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ARTICLE

Overview of the Brazing of Carbon-Carbon Composites

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Abstract: Since the individual use of C/C composite is limited, the joining with other materials becomes a key technology in its application. Brazing is one kind of joining methods that has been studied most, most mature and most widely used for C/C composite in recent decades. Nonetheless, there are still many deficiencies in the brazing of C/C composite because of the characteristic of the composite itself, which cannot meet the needs of specific conditions. The current brazing situation of C/C composite is reviewed in this paper. The difficulties of joining C/C composites and metals, the bonding systems that has been studied, and the joining mechanisms are included. The brazing methods and basic joining mechanisms of C/C composites are summarized emphatically, mainly including: direct brazing with active filler materials, using micro-nano particles reinforced or stress buffer layer added composite filler materials, surface modifying of C/C composite, changing the interface structure, and so on. The current research breakthroughs and remaining problems are analyzed. The feasibility measures of brazing C/C composite are analyzed and summarized to provide basic reference for subsequent research on the brazing of C/C composite and the improvement of joint performance.

Key words: carbon-carbon composites; brazing methods and mechanisms; interfacial structure; composite brazing filler materials

Carbon fiber reinforced carbon matrix composite (C/C composite) is a new kind of composite material with both functional and structural properties. It has the characteristics of low density (1.5~2.0 g/cm^3), high specific strength, good ablation resistance, excellent friction and wear resistance, good thermal shock resistance, good fatigue resistance and corrosion resistance[1-3]. It is widely used in aerospace, navigation, nuclear energy, biomedicine and other high-tech fields, such as the thermal protection system of the space shuttle vehicle wing's leading edge, the nozzle throat in solid propellant rocket motor, the aircraft brake disc, the artificial joint and other components[4]. It is difficult and expensive to directly prepare large and complex components because of the complex preparation process and long generation cycle of C/C composite. Meanwhile, the material itself has poor plasticity, difficult deformation and poor workability. The individual use of C/C composite is limited to a certain extent. Therefore, realizing high-quality joining between C/C composite and other materials or composites themselves is one of the key technologies that must be solved in its popularization and application.

At present, the joining methods of C/C composites include mechanical joining, adhesive joining, brazing, diffusion bonding and self-propagating reaction connection.

Mechanical joining is usually realized through percussion drilling and fasteners such as bolts and screws. However, the mechanical properties of C/C composites are seriously damaged during machining, and the joints are easy to generate problems such as delamination, slag dropping, significant anisotropy and stress concentration, which affect their joining performance. Moreover, the screws increase the weight of the joining pieces additionally, which is not conducive to light-

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weight character. Adhesive joining requires long time solidification, and the adhesive joint has poor resistance to high temperature and oxidation. The service life of the adhesion agent is short, because with the aging of adhesion agent, the bonding strength decreases. Thus the application of adhesive joining is not wide.

C/C composite has a very high melting point (3600~3800 °C) and cannot be welded using conventional fusion methods. Brazing or diffusion bonding methods are generally used. High temperature self-propagating reaction can also be used to realize the joining of C/C composite, but the joint has many internal defects and lower strength. The process temperature of diffusion bonding technology is much higher, and a certain pressure should be applied. The requirements of equipment of diffusion bonding are relatively high, the process is complex, and the joint strength obtained is more dispersed.

As an economical and reliable method of material joining, brazing is a kind of joining method between C/C composites and metals that has been studied most, most mature and most widely used in recent decades. The brazing process of C/C composites is relatively simple, which can realize mass production, and the joints have good reliability.

1 Difficulties of Brazing C/C Composites

Due to the intrinsic qualities of the material itself, there are some difficulties in the brazing of C/C composites: (1) Most of the commonly used brazing filler metals are difficult to wet or not wet C/C composites during brazing. (2) The thermal expansion coefficient of C/C composite is significantly different from that of other metal materials and brazing filler metals, so the residual stress at brazing joint is large and the joint is easy to fracture. (3) C/C composite itself has a certain number of voids, and the joint is prone to generate porosity and filler penetration phenomenon during brazing. (4) The oxidation resistance temperature of C/C composite is low, so brazing must be carried out under vacuum or protective atmosphere. (5) It is difficult to protect C/C composite from damaging by the brazing process, and to maintain its original properties.

2 Brazing Systems of C/C Composites

Plenty of researches have been conducted on the brazing between C/C composites and metals or non-metals. The brazing systems of C/C composites mainly include C/C composites with C/C composites, high-temperature resistant ceramics (SiC ceramic and Li_2CO_3-Al_2O_3-SiO_2 (LAS) glass ceramic), copper and copper alloys (pure Cu, oxygen free Cu, CuZrCr alloy, CuW alloy, etc.), titanium and titanium alloys (TC4 alloy, TC17 alloy, Ti600 alloy, etc.), Ni-based superalloys (GH3044, GH3128 alloy, GH99 alloy, etc.), intermetallic compounds (TiAl alloy, Ti_3Al alloy), stainless steel, Nb, W and TZM alloy, and so on (as shown in Fig.1). It can be seen that brazing between C/C composites and Ti alloys, Ni-based superalloys, Cu alloys have been widely studied.

Some components brazed between C/C composites and metals have been applied in engineering. For example, in the cooling system of a vertical target of an International Thermo-nuclear Experimental Reactor (ITER) divertor, C/C composite has been selected as the first wall protection material to protect the inner copper alloy cooling pipe due to its excellent comprehensive properties (Fig.2)[5]. The reliable joining between C/C composite material and OF-Cu is realized through brazing. The temperature control system heat transfer components of the deep space exploration equipment and

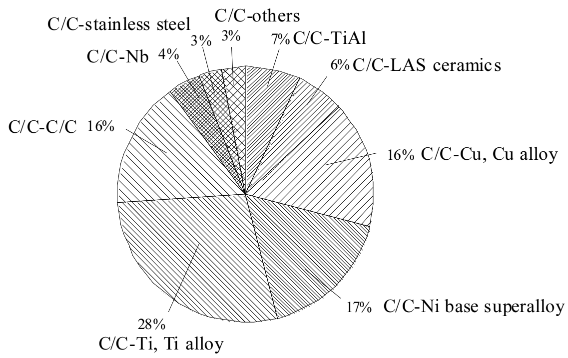

Fig.1 Brazing systems of C/C composites

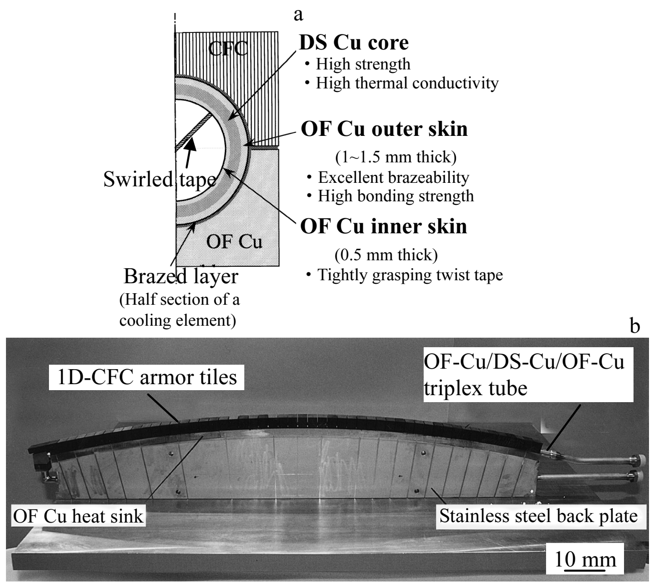

Fig.2 Application of C/C composite and Cu brazing component: (a) concept of a divertor target cooling structure with OF-Cu/DS-Cu/OF-Cu triplex tube brazed between saddle type ID-CFC armor titles and (b) a 1500 mm long vertical divertor target mock-up for ITER[5]

spacecraft can be manufactured by brazing C/C composite plates and titanium tubes (Fig.3)[6]. However, most of joining studies between C/C composites and metals are still in the experimental research stage because of the limitations of the working environment. Meanwhile, there are still many key technologies that need to be broken through before actual engineering application.

3 Methods and Mechanisms of Brazing C/C Com- posites

At present, in the research on brazing of C/C composites, the methods of active brazing filler metals, composite brazing filler metals, adding interlayers, material surface modification and multi-dimensional design are usually adopted to obtain high-quality joints. According to the brazing operation process, it can be generally divided into direct brazing and indirect brazing.

3.1 Direct brazing of C/C composites

Direct brazing, also known as active brazing, is the most widely used method for brazing carbon materials. It is to add some strong carbides generating elements (such as Ti, Zr, Cr, Si, Hf, Pd, V and Nb) with great affinity for carbon to other metals (such as Ag, Cu, Au, and Ni) to form the active brazing filler metals. The interfacial reaction between carbon and the active elements in brazing filler metals is used to improve the wettability of brazing filler metals on carbon materials and form a reliable joint.

When brazing C/C composite, it requires not only the wettability of brazing filler metal on the base material, but also the chemical reaction between brazing filler metal and C/C composite to form a higher metallurgical bonding joint[7]. So far, the commonly used brazing filler metals include Ag-based, Cu-based, Ti-based, Ni-based, micro-nano ceramic particles reinforced composite filler metals and added metal layers composite filler metals (Fig.4).

3.1.1 Active brazing filler materials

In order to improve the wettability of brazing filler metals on C/C composites, active metal elements such as Ti, Cr and V which can react with C to form carbides are generally added to brazing filler metals, such as Al-Ti[8], Ag-Cu-Ti[9,10], Cu-Ti[11], Cu-Cr[12], Ni-Ti[13], Ni-Cr-P[14], Ni-Cr-Si-B[15], Cu-(22~32)Pd-(6~12)V[16] brazing filler metals. The active metal can react with C in the brazing process to generate carbide reaction layer, and the metallurgical bonding formed at the joint improves the joint strength. Some scholars pointed out that pure copper is very difficult to wet on C/C composite surface, whose wetting angle is 137°~140°, and the wettability of Cu on C/C composites is improved greatly after adding Ti element[17,18]. Li[19] through experiments showed that under the condition of 1077 °C heat preservation 5 min, the wetting angle of Cu-12.6 at% Ti brazing filler metal on C/C composite surface was close to 0°. The reason is that after adding Ti element into Cu-based brazing filler metals, Ti and C will have a chemical reaction to generate TiC, improving the wettability of brazing filler metal on the base material.

Fig.3 Advanced C/C composite radiator[6]

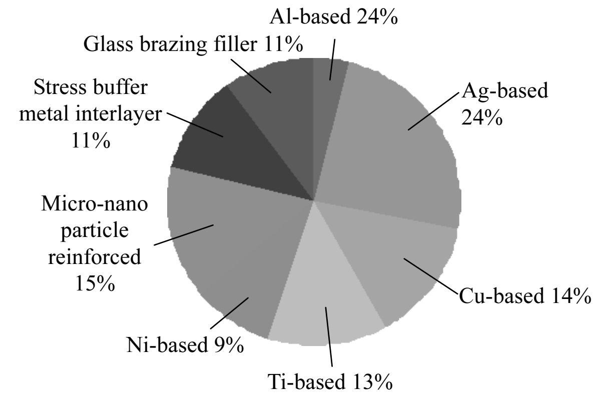

Fig.4 Brazing filler metal systems used to join C/C composites

Ag-Cu-Ti, Cu-Ti active brazing filler materials are the mainstream filler material systems for brazing C/C composites. They are usually used to join C/C composites themselves, C/C composites and Cu alloys, C/C composites and Ti alloys, etc. However, the joints prepared by these brazing filler metals cannot bear high temperature, so the excellent high temperature performance of C/C composites cannot be reflected. Therefore, the research of high-temperature brazing filler alloy or interlayer is still a hot research direction that needs to be conducted for C/C composites in the application of aviation and aerospace. The joints using Ni-based brazing filler metals are of high service temperature, so Ni-based brazing filler metals are usually used to connect C/C composites and superalloys, W, TZM alloy, Nb and other refractory metals.

3.1.2 New composite brazing filler materials

When the content of active elements in brazing filler metal is too high and the joining temperature is too high, abundant brittle intermetallic compounds will be generated in the connection layer, which is unfavorable to the performance of the joint. It is necessary to control the interface structure when joining C/C composite, especially the thickness of the brittle intermetallic carbide layer at the interface, which can be adjusted through changing the composition of the brazing filler metal. In consequence, the development of new composite brazing filler metals has been widely concerned by scholars.

Composite brazing filler metals used in joining C/C composites are mainly divided into two categories: First, adding micro-nano scale reinforcing particles, such as micro SiC[7], graphene nanosheets[20-22], carbon nanotubes[23,24], nano Al_2O_3[25], micro Si_3N_4 or SiO_2[26], micro diamond particles[27], BN[28], TiB_2[29], etc., to prepare dispersion reinforced composite brazing filler metals. The second is to add a metal interlayer to cushion stress. Depending on the different ways of stress relief, it can be divided into soft interlayer (such as copper[30-32], aluminum and nickel) and hard interlayer (such as niobium[33-35], tungsten, molybdenum[36,37]). The soft interlayer generally has lower elastic modulus but better plasticity. Stress can be reduced through the elasticity, plasticity and creep deformation of the metal during the joining process[38]. The elastic modulus of hard interlayer is generally high, and the thermal expansion coefficient is low, which is close to that of the carbon material. When joining, the residual thermal stress in the joint can be transferred to the interlayer[36].

There are three main functions of the additives, whether it is micro-nano reinforced particle or stress buffer metal interlayer. The first is to improve the wettability of brazing filler metal on the composite surface. The second is to decrease the difference of thermal expansion coefficient between materials to reduce the residual thermal stress of the joint. Moreover, the added reinforced particles or intermediate layer materials can also react with the active elements in brazing filler metals or C in composite materials, inhibit the growth of the reaction layer, reduce the thickness of the generated brittle carbide layer, and prevent the excessive thickness of the reaction layer from affecting the joint strength.

In addition, multilayer gradient design of brazing alloys can also be used for joining C/C composites and metals. For example, to solve the problem of large difference in thermal expansion coefficient between carbon material and pure copper, Matsuda et al[39] proposed the idea of using gradient transition layer, designed and prepared Cu-Fe-Cu/Mo/Fe-Cu-Ag multilayer intermediate layer system (Fig.5), and obtained good joints by using traditional brazing technology. Cao et al[40] used $(Ti/Ni/Cu)_f$ multi-foil filler to braze C/C composite and TiAl alloy. The brazing joint displayed reliable interface bonding and excellent mechanical properties at high temperature. The research showed during brazing process, low-melting-point eutectic liquid-phase structure is firstly formed through the contact reaction at the Ti/Ni interface, and then complete melt and diffusion of the filler occurred afterwards with Cu element dissolving. When the joint of the C/C composite and the TiAl alloy was brazed at 980 °C for 10 min, the shear strength reached the maximum 18 MPa at room temperature, and increased to 22 MPa at high temperature about 600 °C.

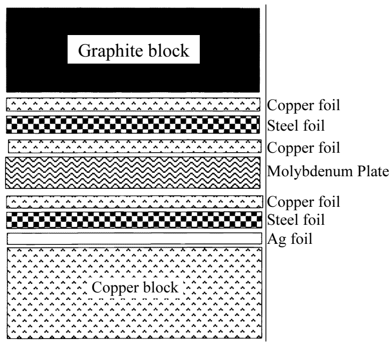

Fig.5 Regions for C-Cu brazing[39]

3.1.3 Glass brazing filler materials

The glass material has a series of advantages such as adjustable coefficient of thermal expansion, good thermal stability and thermal shock resistance[41,42], which has been applied in the joining of C/C composites themselves, C/C composites and ceramics. However, since most glass and glass-ceramic materials are difficult to wet the surface of carbon materials[43], it is difficult to give full play to its advantages in joining. Surface treatment of C/C composites is usually required to improve wettability between brazing filler metals and base materials. When using glass brazing fillers to join C/C composites, fillers are mainly concentrated in inorganic mixed powders composed of SiO_2, B_2O, Al_2O_3, MgO, ZnO, and other oxides. The joining systems are also mainly the joining between C/C composites themselves, and between C/C and glass-ceramic materials.

3.2 Indirect brazing of C/C composites

Indirect brazing of C/C composites usually involves depositing a metal film on the surface or generating a thin carbide layer before brazing. The surface treatment methods reported in previous studies mainly include three types: Firstly, the surface is densified by carbonization after coating organic matters such as resin, or by metallization using metals like Mo, W, Ni, Cu, and Cr. The second is to generate carbide layer on the surface through Cr, Ti, Mo, Ta, etc. metal elements. The third is to use SiC and other materials on the surface to modify the surface of C/C composites by ceramic coatings.

When using 49Ti-49Cu-2Be brazing filler metal to join low density C/C composite and copper, Lauf et al[44] modified the surface of C/C composites before brazing. The surface of C/C composite was coated with a layer of organic resin and carbonized at 1600 °C in vacuum or argon. A dense carbon layer was generated on the surface of C/C composite for modification, so as to improve the compactness of the carbon layer on C/C surface and prevent the infiltration of melted brazing filler. The strength of brazed joint after surface modification is much higher than that of C/C composite itself.

Li et al[45] diffused and deposited metal gradient layer on the surface of C/C composite to achieve reliable bonding between

C/C composite and Ti alloy. That is, the gradient layer close to C/C composite material was one or any two kinds of Ni, W, Ta, Mo, Ru; the gradient layer close to the brazing seam was Ti alloy layer; then C/C composite and Ti alloy were brazed together using Ti-based or Ag-based brazing filler metal. Prior to brazing, a 3 μm thick Ni layer was deposited on the surface of C/C composite by surface modification, and then a 2 μm thick TC4 alloy layer was deposited on Ni layer to form the metal gradient layer. The shear strength of the joint after brazing was up to 48 MPa.

Appendino et al[46] modified C/C composite surface with group VIB (Cr, Mo, W) transition metals. The metals were deposited by the slurry technique on the C/C composite surface and a heat treatment initiated the solid-state reaction between the metals and the composite. Carbides Cr_7C_3, $Cr_{23}C_6$, Mo_2C and WC were detected after surface modification. The carbide layers, obtained after the modification by Mo and Cr, were continuous and had a thickness of 15~20 μm, while the W carbide coating appeared as a non-coherent layer. The C/C composite with carbides formed on the surface was easy to be wetted by molten copper. The pure copper on chromium carbide-modified C/C composite showed a final equilibrium contact angle of 40°, while on the molybdenum and tungsten carbide modified surfaces the contact angle was 35° and 30°, respectively. The chromium carbide-modified C/C-Cu samples had an average shear strength of 33±4 MPa. The molybdenum carbide-modified C/C-Cu samples showed lower shear strength: 6±3 MPa. In the case of the WC modified CFC-Cu, the morphological analysis showed a porous carbide layer that corresponded to a weak interface: no mechanical tests were done for these samples. Then, the chromium modification seems to be the best solution to have a good wettability and a strong interface.

Shi et al[47] realized a firm bonding between C/C composites and GH3044. To improve the bonding strength between C/C composites and Ni-based superalloy (GH3044), SiC nanowires-toughened SiC coating and Ni71CrSi brazing filler were produced as the transition layer and interlayer by a three-step technique of CVD, pack cementation and brazing methods. SiC coating has a medium coefficient of thermal expansion (CTE) between C/C composites and superalloy, which could relieve the CTE mismatch between C/C and GH3044. Moreover, the joint was significantly strengthened and toughened by SiC nanowires. The shearing strength of the C/C-GH3044 joint is 54.4 MPa, which is 60% higher than that of the joint without SiC nanowires. The strengthening mechanism is attributed to the changing of crack propagation and strong pinning effect. Meanwhile, the joint without SiC surface modification was failed, and no mechanical strength was detected[48].

Indirect brazing process is complex and its application is limited. The shear strength of joints is usually higher than that of joints without surface treatment. Nevertheless, when the connection between the surface treatment layer and C/C composites is poor, the fracture occurs in the surface treatment layer during shear test, while the strength of the joint decreases instead[49].

3.3 Brazing interface structure adjustment of C/C composites

The interface structure of base materials has significant effect on wettability and thermal stress while brazing. When liquid brazing filler metal has good wettability on the base material surface, the wettability of brazing filler metal is better with the increase of surface roughness. Changing the shape of brazing interface will result in the change of residual stress.

Multi-dimensional design of C/C composite joint surface can increase the joint area, enhance the mechanical nailing effect, reduce the joint residual stress and improve the joint strength. For example, Xiong et al[50] manufactured rectangular step sections on C/C composite surface by machining method (Fig.6a) before brazing. The interface integrity and strength of the slotted joint were greatly improved and there was no macroscopic crack defect after brazing (Fig.6b, 6c), and the shear strength of the joint increased from 20.68 MPa to 41.63 MPa.

Shen et al prepared ordered conical holes with depth of 300~1000 μm on C/C composite surface by laser micromachining before brazing C/C composites and metals. TiH_2 powders and Cu-3.5Si brazing filler metal were used to join surface machined C/C composite and CuCrZr alloy. The room

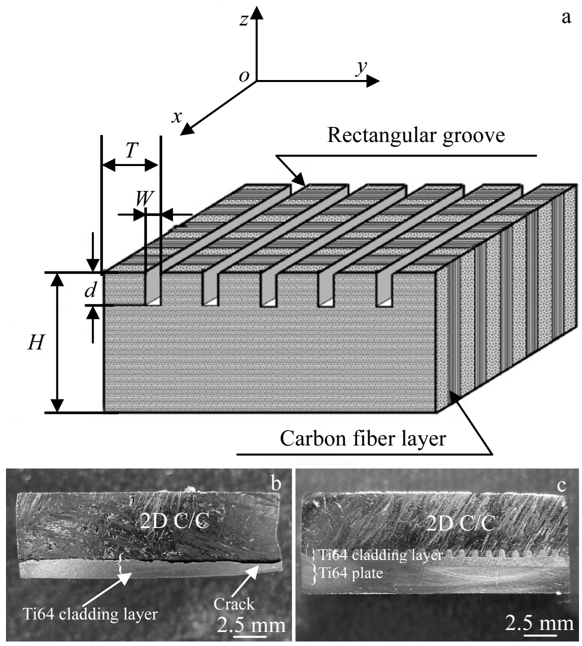

Fig.6 Macrostructure of 2D C/C-Ti64 interface: (a) a schematic of the rectangular-grooves structure on C/C composite, (b) the sample with flat interface, and (c) the sample with rectangular wave interface[50]

temperature shear strength of the brazed joint was 79 MPa, which was much higher than that of without surface treatment (44 MPa)[51]. Ag-28Cu brazing filler foil with a thickness of 250 μm and 2 mm thick Al_2O_3 interlayer was used to braze C/C composite and Ni-based superalloy. Three groups of comparative tests were conducted under the brazing specification of 910 °C and 10 min insulation: (1) Brazing directly with Ag-28Cu filler foil; (2) Brazing with Ag-28Cu filler foil and Al_2O_3 interlayer; (3) Brazing with Ag-28Cu filler foil and Al_2O_3 interlayer and laser punching treatment on C/C composite surface before brazing. The bending strength of three groups of brazed joints after brazing was 16, 33 and 73 MPa, respectively. It indicates that adding Al_2O_3 interlayer and machining C/C composite surface can improve the strength of brazing joint[52].

Guo et al[53] brazed C/C composite to the ReneN5 single crystal superalloy using the AgCuTi filler metal with the flat and zigzag triangular groove interfaces. Triangular grooves were formed on C/C composites surface by laser processing (Fig.7a), and the shear strength of joints increased from 18.7 MPa to 31 MPa (Fig.7b)[53]. The shear strength of the joint with the zigzag triangular groove interface was improved greatly compared to that of the joint with a flat interface. The improvement stems from the reduced residual stress, extended joint area, and enhanced pinning effect.

Xu[54] used TiNi filler metal to join high-density C/C composite and Nb. The joint fractured in the C/C near-braze area after brazing, and the joint strength was only 17 MPa. In order to realize reliable joining, several methods were adopted to fabricate 3-D surface state on high density C/C, including laser machining, needle puncturing and mixture method of laser machining and needle puncturing. By fabricating 3-D interface, the joint strength was obviously improved. With laser machining treatment, the front end of the 3-D interface hole was loose under the ablation of laser beam while brazing, so the infiltration of the filler metal formed a good bond. There was exposed C fiber on the hole wall when the needle was inserted. A good bonding interface can also be obtained through the "nailing" effect of filler penetration. Using laser machining holes and needle puncturing simultaneously to prepare composite holes on C/C composite surface combined the advantages of the above two, and the shear strength of joints increased from 17 MPa to 92 MPa, which was 440% higher than that of flat interface. Thus it can be seen that, reasonable design of joint interface structure can greatly improve the joint strength.

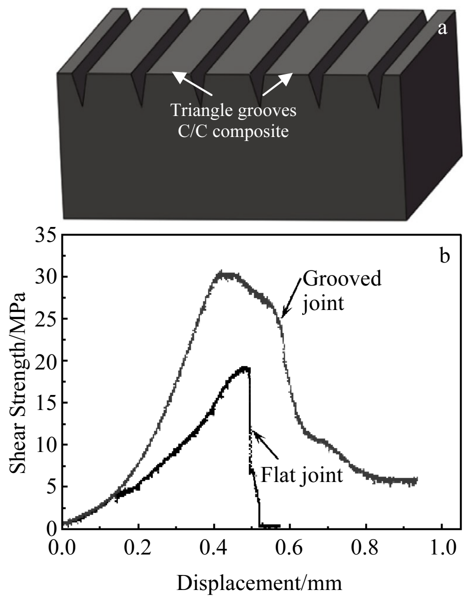

Fig.7 Structure of zigzag triangle grooves on C/C (a) and shear strength curves of the joint with different interfaces (b)[53]

Wang et al[55] studied vacuum brazing between C/C composites and TiAl alloys with Ag-Cu-Ti filler metal. A new method of making holes on C/C composite surface was introduced. More brazing materials can flow into the C/C composite after drilling, which played a role of nailing the C/C composite, so it is conducive to the improvement of joint performance. The joint strength increased from about 10 MPa to 26.4 MPa. The infiltration interface that was derived by surface puncturing can significantly strengthen joints and improve the impact resistance properties of the joint.

Zhang et al[56, 57] brazed pure copper and C/C composite in vacuum environment with AgCuTi filler metal. A conical interface structure was designed to further reduce the residual thermal stress of the joint. The finite element analysis showed that the residual stress in C/C composites adjacent to the cone can be reduced effectively by transferring most residual stress to AgCuTi braze filled in the cone. When the height and diameter of cone were 2 and 3.2 mm, respectively, the average value and maximum value of strength was 52 and 63 MPa, 2.57 and 2.67 times higher than that of the flat interface, respectively. Compared to adding interlayer, an appropriate conical interfacial shape is more beneficial to decrease the residual thermal stress, which can promote the performance of connection between C/C composite and Cu.

In conclusion, multi-dimensional design of joining surface exhibits more obvious strengthening effects on the joint strength. The factors that have induced mechanical strengthening of the zig-zag interfacial structure may include the enlarged joining area and strong pinning effect of the braze spikes. The machined composites surface directly resulted in a larger joining area than that of a flat joint by creating a 3-D transition region between the composites and braze after the holes were fully filled with brazing filler metals. Moreover, the dispersing filler material spikes into the composites obviously strengthened the joint through the pinning effect.

4 Conclusions and Prospects

The current brazing situation of C/C composite is summarized in this paper. The brazing methods and basic joining mechanisms of C/C composite are introduced emphatically. At present, the brazability problem of C/C composites has been basically solved. The joining technology of C/C composites is still in its infancy, and joining technology and process stability are not yet mature. The strength of the joining workpieces is not high, and the strength value is highly dispersive, which is still far from practical application. To obtain high strength and reliable joints, the point is to solve the wetting problem of the interface and to control the interface reaction. Possible solutions include: (1) adjusting process parameters and controlling the composition and thickness of interface reaction layer; (2) machining C/C composites surface to increase the contact area of the joint and improve the bonding strength; (3) applying new composite brazing filler metals or stress buffer interlayers is one of more effective methods. It is believed that with the further development of C/C composites, high temperature resistance, low cost and high quality joining technologies will be developed.

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碳/碳复合材料钎焊连接概况

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摘　要：碳/碳复合材料单独使用受到一定的限制，与其它材料的连接成为其应用的一大关键技术。钎焊是近几十年内，研究最多、最成熟、应用最广泛的一种 C/C 复合材料与金属的连接方法。由于材料本身的特性，C/C 复合材料的钎焊仍存在很多的不足，不能满足特定条件下的使用需求。本文综述了 C/C 复合材料的钎焊连接概况。包括 C/C 复合材料与金属材料钎焊的连接难点、已研究的连接体系和接头连接机理。重点归纳了 C/C 复合材料的钎焊方法和基本原理，主要包括：采用活性金属钎料直接钎焊；采用微纳颗粒增强复合钎料或添加应力缓冲层复合材料；对 C/C 复合材料进行表面改性处理；及改变界面结构等方面。分析了目前研究已经取得的突破进展及仍然存在的问题，并分析和总结了 C/C 复合材料钎焊的可行性措施，对后续 C/C 复合材料钎焊的研究及连接性能的提高作出基础性参考。

关键词：碳/碳复合材料；钎焊方法和机理；界面结构；复合钎焊材料

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