Research Article

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Effect of bonding time on the microstructure and mechanical properties of graphite/Cu-bonded joints

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Abstract: The increasing serious heat dissipation problem faced by electronic equipment used in airplanes makes studies on new heat dissipation components urgent. The diffusion bonding of graphite and Cu was successfully realized by a Ti/Ag/Nb interlayer at the bonding conditions applied (850°C, 4 MPa with bonding time varying from 30 to 90 min). The influence of bonding time on the microstructure and mechanical properties of the joints was studied. When the bonding time was 30 min, the typical microstructure of the joints was as follows: graphite/TiC reaction layer/residual Ti layer/Ti₂Ag layer/TiAg/Ti[Ag,Nb]/Cu. As the bonding time increased, the thickness of the residual Ti layer decreased, whereas the thickness of the Ti[Ag,Cu] layer increased. When the bonding time was extended to 90 min, Ti–Cu intermetallic compounds were detected adjacent to the Cu. The shear strength showed an increasing trend with the extension of the bonding time. When the bonding time was 90 min, the maximum shear strength of the joints was 13.4 MPa.

Keywords: diffusion bonding, microstructure, graphite, Cu

1 Introduction

In recent years, with the continuous development of aviation technology, the data and commands that an airplane's electronic equipment need to process have become more and more complex [1]. This promotes the rapid development of semiconductor and microelectronic technology, miniaturization, weight reduction, high integration, and high frequency which have gradually become the trend in the development of microprocessor chips [2]. However, while the operating frequency of electronic equipment continues to increase, power density and heat generation are getting higher and higher [3]. Due its high thermal conductivity, excellent thermal shock resistance, and fatigue resistance, graphite has been used in the radiator components of electronic equipment [4]. Generally, graphite needs to be bonded with metals (Cu, Al) to form a radiator.

As we all know, graphite has a high melting point (3,550°C) and cannot be solid-solved with Cu or form any stable carbides with Cu. Therefore, in this case, fusion welding and direct diffusion bonding methods have been abandoned. Recently, various bonding techniques have been used to bond graphite and metal, such as brazing [5,6], solid-state diffusion bonding [7], and transient liquid phase diffusion bonding [8]. Among these joining methods, solid-state joining has attracted extensive attention due to its advantages such as no solidification structure in the bonding area, no pores, narrow joint interface, and large-area plate bonding [9]. Due to the large difference in the thermal expansion coefficient between graphite and Cu, the high residual stress at the joint deteriorated the performance of the joint. It is necessary to use metal materials with good plasticity as the stress release area to improve reliability of the joint.

In this study, a novel Nb/Ag/Ti composite interlayer was designed to join Cu and graphite. In the composite

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interlayer, Ti is the strong carbide-forming element, which is the key to realize the bonding between Cu and graphite. Ag has excellent ductility and can be used to relieve the stress caused by the large difference in the coefficient of thermal expansion between Cu and graphite. According to the Cu–Ag binary phase diagram [10], the eutectic reaction of Cu and Ag occurs at 775°C. So a large amount of the liquid phase will be formed above 800°C. However, large amounts of Ti dissolves in the AgCu eutectic liquid phase and reacts with Cu to generate a large number of brittle Ti–Cu intermetallic compounds [11]. This is harmful to the mechanical properties of the joint. Nb has a body-centered cubic structure, while Cu and Ag are face-centered cubic structures. Due to the difference in the crystal structure, the solubility between Nb and Ag or Cu is limited [12]. Hence, Nb is introduced as a barrier layer into the composite interlayer to avoid the generation of AgCu eutectic liquid phase and Ti–Cu intermetallic compounds.

In this study, the Ti/Ag/Nb composite interlayer was used to bond graphite and Cu at 850°C for different bonding time. The microstructure, interface evolution mechanism, and shear strength were investigated to clarify the bonding between the microstructure and mechanical properties of the joint during different bonding times.

2 Materials and experimental procedures

The high-strength graphite used in the experiment was obtained from the Beijing Jinglong Special Carbon Technology Co., Ltd. The commercially available Cu was provided by the State Key Laboratory for Advanced Metals and Materials, Beijing, China (99.9 wt%). The graphite was cut into the samples with sizes of 3.5 mm × 3.5 mm × 3 mm for shear strength test and 10 mm × 10 mm × 3 mm for observation of the microstructure, and the dimensions of Cu were

15 mm × 10 mm × 2 mm. The surface to be bonded was ground by SiC papers with grit sizes of 800#, 1,200#, and 2,000# sequentially. Then it was polished using 1 µm diamond pastes and cleaned ultrasonically for 20 min in acetone.

The composite interlayer was composed of Ti (8 µm)/Ag (6 µm)/Nb (4.8 µm), in which Ti and Ag were prepared by commercial foil and Nb was obtained by magnetron sputtering. The Ti and Ag foils were sandwiched between the graphite and Cu coated with Nb layer, as shown in Figure 1(a), and then the assembly was placed in a graphite fixture. A pressure of 5 MPa was applied to the bonding assembly to make the interlayer come in close contact with the substrate. The bonding process was performed using a vacuum furnace, and the vacuum atmosphere of 3×10^{-3} to 5×10^{-3} Pa was maintained during the bonding process. At first, the furnace was heated to 800°C at a rate of 20°C·min⁻¹ and kept for 20 min to homogenize the temperature in the furnace chamber. Then the temperature was increased to the bonding temperature at a rate of 5°C·min⁻¹, and isothermally soaked for 30, 60, and 90 min. Finally, the bonding sample was cooled down to 400°C at the rate of 5°C·min⁻¹ and then cooled in a furnace.

In order to characterize the microstructure of the joint, a scanning electron microscope (MERLIN Compact, ZEISS) equipped with an energy dispersive spectrometer (EDS) was used to observe the cross-sections of the joints. X-ray diffraction (XRD, D8-ADVANCE) was used to identify the phases in the joints. The specimens of the XRD test were cut parallel to the joint surface and then ground to expose the joint interface to be tested. The shear strength of the joints was measured by the Instron-5967 universal material testing machine. The bonded samples were assembled according to Figure 1(b), and then the shear test was carried out by applying pressure on Cu through the pressure head of the Instron-5967 universal material testing machine. The samples were loaded by the testing machine at a constant speed of 0.5 mm·min⁻¹. At least four samples were used to average each set of experimental data.

Figure 1: Schematic diagram: (a) assembling bonding parts and (b) shear test experiment.

3 Results and discussion

3.1 Microstructure characterization of the bonded joints

Figure 2 shows the microstructure and elemental distribution of the graphite/Cu joint bonded with Ti/Ag/Nb composite interlayer at 850°C for 30 min. As shown in

Figure 2(a) and (c–f), a joint without any cracks or pores was obtained. The joint could be divided into six zones. Zone I consisted of a continuous reaction layer close to the graphite. Zones II and IV were composed of dark gray phase and light gray phase respectively, while Zone III was the diffusion transition region between Zones II and IV. Zone V was made up of irregular dark gray phases, in which a small amount of white phase was dispersed. Zone VI was composed of the light gray phase, which

Figure 2: Microstructure (a), enlarged view of blue box 1–4 (c–f), and elemental analysis results (b, g–k) of the graphite/Cu joint bonded with Nb/Ag/Ti composite interlayer at 850°C for 30 min.

Table 1: Results of EDS at different positions shown in Figure 2 (at%)

Point	C	Ti	Ag	Nb	Cu	Possible phase
A	22.56	77.44	—	—	—	TiC
B	—	99.73	0.27	—	—	Ti
C	—	73.35	23.61	2.65	0.39	Ti + TiAg/Ti ₂ Ag
D	—	53.43	42.58	3.19	0.80	TiAg/Ti(Ag)
E	—	87.92	4.51	6.90	0.67	Ti[Ag,Nb]
F	—	—	16.94	83.06	—	Nb or Ag

was preliminarily inferred to be the Nb layer. The results of the EDS line scanning of the graphite/Cu joint are shown in Figure 2(b). It could be seen from the figure that the scanning results of Ti and C presented an X-shaped cross line, indicating that the Ti had diffused into the graphite as an active element and had reacted with the graphite at the interface. An obvious inter-diffusion process occurred between Ti and Ag. The atomic ratio of Ti and Ag in Zone IV was 1:1, and Ti was enriched between the TiAg and Nb layers. The Ti and Cu contents dropped sharply in Zone VI, indicating that the Nb element effectively weakened the diffusion of Ti and Cu. Figure 2(g)–(k) also confirmed this phenomenon.

The EDS results of each point in Figure 2 are shown in Table 1. XRD was carried out to further analyze the phases in the joint. The graphite near the joint interface was cut off and then ground until two positions were exposed (as shown in Figure 3(a)). Figure 3(b) and (c) shows the corresponding XRD patterns of different positions. Phase A is mainly composed of C and Ti, which was speculated to be TiC. From the XRD results of the graphite/composite interlayer (Plane A), TiC can be discovered. The dark phase with 99.73 at% Ti was the residual Ti layer. The TiAg phase detected in the XRD results of Plane B in Figure 3(c) also confirmed this result. Around 73.35 at% Ti and 23.61 at% Ag were detected in phase C, and it may be Ti₂Ag or Ti + TiAg. The EDS result of the off-white phase D showed that the ratio of the Ti content and Ag content in Zone IV was close to 1:1, which was inferred to be composed of the TiAg phase. The dispersive white phase E was composed of 16.94 at% Ag and 83.05 at% Nb, indicating that it was an Ag or Nb precipitated phase. The charcoal gray phase F was mainly composed of Ti, with a small amount of Ag and Nb, indicating that phase F is the Ti[Ag,Nb] solid solution. Therefore, it can be concluded that the typical microstructure of the joint is graphite/TiC/residual Ti layer/Ti + TiAg/TiAg/Ti[Ag,Nb]/Nb/Cu.

Figure 3: Schematic of the positions of graphite/Cu joints (a) and the corresponding XRD analysis: (b) Plane A and (c) Plane B.

3.2 Effect of bonding time on the microstructure of graphite/Cu joints

Figure 4 displays backscattered electron images of the graphite/Cu joints bonded by Ti/Ag/Nb composite interlayer at 850°C for 60 and 90 min, respectively.

It can be observed that the thickness of the residual Ti layer (Zone II) got reduced while the thickness of the enriched Ti layer (Zone V) increased with the increase in bonding time. Moreover, for the bonding time of 60 min, it was found that the widely distributed white phase in the Ti-enrichment layer had increased (Figure 4(a)). A new dark phase layer (Zone V₂) was observed at the position between Zone V₁ and Zone VI, and the compositions of phase A showed that it consisted of 55.64 at% Ti, 0.13 at% Ag, 42.28 at% Nb, and 1.95 at% Cu. From this, it was estimated that phase A is a Ti[Nb] solid solution.

When the bonding time was increased to 90 min, Zone III was obviously widened and its composition had changed. In Zone V, the white phase appeared agglomerated, and a large number of lamellar white phases were observed at the interface. Between Zones V and IV, continuous dendrites appeared and grew from Zone V₁ to Zone IV gradually. A significant diffusion layer appeared between the Nb layer and Cu. The element distribution was shown in Figure 4(c)–(g). From Figure 4(g), it can be clearly observed that Cu was mainly distributed in Zones III and V. Figure 4(c) shows that the Ti diffused through the Nb layer to Cu, thus forming Zone VII.

An EDS analysis was performed on each phase in the joint, and the results are shown in Table 2. The dark gray phase B in Figure 4(b) was consistent with phase B in Figure 2(d), both of which were residual Ti interlayers. The light gray phase C in the joint was composed of

Figure 4: Microstructure of the graphite/Cu joint bonded with Ti/Ag/Nb composite interlayer at 850°C: (a) 60 min, (b) 90 min, and elemental analysis results (b–g) of the joint for 90 min.

Table 2: Results of EDS at different positions shown in Figure 4 (at%)

Point	Ti	Ag	Nb	Cu	Possible phase
A	55.64	0.13	42.28	1.95	Ti(Nb)
B	63.03	11.35	18.69	6.93	Ti[Ag,Nb] + Ti-Cu
C	49.04	38.57	10.13	2.26	TiAg
D	46.28	28.39	7.53	17.80	TiAg + Ti-Cu
E	—	58.75	15.86	25.39	Ag solid solution (Ag(s, s))
F	44.22	9.51	6.04	40.23	Ti-Cu
G	61.03	0.32	7.33	31.32	Ti ₂ Cu
H	15.70	0.02	9.15	75.13	TiCu ₄

63.03 at% Ti, 11.35 at% Ag, 18.69 at% Nb, and 6.93 at% Cu, which was speculated to be Ti[Ag,Nb] + Ti-Cu. The light gray phase D in the dendrite structure was mainly composed of Ti, Ag, and Cu. It was speculated that the dendrite structure was composed of the Ti-Cu compound. Combined with the element distribution and EDS analysis, it can be inferred that white phase E was an Ag precipitated phase. The light gray phase F and dark gray phase G in Zone V ($V_1 + V_2$) were mainly composed of Ti and Cu, and the atomic ratios of Ti and Cu were 1:1 and 2:1, respectively. According to the Ti-Cu phase diagram [13], it can be inferred that the TiCu phase and the Ti₂Cu phase were formed. The light gray phase H in Zone VII was composed of Ti and Cu. The atomic ratio of Ti to Cu was close to 1:4, and it was speculated that the phase was TiCu₄.

3.3 Effect of bonding time on the mechanical properties of graphite/Cu joints

Figure 5 shows the shear strength of the graphite/Cu joints at different bonding times. It can be clearly seen that as the bonding time increased, the strength of the joint showed an upward trend. When the bonding times were 30 and 60 min, the shear strength of the joints were 10.7 and 11.9 MPa, respectively. When the bonding time was increased to 90 min, the joint strength reached the maximum value of 13.4 MPa.

By observing the fracture surface of the different bonding times, as shown in Figure 6, three types of failure modes were observed after the shear strength test. The first type is shown in Figure 6(a) and (a₁) ($t = 30$ min), which was cracking along the area between the Ti-rich layer (Zone V) and the Nb layer, indicating that this area was relatively weak. It can be observed from

Figure 6(b) and (b₁) that the phenomenon of interlayer peeling was found at the fracture position of the composite interlayer, indicating that the interface strength between the interface layers was weak. When the bonding time increased to 60 min, the fracture began near the TiC reaction layer between the graphite and the residual Ti layer, and then extended to the Cu side, forming a bowed fracture mode. When the bonding time reached 90 min (Figure 6(c) and (c₁)), the fracture process occurred inside the graphite base material. This fracture type indicated strong interfacial strength between each interface layer.

3.4 Microstructure formation and evolution mechanism of graphite/Cu joints

During the bonding process, C in the graphite diffused outward and reacted with the carbide forming Ti as follows [14–17]:

The TiC layer was formed at the interface. The formed TiC layer would be densely and metallurgical-wise bonded to the base materials on both sides, which was the key to realize the bonding between the carbon material and the metal material. In the Ti-Ag diffusion couple, Ti and Ag underwent inter-diffusion processes at different speeds. According to the diffusion theory, in reactive diffusion, the first phase was not formed immediately between the Ti-Ag diffusion couples but went through an inoculation period [18,19]. During the inoculation period, a certain degree of mutual diffusion and

Figure 5: Effect of bonding time on the shear strength of graphite/Cu joints.

Figure 6: The fractography of the graphite/Cu joints at 850°C: (a) 30 min, (b) 60 min, (c) 90 min; (a₁–c₁): enlarged view of the blue box in (a–c).

dissolution would occur between the diffusion couples in the area where the interface was extremely thin. Ag[Ti] saturated solid solution had formed around the defects of the Ag crystal structure. In the lattice defects with high Ti content, excessive Ti promoted the transformation of the Ag lattice-type to form a new phase crystal nuclei [20,21]. The crystal nuclei of the intermetallic compound grew laterally along with the joint interface, and the grown intermetallic compound formed a whole and turned to normal longitudinal growth. In this study, based on the interface (Figure 2(a)), it can be inferred that Zone II was the first intermetallic compound phase zone, and its phase was Ti_2Ag . When the Ti_2Ag layer grew to a certain thickness, the second phase TiAg would nucleate and grow.

After all the Ag was transformed, Ti continued to diffuse to the Nb side driven by the concentration gradient, passing through the TiAg and getting collected on the Nb side. This may be related to the supersaturated precipitation of Ti in the Ag phase and the hindering effect of Nb on Ti diffusion. According to the Ti–Nb phase diagram [22], the Ti–Nb system displays considerable mutual solubility and no formation of intermetallic compounds. So Ti[Nb] gets formed in the Ti-rich layer near the Nb side. As the bonding time increased ($t = 60$ min), the Ti[Nb] layer continued to thicken to form Zone V₂ in Figure 4(a). Although the formation of harmful intermetallic compounds (Ti–Cu compounds) was avoided, the Nb–Cu system is actually immiscible and its solubility is negligible, resulting in weak binding between the Nb–Cu interface. The Ag/Nb double interlayers reasonably regulated the diffusion

path of Ti. For the joints with bonding time of 30 min, the mutual diffusion degree of Ti and Nb was insufficient, and no continuous Ti [Nb] layer was formed. In the shear test, the interface between the rich-Ti layer and Nb layer was a weak interface, resulting in the fracture process at this interface. When the bonding time was extended to 60 min, the obvious Ti[Nb] layer was formed, which strengthened the bonding between the Ti-rich layer and the Nb layer. The strength of each interface layer in the joint was equivalent, which caused the joint to fracture from the graphite matrix to the Cu matrix.

In addition, when the bonding time reached 90 min, the effect of Nb on Ti and Cu diffusion was reduced, and long-distance diffusion of Cu occurred. Due to the negative mixing enthalpy between Cu and Ti, the two had a tendency to form intermetallic compounds [23,24]. Therefore, when Cu diffused into the Ti-rich layer and exceeded its saturated solid solubility in Ti, the lattice transformation of Ti would be induced. It caused part of the Ti in the Ti-rich layer to transform into Ti–Cu intermetallic compounds.

At the same time, Cu could even pass through the TiAg layer, transforming part of the Ti in Zone III into Ti–Cu intermetallic compounds. In the meantime, Ti passed through the Nb layer and reacted with part of the Cu to form a scallop-like Ti–Cu intermetallic compounds layer. Therefore, three continuous Ti–Cu intermetallic compound layers were observed in joints. The formation of Ti–Cu compounds can be expressed by the following equation [25]:

According to the equations, four kinds of compounds (Ti_2Cu , Ti_3Cu_4 , TiCu , TiCu_4) are present, and the free energies of formation of these Ti–Cu compounds at 850°C are all negative, which indicates that all the four compounds are thermodynamically favorable. The generated Ti–Cu intermetallic compound had a pinning effect at the interface and enhanced the interface bonding between various interfaces. The shear strength of the joint was improved, causing the fracture to occur inside the graphite.

4 Conclusion

Graphite and Cu were successfully bonded by using the Ti/Ag/Nb interlayer at 850°C for different holding times. The following conclusions can be drawn:

- (1) A continuous TiC reaction layer was formed at the graphite/interlayer interface. During the bonding time of 30 min, the typical microstructure of the joints was as follows: graphite/TiC reaction layer/residual Ti layer/Ti–Ag diffusion layer/TiAg/Ti[Ag,Nb]/Cu.
- (2) As the bonding time increased, the thickness of the remaining Ti layer decreased, while the thickness of Ti[Nb] increased. However, when the holding time was increased to 90 min, Ti–Cu intermetallic compounds were detected in multiple interface layers of the graphite/Cu joint.
- (3) The shear strength increased nonlinearly with the extension of the bonding time. The maximum shear strength of the joint reached 13.4 MPa during the bonding time of 90 min, and the fracture position is inside the graphite matrix.

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