Review Article

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A review on filler materials for brazing of carbon-carbon composites

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Abstract: It is needed to join C/C composite to other materials since its individual use is limited. Brazing is a method to join C/C composite that has been studied most, maturest and most widely used in recent decades. The quality of a brazed joint is largely determined by the intermediate layer material. It is significant to choose filler materials reasonably. C/C composite is difficult to be wetted by common brazing filler materials. Moreover, there is a large difference in the coefficient of thermal expansion between C/C composite and metals. At present, there is no brazing filler alloy exclusively recommended for commercial C/C composites and metal brazing. Usually, active elements are added into filler metals to improve the wettability of them on C/C composite surface. The existing research includes Al-based, Ag-based, Cu-based, Ti-based, Ni-based brazing filler metals, and so on. In addition, various particle reinforced composite filler materials and stress buffer metal interlayer added composite filler materials have been studied for brazing C/C composite. The summarization of the overview on the application of intermediate filler metals is made in this paper. The basic reference basis is provided for the subsequent brazing filler metals development and joint performance improvement for C/C composite brazing.

Keywords: Carbon-carbon composite, Common brazing filler metals, Active brazing filler metal, Surface treatment of C/C composites, Composite brazing filler metal

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1 Introduction

Carbon fiber reinforced carbon matrix composite, also known as carbon/carbon composite (C/C composite) is a new composite material with both functional and structural properties. It is characterized by low density, high specific strength, high specific stiffness, high specific modulus, good thermal conductivity, good fracture toughness, good ablation resistance, good thermal shock resistance, excellent friction and wear resistance, good fatigue resistance and corrosion resistance, etc. [1, 2]. It is widely used in aerospace, navigation, nuclear energy, medical, military project and other high-tech fields [3]. C/C composite has been developed rapidly since its appearance, and with the rapid development of low-cost rapid preparation technology and high-temperature oxidation resistance technology, more and more attention will be paid to C/C composites.

While expanding its application fields, the individual use of C/C composite is limited to a certain extent. 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 composites. Meanwhile, the plasticity, deformation and workability of C/C composite are poor. Generally, in order to reduce costs and improve production efficiency, the economical and feasible solution is to join C/C composites to metals or other materials without affecting the structural performance, while giving

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play to their own advantages, so as to meet the processing requirements of special size and shape components.

So far, the joining methods of C/C composites include mechanical joining [4, 5], adhesive joining [5, 6], brazing, diffusion bonding and high-temperature self-propagating reaction, etc. There are some limitations in both mechanical and adhesive joining. C/C composite cannot be joined by the conventional fusion welding methods since its high melting point (3600°C – 3800°C). 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, maturest and most widely used in recent decades.

In the brazing process of C/C composite, the melted brazing filler metal can not only wet the matrix, but also disperse the joint pressure evenly at the interface. The thermal stress concentration phenomenon caused by the mismatch of the thermal expansion coefficient was alleviated. Meanwhile, the brazed parts are joined through the molten brazing filler metals. The properties of the braze joints are determined by the properties of the brazing filler metals and the interaction between them and base materials to a great extent. C/C composite should be well wetted by the brazing filler metals. Moreover, the properties of brazing joints are also related to the composition of joint products, the structure of compounds, the structure of matrix material interface, and difference of thermal expansion coefficient of brazed parts. Therefore, the quality of brazed joint is largely determined by the brazing filler metal. It is significant to choose filler materials reasonably for improving the reliability of the joint. At present, there is no brazing filler alloy exclusively recommended for commercial C/C composites and metal brazing [7]. Many experts and scholars have studied the joining of C/C composites with different filler metals. The summarization of the overview on the application of brazing filler metals is made in this paper. The basic reference basis is provided for the subsequent brazing filler metals development and joint performance improvement for C/C composite brazing.

2 Common brazing filler metals

2.1 Al-based brazing filler metals

When brazing C/C composite, it requires not only the wetting ability of brazing filler metal on the workpiece but also the chemical reaction between filler and C/C composite to form high strength metallurgical bonding [8]. Currently, the commonly used filler metals include Al-based, Ag-based, Cu-Ni-based brazing filler metals, etc.

Aluminum and aluminum alloys occupy a unique position in modern industrial materials due to their low density, high thermal conductivity and electrical conductivity. Al-based brazing filler metal is characterized by low cost, low melting point and low operating temperature. When applying in brazing of C/C composite, Al from the brazing filler metal can react with C from C/C composite to generate Al_4C_3 compound, so as to realize high strength joining of C/C composite. However, the existence of Al elementary substance in the joint makes it unsuitable to be used at high temperature. The applications of Al-based brazing filler metals in joining of C/C composites are shown in Table 1.

Although Al and C can react to form Al_4C_3 compound, Al can wet carbon materials only at high temperature. Therefore, it is generally preferred to add active element Ti to increase the wettability of the brazing filler metal on C/C composites' surface. On the other hand, Ti can react with C to form TiC intermetallic compounds, further improving the bonding strength. However, the addition amount of Ti should not be too much to avoid the brittle TiC layer is too thick, which will affect the interface joining strength.

2.2 Ag-based brazing filler metals

Ag-based brazing filler metal has the advantages of moderate melting point, good manufacturability, good strength, good toughness, high electrical conductivity, high thermal conductivity and good corrosion resistance. It is a kind of widely used brazing filler metal. The main alloying ele-

Table 1: Al-based brazing filler metals used in joining of C/C composites

Composition of brazing filler metal (wt. %)	Base materials	Brazing temperature ($^{\circ}\text{C}$) Holding time (min)	Strength (MPa)	References
Al	C/C-C/C	1000/45	10 (Shear)	[9]
Al-Ti	C/C-C/C	1110/10	12.3 (Shear)	[10]
Al-15Ti	C/C-C/C	1100/10	16 (Shear)	[11]
Al-14Ti	C/C-C/C	1050/10	14.7 (Tensile)	[12]

Table 2: Ag-based brazing filler metals used in joining of C/C composites

Composition of brazing filler metal (wt. %)	Base materials	Brazing temperature (°C) Holding time (min)	Strength (MPa)	References
Ag28Cu	C/C-TC4	850/10	38 (Shear)	[16]
Ag28Cu	C/C-TC4	880/10	33 (Shear)	[17]
Ag-Cu-2Ti	C/C-CuCr	850/5	16 (Shear)	[18]
Ag-Cu-2Ti	C/C-CuW	850/5	13 (Shear)	[18]
Ag-26.4Cu-4.6Ti	C/C-C/C	900/10	22 (Shear)	[19]
68.8Ag-26.7Cu-4.5Ti	C/C-Ti	910/5	38 (Bending) 0.24±0.09 (Tensile)	[20–22]
Ag-26.7Cu-4.6Ti	C/C-TC4	910/10	25 (Shear)	[23–25]
68.8Ag-26.7Cu-4.5Ti	C/C-(Cu-Mo-Cu)	(915~920)/5	—	[26, 27]
63Ag-34.3Cu-1Sn-1.75Ti	C/C-(Cu-Mo-Cu)	(821~826)/5	—	[26]
63Ag-35.3Cu-1.75Ti	C/C-(Cu-Mo-Cu)	(830~835)/5	—	[27]
63Ag-32.25Cu-1.75Ti	C/C-Graphite foam-Ti tube	820/5	12.4 (Shear) 11.8 (Tensile)	[28]
Ag-26.7Cu-4.6Ti	C/C-TiAl	900/10	12.9 (Shear)	[29]
Ag26Cu2Ti	C/C-K24 (Ni-based)	880/10	16 (Shear)	[30]
Ag-32.25Cu-1.75Ti	C/C-Pure Ti	830/5	24 (Shear)	[31]
70Ag28Cu2Ti	C/C-TC4	860/10	28 (Shear)	[32]
Ag-27Cu-3.5Ti	C/C-C/C	900/10	20 (Shear)	[33]
Ag-26.7Cu-4.6Ti	C/C-TC4	900/10	—	[34]
Ag-26.7Cu-4.5Ti	C/C-C/C	850/30	26.7 (Shear)	[35]
Ag27Cu3Ti	C/C-Rene N5	910/10	Shear: 18.7 (Flat), 31 (zigzag grooved)	[36]
68.83Ag-26.77Cu-4.4Ti	C/C-TC17	860/15	24±1 (Shear)	[14]
Ag-68.8Cu-4.5Ti	C/C-Cu	910/10	Bending: 14 (Flat), 52 (Conical interface)	[37]
Cu-28Ag-2Ti	C/C-OF Cu	850/10	22 (Shear)	[38]
Ag-(10,20, 30, 40)Ti	C/C-GH3044	(990~1080)/(10~90)	45.8 (Shear) (Ag-20Ti; 1020/30)	[39]

ments of Ag-based brazing filler metal include Cu, Zn, Cd and Sn, etc. Since the addition of Cu can reduce the melting temperature of silver without forming brittle phases, Cu is the most important alloying element. Brazing filler metals with Ag-Cu alloy as the matrix, assisted adding Zn, In, Mn, Ni, Si, Sn, Ti and other metal elements possess the advantages of good brazing temperature controllability [13], good wettability with the joined parts, and strong filling gap ability, and so on. Compared with Ni-based or Ti based brazing filler metal, the brazing temperature of Ag-based

brazing filler metal is lower. The applications of Ag-based brazing filler metals in joining of C/C composites are shown in Table 2.

It can be seen from Table 2 that the Ag-based brazing filler metals used for brazing C/C composites are mainly Ag-Cu eutectic alloy and Ag-Cu-Ti brazing filler metals. Ag-Cu-Ti brazing filler metal is one kind of relatively mature and most important active brazing filler metals in the active brazing method. Its low melting temperature makes it is suitable for medium and low temperature. Ag-Cu-Ti used in

Figure 1: Back-scattered electron image of C/C composite/AgCuTi/TC17 joint.

brazing C/C composites is generally based on Ag-Cu eutectic and added trace Ti element. The content of Ti ranges from 1.75 wt.% to 4.6 wt.%. During brazing process, interfacial metallurgical bonding was formed thanks to the elements diffusion and chemical reaction (Figure 1) [14]. The content of element Ti and Cu indicates that diffusion and reactions occur between base material and brazing filler. According the thermodynamic data [15], the Gibbs free energy of TiC formation are represented as follows:

The Gibbs free energy of forming TiC calculated by Equation (1) is about $-172.2 \text{ kJ/mol} \sim -171.2 \text{ kJ/mol}$ at $820^\circ\text{C} \sim 920^\circ\text{C}$. Therefore, element Ti and C can react with each other while using Ag-Cu-Ti fillers to brazing C/C composite.

Ag-based brazing filler metals are commonly used for brazes between C/C composites and Ti alloys, Cu alloys and Ni-based superalloys. The strength of brazing joint varies with different material composition and structure. Most of the shear strength of the joints was above 20 MPa, which was close to the interlaminar shear strength of C/C composites.

2.3 Cu-based brazing filler metals

Cu-based brazing filler metal has the characteristics of relatively high high-temperature bearing capacity, good strength, good plasticity and relatively low price. Researches showed that Cu-based brazing filler metals were widely used in brazing of C/C composites, carbon-based ceramic composites and metals. Some scholars pointed out that C/C composite does not bond well with pure Cu since

Figure 2: Contact angle, θ , of the (Cu-12.6 at% Ti) alloy on the glassy carbon substrate as a function of time at 1350K.

the wetting angle of molten copper on carbon substrates is very high, approximately 137° – 140° [40, 41]. However, the surface wetting on C/C composites was greatly improved by Cu after the addition of Ti element. Li proved that the wetting angle of Cu-12.6Ti (at. %) on carbon materials was close to 0° under the condition of 1077°C heat preservation 5 min [42]. The reason is that after adding Ti element into Cu-based brazing filler metal, Ti and C will react to generate TiC, improving the wettability of the brazing filler metal on base materials (Figure 2). Between the instantaneous process of the pure copper on the same substrate (Figure 3) to 50° and the process of the reduction in θ from 50° to the stationary value of zero, which proceeds quite quickly, there is an intermediate plateau of θ at a value

Figure 3: Contact angle, θ , of pure copper on glassy carbon versus temperature.

Table 3: Cu-based brazing filler metals used in joining of C/C composites

Composition of brazing filler metal (wt. %)	Base materials	Brazing temperature ($^{\circ}\text{C}$) Holding time (min)	Strength (MPa)	References
Cu-30Ti	C/C-Cu	930	7 (Shear)	[18]
Cu15Ti	C/C-C/C	1050/30	21 (Shear)	[43, 45]
Cu-14Ti	C/C-C/C	1200/10	—	[44]
Cu-Ti (TLP-DB) (0.4, 0.6, 0.8, 0.9, 1.0, 1.1)mm Cu +0.1mmTi	C/C-Nb	1050/30	28.6 (Shear) 0.9mmCu +0.1mmTi	[46]
Cu-50Pb	C/C-Cu	1150/40	1.5 (Shear)	[47]
99Cu-1Cr	C/C-W	1155/5	24.7 (Shear)	[48]
Cu-(15~25)Mn- (8~14)Ti	C/C-Stainless steel	(850~950)/(2~10)	16 (Shear)	[49, 50]
Cu-2Al-3Si-2.3Ti	C/C-OF Cu	1030 $\pm$ 2	20.2 (Tensile)	[51]
92.8Cu-3Si-2Al-2.2Ti	C/C-Ti	1040/5	0.27 $\pm$ 0.12 (Tensile)	[20–22]
92.8Cu-3Si-2Al-2.25Ti	C/C- (Cu-Mo-Cu)	(1040~1045)/5	—	[26]
Cu75Pt25	C/C-Nb	1160/20	Shear: 26.7(RT), 17.6(600 $^{\circ}\text{C}$)	[52]

of 50° for about 3min, indicating the existence of an incubation period needed to initiate the chemical reactions between the absorbed titanium and carbon. The kinetics and mechanism of chemical reactions developing at interfaces are important because the reactive wetting process depends on represents the first important step towards the understanding of the reactive wetting phenomena.

The applications of Cu-based brazing filler metals in joining of C/C composites are shown in Table 3.

Since C and Cu can react to form solid solution, a diffusion layer with gradual composition will be formed when Cu diffuses to C/C composite and C diffuses to the brazing joint. The diffusion layer has better compatibility with C/C composite than that of the brazing filler metal with C/C composite [43]. Adding Ti in copper matrix, Ti elements will be enriched towards the interface and form TiC because of the action of chemical and physical adsorption, during which the interfacial tension decreases. Therefore, the wettability of Cu-based brazing filler metal on C/C composite will be improved by adding Ti elements. The brazing filler alloys also can penetrate into the pores of C/C composite by capillary force to form a strong interface bonding [44].

2.4 Ti-based brazing filler metals

The corrosion resistance and high temperature strength of Ti-based brazing filler metals are superior compared with Al-based or Ag-based brazing filler metals. The Ti-based brazing filler metals used for brazing carbon materials are mainly binary or ternary alloys of Ti. Although pure Ti can also be used for brazing carbon materials, a very thick carbide layer will be generated at the joint due to the strong

Figure 4: The formation model of the C/C-C_f/SiC composite joint.

Table 4: Ti-based brazing filler metals used in joining of C/C composites

Composition of brazing filler metal (wt. %)	Base materials	Brazing temperature (°C) Holding time (min)	Strength (MPa)	References
49Ti-49Cu-2Be	C/C-Metals (Cu, steel, etc.)	980/5	—	[53]
Ti-Ni-Si (1:1:4) PTLP	C/C-C/C	1150/45 30MPa	23.58 (Shear)	[54]
Ti-SiC-Si-C (3:1:1:1) vacuum hot pressing	C/C-C/C	1600/6 30MPa	38.09±5.09 (Shear)	[55]
70Ti15Cu15Ni	C/C-Cu	1000/10	24 (Shear)	[56, 57]
70Ti15Cu15Ni	C/C-(Cu-Mo-Cu)	975-980/5	—	[26]
70Ti15Cu15Ni	C/C-Ti	975/5	0.33±0.13 (Tensile)	[20–22]
Ti35Zr10Ni15Cu	C/C-(TiBw-TC4)	900/10	13.6 (Shear)	[58]
50Ti50Ni	(low density) C/C -Nb	1200/20	Shear: 44(RT), 36 (600°C)	[59]
50Ti50Ni	(high density) C/C -Nb	1200/20	Shear: 17 (Flat), 92 (Conical interface)	[59]
Ti-Ni-Cu (1:1:1)	C/C-TiAl	980/10	18 (Shear)	[60]
TiSi ₂	C/C-C/C	1490/2	Shear: 34.4±9.6 (1164°C)	[61]
Ti-Ni-Ti (1:3:1)	C/C-C _f /SiC	960/30 0.5MPa	44.7 ± 12.8(Shear)	[62]

reaction between Ti and C. Moreover, the coefficient of thermal expansion of pure Ti is high, which is easy to cause cracks in carbon materials. Adding a small amount of Cr and Ni to Ti matrix can reduce the melting point of Ti-based brazing filler metal and improve its wettability (Figure 4). In addition, 49Ti-49Cu-2Be [53], Ti-Ni-Si [54] and Ti-Si-SiC-C [55] can be used for brazing C/C composites as well. The applications of Ti-based brazing filler metals in joining of C/C composites are shown in Table 4.

2.5 Ni-based brazing filler metals

At present, when joining C/C composites, Ag-based or Cu-based brazing filler metals are widely used. However, the service temperature of Ag-based or Cu-based brazing filler metals is lower, which cannot guarantee reliable joining strength while above 500°C. It is unable to reflect the excellent high-temperature performance of C/C composite. For example, the joint using Ag-based or Cu-based brazing filler metal cannot meet the high temperature service requirements when C/C composite is used in the engine nozzle. Therefore, it is necessary to search and develop brazing filler metals which meet the requirements of high temperature performance.

Ni-based brazing filler metal is used for brazing workpiece that works at high temperature. It has excellent corrosion resistance and heat resistance. The bearable servicing temperature of the brazing joints using Ni-based brazing filler metals can reach up to 1000°C. The brazing joints using Ni-based brazing filler metals also have satisfactory performance in liquid oxygen, liquid nitrogen and other low temperature media. Whereas, the melting point of nickel

Figure 5: Gibbs free energy calculation results of carbides.

Table 5: Ni-based brazing filler metals used in joining of C/C composites

Composition of brazing filler metal (wt. %)	Base materials	Brazing temperature (°C) Holding time (min)	Strength (MPa)	References
Ni-33Cr-24Pd-4Si	C/C-Cu	1210	6 (Shear)	[18]
Ni-11Cr-10P	C/C-Cu	935	7 (Shear)	[18]
Ni-Zr-Cr	C/C-C/C	1270/10	10 (Bending)	[63]
MBF-20 Amorphous (Ni-6.48Cr-3.13Fe- 4.38Si-3.13B)	C/C-Ti	1045/4	—	[64]
MBF-30 Amorphous (Ni-4.61Si-2.8B-0.02Fe)	C/C-Ti	1080/4	—	[64]
BNi-2 Ni-(6~8)Cr-(2.5~3.5)Fe- (4~5)Si-(2.75~3.5)B	C/C-GH99	1170/60	Shear: 35.4(RT), 15.3(800°C), 8.6(1000°C)	[65]
Ni-Si	C/C-GH3128	1160	12.6 (Shear)	[66]
80Ni-20Ti PTLP	C/C-GH3044	1030/30 4.5(MPa)	9.78 (Shear)	[67]
BNi-2 (Ni-7.0Cr- 3.0Fe-4.5Si-3.1B)	C/C-GH3128	1170/60	24 (Shear)	[68]
BNi68CrWB	C/C-GH600	1150-1200/10	Shear: 49.9(RT), 21.6(800°C)	[69]
NiCrSiBFe Amorphous	C/C-Ni-based superalloy	1170/60	Shear: 35 (RT), 15 (800°C), 9(1000°C)	[70]

is high. The thermal strength of nickel is low. The reaction ability of nickel with carbon materials is weak. Alloying elements should be added to reduce its melting point and improve its thermal strength.

NiP and NiCrP are two kinds of Ni-based brazing filler metals with lowest melting points. They are both able to join materials tightly because of the eutectic structure and excellent fluidity. The main functions of Cr in NiCrP are solid solution strengthening by forming solid solution with Ni, and improving the antioxidant capacity of it. Cr is also used as active element to react with carbon material to form carbide layer. The Gibbs free energy for the formation of Cr-C compound is shown in Figure 5. The applications of Ni-based brazing filler metals in joining of C/C composites are shown in Table 5.

It can be seen from Table 5 that Ni-based brazing filler metal is mainly used for the joining between C/C composite and Ni-based superalloy. With the development of materials, amorphous active Ni-based brazing filler metals are increasingly used in brazing between C/C composites and metals. Through comparison, it is found that the strength of

C/C composite joints using Ni-based brazing filler metals is generally lower than that of joints with Ag-based, Cu-based and Ti-based brazing filler metals.

3 Surface treatment of C/C composites before joining

Indirect brazing is another method that has been used to join C/C composites and other materials. Indirect brazing of C/C composites usually involves depositing a metal film on the surface or generating a thin carbide layer before brazing (Figure 6). Metals used for surface metallization generally include: Mo, W, Ni, Cu, Cr, etc. The elements commonly used to generate carbide surface layer are Cr, Ti, Mo, Ta, etc. In addition, the in-situ synthesis of carbon nanotubes on the C/C composite material surface before brazing can also improve the wettability of the filler metal on composite material surface, which is beneficial to the strength of the joint. However, indirect brazing process is complex and

Figure 6: Wettability of pure copper on CFC substrate (1100°C for 30 min).

Table 6: Surface treatment before joining and brazing filler metals used in joining of C/C composites

Composition of brazing filler metal (wt. %)	Surface treatment of C/C composites	Base materials	Brazing temperature (°C) Holding time (min)	Strength (MPa)		References
				Surface treatment	Without treatment	
Ag26.7Cu4.6Ti	Cr-C compound layer was formed	C/C-Ni-20Co-18Cr	990/15	Fracture occurred on C/C side.	—	[53]
87.75Cu12Ge0.25Ni Gemco	Cr-C composite layer	C/C-OFHC Cu-CuCrZr	(970 980)/30 995	34±4 (Shear)	—	[71, 72]
—	carbide layer (Cr, Mo, W)-C	C/C-Cu	1100/60	Shear: 33±4 (Cr), 11±2 (Mo)	—	[73–75]
Ti23Cu11Zr9Ni foil + Cu foil 20, 40, 100 (μm)	Cr-C composite layer	C/C-TC4	960/20	39±8.5 (Shear) 40μm Cu	15±2 (Shear)	[76]
Ti23Cu11Zr9Ni powder Ti or Ag based	Cr-C composite layer 3μmNi+ 2μmTC4	C/C-TC4	960/20	52±6 (Shear)	15±2 (Shear)	[77]
Cu-3.5Ti 2mm OFHC Cu	Depositing TiH2	C/C-Ti alloy	(800–850)/15	48 (Shear)	—	[78]
		C/C- Cu- CuCrZr	800/30 5MPa	Shear: 44 (Flat), 79 (Conical interface)	—	[79]
		C/C-Nb	1110/20	19 (Shear)	—	[80]
		C/C-Ti	877/5	15.3 (Shear)	—	[12]
		C/C-GH3128	1170	22.5 (Shear)	0 (Shear)	[81]
Zr64Ni36 Ti20Zr15Cu5Ni Ni-Ti	Rhenium (Re) coating SiC modification coating SiC surface modification coating SiC surface modification coating SiC surface modification coating SiC nanowires -toughened SiC coating modified	C/C-C	1600/6 30MPa	22.64±2.61 (Shear)	38.09±5.09 (Shear)	[55]
		C/C-GH3044	1180/30	35.08 (Shear)	Joint failed	[82]
		C/C-GH3044	1180/30	54.4 (Shear)	—	[83]
		C/C-Ti600	930/10	Shear: 73(RT), 41(600°C)	33(RT), 25(600°C)	[84]
Cu-46Zr	φ15.7nm × 2μm CNT were in situ formed	C/C-Nb	950/10	41 (Shear)	17 (Shear)	[85]
22Ti78Cu	φ18.9nm × 2μm CNT were in situ formed	C/C-GH3044	1030/30 0.3MPa	32.09 ± 1.98 (Shear)	23.47 ± 1.15 (Shear)	[86]
Ti/Ni/Cu/Ni (1:1:20:1)	Pre micro-oxidation and partial transient liquid phase (PTLP) process	C/C-C/C	1400/10	Shear: 26(RT); 36(1200°C)	<10 (Shear)	[87]
Ti-Si eutectic alloy Ti14Si86	Pre-infiltration Ti-Si	C/C-Nb	880/10	Shear: 62(RT); 48(500°C)	29 (Shear)	[88]
Ag27.6Cu1.5Ti	pre-oxidation and in situ growth of CNT

Figure 7: Shear strength of Nb-C/C joint brazed at 880°C for 10 min under different surface modification conditions of C/C composites at different test temperatures.

its application is limited. The researches on joining of C/C composites through surface treatment before joining are shown in Table 6.

It can be seen from Table 6 that the shear strength of joints obtained by brazing C/C composites after surface treatment is usually higher than that of joints without surface treatment (Figure 7). 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, the strength of the joint decreases instead. The strength of joints between surface treated C/C composites and other materials is related to the composition and thickness of surface coating, the bonding strength between C/C composites and coating layer, and chemical reaction between coating and the brazing filler metal.

4 Composite brazing filler metals

In recent years, the single brazing filler metals cannot meet the demand of application in some aspects. In consequence, the development of new composite brazing filler metals has been widely concerned by scholars.

4.1 Micro-nano particles reinforced composite brazing filler metals

The performance of joint is significantly affected by the thickness of intermetallic compound layer. It is necessary to control the interface structure when joining C/C composite, mainly the thickness of the brittle intermetallic carbide layer at the interface, which can be adjusted through changing the composition of the brazing filler metal.

It has been found that micron-sized or nano-sized particles are beneficial to improve the wettability of filler metals on C/C composite (Figure 8) [92], inhibit the growth of the reaction layer and alleviate the mismatch of thermal expansion coefficient between the brazed seam and base material, thus reducing the residual thermal stress (Figure 9) [94] and improving the mechanical properties of the joint (Figure 10) [92]. Therefore, manufacturing composite brazing filler metal with particle dispersion enhancement through adding micro-nano ceramic particles becomes an effective method to join C/C composite. The applications of micro-nano particles reinforced composite brazing filler metals in joining of C/C composites are shown in Table 7.

Figure 8: Variations of drop height and contact angle with different GNS contents.

Table 7: Micro-nano particles reinforced composite brazing filler metals used in joining of C/C composites

Composition of brazing filler metal (wt. %)	Composition and content of reinforced particles	Base materials	Brazing temperature (°C) Holding time (min)	Composite brazing filler metals	Strength (MPa)	Additive free	References
Ag26.7Cu4.6Ti Ag-28Cu	4.6µm SiC (0.35) vol. % 2mm Al ₂ O ₃ plate	C/C-TC4 C/C-Ni-based superalloy	910/10 910/10	29 (Shear) 15vol.% SiC Bending: 33(Flat), 73 (zigzag interface)	25 (Shear) 16 (Bending)		[8] [89]
Ag26.7Cu4.5Ti	20nm Al ₂ O ₃ 0.3 (wt. %)	C/C-TC4	880/10	27.8 (Shear) 0.3wt. % Al ₂ O ₃	19.5 (Shear)		[90]
Ag26.7Cu4.5Ti	GNS ^[d] 0, 0.1, 0.3, 0.5, 0.8(wt. %)	C/C-TC4	900/10	30.2 (Shear) 0.3wt. % GNS	19.5 (Shear)		[91, 92]
Ag26.7Cu4.5Ti	25µm diamond 5, 15, 25 vol.%	C/C-316 stainless steel	900/10	53 (Shear) 15vol. % diamond	26 (Shear)		[93]
Ag-28Cu	<2µm 4wt.% Si ₃ N ₄ or SiO ₂	C/C-TC4	850/5	Shear: 45±3.1(Si ₃ N ₄), 41±2.1(SiO ₂)	18±2.5 (Shear)		[94]
AgCu eutectic+ 50µm Cu foil Cu-50Ni	60µm TiH2 powder 2 8µm TiB2 3wt. %	C/C-Cu C/C-TiBw/TC4	930/25 950/10	30 (Shear) Shear: 18.5(RT ^[b]), 34.5(400°C), 10(700°C)	— 8.4 (RT Shear)		[95] [58, 96]
Cu-50Ni	0.5 0.8µm SiC 3wt. %	C/C-TiBw/TC4	950/10	Shear: 22.5(RT), 35.4(400°C), 6(700°C)	8.4 (RT Shear)		[58]
Cu-50TiH2	BN 2wt. %	C/C-CuCrZr	950/10	—	—		[97]
Ti-23Cu-11Zr-9Ni	φ(1.2)µm GNS 1wt. %	C/C-TC4	940/10	42±3 (Shear)	22±4 (Shear)		[98]
Ti-23Cu-11Zr-9Ni	φ(20.30)nm × (1.2)µm CNT ^[c] 0.5, 1.0, 3.0 (wt. %)	C/C-TC4	940/10	38±2 (Shear) 1wt. % CNT	22±4 (Shear)		[99]
BNI-2	TiH2 1, 3, 8(wt. %)	C/C-GH99	1170/60	Shear: 40(RT), 10(800°C), 10(1000°C) 5wt. % TiH2	35.4 (RT), 15.3(800°C); 8.6(1000°C)		[65, 100]
Pyrocarbon PyC	CNT/PyC interlayer	C/C-C/C	—	19.2 (Shear)	9.4 (Shear)		[101]
Ag26.7Cu4.5Ti(TiH2)	GNP ^[d] 0.05 0.6 wt. %	C/C-Ti3Al	880/15	26.7 (Shear) 0.1wt. % GNP	19.1 (Shear)		[102]

^[d] GNS: graphene nanosheets^[b] RT: room temperature^[c] CNT: carbon nanotube^[d] GNP: graphene nanoplatelet

Figure 9: Distribution of equivalent Von Mises stress in joints brazed with (a) pure AgCu, (b) AgCu+Si₃N₄, and (c) AgCu+SiO₂, and residual stress distribution of the middle cross-section in C/C composites.

Figure 10: Effect of GNS content on the shear strength of joints (T=880°C, t=10min).

4.2 Stress buffer metal interlayer added composite brazing filler metals

While joining C/C composites to metals, one of the important factors hindering joint performance is the large difference of thermal expansion coefficient between them (Figure 11). In order to alleviate the joint thermal stress and improve the strength of the joint, the method of inserting metal interlayer into the joint is generally adopted. Depending on the different ways of stress relief, it can be divided into soft interlayer (such as copper, aluminum and

Figure 11: Effect of thickness of interlayer on shear strength at room temperature of the joints.

nickel, etc.) and hard interlayer (such as niobium, tungsten, molybdenum, etc.). 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 [23] lastic 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 [103] applications of stress buffer metal interlayer added composite brazing filler metals in joining of C/C composites are shown in Table 8.

Table 8: Stress buffer metal interlayer added composite brazing filler metals used in joining of C/C composites

Composition of brazing filler metal (wt. %)	composition and specification of intermediate layer	Base materials	Brazing temperature (°C) Holding time (min)	With interlayer	Strength (MPa)	Interlayer free	References
Ag26.7Cu4.5Ti	Nb 0.1mm	C/C-1Cr18Mn15N Stainless steel	900/10	72.2 (Bending)	—	—	[104]
Ag68.8Cu4.5Ti	Nb 0.3mm, 1mm	C/C-Cu	910/10	Bending: 37.39 (Flat), 52(Conical interface) 1mm Nb	14.42 (Bending(Flat))	—	[105, 106]
Ag26.7Cu4.6Ti	0.3mm graphene-foam Cu (G-Cu)	C/C-Nb	880/10	Shear: 43 (G-Cu)	13 (Shear)	—	[107]
Ag26.7Cu4.6Ti	0.3mm C-Cu foam; (1, 3, 5, 10) C-Cu (wt. %)	C/C-Nb	880/10	52.8 (Shear) 3C-Cu (wt. %)	12.2 (Shear)	—	[108]
93Cu2Ti2Al3Si (Cu-ABA) 87.75Cu12Ge0.25Ni (Gemco)	Mo (1~2)mm	C/C-CuZrCr C/C-CuABA-Mo-Gemco- CuZrCr	1035/18	—	—	—	[109, 110]
Ti-37.5Zr-15Cu-10Ni Amorphous	Cu (0.2, 0.3, 0.5, 0.7, 0.9) mm + Mo (0.1mm)	C/C-TC4	900/5	Shear: 21(RT), 27(650°C) (0.3mmCu+ 0.1mmMo)	7 (RT Shear)	—	[23, 111, 112]
Fe-42Ni-20Cr-(10~12)(Si,P)	(100 500)µm Nb + φ100µm Mo	C/C-Inconel-600	1080/5	11.2 (Shear) 1µm Nb	—	—	[113]
87.75Cu12Ge0.25Ni (Gemco)	The brazing foil has been coated by Cr.	C/C-Cu	980/15	23 (Shear) 3µm Cr	—	—	[114]

Table 9: Glass brazing filler materials used in joining of C/C composites

Composition of brazing filler metal (wt. %)	Surface treatment of C/C composites	Base materials	Brazing temperature (°C) Holding time (min)	Max Strength (MPa)	References
SB Borosilicate glass	—	C/C-C/C	1020/45, 1200/45	—	[9, 118, 119]
ZBM Glass ^[a]	—	C/C-C/C	1200/45	—	[9, 118, 119]
SABB Glass ^[b]	β -SiC surface modification coating	C/C-C/C	1300/60	30 (Shear)	[117]
Borosilicate glass ^[c]	α -SiC surface modification coating	C/C-C/C	1400/30	26 (Shear)	[120]
LMA Glass ^[d]	SiC surface modification coating and SiC whiskers	C/C-LAS Glass-ceramic ^[f]	(1200~1300)/30	Shear: 12.17 (coating), 19.91 (whisker)	[121]
VHPT ^[e]	SiC-MoSi ₂ surface modification coating	C/C-LAS Glass-ceramic	(20~25)MPa	26.21 (Shear)	[122]
Y ₂ O ₃ -Al ₂ O ₃ -SiO ₂ -TiO ₂	SiC-MoSi ₂ surface modification coating	C/C-LAS Glass-ceramic	1250/30 20MPa	30 (Shear)	[115]
MAS Glass ^[g]	SiC surface modification coating	C/C-LAS Glass-ceramic	1200/15 20MPa	26.21 (Shear)	[116]
VHPT	SiC surface modification coating	C/C-LAS Glass-ceramic	1200/15 20MPa	26.07 (Shear)	[123]
YAST glass	MWCNT ^[h] in-situ formed, directly added	C/C-LAS Glass-ceramic	1150/45 20MPa	In-situ	[124]
VHPT	SiC nanowire reinforced SiC surface modification coating	C/C-LAS Glass-ceramic	1250/45 30MPa	38.16 ± 4.99 (Shear)	[125]
MAS Glass	CNT reinforced SiC surface modification coating	C/C-LAS Glass-ceramic	(1220~1250)/30 25MPa	31.84 ± 3.07 (Shear)	[125]

^[a] SB Borosilicate glass: 80SiO₂-20B₂O (mol. %); ZBM Glass: 4.53SiO₂-29.48B₂O-4.04 Al₂O₃-50.46ZnO-2.37Na₂O-9.12MgO (mol. %)

^[b] SABB Glass: 70.4SiO₂-2.1Al₂O₃-17.5 B₂O₃-10.0BaO (wt. %)

^[c] Borosilicate glass: 50~75 SiO₂, 10~25 Al₂O₃, 5~10 CaO (wt. %)

^[d] YAST Glass: Li₂O-MgO-Al₂O₃-SiO₂

^[e] VHPT: Vacuum hot-pressing technology

^[f] LAS Glass-ceramic: Li₂CO₃-Al₂O₃-SiO₂

^[g] MAS Glass: MgO-Al₂O₃-SiO₂

^[h] MWCNT: Multi-walled carbon nanotube

5 Glass brazing filler materials

Compared with other materials, glass has incomparable properties. The thermal expansion coefficient of glass material can be adjusted in a wide range. By adjusting the composition and content of the glass material, it is possible to prepare glass brazing filler that matches the thermal expansion coefficient of the base material. Meanwhile, glass ceramic materials have a series of advantages such as good thermal stability and good thermal shock resistance [115, 116]. Glass materials have been applied in the joining of C/C composite material itself, C/C composites and ceramics. The applications of glass brazing fillers in joining of C/C composites are shown in Table 9.

However, since most glass and glass-ceramic materials are difficult to wet the surface of carbon materials [117], 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 fillers and base materials. In consideration of good physical and chemical compatibility between SiC and C/C composites and glass materials, the surface SiC modification of C/C composites has become a research trend.

As can be seen from Table 9, when using glass brazing fillers to joining C/C composites, fillers are mainly concentrated in inorganic mixed powders composed of SiO_2 , B_2O_3 , 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. A certain amount of pressure was needed during the joining process, so as to discharge the gas, which produced by glass brazing filler due to excessive temperature, from the welding seam, and reduce the occurrence of defects.

6 Other kinds of brazing filler materials

Except for the above-mentioned brazing filler materials, the Si slice and Mg_2Si powder can also be used to realize the joining of C/C composite itself. The average shear strength of joints was 22 MPa and 5 MPa, respectively [9]. The shear strength at room temperature of the joints was 20–24 MPa when brazing C/C composite itself using Au-23Pd-22Ni-(6–12)V or Au-8Cu-30Pd-20Ni-(6–12)V. The C/C composite itself, C/C composite and TZM, C/C composite and W, C/C composite and Nb were brazed by Cu-(22–32)Pd-(6–12)V brazing filler metal. The shear strength of the joints at room temperature can be up to 30.6 MPa, 36.9 MPa, 44.8 MPa

and 21.9 MPa, respectively [126]. The shear strength of the joint of C/C composite brazed with W-(18–22)Co-(8–15)Cr-(14–21)Ti was 30 MPa at room temperature. After conical surface designing of C/C composite, the shear strength was increased to 45 MPa [127]. When using B powder as intermediate material to join C/C composite and the brazing parameter was 1995°C/15min, the maximum shear strength (18.6 ± 1.9 MPa) was obtained at 1600°C [61].

7 The effect of brazing parameters on joint properties

The brazing of C/C composite is mainly achieved by the chemical reaction among C element and the chemical elements in the intermediate layer material to form a metallurgical bonding. The strength of the joint depends largely on the type, size, and distribution of the compound, that is, the reaction degree in the interface. The insufficient reaction will lead to the weakening of interfacial bonding, while excessive reaction will destroy the performance of the joint by forming more brittle compounds at the interface. It is necessary to control the interface structure when joining C/C composite, mainly the thickness of the brittle intermetallic carbide layer at the interface, which can be adjusted through selecting appropriate process parameters. Therefore, the influence of brazing process parameters, such as temperature, heating/cooling rate and holding time, on joint strength should also be considered after the brazing material is selected.

The selection of brazing temperature should be based on the solidus and liquidus temperature of the chosen filler metal as the most important reference index. When brazing C/C composite, in order to form a metallurgical bonding, adequate temperature is required to melt the filler metal and cause a chemical reaction at the interface. However, when the brazing temperature is too high, large number of brittle compounds will be generated in the reaction layer, which is not good for the properties of the joint. Large temperature gradient and internal stress will be caused in the base materials and joint since excessive heating or cooling rate during brazing. Excessive residual stress can seriously reduce the strength of the joint and even cause cracks at the interface between C/C composite material and the intermediate layer materials. Adequate holding time can make sure the occurrence of metallurgical reaction and a firm joint. With the extension of holding time, the brazing joint structure tends to be homogenized. Whereas, if the holding time is too long, the thickness of the compound reaction

layer will be increased and the performance of the joint will be reduced.

The formation of the braze joint is the result of the synergistic effect of various factors, such as temperature, heating or cooling rate, and holding time. Therefore, a robust joint can only be obtained under the appropriate process conditions. For the brazing of C/C composite, different process parameters are needed when the joining systems and filler materials are different. Especially for the joining of heterogeneous materials, the interfacial metallurgical behavior of different materials is significantly different, and the types and distribution of the interface phases will become more complex, and the interface structure will be more difficult to explore. Generally, a large number of experiments or finite element simulation auxiliary methods should be used to explore the best process parameters.

8 Conclusions and prospects

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 carbide are generally added to brazing filler metals, such as Al-Ti, Ag-Cu-Ti, Cu-Ti, Cu-Cr, Ni-Ti, Ni-Cr-P, Ni-Cr-Si-B brazing filler metals mentioned above. 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.

Ag-based, Cu-based and Ti-based brazing filler metals are commonly used in joining of C/C composites. They are usually used to join C/C composite itself, C/C composite and Cu alloy, C/C composite and Ti alloy, etc. However, the joints prepared by these brazing filler metals cannot bear high temperature. Therefore, the excellent high temperature performance of C/C composites cannot be reflected. The joints used 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.

The research and development of composite brazing filler metals improves the shear strength of joints on the basis of traditional brazing filler metals. Composite brazing filler metals used in joining C/C composites are mainly divided into two categories: First, adding micro-nano scale reinforcing particles, to prepare dispersion reinforced composite brazing filler metals. The second is to add a metal interlayer to cushion stress. 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 other is to decrease the difference of thermal expansion coefficient between materials to reduce the residual thermal stress of the joint. Moreover, the thickness of brittle carbide layer can be reduced by adding particles or interlayer materials, which avoided the bad influence of thick reactive layer on joint strength.

At present, the joining technology of C/C composites is still in its infancy, and joining technology and process stability are not yet mature. The brazing filler metals used in C/C composite joining are not systematic. There are few studies on brazing filler metals which can be used in high temperature. Possible solutions include: (1) adjusting the composition of brazing filler metal and controlling the composition and thickness of interface reaction layer; (2) developing high-temperature resistance brazing filler metals which can reflect the excellent high-temperature performance of C/C composite; (3) experimenting and developing appropriate glass brazing fillers; (4) applying new composite brazing filler metals or stress buffer interlayers. It is believed that with the further development of C/C composites, high temperature resistance, low cost and high quality joining technologies will emerge as the times require.

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