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Effect of brazing temperature on microstructure and mechanical properties of Si_3N_4/Si_3N_4 joints brazed with Ag–Cu–Ti + Mo composite filler

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Abstract Mo particles have been introduced into Ag–Cu–Ti brazing alloy for the joining of Si_3N_4 ceramic. Effect of brazing temperature on microstructure and mechanical properties of the joints were investigated. The result shows that a continuous reaction layer which is composed of TiN and Ti_5Si_3 was formed at the Si_3N_4/braze interface. The central part of the joint was composed of Ag-based solid solution, Cu-based solid solution, Mo particles, and Cu–Ti intermetallic compounds. By increasing the brazing temperature, both the thickness of the reaction layer and amount of Cu–Ti intermetallic compounds in the joint increased, being beneficial for the joint strength. Whereas, the reaction between Ti and Si_3N_4 ceramic proceeded excessively and more Cu–Ti intermetallic compounds were precipitated in the joint while elevating the brazing temperature to 950 °C, leading to deterioration of the bending strength. The maximal bending strength reached 429.4 MPa at 900 °C for 5 min when the Si_3N_4 ceramic was brazed with Ag–Cu–Ti + Mo composite filler.

Introduction

Silicon nitride ceramics with high strength, Young's modulus, hardness, corrosion resistance, and wear properties are widely used in all fields of industry. However, it is difficult to produce large-sized or complex-shaped ceramic components without any defects due to poor workability and low ductility of silicon nitride ceramics [1, 2]. Therefore, it is necessary to join small or simple ceramic pieces to form large-size or complex-shape ceramic components [3–6]. Robust assembly and integration technologies play an essential role in fabricating ceramic joints [7]. Among the joining approaches, active metal brazing is a simple and cost-effective method to join both ceramics to themselves and ceramics to metals [8]. It uses braze powders, pastes, ribbons, or wires, which are melted, allowed to wet and spread in the joint region, and solidify to form sound joints [9]. However, all joining techniques must take into account the difference in the coefficient of thermal expansion (CTE) between the brazing alloy and the ceramic. This CTE mismatch of joining materials can result in high critical residual stresses at the interface, which will reduce the joint strength, and in some cases, lead to failure during or after the joining process [10, 11].

In recent decades, many kinds of filler alloys have been developed to join Si_3N_4 ceramic, among which is Ag–Cu–Ti brazing alloy, which was developed earlier and has been produced on an industrial scale [12]. However, large residual stress can be caused in ceramic near the joint interface due to large CTE mismatches between the Si_3N_4 ceramic and Ag–Cu–Ti brazing alloy [13–15]. The problem can be alleviated by adding a material with low CTE (particles or fibers) to the brazing alloy. The addition of ceramic particles or fibers into brazing alloy, such as carbon fibers [16], SiC [17], and WC [18], has shown significant improvements in the joint flexural strength. Zhu et al. [19, 20] reported that the addition of 12 vol.% short carbon fibers to 63Ag–34Cu–2Ti–1Sn (wt%) brazing alloy had resulted in up to 30% improvement in the shear/tensile joint strength of the stainless steel/alumina joints. Unfortunately, few studies have been carried out on metal particles as additive phases and scant information is available on the brazing response of Si_3N_4 ceramics to themselves by using the composite filler.

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In the work, Si_3N_4 ceramic was joined to itself by using Ag–Cu–Ti + Mo composite filler. Mo particles serve to decrease the CTE of the brazing alloy. Besides, it is also a reinforcement which can increase the overall strength of the brazing layer. Effect of brazing temperature on microstructure and mechanical properties of the joints were investigated.

Materials and experimental procedures

Si_3N_4 ceramic used in this investigation was hot pressed with MgO and Al_2O_3 additives. The ceramics were sliced into 3 mm × 4 mm × 17 mm pieces. The brazing surfaces (3 mm × 4 mm) of the ceramic pieces were coarsely ground on SiC abrasive papers and then polished with 0.5 μm diamond paste. The composite filler consist of 69.12Ag–26.88Cu–4Ti (wt%) alloy powder with a particle size of 50 μm and Mo particles with an average diameter of 10 μm. The volume fraction of Mo particles in the composite filler was designed as 5%. To prevent segregation and aggregation of each component, mechanical alloying was used to prepare the Ag–Cu–Ti + Mo composite filler. The starting powder mixtures were high-power ball milled for 2 h in a planetary ball mill (QM-3SP04, Nanjing University Instrument Plant, China) with a ball to powder mass ratio of 10:1. Then, a small amount of binder was added to the powder mixture for making a composite paste. Before assembling, the Si_3N_4 ceramics were cleaned with acetone in an ultrasonic bath for 30 min. The composite brazing paste was placed between two Si_3N_4 samples and a normal load of 0.30–0.40 N was applied to the assembly to hold them together. The assembly was heated in a vacuum furnace to the brazing temperature (840–950 °C), isothermally soaked for 5 min at the brazing temperature, and cooled to room temperature at a rate of 5 °C/min.

The strength of the butt joint was measured by a three-point bending test with a cross-head speed of 0.5 mm/min. An average of at least three samples was used to determine the bend joint strength for each joining condition. The brazed joints were mounted in epoxy, ground, polished, and examined with a scanning electron microscope (SEM) coupled with energy dispersive spectrometer (EDS) on a Hitachi 4700 system (Tokyo, Japan).

Results and discussion

Microstructure characterization of the Si_3N_4/Si_3N_4 joints

Figure 1 shows microstructure and corresponding elements' area distribution images of a Si_3N_4/Si_3N_4 joint brazed with Ag–Cu–Ti + 5vol.%Mo composite filler at 950 °C for 5 min. The well-bonded joints reveal the composite filler shows good wetting and intimate contact to the Si_3N_4 ceramic. As can be seen from Fig. 1b, Mo particles distribute uniformly in the brazing layer. A continuous reaction layer with an average thickness of 3.8 μm exists between the Si_3N_4 ceramic and the braze (zone A in Fig. 1a). The reaction layer is mainly composed of Ti as shown in Fig. 1f, indicating that Ti has been concentrated at the Si_3N_4/brazing alloy interface. According to composition analysis, the reaction layer which is made up of 18.49 at.% Si, 68.51 at.% Ti, and 13 at.% N should be composed of a bi-layer system of TiN and Ti_5Si_3 phase. According to Ag–Cu binary phase diagram, Ag–Cu eutectic melts at 780 °C. During brazing, Ag–Cu brazing alloy melts at 780 °C. Then, Ti begins to be dissolved in the liquid gradually. The entire Ag–Cu–Ti brazing alloy is in liquid state when the temperature reaches 950 °C. Ti, which was dissolved in the melt, will diffuse towards Si_3N_4 ceramics and react with them to form TiN reaction layer. The free energy of formation for Si_3N_4 and TiN is represented as following [21]:

$$\Delta G_f^0(Si_3N_4)(KJ/mol) = -361.9 + 0.1575T$$

$$\Delta G_f^0(TiN)(KJ/mol) = -672.6 + 0.1865T$$

It can be found that the free energy of formation for Si_3N_4 and TiN are both negative at 950 °C. Moreover, the free energy of formation for TiN (−444.5 kJ/mol) is lower than that for Si_3N_4 (−169.3 kJ/mol), indicating that TiN is more stable at the brazing temperature. So Ti will react with Si_3N_4 ceramic to form TiN by the following reaction [22–26]:

$$Si_3N_4(s) + 4Ti\ (l) = 4TiN\ (s) + 3Si\ (s) \quad (1)$$

$$\Delta G^0(KJ/mol) = -1356 + 0.199T$$

Due to the negative systematic free energy (−1121.6 kJ/mol) at the brazing temperature, the reaction will proceed continuously until TiN reaction layer becomes thick enough to impede Ti diffusing towards Si_3N_4 ceramic further. During the formation of TiN reaction layer, Si is released and diffuses into the molten braze. Ti_5Si_3 phases are formed adjacent to the TiN reaction layer by the following reaction [22, 23, 25]:

$$5Ti\ (s) + 3Si\ (s) = Ti_5Si_3(s) \quad (2)$$

$$\Delta G^0(KJ/mol) = -194.14 + 0.0167T$$

Thermodynamic calculations from the free energy of formation for Ti_5Si_3 (−173.7 kJ/mol) confirm its stability. Consequently, the Si_3N_4 ceramic/braze interface consists of an inner TiN layer near the Si_3N_4 ceramic and an outer Ti_5Si_3 layer adjacent to the brazing alloy. In the research, it is difficult to distinguish the two reaction layers clearly

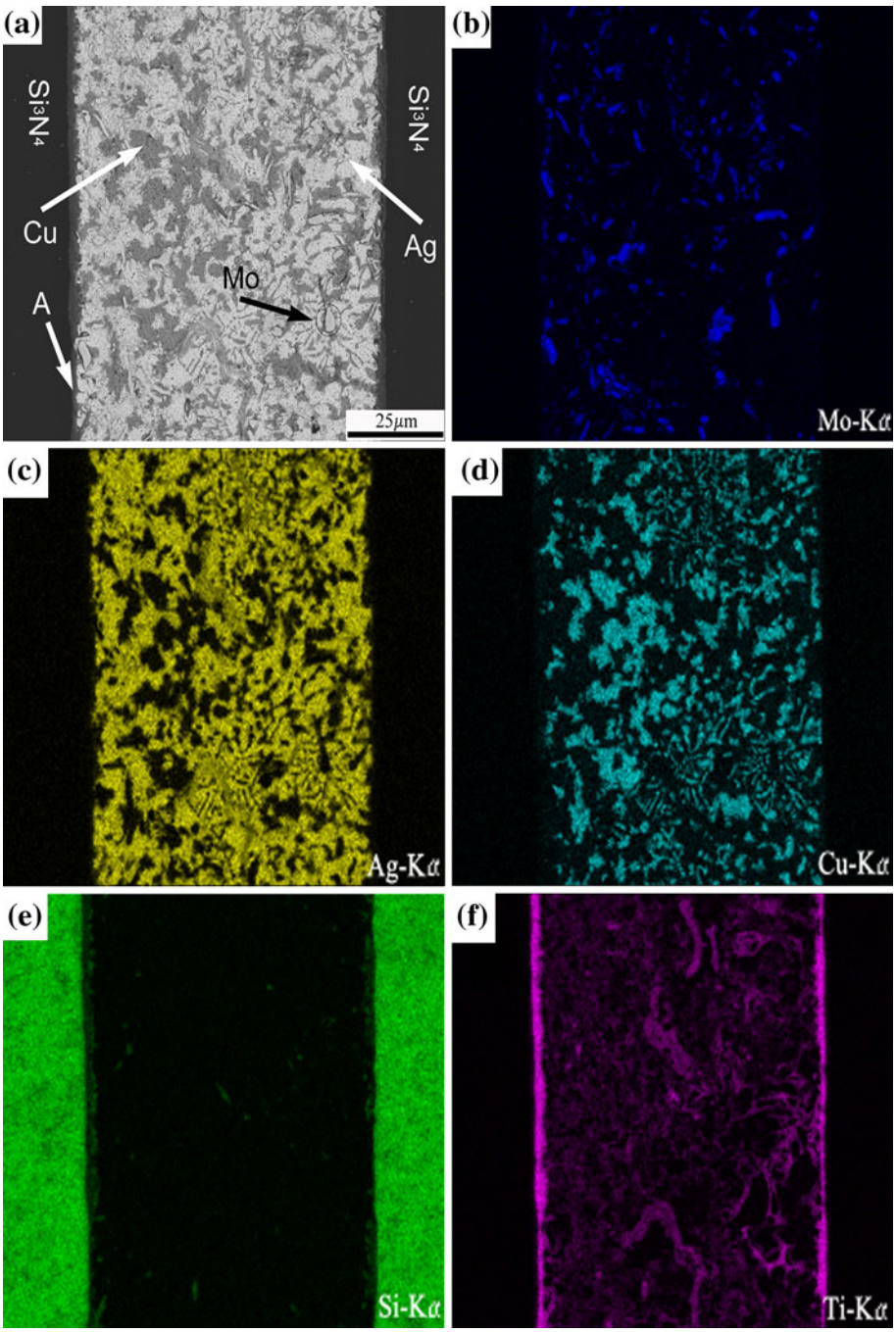

Fig. 1 Morphology and elemental analysis results of the Si_3N_4/Si_3N_4 joint brazed with $(Ag_{72}Cu_{28})_{96}Ti_4$ + 5vol.%Mo composite filler at 950 °C for 5 min

because they show little differences in the back-scattered micrograph.

The central part of the joint was composed of bright Ag-based solid solution, gray Cu-based solid solution, Mo particles, and some reaction phases. When Ag–Cu–Ti brazing alloy melts, Mo particles remains in solid state during brazing. The interaction between Ag–Cu–Ti and Mo has an impact on the composite filler accommodating residual stresses, which will affect the joint strength eventually. So, it is necessary to investigate the interaction between the brazing alloy and Mo particles.

Figure 2 presents back-scattered micrograph of reaction products in the joint and corresponding elements' line scans around Mo particles. Interfaces between the brazing alloy and Mo particles show intimate physical bonding. No cracks or pores were found in the joint. The gray part across the scanning line is rich in Mo, and no other elements are found in the area. They were determined to be

Mo particles. Ti concentration fluctuates where Mo particles contact the brazing alloy, as shown in Fig. 2b, which will be elucidated in the following section. There are also Cu–Ti intermetallic compounds detected in the joint, as demonstrated in Fig. 2a. Combining with Cu–Ti binary phase diagram [22], Ti could react with Cu to form Cu–Ti intermetallic compounds owing to their strong mutual affinity.

As can be seen from Fig. 1f, Ti element not only concentrates at the Si_3N_4 ceramic/braze interface, but also distributes in the joint randomly. It is necessary to clarify the form of Ti element in the brazing seam. Figure 3 displays SEM images of different regions in the seam for the Si_3N_4/Si_3N_4 joint brazed with $(Ag_{72}Cu_{28})_{96}Ti_4$ + 5vol.%Mo composite filler at 900 °C for 5 min. The black area, as shown in Fig. 3a, was caused by desquamation of Mo particles during polishing due to dissimilar hardness of Ag–Cu and Mo particles. Moreover, a quantitative overview of chemical analysis for different regions is provided in Table 1, which demonstrates various kinds of Cu–Ti intermetallic compounds may occur at optional points in the joint. As shown in Fig. 3, tiny precipitates are distributed randomly in the Ag and Cu-based solid solution. The precipitate, such as region A, is enriched with Ti (~38.01 at.%) and Cu (~57.71 at.%) and a trace amount of Ag. The stoichiometric ratio between Cu and Ti of the precipitate indicates it is almost a Cu_3Ti_2 intermetallic compound. It is worth noting that there are also CuTi (labeled B) and Cu_4Ti_3 (labeled C) intermetallic compounds detected in the joint, as shown in Fig. 3b and c. Ti possesses two typical allotropic structures, existing in the form of α-Ti at low temperatures and transforming to β-Ti when the allotropic transformation occurs at 882.5 °C. Based on Mo–Ti binary phase diagram [27], β-Ti and Mo are infinitely miscible in the temperature range of β-Ti. However, the maximal solubility of Mo in (–Ti is less than 1 wt%, at which level (–Ti cannot be dissolved in Mo. Thus, Ti element in the composite filler was β-Ti at the brazing temperature (900 °C). They diffused towards and reacted with Si_3N_4 ceramics continuously. Besides, partial Ti element was absorbed by Mo particles and they began to be dissolved mutually. The transformation of β-Ti → α-Ti occurred when the temperature reached 882.5 °C during cooling. Thus, the Ti element that was dissolved in the Mo particles previously was precipitated due to the lower solubility between α-Ti and Mo in the subsequent cooling. They could react with Cu to form Cu–Ti intermetallic compounds as the temperature decreased. The varieties of Cu–Ti intermetallic compounds are dependent on the composition ratio of Ti and Cu during reaction [28–30].

Effect of brazing temperature on microstructure of the Si_3N_4/Si_3N_4 joints

Figure 4 displays back-scattered electron images of Si_3N_4/Si_3N_4 joints brazed using Ag–Cu–Ti + 5vol.%Mo composite filler at 840, 900, and 950 °C for 5 min. Sound joints without voids or cracks were obtained along all joints for the brazing temperatures investigated. As per the explanation above, the reaction layer at the Si_3N_4/braze interface consisted of an inner TiN layer near the ceramic and an outer Ti_5Si_3 layer adjacent to the brazing alloy, as shown in Fig. 4d–f. It is also observed that the thickness of the total reaction layer increases with elevating the brazing temperature. At the lower brazing temperature (840 °C), the average thickness of the total reaction layer (TiN layer and Ti_5Si_3 layer) is only 1.2 μm and moreover, it could not be formed at local area due to insufficient reaction between Ti and Si_3N_4 ceramic, as indicated in Fig. 4d. When elevating the brazing temperature to 900 °C, the reaction layer (TiN layer and Ti_5Si_3 layer) at the Si_3N_4/braze interface becomes more continuous and grows to 1.4 μm. Further increasing the brazing temperature to 950°, the reaction layer (TiN layer and Ti_5Si_3 layer) grows further and reaches 3.8 μm. Increasing brazing temperature favors the diffusion of Ti and the reaction between Ti and Si_3N_4

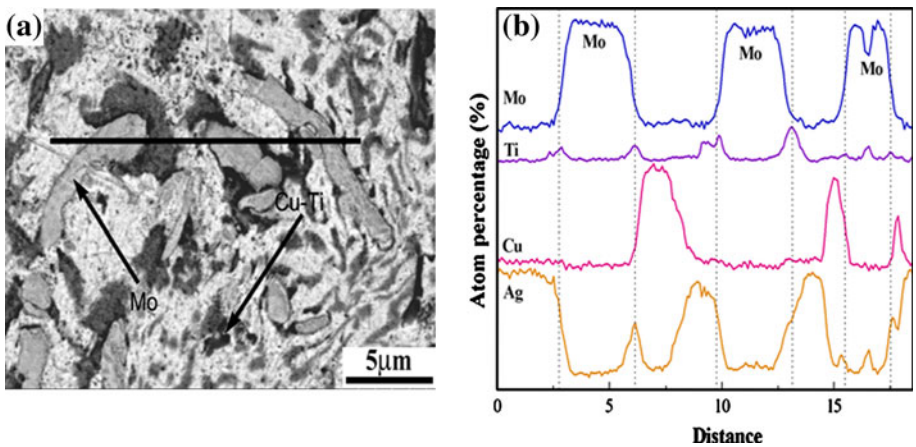

Fig. 2 Morphology of the joint (**a**) and elemental distributions (**b**) of Mo, Ag, Cu and Ti elements along the *black line* in **a**

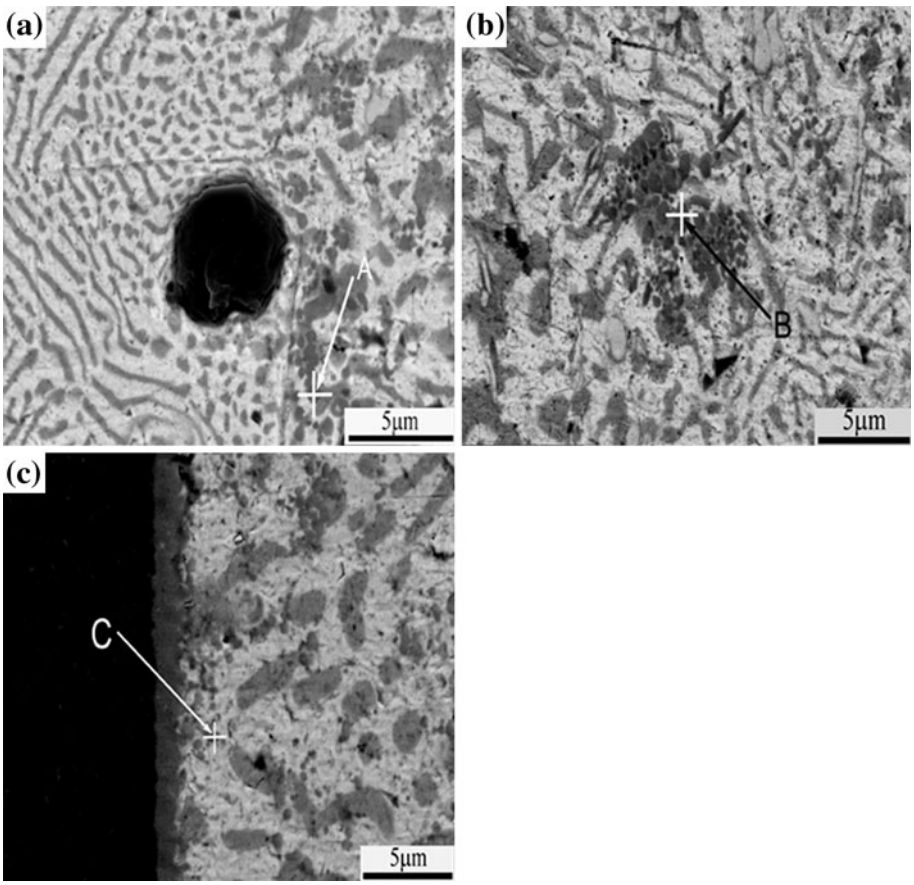

Fig. 3 Back-scattered electron images of the Si_3N_4/Si_3N_4 joint brazed with $(Ag_{72}Cu_{28})_{96}Ti_4$ + 5vol.%Mo composite filler at 900 °C for 5 min

Table 1 Compositions of the points A, B and C shown in Fig. 3

Point	Composition (at.%)			Phase
	Ag	Cu	Ti	
A	4.28	57.71	38.01	Cu_3Ti_2
B	6.36	48.23	45.41	CuTi
C	–	56.26	43.74	Cu_4Ti_3

ceramic as diffusion is thermally activated processes. High brazing temperature results in substantial mass transport across the interface and faster growth of the reaction zone, favoring increase of thickness of the reaction layer. In addition, the higher the brazing temperature is, the longer the Ti element stays in β-Ti stage. Thus, more Ti element and Mo particles were mutually dissolved during brazing, resulting in more Ti element were expelled into the braze during the subsequent cooling. Ti could react with Cu. So, more fine Cu–Ti intermetallic compounds are present in the joint at the higher brazing temperature, as compared Fig. 4c with a, b.

The volume fraction of Ag–Cu eutectic structure in the joint decreases with increasing brazing temperature, as demonstrated in Fig. 4a–c. Formation of Cu–Ti intermetallic compounds in the joint resulted in the depletion of Cu content in the braze. The composite filler's chemical composition was deviated from Ag–Cu eutectic to hypoeutectic. Besides, more Ti element was dissolved around Mo particles at the higher brazing temperature and more Ti element was precipitated during solidification. Ti element can react with Cu, and increased consumption of Cu content in the composite filler will lead to serious deflection of Ag–Cu eutectic composition. No eutectic structure was detected at 950 °C.

Effect of brazing temperature on bending strength of the Si_3N_4/Si_3N_4 joints

Figure 5 shows the bending strength of the specimens brazed at different brazing temperatures for 5 min. It can be seen from the picture that the brazing temperature influences the bending strength of joints seriously. When the brazing temperature is 840 °C, the bending strength of the joint is 330.1 MPa. The joint strength increases rapidly while elevating the brazing temperature to 900 °C, at which the maximal strength (429.4 MPa) is received. The joint strength decreases to 299.1 MPa when the brazing temperature is elevated to 950 °C. Variation of the brazing temperature influences the joint strength in two aspects.

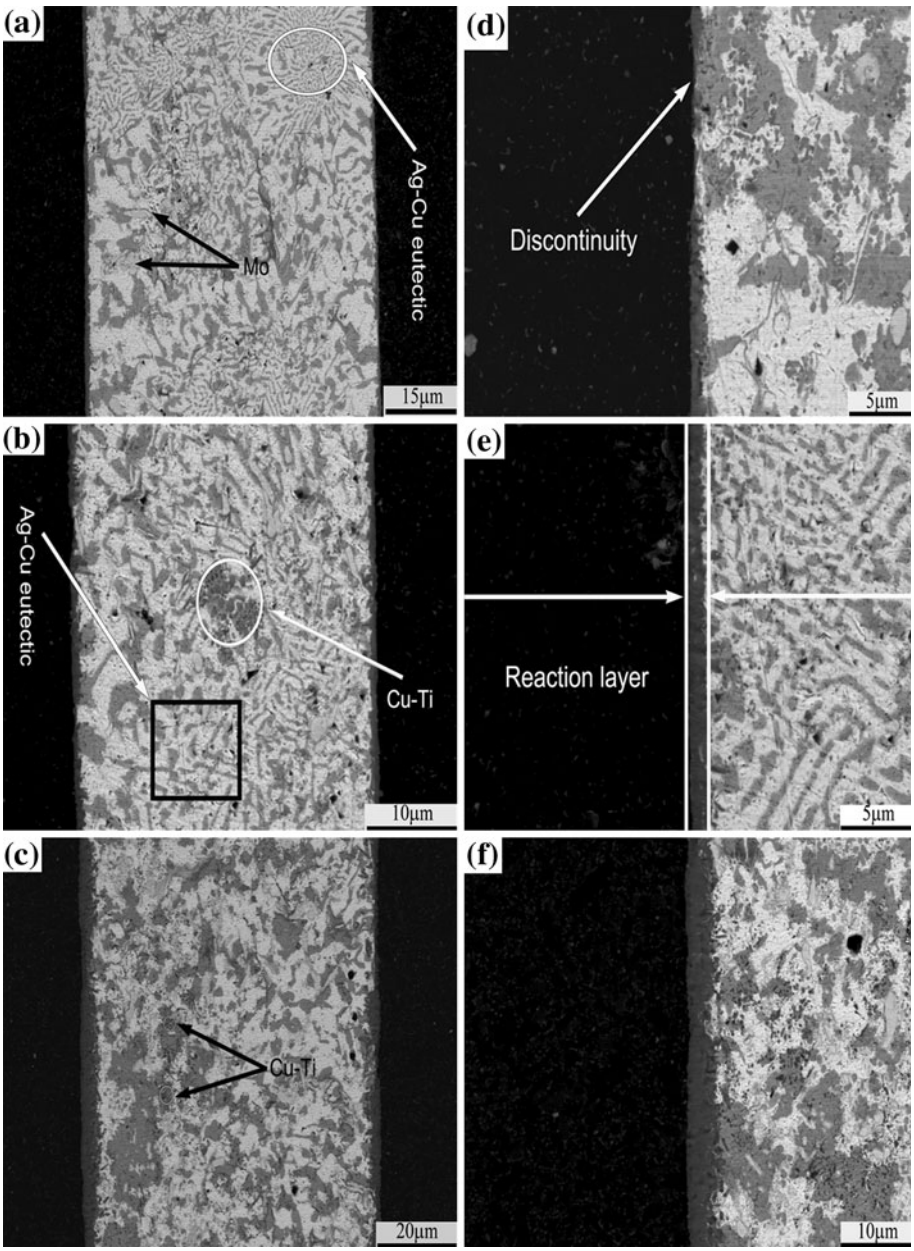

Fig. 4 Morphologies of Si_3N_4/Si_3N_4 joints brazed by Ag–Cu–Ti + 5vol.%Mo composite filler at different temperature for 5 min: **a** 840 °C; **b** 900 °C; **c** 950 °C; **d** magnified reaction layer in **a**; **e** magnified reaction layer in **b**; **f** magnified reaction layer in **c**

Firstly, brazing temperature influences the thickness of the Si_3N_4/braze interfacial reaction layer, which will affect joint strength directly. When the brazing temperature is lower, Ti element diffuses slowly and cannot sufficiently react with Si_3N_4 ceramic to form a strong interfacial bond due to the liquid brazing alloy's poor fluidity, resulting in lower strength. Higher brazing temperature promotes the growth of the reaction layer. According to the literatures [31], it is essential that a suitable thickness of interfacial reaction layer is formed at the Si_3N_4/ braze interface, because the reaction layer can decrease the thermal residual stresses gradient between Si_3N_4 ceramic and brazing alloy. However, the joint strength will be deteriorated when the thickness of the reaction layer exceeds a certain value (the optimum thickness of the reaction layer is 1.4 μm when the maximum joint strength is achieved in this study). Interfacial reaction products are new phases (TiN and Ti_5Si_3) to the Si_3N_4 substrates, and have different coefficients of thermal expansion. A thick and complicated reaction layer could cause great stress in the joint, lowering the joint strength. An appropriate reaction layer can be obtained by adjusting the brazing temperature during brazing.

Secondly, brazing temperature also affects the amount of Cu–Ti intermetallic compounds that were precipitated in the joint. The higher the temperature is, the more the Cu–Ti intermetallic compounds will be precipitated. It is beneficial that a certain amount of Cu–Ti intermetallic compounds exist in the joint owing to their lower CTE, which

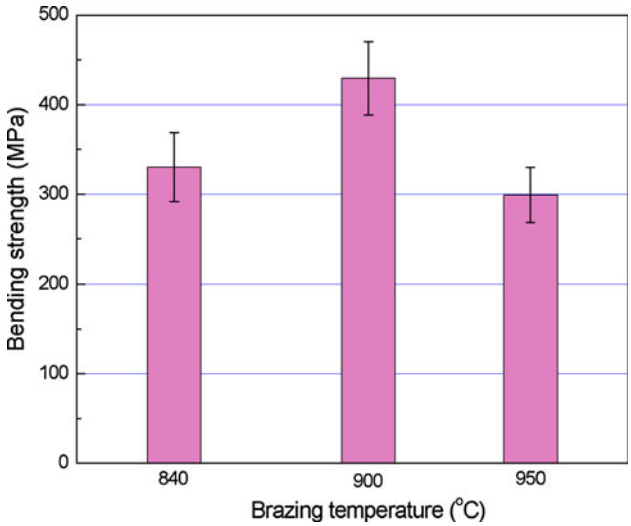

Fig. 5 Effect of brazing temperature on bending strength of the joint

will lower the CTE mismatch between the Si_3N_4 ceramic and the brazing alloy, enhancing the brazed joint quality. Whereas, an abundant presence of these brittle intermetallics in the joints will impair the ability of Ag and Cu-based solid solution to plastic deformation, which will be detrimental to relax thermal residual stresses generated during brazing, resulting in deterioration of the bonding strength.

Influenced by above two aspects, when the brazing temperature is 840 °C, a thin reaction layer was formed, which presented a poor load-carrying ability and led to lower bond strength. The maximum joint strength (429.4 MPa) is received at 900 °C. Two factors contribute the maximal strength. An appropriate interfacial reaction layer was formed at the Si_3N_4/braze interface. Meanwhile, a certain amount of Cu–Ti intermetallic compounds were precipitated in the joint. These Cu–Ti intermetallic compounds decreased CTE of the composite filler further and accommodated the thermal residual stresses. As the brazing temperature rose to 950 °C, the reaction between Ti and Si_3N_4 ceramic proceeded more adequately and the interfacial reaction layer was thickened. The load-carrying ability of the interface may also be weakened when its thickness exceeds a certain value. Furthermore, more Cu–Ti intermetallic compounds were precipitated in the joint at 950 °C. Excessive Cu–Ti intermetallic compounds in the joint will also be detrimental to

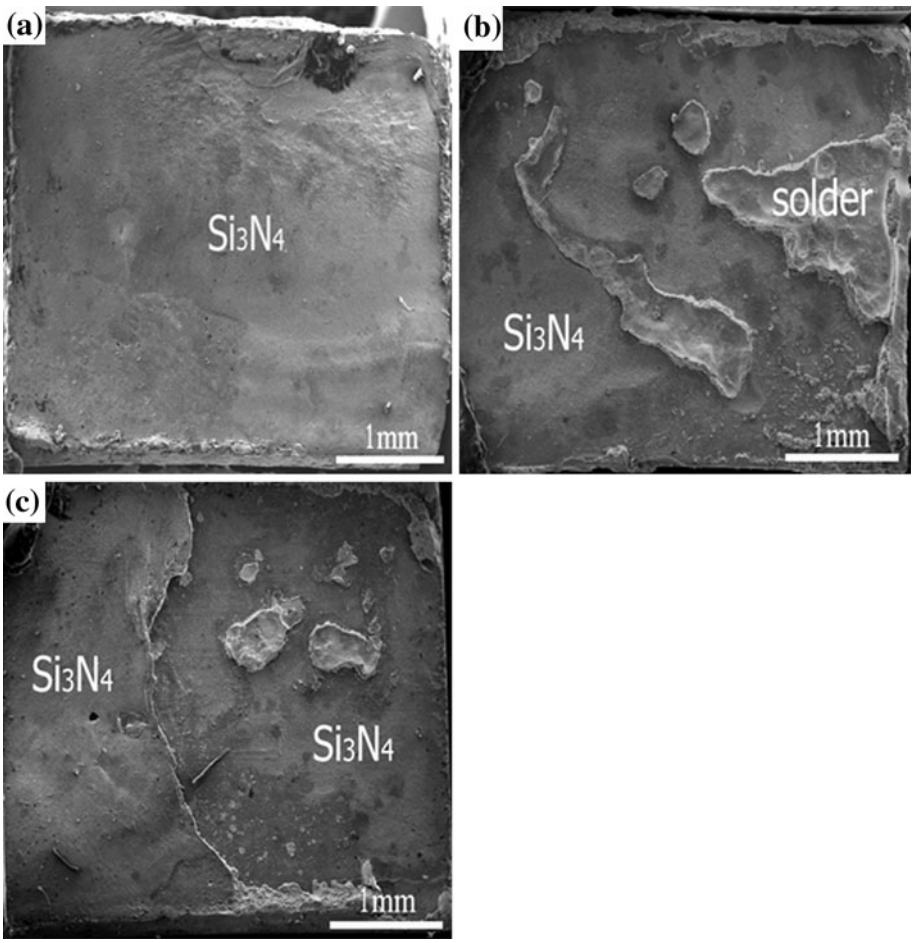

Fig. 6 Fractography of Si_3N_4/Si_3N_4 joints brazed by Ag–Cu–Ti + 5vol.%Mo composite filler at different temperature for 5 min: **a** 840 °C, **b** 900 °C, **c** 950 °C

the joint strength. Consequently, the joint strength was decreased at 950 °C.

Typical fracture surfaces of the joints were observed after three-point bending tests by SEM. The fracture can be divided into three types, as shown in Fig. 6. The first type is shown in Fig. 6a, in this case brazed at 840 °C, fracture started in the ceramic adjacent to the Si_3N_4 ceramic/braze interface, then propagated through the Si_3N_4 ceramic in a direction normal to the largest tensile stresses, creating a dome-shaped fracture surface. Figure 6b is another typical fracture, in this case brazed at 900 °C, the sample was fracturing randomly, partly in the ceramic and partly in the brazing seam. This kind of fracture type indicates the strongest joint strength. Fracture initiated in Si_3N_4 ceramic near the interface and propagated across the interface into another ceramic, is termed as the third fracture type, as indicated in Fig. 6c.

The Si_3N_4 ceramic was also brazed with Ag–Cu–Ti brazing alloy at 900 °C for 5 min and an average three-point bending strength of 200 MPa was obtained. The maximal bending strength (429.4 MPa) was received when the joint was brazed with Ag–Cu–Ti + 5vol.%Mo composite filler, which is 114.7% higher than the average strength for the case without Mo particles addition. This part will be summarized in the future.

Conclusion

The Si_3N_4 ceramic was successfully brazed to itself by using Ag–Cu–Ti + 5vol.%Mo composite filler at different brazing temperatures for 5 min. The following conclusions can be drawn.

(1) The Si_3N_4 ceramic/braze interface consists of an inner TiN layer near the ceramic and an outer Ti_5Si_3 layer adjacent to the brazing alloy. The central part of the joint is composed of Ag-based solid solution, Cu-based solid solution, Mo particles and Cu–Ti intermetallic compounds.

(2) The reaction layer at the Si_3N_4 ceramic/braze interface was thickened and more fine Cu–Ti intermetallic compounds were precipitated in the joint with increasing the brazing temperature. Moreover, the volume fraction of Ag–Cu eutectic structure in the joint was decreased at higher brazing temperature. No Ag–Cu eutectic structure was detected in the joint at 950 °C.

(3) The maximum bending strength reaches 429.4 MPa when the Si_3N_4/Si_3N_4 joints were brazed with Ag–Cu–Ti + 5vol.%Mo composite filler at 900 °C for 5 min. An appropriate interfacial reaction layer at the Si_3N_4/braze interface and a certain amount of Cu–Ti intermetallic compounds in the joint contribute the maximal joint strength. When the brazing temperature was 840 °C, the thin reaction layer demonstrated a poor load-carrying ability, and led to lower bond strength. As the brazing temperature rose to 950 °C, excessive Cu–Ti intermetallic compounds in the joint deteriorated the joint strength.

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