

Article

Process and Mechanism of Surface Brazing of Graphene on Aluminum Nitride

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Abstract

In order to enhance the heat dissipation of a chip, this work investigates the enhancement of the thermal homogenization effect of a ceramic substrate with a high-thermal-conductivity graphene material to improve the interfacial heat transfer performance. AgCuTi-activated brazing material is used to connect the graphene film/AlN. The mechanism of the influence of brazing temperatures on the microstructure and thermal conductivity of joints is discussed. The thermal conductivity of the graphene/AlN double layer composite brazed at 890 °C for 10 min holding time was the highest at 482.3 W m⁻¹ K⁻¹. This study provides a new solution for the application of AlN ceramics in high-heat-flow scenarios.

Keywords: brazing; thermal conductivity; interfacial bonding; graphene; Aluminum nitride



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1. Introduction

Thin-film thermopile heat flow sensors, fabricated using advanced micro–nano processing technology, represent a cutting-edge solution for thermal parameter measurements in extreme environments [1]. Characterized by their exceptional responsiveness, these miniature sensors are engineered to capture and analyze rapid transient changes in high-energy heat flow with unprecedented precision [2,3]. Widely applicable across diverse and challenging thermal scenarios, such sensors have been successfully deployed in critical applications, including high-efficiency heat field destruction measurements, aircraft surface thermal environment monitoring, and engine heat flow characterization [4,5].

Aluminum nitride (AlN) ceramics have become an important material in the field of packaging and the thermal management of high-power electronic devices, owing to their excellent thermal conductivity [6] and electrical insulation [7]. However, in large-heat-flow-density-scenarios, the thermal diffusion performance of a single material often fails to meet the demand for rapid homogeneous heat transfer [8]. In recent years, graphene, a two-dimensional material with ultrahigh thermal conductivity and excellent mechanical properties, has shown great potential in the field of thermal management [9,10]. The preparation of a graphene film on the surface of AlN ceramics through the study of brazing technology can not only give full potential to the high thermal conductivity characteristics of graphene [11,12], but also effectively compensate for the thermal bottleneck of AlN ceramics in the area of localized heat flow concentration [13]. In a large heat flow test,

a graphene film on the surface of AlN ceramics can significantly enhance the thermal homogenization effect of the material [14]. The high thermal conductivity of graphene accelerates the lateral diffusion of heat on the ceramic surface [15], reduces the formation of hot spots, and thus improves the overall heat transfer efficiency [16]. In addition, the good interfacial bonding between graphene and AlN ceramics can effectively reduce the interfacial thermal resistance [17,18] and further enhance the thermal transport properties of the composite material. This provides a new solution for the application of AlN ceramics in high-heat-flow scenarios.

In the current work, an Ag-Cu-Ti active brazing alloy was used to bond a graphene film to AlN ceramics. The characteristic interfacial microstructures and reaction products at the interfaces of the brazed joints were analyzed. Moreover, this work further evaluated how temperature variations during the brazing process influenced both the microstructure and thermal conductivity of the brazed joints.

2. Materials and Methods

Before the brazing process commenced, the surfaces of the AlN base material and Ag-Cu-Ti foil (0.5 mm thick) were abraded using sandpaper (500–1000 grit) and subsequently subjected to ultrasonic cleaning in an ethanol solution for 10 min. Subsequently, the graphene film, Ag-Cu-Ti foils, and AlN base materials were assembled in the order depicted in Figure 1. Graphite blocks applied a constant pressure of approximately 0.1 MPa to secure the stacked components during the joining operations. Vacuum brazing was performed in a controlled environment maintained at 5×10^{-3} Pa, with thermal processing parameters including gradual heating ($10^\circ\text{C}/\text{min}$ ramp rate) to target temperatures between 830 and 920°C . Isothermal holding at the peak temperature lasted 10 min before controlled cooling commenced at $5^\circ\text{C}/\text{min}$ until the ambient temperature was reached.

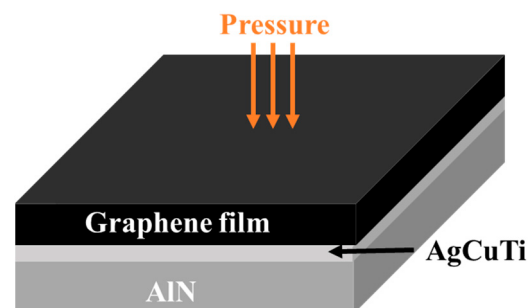


Figure 1. Schematic diagram of brazing experiment.

The interfacial microstructures of the joints fabricated under varying process conditions were analyzed using a scanning electron microscope (SEM, Zeiss Merlin Compact, Oberkochen, Germany) integrated with energy-dispersive X-ray spectroscopy (EDS, Oxford, Oxford, UK). The relative error in the atomic percentage of the element points scanning position was typically between $\pm 2\%$ and $\pm 5\%$. Thermal diffusivity assessments were conducted by the flash thermal conductivity meter (NETZSCH LFA 467 instrument, Seelbach, Germany). The laser flash methodology for thermal conductivity determination requires the multiplicative combination of three key material properties, as presented in Equation (1) [19].

$$\lambda = \alpha \times \rho \times C_p \quad (1)$$

where α denotes thermal diffusivity, ρ represents the sample density, and C_p stands for the specific heat.

3. Results and Discussion

3.1. Interfacial Microstructure of the Brazing Joints

Figure 2 presents the characteristic microstructure of the graphene/AlN joint fabricated through AgCuTi-assisted brazing at 860 °C for a duration of 10 min. The cross-sectional overview in Figure 2a demonstrates the interfacial connection between the graphene and ceramic substrate without obvious unsoldered areas or defects. EDS analysis was used to identify the chemical composition of the reaction products, and the corresponding data are presented in Table 1. The metallic matrix appearing as bright zones (Phase E) predominantly consists of a silver-rich solid solution (Ag (s. s)), while the intermediate gray regions (Phase F) represent a copper-based solid solution (Cu (s. s)) according to reference [20]. A magnified image of the AlN-side reaction interface in Figure 2f reveals three distinct metallurgical phases. Notably, the interfacial region near the AlN substrate demonstrated a tri-layered architecture, designated as B, C, and D in Figure 2f. The compositional analysis in Table 1 indicates that these layers correspond to intermetallic compounds: layer B comprises Ti_3Al , layer C contains AgTi, and layer D consists of CuTi phases. The formation of the Ti_3Al layer can be attributed to the diffusion of Ti into the AlN substrate [21]. Figure 2g shows the reaction zone between the AgCuTi filler and graphene. According to the elemental distribution, the reaction zone was composed of TiC. A comprehensive analysis revealed the multi-layered interfacial architecture in AlN/graphene joints processed at 860 °C for 10 min: $\text{AlN}/\text{Ti}_3\text{Al} + \text{AgTi} + \text{CuTi}/\text{Ag (s, s)} + \text{Cu (s, s)} + \text{TiCu}/\text{TiC}/\text{graphene}$.

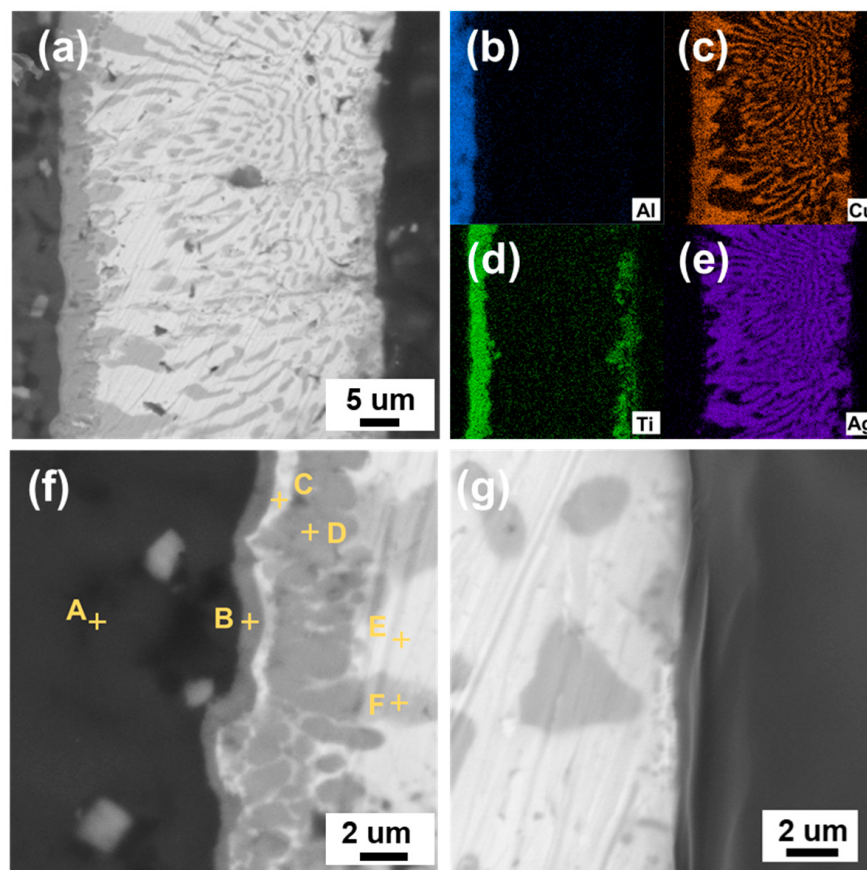


Figure 2. SEM images and elemental distribution of the graphene/AlN joint: (a) the entire joint; (b–e) elemental distribution of Al, Cu, Ti, and Ag; (f) interface morphology between AlN and brazing seam; (g) interface morphology of brazing seam and the graphene.

Table 1. Atomic percentages of element point scanning position in Figure 2f.

Spots	N (at%)	Al (at%)	Ti (at%)	Cu (at%)	Ag (at%)	Possible Phase
A	7.57	92.10	0.06	0.28	0.00	AlN
B	11.43	16.04	62.47	4.56	5.53	Ti ₃ Al
C	0.00	10.02	34.64	17.38	37.96	AgTi
D	0.00	12.98	48.39	35.54	3.10	CuTi
E	0.00	0.36	0.13	9.05	90.46	Ag (s, s)
F	0.00	5.43	0.90	90.91	2.76	Cu (s, s)

During brazing, Ti in the AgCuTi brazing filler metal plays a key role. At high temperatures, Ti exhibits strong reactivity, enabling it to undergo chemical reactions with Al in AlN and C in graphene. At the interface between the brazing alloy and AlN, Ti participates in reactions to form a reaction layer composed of Ti₃Al + AgTi + CuTi. This compound effectively enhanced the bonding strength between the brazing alloy and AlN. At the interface between the filler metal and graphene, Ti reacts with C to form TiC. The formation of these compounds improved the wettability and bonding strength between the filler metal and graphene [22]. The formation of the reaction phase enables atomic-level bonding between graphene and AlN, creating an efficient heat transfer pathway.

3.2. Effect of Brazing Temperature on the Interfacial Microstructure of Brazed Joint

Figure 3 presents the interfacial morphology evolution of the AlN/graphene joints brazed at various temperatures for 10 min. Notably, the composite reaction zone comprising Ti₃Al + AgTi + CuTi phases demonstrated a progressive thickness expansion with an elevated processing temperature. The employed Ag-Cu-Ti filler has a high activity of Ti element. During the brazing process, Ti migrates to both sides and accumulates at the two interfaces [23]. Nevertheless, thermal exposure at 920 °C induced structural defects manifested as fracture propagation within both the reaction layer and the neighboring ceramic matrix, as shown in Figure 3d. Owing to the difference in the thermal expansion coefficients of graphene and the brazing reaction layer, a high residual stress is generated during the cooling process [24,25]. As the brazing temperature continued to rise, the residual stress increased, ultimately leading to the fracture of the graphene interlayer.

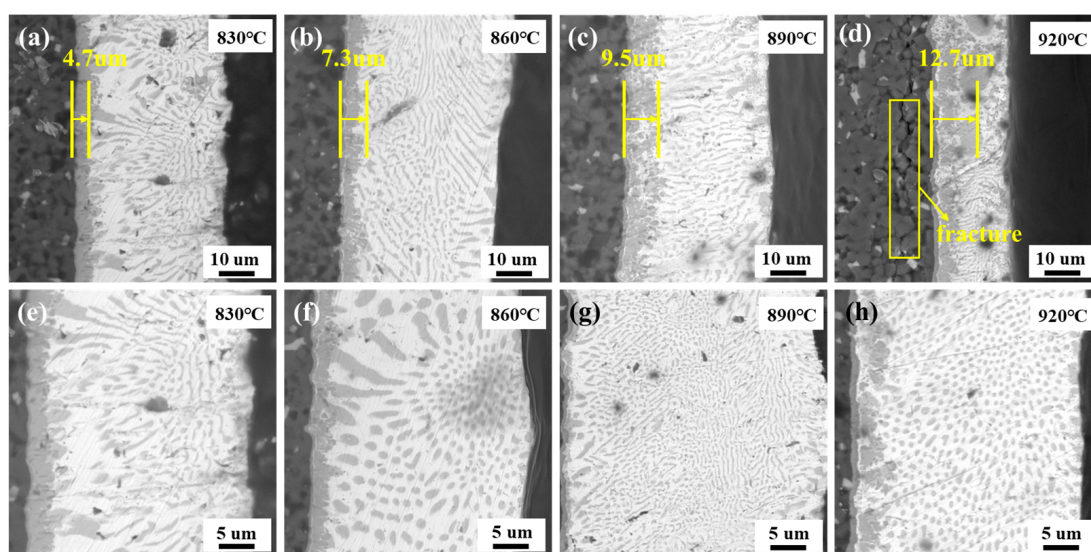


Figure 3. SEM morphologies of the brazed joints at different temperatures: (a) 830 °C; (b) 860 °C; (c) 890 °C; (d) 920 °C; (e–h) brazing seam at 830 °C, 860 °C, 890 °C, and 920 °C.

3.3. Effect of Brazing Temperature on the Thermal Conductivity of Brazed Joint

Figure 4 illustrates the relevant thermal performance indices of the graphene/AlN joint at different temperatures. As shown in Figure 4a, the variation in the brazing temperature influenced the specific heat of the graphene/AlN joint. The test results in Figure 4 show that the thermal conductivity of the samples gradually increased with the brazing temperature, reaching a maximum at a brazing temperature of 890 °C. The thermal conductivity also exhibited an upward trend with an increase in the brazing temperature. However, owing to the high brazing temperature, the graphene interlayer ruptured. Although the width of the brazed seam can clearly be seen to decrease significantly in the SEM images, the detrimental effect of the base material fracture on the thermal conductivity is more pronounced [26]. Consequently, the thermal conductivity of the samples brazed at 920 °C was lower than that of the samples brazed at 890 °C.

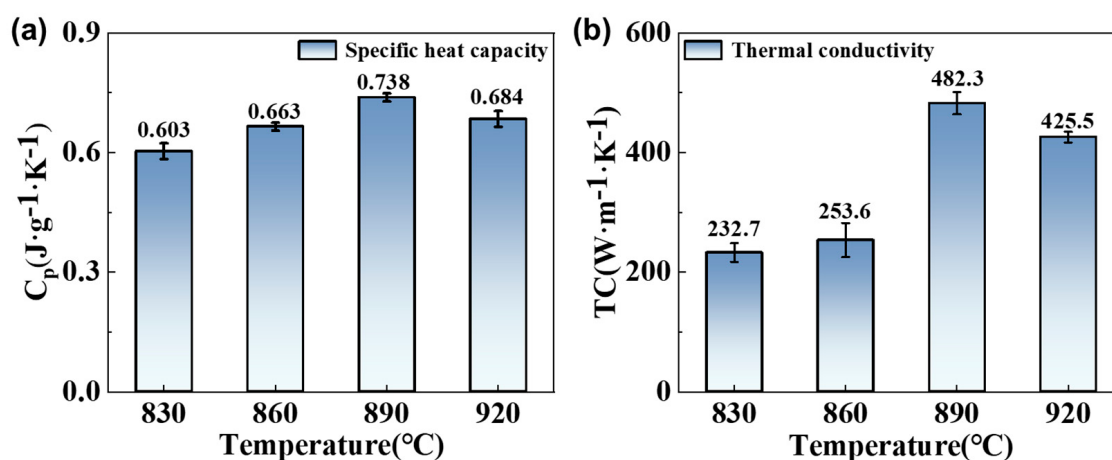


Figure 4. The thermal properties test of samples with different brazing temperatures. The thermal parameters involve (a) specific heat, (b) thermal conductivity.

Data analysis revealed that the brazing temperature exerted a substantial influence on the thermal conductivity. Compared with the thermal conductivity of AlN alone, the final thermal conductivities of the samples brazed at different temperatures were all enhanced. This indicates the outstanding heat dissipation performance of the graphene layer, which is consistent with the anticipated experimental hypotheses.

An increase in temperature promoted the diffusion of Ti, resulting in an increase in the thickness of the reaction layer. Within a certain temperature range (830–890 °C), the increase in the reaction layer thickness and structural optimization facilitated phonon transmission, thereby enhancing the thermal conductivity [27]. However, at excessively high temperatures (920 °C), excessive element diffusion leads to a non-uniform reaction layer composition, and the residual stress results in pores and cracks. These defects hinder phonon transmission, thereby decreasing the thermal conductivity [28,29].

In the rise and fall temperature profile test, the infrared thermal imaging of the samples at different brazing temperatures was captured using an infrared thermal imager, and the results are shown in Figure 5. In the rising temperature test, a brighter color corresponded to a higher surface temperature. From the beginning of the warming, all six samples were at room temperature, and as the warming proceeded, sample (890 °C) had the fastest temperature response owing to its highest thermal conductivity, had the highest surface brightest temperature at the same moment, and was already almost close to the equilibrium temperature at 5 s. At 60 s, all samples reached equilibrium temperature. In the case of a constant temperature of the bottom heat source, the surface temperature of the sample of 890 °C was the lowest. This enhancement of the interfacial structure facilitated more

efficient phonon heat conduction. The optimal thermal performance, characterized by the maximum thermal conductivity, was achieved at a brazing temperature of 890 °C. During the cooling stage, the surface temperature of all samples gradually decreased with the cooling process, and the surface gradually became darker. Similarly, the sample cooled most rapidly at 890 °C, the color darkening was the most obvious, and the surface temperature was close to the room temperature at 135 s.

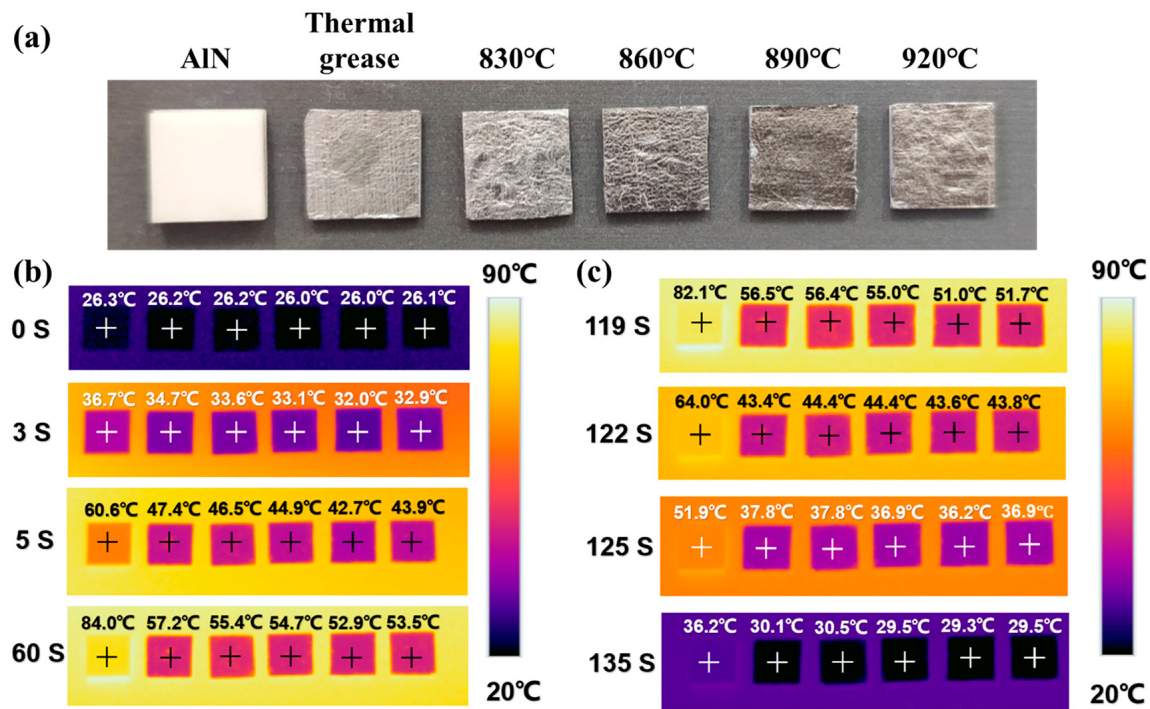


Figure 5. Infrared images of samples: (a) thermal grease and different brazing samples; (b) the rising temperature test; (c) falling temperature test.

Brazing offers a significant advantage over thermal grease for joining graphene films to AlN ceramic substrates. The resulting metallurgical bond minimizes the interfacial thermal resistance, facilitating more efficient heat transfer through the graphene layer and consequently enhancing the overall thermal performance of the component. The microscopic interfacial structure analysis shows that brazing causes the graphene film and AlN ceramic substrate to realize close interfacial bonding, eliminating the tiny air gaps that existed at the interface of the original mechanical connection and improving the overall heat dissipation performance of the component [30].

4. Conclusions

This research aimed to elucidate the mechanism of microstructural evolution in graphene/AgCuTi/AlN brazed joints. In addition, the correlation between the microstructure and thermal conductivity of the brazed joint was explored. The key findings are as follows.

(1) During brazing, the Cu and Ti components from the liquid Ag-Cu-Ti alloy diffuse and interact with graphene. The composite reaction zone comprising Ti_3Al + AgTi + CuTi phases demonstrated progressive thickness expansion with an elevated processing temperature.

(2) The good interfacial bonding of graphene with AlN effectively enhances the thermal conductivity. However, when the brazing parameters exceeded 890 °C and 10 min, several cracks appeared in the graphene interfacial reaction layer.

(3) The thermal conductivity test and infrared images of the samples showed that the graphene/AlN composite substrate welded at 890 °C for 10 min exhibited the highest thermal conductivity, reaching $482.34 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$. Interface damage caused a decrease in the thermal conductivity when the temperature continued to rise to 920 °C.

Author Contributions: W.L.: Investigation, Methodology, Software, Writing—original draft. Z.W.: Formal analysis, Methodology, Writing—original draft. X.W.: Software, Investigation. D.K.: Conceptualization, Supervision, Writing—review and editing. C.X.: Methodology, Validation. Y.Y.: Investigation, Data curation. J.L.: Formal analysis, Validation, Writing—review and editing. All authors have read and agreed to the published version of the manuscript.

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