Brazing Si_3N_4 Ceramic to Molybdenum Using an Ag-Cu-Ti Filler

The microstructure and interface formation mechanism of the $\text{Si}_3\text{N}_4/\text{Mo}$ joint was analyzed

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ABSTRACT

A Si_3N_4 ceramic was successfully joined to molybdenum (Mo) using an Ag-Cu-Ti filler alloy. The interfacial microstructure of the $\text{Si}_3\text{N}_4/\text{Ag-Cu-Ti}/\text{Mo}$ joint was investigated by scanning an electron microscopy, energy dispersive spectrometer, and x-ray diffraction. The results showed the joint brazed at 900°C for 10 min was smooth, and there were no holes and cracks at the interface. A continuous reaction layer, which is composed of TiN and TiSi_2 , was formed near the Si_3N_4 ceramic, with TiN being located near the ceramic. The central part of the joint was composed of Ag- and Cu-based solid solutions. At the side near the Mo metal, there was a formation of the MoTi solid solution. The typical structure of the $\text{Si}_3\text{N}_4/\text{Mo}$ joint was $\text{Si}_3\text{N}_4/\text{TiN} + \text{TiSi}_2$ reaction layer/Ag(s,s) + Cu(s,s)/MoTi/Mo. Because TiN and TiSi_2 compounds are generated on the ceramic side, the microhardness of the reaction layer on the ceramic side was decreased but still much higher than the hardness of the brazing seam and the Mo base material. The shear strength of the brazed joint was 204 MPa at room temperature.

KEYWORDS

- Si_3N_4 Ceramic • Molybdenum • Brazing
- Interfacial Microstructure • Formation Mechanism

Introduction

Si_3N_4 ceramic has attracted considerable interest in recent years primarily due to its high thermal conductivity, low dielectric loss, good thermal shock resistance, high bending strength, and fracture toughness, so it is considered a potential high-power heat dissipation and packaging material (Ref. 1). However, the hardness and brittleness make it difficult to obtain large-sized and complex components. Therefore, joining Si_3N_4 to metallic materials is important to satisfy its application.

There have been some methods, such as diffusion bonding (Ref. 2), transient liquid phase (Ref. 3), and brazing bonding (Ref. 4), proposed for ceramic metal or joining.

Among these methods, brazing bonding has attracted extensive attention due to its simplicity and cost effectiveness (Ref. 5) and has become the most commonly used reliable method to connect ceramics and metals. It has been reported that Si_3N_4 ceramic has successfully brazed together with copper (Ref. 6), TC4 alloy (Refs. 7, 8), TiAl alloy (Refs. 9–11), Invar alloy (Refs. 12–14), and 42CrMo steel (Refs. 15–17).

Molybdenum (Mo) has good thermal conductivity and low coefficient of thermal expansion (CTE). Hadian et al. (Ref. 18) used two Ni-based brazing alloys to braze Mo and Si_3N_4 . Mo, Si, and N diffused from the parent materials. Mo compounds were formed at the $\text{Si}_3\text{N}_4/\text{filler metal}$ interface, resulting in brazing joints in the ceramic/filler degumming at the interface. Peteves and Nicholas (Ref. 19) used Mo as a middle layer to braze Si_3N_4 ceramics. Mo was more successful as an interlayer than Cu or Nb, with its CTE matching the ceramic and affecting less the reactive route for the joint formation. Recently, research has focused on adding Mo particles as a reinforcing phase to the brazing filler metal to reduce the CTE mismatch between the ceramic base material and brazing filler metal (Ref. 20), improving the joint strength. However, there are few reports on the joining of Mo/ceramic assemblies using an Ag-Cu-Ti filler. Therefore, it is of great significance to study the brazing process and connection mechanism of Mo and Si_3N_4 ceramics.

In this work, Si_3N_4 was joined to Mo using the traditional Ag-Cu-Ti brazing filler. The microstructure of the $\text{Si}_3\text{N}_4/\text{Mo}$ brazed joint was studied by means of metallographic observation, scanning electron microscopy (SEM), and x-ray diffraction (XRD). At the same time, microhardness tests were performed. The interface formation mechanism brazing parameters on the interfacial microstructure and mechanical properties were also analyzed.

Experimental Procedure

Materials

The materials used in this study were a Mo block and Si_3N_4 pieces. Both Mo, with a purity of 99.9 wt-%, and

Fig. 1 — Schematic diagram of the sample clamping structure.

Fig. 2 — Brazing temperature curve.

Fig. 3 — Schematic diagram of the shear test specimen.

Fig. 4 — Metallographic microstructure of the $\text{Si}_3\text{N}_4/\text{Mo}$ joint.

Table 1 — Main Properties of Ceramics and Molybdenum (Mo) at Room Temperature

Material	Modulus of Elasticity (GPa)	Thermal Expansion Coefficient (10^{-6}K^{-1})	Melting Point ($^{\circ}\text{C}$)	Thermal Conductivity (W/m·K)	Density ($\text{g}\cdot\text{cm}^{-3}$)
Si_3N_4	300	3.4	—	80	3.2
Mo	330	5.35	2617	138	10.2

Table 2 — The Main Performance of Ag-27Cu-3.3Ti

Material	Ingredient (wt-%)	Melting Point ($^{\circ}\text{C}$)	Thickness (μm)
Ag-Cu-Ti foils	69.7Ag + 27Cu + 3.3Ti	780 ~ 805	100

Si_3N_4 , with a density of 3.2 g/cm^3 , were supplied by Shanghai Institute of Ceramics, Chinese Academy of Sciences. The Si_3N_4 and Mo samples gave the dimensions of $6 \times 6 \times 1$ and $9 \times 6 \times 6 \text{ mm}^3$, respectively. The filler metal of $100 \mu\text{m}$ Ag-27Cu-3.3Ti wt-% foil was processed into a square sheet with a side length of $6 \times 6 \text{ mm}^2$ as the faying surface of the joint. The properties and performance of the base materials and filler are shown in Tables 1 and 2.

Joining Process

The joining surfaces of the Mo and Si_3N_4 samples were treated by polishing with a #400 sandpaper followed by ultrasonic cleaning in acetone for 8 min.

The schematic diagram of the brazed joint is shown in Fig. 1. Vacuum brazing of Mo to Si_3N_4 was carried out in a MOV-443-type furnace under a vacuum of $2.0 \times 10^{-4} \text{ Pa}$. The

Fig. 5 — Secondary electron imaging of brazed $\text{Si}_3\text{N}_4/\text{Mo}$ joints brazed at 900°C for 10 min: A — Overall morphology; B — $\text{Si}_3\text{N}_4/\text{Ag-Cu-Ti}$ interface area; C — $\text{Ag-Cu-Ti}/\text{Mo}$ interface area.

temperature curve is shown in Fig. 2. The assemblies were heated from room temperature to 300°C at a rate of $20^\circ\text{C}/\text{min}$ and held for 10 min. Subsequently, they were heated up to 900°C at a rate of $10^\circ\text{C}/\text{min}$. After holding for 10 min at the brazing temperature of 900°C , the assemblies were forced to cool to 300°C at a rate of $5^\circ\text{C}/\text{min}$ with argon. Finally, the joint cooled with the furnace-to-room temperature in vacuum. During brazing, a self-made fixture was used to apply a certain pressure to prevent the sample from moving when the brazing material melted.

Microstructural Characterization

After brazing, the sample was first embedded by a cold inlay method. Then, the connection surface was polished with different grades of metallographic sandpaper, and the brazed joint was etched by mixed acid ($\text{HCl}:\text{HNO}_3:\text{H}_2\text{O} = 2:1:3$) to facilitate the observation of the metallographic structure under a VHX-500F digital microscope. Before observation, the surface of the joint was wiped with absolute ethanol. Metal salt produced during the etching process can be dissolved in water without introducing other impurities.

The interfacial zones of $\text{Si}_3\text{N}_4/\text{Mo}$ joints were characterized by a XL30FEG SEM equipped with an energy dispersive spectrometer (EDS). The phase identification of the interfacial zones in the joints was conducted by a D8 ADVANCE XRD spectrometer (choose $\text{Cu K}\alpha$ radiation, set current to 40 mA, set acceleration voltage to 40 kV, and set scan speed to $10^\circ\text{C}/\text{min}$).

The microhardness test for the $\text{Si}_3\text{N}_4/\text{Mo}$ joint was performed with a HV-1000 microhardness tester. The test pressure at the weld was 25 gf; the test pressure at the Mo and ceramic sides were 100 and 500 gf, respectively; and the holding time was 15 s. The shear strength was performed by a WDW-10 universal testing machine at a constant speed of 0.5 mm/min. Figure 3 shows the shear test specimen used in this experiment.

Results and Discussion

Brazing Joint Morphology

Figure 4 shows the metallographic microstructure of the $\text{Si}_3\text{N}_4/\text{Mo}$ joint brazed with the Ag-Cu-Ti filler at 900°C for 10 min. It can be seen that the filler had good contact with the base materials on both sides, and there were no obvious cracks or holes in the joint. Between Si_3N_4 and the Ag-Cu-Ti filler, there is a gray interfacial reaction Layer I with a thickness of about $3.8\text{ }\mu\text{m}$, indicating that the filler reacted with Si_3N_4 . Layer II is part of the Ag-Cu-Ti filler, mainly composed of a dark and large, light-colored phase. At a high temperature, the filler melted and overflowed to both sides, making the thickness of the intermediate layer slightly lower than the original thickness of the filler. Layer III is the interface layer of the Ag-Cu-Ti/Mo.

Typical Interfacial Microstructure of $\text{Mo}/\text{Ag-Cu-Ti}/\text{Si}_3\text{N}_4$ Brazed Joints

Figure 5 is the secondary electron imaging diagram of microstructure morphology of the $\text{Si}_3\text{N}_4/\text{Mo}$ joint, and Fig. 5B and C is a magnified micrograph of regions 1 and 3, respectively. Region 1 is the transition reaction layer between Si_3N_4 and the Ag-Cu-Ti filler. From region 2, it can be seen that the middle filler layer is a eutectic structure, and region 3 is a relatively

Fig. 6 — Typical microstructure and elements distribution of the $\text{Si}_3\text{N}_4/\text{AgCuTi}/\text{Mo}$ joint at 900°C for 10 min: A — Typical interfacial microstructure; B–G — element distribution maps of N, Si, Ti, Cu, Ag, and Mo.

flat bonding interface between filler and Mo. To obtain the elemental compositions of regions 1–3, microareas A–F were measured by an EDS, and the results are given in Table 3.

According to the literature (Refs. 21–23), in the microstructure of the $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$ or $\text{Si}_3\text{N}_4/\text{metal}$ joint brazed with the Ag–Cu–Ti filler, a thin Ti–N layer will be formed on the Si_3N_4 ceramic side and the Ti–Si phase will be formed next to the Ti–N layer. The light gray microarea B in region 1 contained N (41.21 at.-%), Ti (31.97 at.-%), and Si (22.47 at.-%), inferred as TiN and Si. The microarea C located in region 1 consisted of Si (47.61 at.-%) and Ti (22.31 at.-%), corresponding to TiSi_2 .

The light gray microareas D and E in region 2 gave elemental compositions of Ag (86.97 at.-%) and Cu (64.77 at.-%), respectively, determined as Cu solid solution (Cu[s,s]) and Ag solid solution (Ag[s,s]). The microarea F in region 3 contained Mo (17.63 at.-%), Ti (21.93 at.-%), Ag (35.16 at.-%), and Cu (25.29 at.-%), inferred as MoTi and AgCu(s,s).

Figure 6 presents a back-scattered electron image of the $\text{Si}_3\text{N}_4/\text{Mo}$ joint brazed with the AgCuTi alloy at 900°C for 10 min and the corresponding element distribution maps throughout the joint. It can be seen that during the brazing process, the active element Ti diffused from the middle of the filler to the base materials on both sides, and the Ti content on the Si_3N_4 side was higher than that on the Mo side, indicating that the Ti element diffused toward the Si_3N_4 side. Mo did not diffuse from the base material to the filler and ceramic side.

The phase identification of the interfacial zone in the

Table 3 — Chemical Compositions and Possible Phases of Each Spot Marked in Fig. 5 (at.-%)

Spot	Chemical Compositions (at.-%)						Possible Phases
	Si	N	Mo	Ag	Ti	Cu	
A	55.14	44.86	—	—	—	—	Si_3N_4
B	22.47	41.21	—	00.68	31.97	03.66	TiN + Si
C	47.61	05.17	—	16.65	22.31	08.24	TiSi_2
D	03.95	01.19	—	86.97	00.67	07.22	Ag (s,s)
E	—	08.62	03.59	15.58	07.44	64.77	Cu (s,s)
F	—	—	17.63	35.16	21.93	25.29	MoTi + AgCu (s,s)
G	—	—	91.20	08.80	—	—	Mo

Fig. 7 — XRD analysis of the $\text{Si}_3\text{N}_4/\text{Mo}$ joint.

$\text{Si}_3\text{N}_4/\text{Mo}$ joint was performed by an XRD, and the results are shown in Fig. 7. It can be seen that there were TiN, TiSi_2 , and MoTi solid solutions in the brazing joint. Combined with the analysis of the XRD and EDS results, the interface structure of the brazed joint was indicated as $\text{Si}_3\text{N}_4/\text{TiN} + \text{TiSi}_2/\text{Ag(s,s)} + \text{Cu(s,s)}/\text{MoTi}/\text{Mo}$.

The microhardness distribution along the $\text{Si}_3\text{N}_4/\text{Mo}$ joint brazed with Ag-Cu-Ti is depicted in Fig. 8. It can be seen from the figure that the microhardness of Mo after brazing was about 300 HV. The average hardness values of the filler layer were lower than those of base materials.

However, the microhardness of Si_3N_4 ceramics was very high, about 1800 HV. During the brazing process, the TiN + TiSi_2 compound was formed due to the reaction between Ti and Si_3N_4 ceramic, which reduced the hardness of the reaction layer on the ceramic side, about 1500 ~ 1800 HV, but was still much higher than the microhardness at the brazing seam and Mo. Then, the shear strength of the joint brazed with AgCuTi was 204 MPa at room temperature.

Interface Formation Mechanism of Joint

According to the above analysis, the microstructure formation process of Si_3N_4 ceramics and Mo joints brazed with Ag-Cu-Ti filler can be summarized into several stages, as shown in Fig. 9.

1) During the brazing process, when the heating temperature reached the melting point of the filler, the filler melted transit into a liquid phase, the active Ti element diffused to the base materials on both sides under the driving force of the chemical potential difference, and the Mo element in the metal dissolved into the filler, as shown in Fig. 9A.

2) As shown in Fig. 9B, as brazing proceeded, the Ti element diffused to the ceramic side reacted with the Si_3N_4 ceramic matrix as follows (Ref. 24):

$$\Delta G^0 \text{ (J/mol)} = -613000 + 40.8T$$

When the temperature reached 900°C (1173 K), TiN began to nucleate in the local region of the ceramic surface, and the generated Si element diffused into the filler under the driving of a concentration gradient. While at the Mo interface, the dissolved Mo element formed a solid solution with the Ti element diffused to the interface.

3) As shown in Fig. 9C, the microstructure of the joint evolved further with the interfacial reaction between the filler and the base materials. At the $\text{Si}_3\text{N}_4/\text{Ag-Cu-Ti}$ interface, the initially formed TiN grains gradually grew and connected to each other, eventually forming a continuous TiN interfacial reaction layer on the ceramic surface. At the same time, the Si element released by the reaction gradually diffused into the liquid filler, and the filler region near the TiN interface reaction layer reacted with the Ti enriched in the interface region as follows (Ref. 24):

$$\Delta G^0 \text{ (J/mol)} = -140200 + 5.44T$$

4) In the cooling process after brazing, the high melting point Cu precipitated first, the low melting point Ag precipitated and dissolved certain Ti elements, and finally formed Cu- and Ag-based solid solutions, as shown in Fig. 9D. The microstructure of the joint was $\text{Si}_3\text{N}_4/\text{TiN} + \text{TiSi}_2/\text{Ag(s, s)} + \text{Cu(s, s)}/\text{MoTi}/\text{Mo}$.

Conclusion

Reliable brazing of Mo and Si_3N_4 ceramic was achieved by using an Ag-Cu-Ti filler. The brazed joints obtained by

Fig. 8 — Microhardness of brazed joints with different inter-layers.

Fig. 9 — Schematic diagram of the formation of the Si₃N₄/Mo joint.

holding 10 min at 900°C were well connected, and there were no holes and cracks at the interface. The interfacial microstructure and joining properties of the Si₃N₄/Ag-Cu-Ti/Mo brazed joint were investigated in this study. Primary conclusions are summarized as follows:

1) The whole brazing joint is mainly comprised of three parts: the interface reaction layer near the ceramic side is composed of TiN and TiSi₂, the middle of the brazing joint is Cu-based and Ag-based solid solution, and the formation of MoTi solid solution is near the Mo side. The typical structure of the Si₃N₄/Mo joint is Si₃N₄/TiN + TiSi₂ reaction layer/Ag(s,s) + Cu(s,s)/MoTi/Mo.

2) In combination with the composition distribution and data analysis, TiN and TiSi₂ compounds are generated on the ceramic side, so the microhardness of the reaction layer on the ceramic side is decreased but still much higher than the hardness of the brazing joint and the Mo base material.

3) The shear strength of the joint brazed at 900°C for 10 min was 204 MPa at room temperature.

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