Microstructure and Shear Strength of Brazing TiAl/Si₃N₄ Joints with Ag-Cu Binary Alloy as Filler Metal

Duo Dong 1,2, Dongdong Zhu 1,*, Ye Wang 3, Gang Wang 4,*, Peng Wu 4 and Qing He 1

1 Key Laboratory of Air-Driven Equipment Technology of Zhejiang Province, Quzhou University, Quzhou 324000, China; dongduohit@163.com (D.D.); helinqi@163.com (Q.H.)
2 State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 410083, China
3 School of Materials Science and Engineering, Harbin University of Science and Technology, Harbin 150001, China; wangye1984@hrbust.edu.cn
4 School of Mechanical and Automotive Engineering, Anhui Polytechnic University, Wuhu 241000, China; ahpwuwp@163.com
* Correspondence: zhudd8@163.com (D.Z.); gangwang@ahpu.edu.cn (G.W.);
Tel.: +86-570-802-6634 (D.Z. & G.W.)

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Abstract: Vacuum brazing of TiAl intermetallic alloy to Si₃N₄ ceramics was performed using Ag-28Cu (wt.% filler alloy. The brazing joints obtained at different brazing temperatures were studied in this work. The microstructure and the shear strength were analyzed in detail. The results show that the brazed joints could be divided into three regions: AlCu₂Ti reaction layer near the Ti-48Al-2Cr-2Nb alloy, a typical Ag-Cu eutectic structure and a thin continuous TiN + Ti₅Si₃ reaction layer near the Si₃N₄ ceramics. The microstructure varied as the brazing temperature was increased from 1153 K/15 min to 1193 K/15 min. The shear strength of the joints first increased as the brazing temperature increased from 1153 K to 1173 K, and then decreased. The maximum shear strength reached 105.5 MPa at 1173 K/15 min and the mechanism was discussed.

Keywords: TiAl alloy; Ag-Cu; brazing; microstructure; Si₃N₄

1. Introduction

In the last decades, TiAl alloys have received considerable attention due to its higher specific strength, lower density and excellent creep resistance at elevated temperatures, which can be used as potential candidates for high temperature structural applications [1–4]. Recently, Ti-48Al-2Cr-2Nb blades have been used in commercial turbofan engines of General Electric Company [5–7]. However, due to the brittle nature and poor workability, fabricating large scale or complex-shaped components of TiAl alloys has many challenges [8–10].

To utilize the advantages of TiAl alloys, it is necessary to join small TiAl components to other materials. Recently, researchers have studied the vacuum brazing of TiAl alloys to ceramics. So far, various methods for ceramic-metal joining have been commonly used [11–14]. Compared with transient liquid phase bonding, diffusion bonding, etc., vacuum brazing is advanced in simplicity, lower cost and high quality [15,16]. Si₃N₄ ceramic is one of the most widely used ceramics in the thermal machine, heat exchangers, wear resistant elements and wave-transparent materials due to its excellent properties, such as light weight, high resistance to thermal shock, excellent creep resistance and wear properties [17–19]. When used as wave-transparent materials in antenna radomes, it would be brazed to a TiAl intermetallics holder. In fact, many factors would influence the brazing
quality, such as the surface when used as chip resistors [20]. When used for structural applications, the quality of the surface has rarely been studied. However, up to now, the brazing of Si₃N₄ ceramics to Ti-48Al-2Cr-2Nb has rarely been reported.

It is well known that the filler metals play an important role in brazing ceramics to metals. Due to the low melting point and excellent wettability and ductility [21–23], the AgCu based filler alloy was effective in reducing the mismatch of the coefficients of thermal expansion. Correspondingly, the residual thermal stresses were reduced. So, AgCu based filler alloys have been widely used in brazing Si₃N₄ ceramics to metals.

In this work, an Ag-28Cu filler alloy was developed to braze the Si₃N₄ ceramics to a Ti-48Al-2Cr-2Nb alloy. The microstructure of the brazed joints was characterized by X-ray diffraction (XRD) and scanning electronic microscopy (SEM, Hitachi, Tokyo, Japan) equipped with energy dispersive spectroscopy (EDS, Bruker, Karlsruhe, Germany). The joining properties are also studied.

2. Materials and Methods

Si₃N₄ ceramics used in this paper were hot pressed with Al₂O₃ additives. The metal partner used was TiAl intermetallic with composition of Ti-48Al-2Cr-2Nb (at.%). The Si₃N₄ ceramics and TiAl intermetallic was cut into blocks with the dimension of 4 mm × 4 mm × 4 mm and 10 mm × 10 mm × 4 mm, respectively. The Ag-28%Cu filler alloy used in this study was about 50 µm in thickness. Prior to joining, the brazing surfaces were ground on SiC grit papers and polished. All of the polished samples were ultrasonically cleaned by acetone. The Ag-Cu filler alloy was sandwiched between Si₃N₄ ceramic and Ti-48Al-2Cr-2Nb alloy specimens as shown in Figure 1a. A pressure of about 20 kPa was applied on the brazing assembly to ensure close contact of the surfaces by graphite blocks. The assembly was brazed in a vacuum of less than 2 × 10⁻³ Pa. The brazing processes were 1153 K/15 min, 1173 K/15 min and 1193 K/15 min, respectively.

![Figure 1. Schematic drawing of: (a) Brazing assembly; (b) shear test.](image)

The microstructure of the brazed joints was examined by SEM (Hitachi, Tokyo, Japan) equipped with EDS. A thin layer of gold was sprayed before the SEM examination. Subsequently, the SEM scans were carried out with the operation voltages of 15 kV. The phase analysis in the brazing seam was characterized by XRD. The XRD analysis was carried out on the surface of the shear test samples. The shear strength of the brazed joint was carried out on an Instron electronic universal testing machine (Instron, Boston, MA, USA), and the schematic illustration of the room temperature shear test is shown in Figure 1b. Five shear strength samples were tested to obtain the average shear strength.

3. Results and Discussion

3.1. Microstructure of TiAl/AgCu/Si₃N₄ Joints

The typical cross-section of the Ti-48Al-2Cr-2Nb alloy and Si₃N₄ ceramic joint brazed with Ag-Cu filler metal at 1173 K is shown in Figure 2a. It can be seen obviously that a defect-free joint was formed between the base materials and the Ag-Cu filler alloy. The brazing joint can be divided into three...
characteristic regions, as marked in Figure 2a. It can be seen from Figure 2b–f, Ag, Cu, Ti and Al are the major elements in the brazing joint. The zone I is the Cu and Ti rich reaction layer near the Ti-48Al-2Cr-2Nb alloy, the zone II has typical Ag-Cu eutectic structure which can often be observed when ceramics were brazed by Ag-Cu-Ti filler alloy [24,25], where the bright phases are Ag-rich and the dark gray phases are Cu-rich. The zone III is the thin continuous reaction layer at the Si₃N₄ ceramic side.

The magnified images of the regions are given in Figure 3. The phases in the brazing joint were characterized by XRD and the results are shown in Figure 4. It can be seen that the γ-TiAl, Si₃N₄, Ag, AlCu₂Ti, TiN and Ti₅Si₃ phase can be found in the brazing seam. The EDS is also employed to analyze the chemical compositions of the phases. The different phases in the brazing joint are specified as A–H respectively and the detected results and the possible phase are all listed in Table 1. As the XRD analysis was carried out on the surface of the shear test samples and a low volume fraction of phase constitution (less than 5%) may not be detected by XRD, the phase detected by XRD is different from those identified by EDS. The phases marked B, C, and D can be found in the interface between the Ti-48Al-2Cr-2Nb and the Ag-Cu filler (zone I). The white phase marked B is Ag(s,s) incorporating 75.1% Ag, 12.3% Cu, 6.0% Ti, 5.6% Al and 0.9% Cr, which is determined to be Ag(s,s). The atomic ratio of Al, Cu and Ti is about 1:2:1 in phase marked C. According to the Al-Ti-Cu phase diagram, it is AlCu₂Ti phase. Additionally, the dissolution of the γ-TiAl in the Cu-Ag liquid phase will lead to the phase containing AlCu₂Ti and Cu(s,s) precipitated during the solid process: \( L = \text{AlCu}_2\text{Ti} + \text{Cu(s,s)} \), such as phase D. The chemical compositions of the bright phase marked E inside the brazing seam (zone II) can also be identified as Ag-rich. It can also be seen that there are many dark phases marked F formed in Ag(s,s). According to the EDS results, the phase is the Cu rich phase. The chemical composition result of phase G in zone III indicates that Ti atoms diffused to the interface next to the Si₃N₄ ceramics, and formed TiN + Ti₅Si₃ reaction layer via the reaction: \( 9\text{Ti} + \text{Si}_3\text{N}_4 = 4\text{TiN} + \text{Ti}_5\text{Si}_3 \). The Gibbs free energy of this reaction at 1173 K is \(-1297.945 \text{ kJ/mol} + (\Delta G_f \text{ (kJ/mol)} = -1550.14 + 0.215T) \) [26], which confirms the compounds TiN and Ti₅Si₃ are both stable. The TiN + Ti₅Si₃ reaction layer can also be found other studies.

**Figure 2.** Elemental analysis results of the TiAl/AgCu/Si₃N₄ joints brazed at 1173 K. (a) Interfacial microstructure; (b–f) element distribution images of Ag, Cu, Ti, Al and Si.
The thickness of the whole brazing joints is about 85 µm at 1153 K, 63 µm at 1173 K, and 42 µm at 1193 K. The thickness of the brazing joints decreases with the increasing temperature. The main reason is that the TiN + Ti₅Si₃ reaction layer thickened with increasing temperatures. When brazed at 1153 K, a macro-crack can be clearly seen in Figure 5a. It can also be found that the volume fraction of the Cu-rich phase in Ag(s,s) (zone II) decreases.

Further increasing the brazing temperature to 1173 K, the volume fraction of the continuous AlCu₂Ti layer increases greatly and the volume fraction of the Cu-rich phase decreases.

| Phase | Al | Ti | Ag | Cu | Si | N  | Nb | Cr | Possible Phase              |
|-------|----|----|----|----|----|----|----|----|----------------------------|
| A     | 48.0 | 49.6 | -  | -  | -  | 1.4 | 1.0 | γ-TiAl                     |
| B     | 5.6  | 6.0  | 75.1 | 12.3 | -  | 0.1  | 0.9 | Ag-rich                    |
| C     | 23.5 | 26.6 | 0.6  | 47.4 | -  | 0.8  | 1.1 | AlCu₂Ti                    |
| D     | 8.4  | 7.7  | 22.4 | 59.7 | -  | 0.5  | 1.3 | AlCu₂Ti + Cu(s,s)          |
| E     | 4.8  | 3.8  | 71.3 | 13.5 | 2.0 | 3.2  | 0.4 | 1.0 | Ag-rich                    |
| F     | 24.6 | 0.5  | 21.6 | 51.5 | 0.1 | 0.5  | 1.2 | Cu rich phase              |
| G     | 0.2  | 59.4 | 4.7  | 2.3  | 15.6 | 17.5 | 0.1 | 0.2 | TiN + Ti₅Si₃               |
| H     | 5.3  | 3.2  | 1.0  | 1.1  | 52.9 | 35.1 | 0.3 | 1.1 | Si₃N₄                      |

Figure 5 shows the SEM images of the TiAl/AgCu/Si₃N₄ joints brazed at different brazing temperatures for 15 min. It clearly demonstrates that the brazing joints also consisted of three zones: AlCu₂Ti + Ag(s,s) layers (zone I), Ag(s,s) (zone II) and TiN + Ti₅Si₃ reaction layer (zone III). The thickness of the whole brazing joint is about 85 µm at 1153 K, 63 µm at 1173 K, and 42 µm at 1193 K. The thickness of the brazing joints decreases with the increasing temperature. The main reason is that the molten filler metal would be expelled out of the brazing seam at higher brazing temperatures. It can be seen that the TiN + Ti₅Si₃ reaction layer thickened with increasing temperatures. When brazed at 1153 K, a macro-crack can be clearly seen in Figure 5a. It can also be found that the volume fraction of the Cu-rich phase in Ag(s,s), which is beneficial to strengthen to brazing seam, gets to the highest level at 1173 K. Further increasing the brazing temperature to 1173 K, the volume fraction of the continuous AlCu₂Ti layer increases greatly and the volume fraction of the Cu-rich phase decreases.

Figure 4. X-ray diffraction (XRD) results of the fracture surfaces brazed at 1173 K.

Table 1. Chemical analysis (at.%) of the phases marked in Figure 3.
Figure 5. The interfacial microstructures of TiAl/AgCu/Si$_3$N$_4$ brazed joints at different brazing temperatures. (a) 1153 K, (b) 1173 K, (c) 1193 K.

From the analysis above, the formation mechanism of the TiAl/AgCu/Si$_3$N$_4$ brazed joints can be concluded. When the heating temperature reaches the melting point of the Ag-Cu filler alloy during brazing, it began to melt. Ti and Al would diffuse to the brazing seam. The AlCu$_2$Ti layer would form at the TiAl side. At the same time, the Ti atom diffused to the Si$_3$N$_4$ side, which can react with Si$_3$N$_4$ to form TiN + Ti$_5$Si$_3$ reaction layer next to the Si$_3$N$_4$ side. When brazed at 1153 K/15 min, a macro-crack was generated in the brazing seam, which is caused by the lower flowability of the filler alloy. The diffusion rate is also lower, and then the volume fraction of Cu-rich phase in zone II is a bit higher. With increasing the brazing temperature to 1173 K, the macro-crack has disappeared due to higher flowability. Further increasing temperature to 1193 K, more Ti and Al will diffuse to the brazing seam and more Cu would diffuse to the TiAl side at higher brazing temperature, the volume fraction AlCu$_2$Ti phase increases. The thickness of the brazing seam decreases with the increasing temperatures. The reduction was due to the expulsion of molten Ag-Cu filler alloy by capillarity out of the brazing seam at higher temperatures.

3.2. Mechanical Properties of TiAl/AgCu/Si$_3$N$_4$ Brazed Joints

Figure 6 shows the shear strength of the TiAl/AgCu/Si$_3$N$_4$ joints brazed at different temperatures. It can be seen that the highest shear strength was obtained at 1173 K/15 min. The shear strength is about 46.3 MPa at 1153 K, 105.5 MPa at 1173 K and 85.8 MPa at 1193 K. Vacuum brazing of the TiAl intermetallic alloy to Si$_3$N$_4$ ceramics was performed using Ag-Cu-Ti filler alloy in Song’s study [27]. The maximum shear strength is about 14.15 MPa. It can be clearly seen the maximum average shear strength is much higher than previous studies.

According to the microstructural analysis, the brazing seam was mainly composed of AlCu$_2$Ti, Ag(s,s) and TiN + Ti$_5$Si$_3$ reaction layer. When brazed at 1153 K, the shear strength is a bit lower. The main reason is that the diffusion of element is incomplete at low brazing temperature. Macro-cracks were formed in the brazing seam, which can deteriorate the shear strength greatly. When the
brazing temperature increases to 1173 K, the element would diffuse more intensively. The cracks have disappeared in the brazing seam. It can also be seen that more rich Cu phase was found in Ag(s,s), which can strengthen the brazing seam. All these lead to the increasing of shear strength. Further increasing temperature to 1193 K, the Ag(s,s) layer in zone II and the rich Cu phase in Ag(s,s) decreased, then the shear strength shows a slight decrease. Figure 7 shows the fracture modes of the TiAl/AgCu/Si₃N₄ joints. When brazed at 1153 K, the joint fractured along the brazing seam, as shown in Figure 7a. This fracture mode was formed mainly because the macro-defects were formed at 1153 K, the joints showed a weak bonding. When brazed at 1173 K and 1193 K, the fracture ruptured at the substrate, as shown in Figure 7b,c. The results showed that the residual stress was reduced when Ag-Cu filler alloy was used.

![Figure 7. Fracture modes of TiAl/AgCu/Si₃N₄ joint brazed at different temperatures. (a) 1153 K, (b) 1173 K, (c) 1193 K.](image)

4. Conclusions

TiAl intermetallic alloy and Si₃N₄ ceramics were successfully brazed by the Ag-Cu filler alloy. The microstructure and shear strength of the brazed joint at different brazing processes were investigated. The primary conclusions are summarized as follows:

1. A sound joint without cracks or voids can be formed between the base materials and the Ag-Cu filler alloy. The brazing joints consist of three zones: AlCu₂Ti reaction layer, Ag(s,s) and TiN + Ti₅Si₃ reaction layer. The typical microstructure of the brazing joints is the AlCu₂Ti, Ag(s,s), Cu rich phase, TiN and Ti₅Si₃ phase.

2. As the brazing temperatures increases, the thickness of the whole brazing joints decreases which is attributed to the fact that the molten filler metal would be expelled out of the brazing seam at higher brazing temperatures. The reaction layer of TiN + Ti₅Si₃ also thickened with increasing brazing temperatures. When brazed at 1153 K, a macro-crack was generated in the brazing seam, which is caused by the lower flowability of the Ag-Cu filler alloy. At 1193 K, a few micro-cracks can be found in the brazing seam. A defect-free joint was obtained at 1173 K.

3. As the brazing temperatures increases, the shear strength first increased and then decreased. The maximal shear strength of the joints reached 105.5 MPa at 1173 K. Macro- and micro-cracks and the Cu-rich phase can influence the shear strength greatly.

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