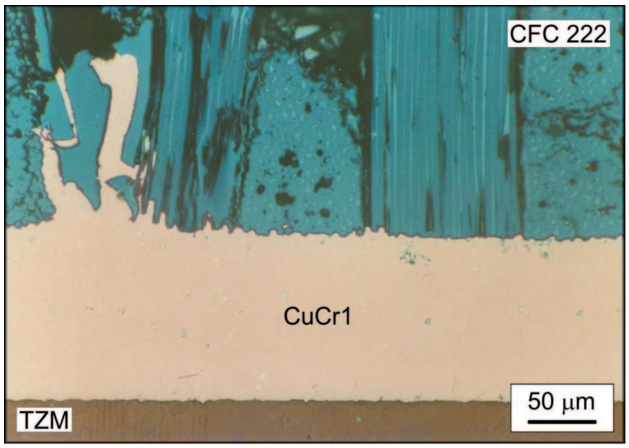

Fig. 12. Brazed joint CFC 222-TZM made with CuCr1 brazing filler metal at controlled slit width of 130 μm

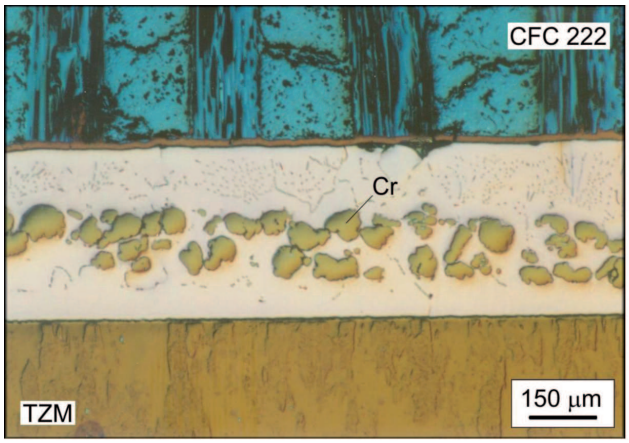

Fig. 13. Microstructure of brazed joint CFC 222-TZM made using two pure copper strips with a layer of Cr powder between them

As a result, a correct brazed joint was obtained, with a gap ca. 130 μm wide. Width of the reactive zone in the joint reached 2 μm. In the zone containing carbide phases, mainly Cr_3C_2, identified was the composition: 96.76 to 98.40 wt.% Cr, 0.85 to 2.83 wt.% Cu and 0.34 to 0.42 wt.% Mo (no carbon analysis).

A brazing paste consisting of Cu and Cr powders joined with a bonding agent was also used for brazing. Granularity of the applied metallic powders was below 150 μm. Chromium content in the paste was changed from 0 through 1 and to 5 wt.%. Using exclusively pure copper powder, with no active chromium, no wettability of graphite was obtained. With 1% Cr in the paste, obtained was the reactive zone nearly 2 μm wide. With 5% Cr, at constant gap 130 μm wide, width of the reactive zone increased and reached ca. 11 μm. In so thick reactive zone, cracks appeared during shrinkage of the joint. Results of brazing the material pair CFC 222-TZM indicate that 1 wt.% Cr is sufficient to obtain a correct brazed joint at satisfactory wettability of graphite. This Cr content is much smaller than that used in the experiments whose results were shown in Fig. 4 above. Correct wetting of graphite, determined by the wetting angle lesser than 30°, occurred there when Cr content alloyed with copper exceeded 20 wt.% [12].

Similar results of brazing as for CuCr1 strip were obtained using a pure copper strip electroplated with chromium. Thickness of Cr coating was so selected that, with use of a copper strip 0.2 mm thick, chromium content amounted to 1.0 wt.%. In this case, thickness of Cr coating reached 2.0 μm.

Successive brazing tests were carried-out using active chromium in form of powder with granulation below 150 μm placed between two strips of pure copper, each 0.1 mm thick. The joint, obtained at correct graphite wetting, is shown in Fig. 13.

In the central part of the joint, visible are large chromium particles with hardness 201 HV 0.015 that were not completely dissolved in copper during brazing. The fact that chromium dissolves in copper is confirmed by frayed and developed grain boundaries of Cr particles. Together with the filler metal matrix, they create a composite structure, which results in unfavourable hardening of the braze. In this case, the used percentage of chromium was too high. The braze matrix includes also tiny chromium particles created as a result of precipitation processes related to decreasing solubility of chromium in copper.

7. Conclusions

- The wettability test of the graphite composite CFC 222 permits selecting copper-based filler metal with 1 wt.% of active chromium for its vacuum brazing with molybdenum alloy TZM.
- The wedge test permits evaluating physicochemical phenomena occurring at brazing materials with diverse physicochemical properties.
- On the grounds of metallographic analysis of a braze structure in the wedge gap, possible is determining a favourable microstructure of the joint, with no hard phases and precipitates, and transferring the selected brazing gap to a parallel joint.
- Brazing dissimilar materials CFC 222 and TZM on the grounds of wedge test analysis leads to selecting a wider brazing gap, 0.1 to 0.13 mm wide. In narrower gaps, disadvantageous joint structure happens, as well as cracks and delaminations of graphite.
- The CFC 222 graphite is satisfactorily wetted by copper-based filler metals with 1 wt.% of active chromium by intermediate wetting the reactive zone containing hard carbide phases, first of all chromium carbide Cr_3C_2. Higher chromium content in

copper-based filler metal leads to a wider hard reactive zone at the interface braze-graphite and to its cracking.

- Chromium, as a component of copper-based filler metals designed for brazing dissimilar materials like graphite composite CFC 222 with molybdenum alloy TZM, can be used not only as an alloying element, but also as a component of a brazing paste or as electroplated coating on a pure copper strip.

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