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Interfacial microstructure and mechanical properties of tungsten carbide brazed joints using Ag−Cu−Zn + Ni/Mn filler alloy

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Abstract: WC−Co hard metal was furnace brazed by Ag−Cu−Zn+Ni/Mn filler alloy using a tube furnace under high-purity argon at 730 °C. The influence of brazing time and gap size of joints was studied. The results revealed the maximum shear strength of (156±7) MPa for samples with 150 μm gap size at a holding time 15 min. The characterization and microstructure of the brazed joints were characterized by SEM, EDS and XRD. The results showed that increasing the time from 5 to 15 min could provide a better chance for the liquid interlayer to flow towards the base metal. However, the formation of some metallic phases such as Mn_3W_3C at brazing time longer than 15 min resulted in decreased shear strength of the joint.

Key words: brazing; tungsten carbide; Ag alloy; filler alloy; microstructure; wetting properties; shear strength

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1 Introduction

The use of cemented tungsten carbide (WC) as a composite material has grown year by year. Excellent wear resistance with proper toughness makes WC appropriate for many applications such as lathe tools, wire drawing dies, and press tools, but despite its expensiveness, it has better performance compared with steel tools. One of the wide applications of tungsten carbide is its use as a tipping material for rock drilling, shaping tools, and for high duty cutting blades [1,2]. Earth boring drill bit bodies are fabricated from either steel or a hard metal matrix material. The matrix materials are usually a composite of ceramic particles like tungsten carbide or titanium carbide infiltrated with a proper binder metal. In this unique infiltration process, the carbide is mixed with a binder metal before it is introduced into the mold. Cobalt is a common binder metal, but nickel and iron are infrequently used [3,4]. The hard WC grains and Co binder as a ductile phase possess good compatibility; therefore, the WC−Co has unique and superior mechanical properties such as hardness, high modulus and wear resistance. These properties make WC−Co cermets to be one of the most widely-used inserts in rock drilling tools as well as metal cutting tools and industrial wear resistant parts like dies, punches, and seals [1,5,6].

In recent years, various bonding technologies such as adhesive gluing, solid state bonding, brazing and welding have been developed to join cemented carbides together or to a base metal. Among these joining methods, brazing has received great attention due to its simplicity, perfect adaptability of part size and shape and high joint strength as well as good repetitiveness and low cost [7,8]. Tungsten carbide is a sintered product usually with cobalt, as the binder, which cements the particles of tungsten carbide together. WC−Co products with different properties are obtained by varying the proportions of WC and Co existed in the material [9].

Wetting of brazed joints is the primary role of any brazing filler alloy to form a joint perfect enough to withstand loads on duty. In a brazing process, the filler alloy is heated above its melting point and flows through the parent material parts due to its capillary properties. Before cooling and formation of solid joints, the molten filler alloy should wet the surfaces of the close-fitting parts [10]. The brazing filler alloys have a further function to perform in WC brazing. The ability of an alloy to wet and bond to tungsten carbide is a function of the composition of the tungsten carbide such as the cobalt content and level of any other metallic carbide added to its composition [11,12]. Although the contact angle test has commonly been used for a smooth and

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chemically homogeneous surface, achieving acceptable superficial wettability in industry is not usually attainable due to the chemical heterogeneity and roughness. Moreover, the wettability of solids with fine dispersion can be a function of various parameters like molecular interactions among active phases in the brazing zone as well as particle size and porosity of the powder bed [13]. There are two general approaches for improving the wettability of cermet; these include the choice of a proper chemical composition enjoying the ability to interact with the filler alloy and surface preparation to induce bonding [14].

In this work, Ag−Cu−Ni−Mn−Zn, as a commercial filler alloy, was applied to joining the two parts of WC−Co cemented carbide together. The main reason for the selection of silver-based brazing alloy was due to its good compatibility with ceramics and uniform compositional variation during the crystallization process [15,16]. It has been reported that the wetting properties of tungsten carbide are significantly reduced when titanium and/or tantalum carbides are present in the product [11]. Recent research works have shown that the presence of nickel in a brazing material provides enhanced wetting of the alloy onto cemented carbide. It is believed that this is due to the close similarity of the physical and chemical properties of nickel and the cobalt as cementing element [12]. As a result of this similarity, these metals demonstrate higher solubility compared with others and the phenomenon of wetting is inextricably bound up with the dissolution of the parent metal by the filler material. It is also believed that the manganese reacts with the free carbon to produce particles of manganese carbide which subsequently is wetted by the molten brazing alloy [17]. The presence of active elements in brazing zone can result in a remarkable chemical reaction in addition to the appearance of new solid compounds which can decrease the system free energy and the wetting angle [8,18]. In this work, the effect of two distinct variables including brazing time and filler alloy thickness have also been considered and their effects on the strength of microstructure of joints have been clearly discussed.

2 Experimental

2.1 Materials

The tungsten carbide cobalt used for the experiments was a commercial one called UG8. UG8 is commonly applied to the body of drilling tools due to its higher fracture toughness that is mainly related to its mass fraction of cobalt in comparison with other drilling tools containing Fe and Ni [19]. The chemical composition of UG8 is listed in Table 1. The Argo-braze 49H filler foil material with a liquidus temperature of 680 °C and solidus temperature of 705 °C obtained from Johnson Matthey Metal Joining Company was applied for brazing tungsten carbide parts. Its composition is listed in Table 2. A Tenacity 6 flux (EN1045 standard in FH12 group) made by Johnson Matthey Metal Joining Company was used in the brazing process. The brown flux powder mixed with warm distilled water in a mass ratio of 2:1 was placed on the surface area of the joint.

Table 1 Chemical composition of tungsten carbide cermet (mass fraction, %)

W	C	Co	Ti	Si	Fe	Cr
75.8	5.87	9.2	8.63	<0.3	<0.2	<0.1

Table 2 Chemical composition of Argo-braze 49H (BAg22) [19] (mass fraction, %)

Ag	Cu	Zn	Ni	Mn
49	16	23	4.5	7.5

2.2 Experimental techniques

A schematic illustration of the brazing specimens is shown in Fig. 1. As seen, the as-received WC−Co was cut into cylindrical samples with a diameter of 12 mm and a height of 150 mm for shear strength test and cubic samples with dimensions of 8 mm × 8 mm × 12 mm and used for microscopic observation. As it is seen from Fig. 1, the gap sizes were controlled by edges, which were created using wire cut in the top of cubic and cylindrical shaped samples. Operation temperature of the flux is in the range of 550−800 °C, using a tube furnace with an internal diameter of 35 mm and a external diameter of 45 mm.

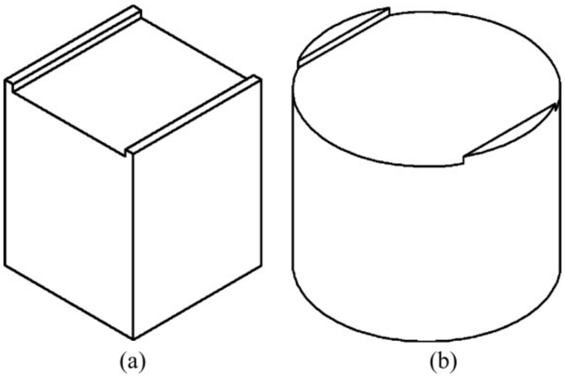

Fig. 1 Schematic illustration of brazing cubic (a) and cylindrical (b) specimens

The bonding surfaces were ground by SiC abrasive papers and then polished. After mechanical preparations, surfaces were rinsed with methanol and then put in 6 mol/L HCl for 20 s. Later, the samples were washed in water thoroughly. Prior to assembling, the specimen was degreased and cleaned with acetone in an ultrasonic bath. Then, flux paste was rubbed onto the joint surfaces to deactivate surface oxidation. The WC−Co parts with a filler alloy foil in between were placed in designed graphite fixtures. The assembly was then placed into a tube furnace under high-purity argon atmosphere by using a sealed push rod system which led the fixtures into the hot zone of the furnace. The heating program used was as follows. The furnace temperature was raised from 25 up to 730 °C via a heating rate of 15 °C/min and was kept at this temperature for a predetermined time for brazing. At the end of the brazing cycle, the fixtures were pulled out of the furnace and cooled to room temperature under a protective atmosphere.

In order to study the influence of brazing time, the process was carried out at 730 °C with a filler alloy thickness of 250 μm for 5, 10, 15, 20 and 30 min. Afterwards, the samples with an optimum brazing time were brazed at 730 °C with different filler alloy thicknesses (i.e., 50, 150, 250, and 400 μm). The shear strength of the brazed joints was measured by a new and specially designed fixture (Fig. 2) and by a universal electronic testing machine. Here, the tensile testing fixture consisted of two carbon steel parts fixed by a stud and bolt, so that can slide over each other in the longitudinal axis. The cylindrical tensile test specimens could be placed in a 10 mm-diameter hole created on each sliding parts of the fixture. These holes are 20 mm in depth, which is 5 mm longer than the height of cylindrical samples. At least, three samples were used for each condition to determine the mechanical properties and microstructure of the brazing joints.

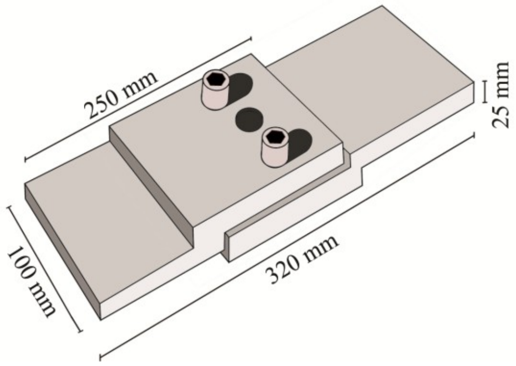

Fig. 2 Designed fixture of shearing strength test

The cross sections of the joints were studied by the field emission scanning electron microscope (FE-SEM) (Mira3, TEScan Company of Czech Republic) equipped with an energy dispersive spectrometer (EDS). Phase identification at the joint interface was also studied by X-ray diffraction (Philips X’Pert Pro) equipped with Cu K$_\alpha$ lamp. The XRD tests were taken from the filler alloy and WC−Co region of fracture surfaces of cylindrical specimens.

3 Results and discussion

3.1 Microstructure

To study the phase changes after brazing, it is required to know the microstructure of materials before the brazing process. The SEM images of WC−Co base material and Ag−Cu−Zn + Ni/Mn (BAg22) filler alloy before brazing are illustrated in Figs. 3 and 4, respectively. The studied cemented carbide is composed of WC and TiC grains in the cobalt binder (Fig. 3). Under high temperatures, the precipitation of fine particles of TiC would prevent the WC grains from enlarging and result in higher strength and hardness of the hard metal [20]. Three distinct phases have been assigned to the filler alloy microstructure in Fig. 4. Except the light-gray phase rich in silver, there are two other regions observed in the microstructure: 1) the scattered dark-gray path rich in copper on the plane rich in silver phase and 2) dark-gray spot phase rich in Ni−Mn (R1-5b/R2-2).

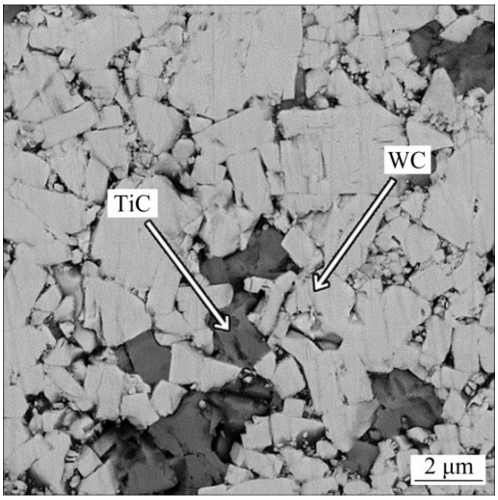

Fig. 3 SEM image of cemented carbide

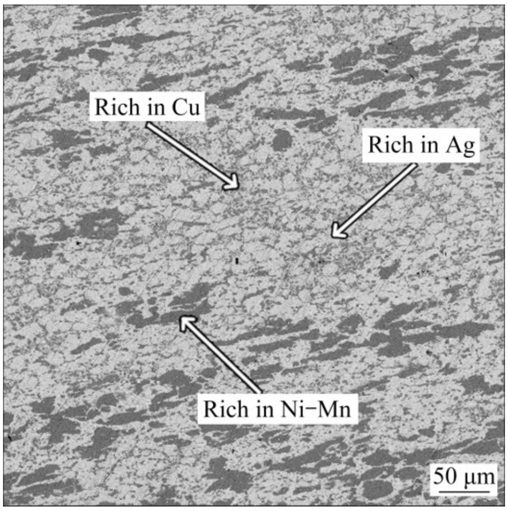

Fig. 4 Phases distinct in SEM image of BAg22 filler alloy

In the next step, the distinct zones in different phases (i.e., rich in silver phase, copper phase and Ni−Mn phase) were analyzed by EDS to provide more information about the composition of three phases and the results are shown in Fig. 5. Higher amounts of zinc are distinguished in the light background of the filler alloy which can refer to the solubility of Zn in silver. The BAg22 filler alloy melts and flows through the adjacent brazing surfaces when its temperature reaches the melting point. As expected, the melted filler alloy wets the surfaces of the substrate with no residual flux. According to the SEM image of Fig. 6, sound joints were obtained without any voids or cracks along the brazing surfaces for most of the samples brazed under the study conditions, revealing the appropriate wettability on the brazing surfaces. Although the brazing filler alloy could melt at the brazing temperature of 730 °C that is higher than the solidus and liquidus temperature of the filler alloy, evaporation of Zn in the braze alloy might cause pores in the bond, mainly due to low melting and boiling points of zinc [21].

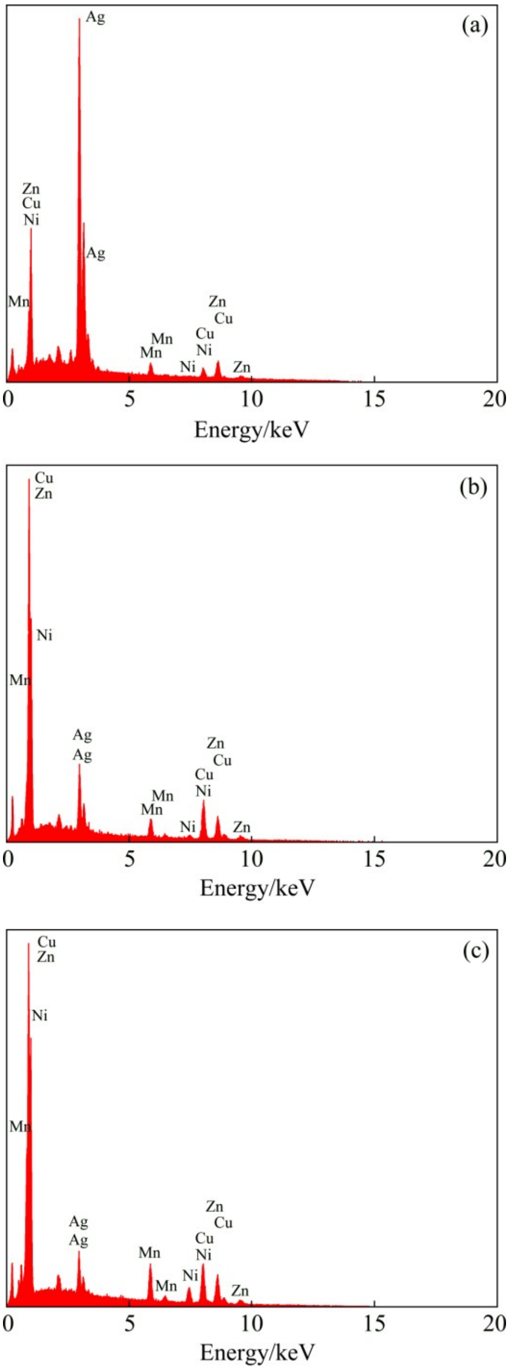

Fig. 5 EDS analysis results of pointed phases in Fig. 4: (a) Rich in silver phase; (b) Rich in copper phase; (c) Rich in Ni−Mn phase

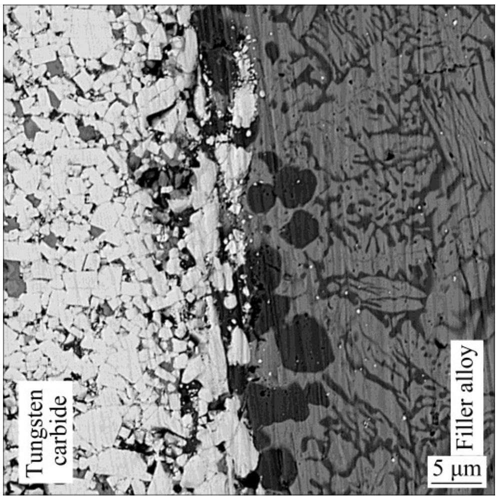

Fig. 6 SEM image of joints brazed at 730 °C held for 15 min with filler alloy thickness of 250 μm

As a result of discontinuity within the brazed joints, the cavities are known to reduce the strength. It can be inferred from Fig. 7 that with increasing the brazing time from 20 to 30 min, some porosities would appear in the brazed zone. The number of cavities can result in premature cracking and reduced strength of the brazed joints. It is noted that the joint strength is not only influenced by porosities but also by the joint microstructure.

The wettability contact angle defines the hydrophobic characteristics of solid surfaces. In fact, a liquid drop places on the surface in such a way that it minimizes the free energy of the system [13]. Figure 8 shows the average contact angle of BAg22 on W matrixes with three specimens. Obviously, the acute contact angle of BAg22 on W matrix is (28±3)°.

As is reported in Ref. [22], in brazing process, the base metal surfaces may dissolute by the molten filler alloy, bringing about changes in their chemical compositions. More time would provide a more efficient flow of the molten filler alloy into the WC particles. Subsequently, by increasing the brazing time, atoms are allowed to diffuse across the interface, so that the WC grain boundaries will be surrounded by the filler alloy. This can be attributed to the absence of the cobalt

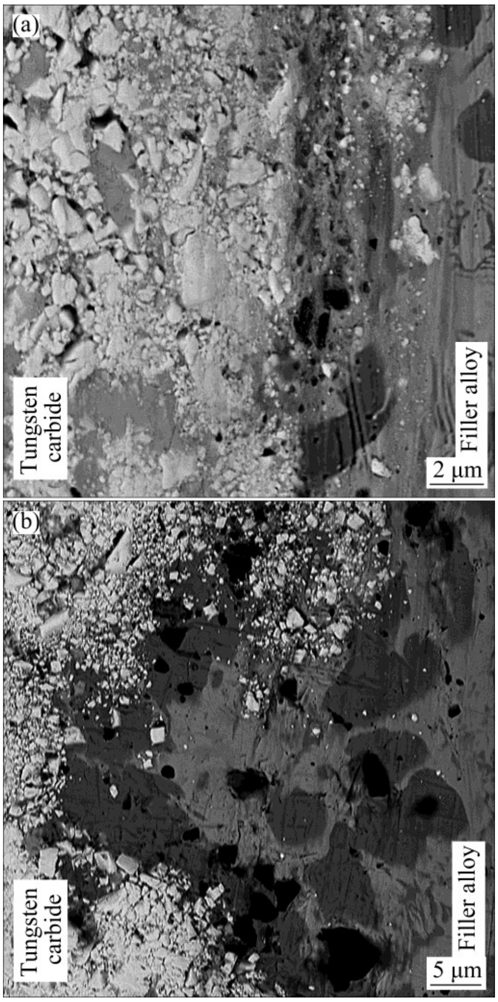

Fig. 7 SEM images of joints brazed at 730 °C with filler alloy of 250 μm in thickness for 20 min (a) and 30 min (b)

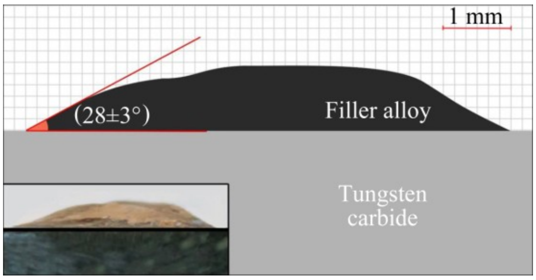

Fig. 8 Contact angle of wetting specimens brazed at 730 °C for 15 min with filler alloy of 150 μm in thickness

binder on the cermet surface (Fig. 7). Indeed, in the cleaning process of the substrate, hydrochloric acid washed away the cermet binder and provided suitable condition for filler alloy to flow through the brazed surfaces. Therefore, one of the main reasons for increasing the strength by holding time can be referred to diffusion.

Actually, as time passes, atoms would have enough time to diffuse across the interface. One of the main elements in improving wettability of cermet surfaces by filler alloy is Ni, which can promote both the wettability and plasticity. Moreover, this element also possesses good solubility in Fe and Co [23]. Thus, due to similar physical and chemical properties with Ni and Co [24], Ni would be a potential candidate for brazing cemented carbide with cobalt binder. Thereby, Ni would revive the properties of the depleted Co, removed during surface cleaning by acid. As can be clearly seen in Fig. 9 as well as in Fig. 6, the dark phases rich in Ni and Mn migrate towards the interface and aggregate there, after brazing. These phases are able to reduce the stress caused by cooling and also make a stiffer joint [23].

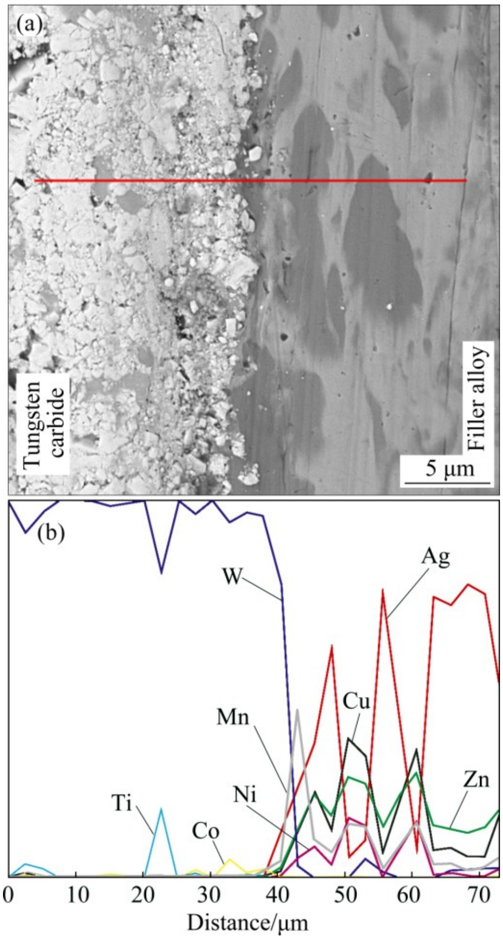

Fig. 9 SEM image (a) and EDS line scanning (b) of joint brazed at 730 °C for 15 min with filler alloy of 150 μm in thickness

Diffusion of elements between the filler material and the cermet substrate is also clearly distinguishable in all brazed samples. As it is seen from Fig. 10, there is a thin diffusive layer devoid of Ti next to the interface of WC−Co/filler alloy in the WC−Co cermet. Here, the thickness of the depleted titanium layer increases with increasing holding time, and hence, it provides more chances for a better diffusion of Ti. It has been reported that TiC, as a low stability phase, is one of the most important components in WC−Co base metals [23].

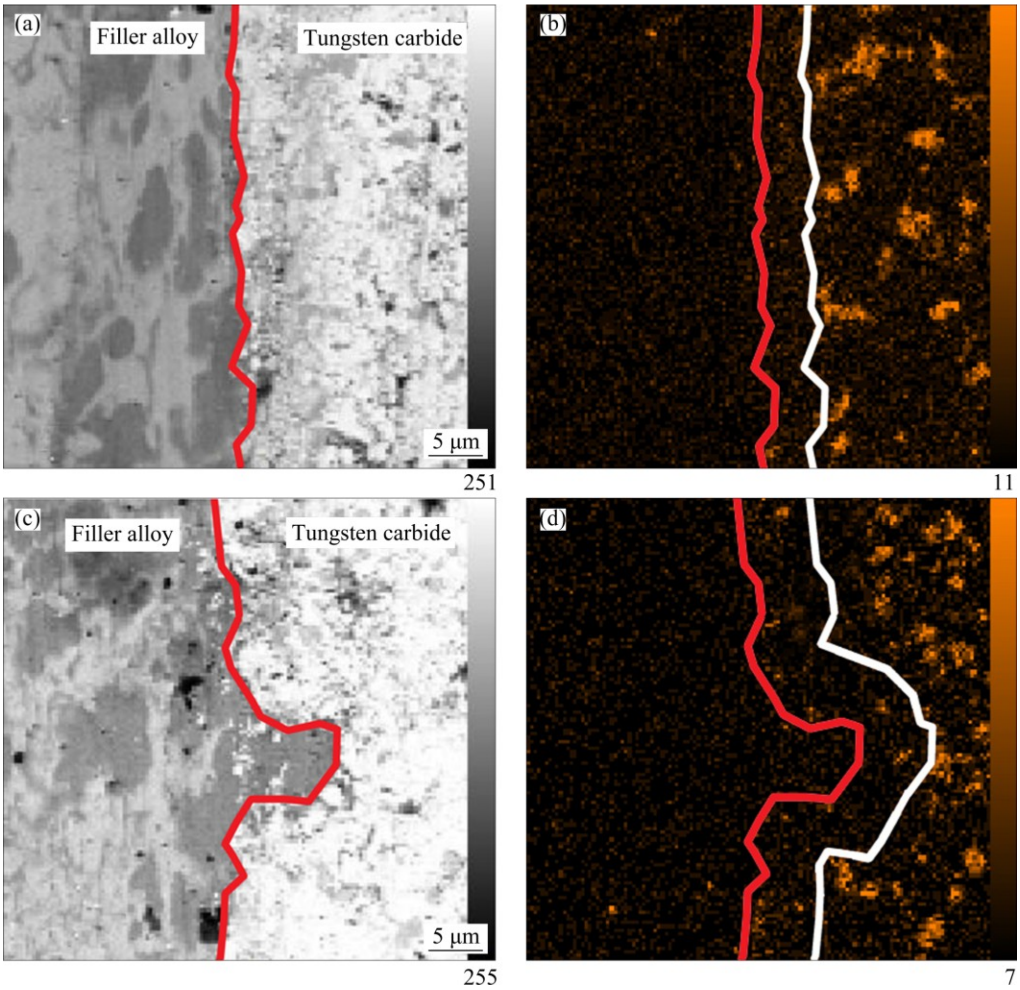

Fig. 10 SEM images (a, c) and EDS elemental mappings (b, d) of joints brazed at 730 °C holding for 15 min (a, b) and 30 min (c, d)

However, titanium tends to yield various compounds with other elements such as copper and form more stable compositions. Furthermore, it should be taken into consideration that titanium has a good solubility in copper, so that the presence of different copper−titanium compounds such as CuTi, $CuTi_2$, $CuTi_3$, Cu_7Ti_2, Cu_3Ti, and Cu_2Ti has been reported in a corresponding Cu−Ti phase diagrams [25,26]. It should be carefully taken into consideration that the process of joining titanium alloys has to be carried out in high vacuum together with precise monitoring to prevent the titanium from reacting with other elements [15]. However, in the XRD analysis of the brazed joint illustrated in Fig. 11, only Cu_3Ti intermetallic compound was detected in the interface zone, which is a result of diffusion processes taken place at the boundaries of WC−Co and filler material. The melting filler alloy requires optimized time to wet the brazing surfaces. So, less time needed would not provide sufficient time to fulfill the wetting process, since it leads to an imperfect joint.

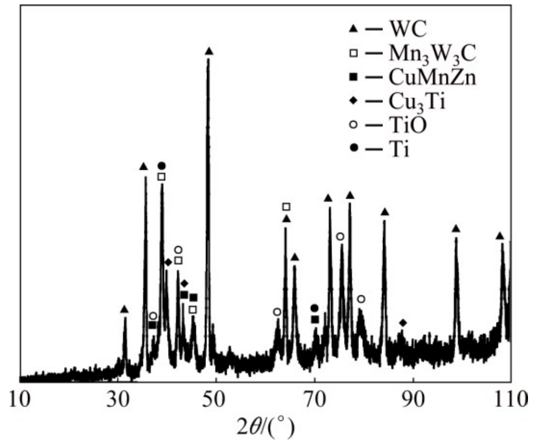

Fig. 11 XRD pattern of joint brazed at 730 °C for 20 min with filler alloy of 250 μm in thickness

However, it is reported that the formation of intermetallic compounds such as Mn_3W_3C, which is possibly increased with elapse of time, would be very detrimental to shear strength of brazing joints [27]. Additionally, the results obtained from XRD analysis (Fig. 11) and EDS line scanning (Fig. 9) also support the formation of Mn_3W_3C phase. Moreover, a point next to the joint surface was analyzed by EDS and the results are shown in Fig. 12, which shows a relatively strong peak for Mn. Although Mn_3W_3C has not been clearly detected in the SEM image of the interface (see Fig. 6), and its existence can be inferred from the sudden rise of Mn content in Fig. 9.

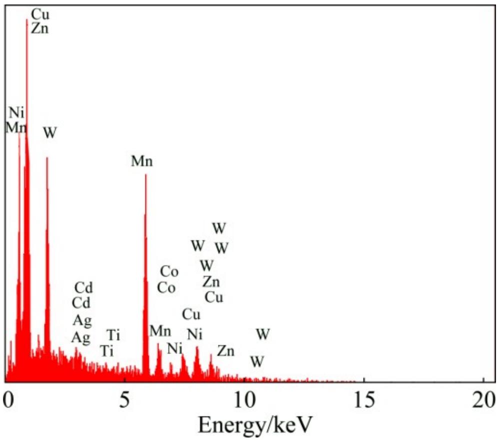

Fig. 12 EDS analysis result of Mn_3W_3C phase

3.2 Joint strength

The WC−Co joints can be properly brazed by using BAg22 filler metal at the brazing temperature of 730 °C. In this part, the effects of time and filler alloy thickness on the joint strength were investigated and the results are shown in Figs. 13 and 14, respectively. As can be seen from Fig. 13, the maximum shear strength of (148±6) MPa was achieved for joints within 15 min and a filler alloy thickness of 250 μm. While it was illustrated that, at a brazing time of 15 min, a maximum shear strength of (156±7) MPa was achieved at a filter alloy of thickness of 150 μm.

However, for a filler material thickness of more than 150 μm, the shear strength of brazing decreased. While, for thinner filler alloys less than 150 μm, the strength is not sufficient. This might be due to the lack of enough flow of the molten filler alloy along the joint surfaces. Meanwhile, for such thin thicknesses, the filler material would not allow the brazing filler to deform in a ductile fashion and that the filler material would not be able to dissipate thermal stresses induced during the cooling process. It should be noted that, from the mechanical point of view, the application of a thinner filler material should ensure a better strength of the brazed joints, but the least thickness does not produce the best strength. Through microscopic images and phase immigration, the influence of brazing time on the shear strength of joints has also been proved. On the other hand, the thick layers of brazing alloys are not favorable as well. In thick filler alloys, the surfaces of the WC−Co base material could not prevent the deformation of the brazing joints. However, the thin layer of the filler alloy which prevents it from deformation is securely bonded to the two surfaces of the parent materials [19]. Generally, the least thickness would provide the best strength, due to the fact that the lower thickness of the filler alloy can lead to easier capillary action at the interface. On the other hand, shrinkage and void defect formation would be less probable during solidification [22].

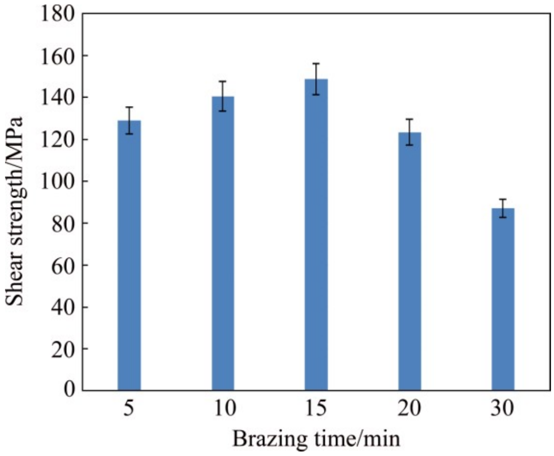

Fig. 13 Effect of holding time on shear strength of joints brazed at 730 °C with filler alloy of 250 μm in thickness

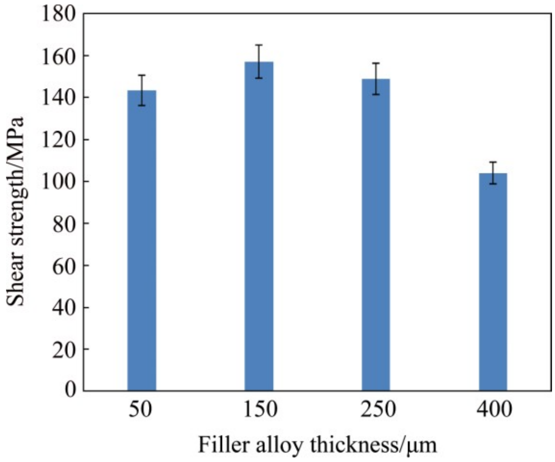

Fig. 14 Effect of filler alloy thickness on shear strength of joints brazed at 730 °C for 15 min

In typical brazed joints, there is usually a thin layer of filler metal bonded to two strong and low ductility parent materials. In this case, under tensile loading, i.e. above the yield point of the brazing filler metal, it is expected that the filler metal begins to deform in the same manner as a tensile test specimen deformed by necking. However, as the thin layer of the filler metal is securely bonded to the two-parent materials, it would not be deformed. Accordingly, the brazed joints fail due to tri-axial stress conditions and show less ductile deformability. So, there would be a brittle mode of fracture [19]. The inappropriate flowability of the molten filler alloy and low wettability of joining surfaces would cause pore formation and result in incomplete penetration of the filler alloy between the contacting surfaces. Thus, shear strength of the joints might be less than what expected [18].

4 Conclusions

1) The results reveal that the maximum shear strength of (148±6) MPa is achieved for joints within 15 min of brazing time and a filler alloy thickness of 250 μm. While, the maximum shear strength of (156±7) MPa is obtained for samples with 150 μm in gap size at a holding time of 15 min.

2) The shear strength increases with increase in brazing time from 5 to 15 min. It is illustrated that more time can provide a better chance for the liquid interlayer to flow towards the base metal. However, it is observed that the WC grain boundaries are surrounded by filler due to cobalt binder depletion on the cermet surface.

3) The shear strength decreases with increasing brazing time from 15 to 30 min. While, some voids are produced at longer time, due to evaporation of Zn content of filler alloy. These voids result in diminished mechanical properties of the joint. The formation of compounds such as Mn_3W_3C at longer holding time induces a destructive effect on the shear strength.

4) The lack of enough molten filler alloy and disability to dissipate thermal stresses during the cooling are two main reasons for decreased shear strength with decreasing the filler metal thickness from 150 to 50 μm.

5) By increasing the filler metal thickness from 150 to 400 μm, the filler alloy cannot lead to easier capillary action at the interface. On the other hand, shrinkage and void defect formation will be more probable during solidification.

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采用 Ag−Cu−Zn + Ni/Mn 合金钎料的碳化钨钎焊接头的界面结构和力学性能

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摘　要：使用管式炉在高纯氩气中 730 °C 下，以 Ag−Cu−Zn + Ni/Mn 合金为钎料焊接 WC−Co 硬质合金。研究钎焊时间和间隙尺寸对接头的影响。结果表明，焊接 15 min，间隙尺寸为 150 μm 的样品的最大剪切强度为 (156±7) MPa。采用 SEM, EDS 和 XRD 手段表征钎焊接头的的特性和结构。焊接时间从 5 min 延长到 15 min，为液体中间层流向基材提供了较好的机会。然而，当焊接时间超过 15 min 时，金属相如 Mn_3W_3C 的形成导致了接头的剪切强度降低。

关键词：钎焊；碳化钨；银合金；钎料合金；显微组织；润湿特性；剪切强度

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