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Brazing mechanisms of the Ti₂AlC joints using a pure Al filler metal

Chengjie Lu¹, Liangbo Sun¹, Jie Zhang^{1*}, Qin Qi¹, Gilles Hug²

¹*School of Materials Science and Engineering, Harbin Institute of Technology,*

Harbin 150001, China

²*Laboratoire d'Etudes des Microstructures, CNRS-ONERA,*

Châtillon 92322, France

Abstract

Brazing of Ti₂AlC ceramics has been successfully performed using a pure Al filler metal, in the temperature range 1023 K - 1173 K and with holding time ranging from 0 min to 30 min. The microstructure of the Ti₂AlC joints was studied, and the mechanical properties of the joints were evaluated by shear strength test. It is observed that the Al filler has weak effect on the stability of Ti₂AlC substrate, and only a small amount of decomposition products including the TiC_x and TiAl₃ compounds can be observed in the joints. In addition, the formation of an inter-diffusion layer in the Ti₂AlC substrate is considered as the major brazing mechanism. The maximum shear strength of the Ti₂AlC joints using the pure Al filler metal is 95 MPa, with an electrical conductivity of $2.21 \times 10^6 \text{ S} \cdot \text{m}^{-1}$, after holding the sample at 1123 K for 10 min.

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* Corresponding author: Tel/Fax: +86 451 86414234

E-mail address: hitzhangjie@hit.edu.cn (J. Zhang).

Keywords: Ti₂AlC; aluminum filler; brazing; mechanical property; electrical conductivity

1. Introduction

The MAX phases, which refer to a group of nanolaminate ternary ceramics with general formula $M_{n+1}AX_n$ (M, transition metal; A, A-group element; X, carbon or nitrogen), have attracted increasing attention in recent years due to their unique combination of metallic and ceramic properties [1, 2]. Among them, the Ti₂AlC phase is one of the most fascinating members with typical 211 structure (i.e. $n=1$), since it possesses the lowest density of all compounds in this class [3], an excellent electrical conductivity [4] and a small friction coefficient [5]. Such characteristics qualify the Ti₂AlC ceramics for many structural and functional applications as, for example, the heat/electron exchanger or high temperature solid lubricant. Numerous efforts have been carried out to synthesize Ti₂AlC ceramics, via various techniques like combustion synthesis (CS) [6], spark plasma sintering (SPS) [7] and self-propagating high-temperature synthesis (SHS) [8]. However, it is still difficult to process dense Ti₂AlC ceramics with high purity, large dimensions and complex shapes, because of the narrow single Ti₂AlC phase domain in the Al-C-Ti ternary phase system [9], which severely limits its applications. Joining technique is one of the convenient and promising solutions to overcome this problem. By achieving the Ti₂AlC joints, the limitation of dimensions can be overpassed, and the component with complex shapes can be obtained by joining small bulks.

Only few papers reporting about the joining of the MAX ceramics can be found in the past years, among which the diffusion bonding technique was mostly adopted. Inspired by the phenomenon that the MAX phases could form various solid solutions like $(M, M')AX$ and $M(A, A')X$, Ganguly *et al.* [10] could successfully bond Ti_3SiC_2 - Ti_3GeC_2 and Ti_2AlC - Nb_2AlC couples using a direct diffusion method. Interestingly, they discover that the Ti_3SiC_2 - Ti_3GeC_2 diffusion couple could form in the temperature range 1473 K - 1773 K, by forming a continuous gradient of $Ti_3(Si, Ge)C_2$ solid solutions. However, the diffusion temperature to obtain the Ti_2AlC - Nb_2AlC couple was much higher, about 1723 K - 1773 K, which was mainly attributed to the strong M-X bonds in the MAX structure that need to break and reconstruct during the diffusion process. Similarly, Yin *et al.* [11] managed to join two samples of Ti_3AlC_2 ceramics by inserting a Si interlayer. The experiments were performed in the temperature range 1573 K - 1673 K, for 120 min. The presence of a $Ti_3(Si, Al)C_2$ solid solution domain was confirmed by XRD, and a maximum flexural strength of the joints of 285 MPa, was obtained after holding the sample at 1673 K for 120 min, accounting for 80% of the strength of the Ti_3AlC_2 substrate.

It can be concluded from the above analysis that extremely high temperature is required to achieve the MAX phase diffusion couple by forming solid solutions, which is considered to be even higher than the decomposition temperature of the ceramic substrate under vacuum [12]. To decrease the processing temperature, as well as to overcome some disadvantages of the diffusion technique, we have performed a study of

brazing Ti_2AlC ceramics using a pure Ag filler metal [13]. The results suggested that the silver filler could diffuse into the Ti_2AlC substrate along the grain boundary, and then formed an $\text{Ag}_3(\text{Ti}, \text{Al})$ solid solution in the interaction area. However, no TiC_x compound, the typical decomposition product of the Ti_2AlC ceramics, was detected in the XRD pattern, suggesting that the interaction effect between the silver filler and the Ti_2AlC substrate would not induce a decomposition behavior of Ti_2AlC ceramics [14]. Finally, the maximum shear strength of the Ti_2AlC joints using pure Ag filler metal was measured to be 132 MPa, and was obtained for a brazing temperature of 1030 °C holding for 5 min.

The goal of this work is to further decrease the brazing temperature of Ti_2AlC ceramics, by using a pure Al filler metal. In addition, it is necessary to characterize the interaction behavior of the filler elements with the Ti_2AlC substrate, to better understand the intrinsic structural features of MAX phases.

2. Experimental procedure

The bulk polycrystalline Ti_2AlC selected in this study was processed by spark plasma sintering (SPS) using Ti, Al and graphite powders. In general, minority phases, including the TiC_x , TiAl_x and Ti_3AlC_2 compounds are always observed in Ti_2AlC compounds, therefore, a detailed characterization of the raw material was performed. A backscattered electron (BSE) micrograph of the Ti_2AlC substrate reveals its morphology as displayed in Fig. 1(a). Two phases with different contrasts can be observed. Firstly, the grey phase labeled A in the figure possesses an average atomic percentage of Ti 49%,

Al 24% and C 27% according to the EDS analysis. It is confirmed to be the Ti_2AlC main phase. Secondly, the light phase labeled B is identified as the TiAl_x intermetallic, with an average atomic percentage of Ti 72% and Al 28%. Afterwards, an X-ray diffraction (XRD) characterization was performed, and the corresponding result is shown in Fig. 1(b). The major phase showing up in the XRD pattern is the Ti_2AlC compound, and the minor phase is determined to be the Ti_3Al compound, corresponding to the light phase B discovered in the BSE morphology. In order to obtain the Ti_2AlC brazing joints with excellent heat/electron conductivity, the pure Al filler metal has been selected in the present work. In addition, the brazing filler with high Al content might be favorable for maintaining the stability of the Ti_2AlC substrate during the brazing process, since the depletion of Al atoms in the Ti_2AlC structure is considered to be the major cause of the decomposition behavior [15]. Although Ti_2AlC joints brazed using a pure Al filler metal might not be applicable in many high-temperature areas, it is still a convenient way to achieve the Ti_2AlC ceramic components which serve at room or intermediate temperature.

The bulk Ti_2AlC ingots were cut into samples with dimensions of $4\times4\times4\text{ mm}^3$ for microstructural observation, and samples of $4\times4\times10\text{ mm}^3$ for shear strength testing and electrical conductivity test. The surface of the specimens was ground using the 800-grit SiC paper, followed by the mechanical polishing using the $0.5\text{ }\mu\text{m}$ diamond paste. Commercial pure Al foils $270\text{ }\mu\text{m}$ thick were cut into pieces with dimensions of $4\times4\text{ mm}^2$, and then etched in a 10 wt. % NaOH solution and a 5 vol. % HNO_3 solution, to

remove the native Al_2O_3 oxide scale. Afterwards, the Al foils were cleaned with water, and then preserved in ethanol. Two Ti_2AlC rectangular specimens together with a pure Al foil were assembled in a sandwich structure: $\text{Ti}_2\text{AlC}/\text{Al}/\text{Ti}_2\text{AlC}$, and then placed in a graphite jig, on which a constant load of 5 MPa was applied to promote the contact of brazing filler with the ceramic substrates. A set of brazing experiments were performed under vacuum in a furnace, at brazing temperatures of 1023 K, 1073 K, 1123 K and 1173 K, and holding times of 0 min, 10 min and 30 min. A heating rate of $10\text{ K}\cdot\text{min}^{-1}$ was used while a slower cooling rate of $5\text{ K}\cdot\text{min}^{-1}$ was set in order to reduce the thermal stress in the joints that may form during the cooling period.

After the brazing experiments, shear strength tests were firstly conducted to obtain the optimal brazing parameters. The measurements were performed using a universal testing machine (Instron 5569) at room temperature, with a constant loading speed of $0.5\text{ mm}\cdot\text{min}^{-1}$. Three samples were tested for each brazing parameter. Furthermore, a special designed jig was designed for this work [16] in order to minimize the influence of friction on the results of the tests: the jig was able to adjust the gap width accordingly. Considering the excellent conductivity of both the Ti_2AlC substrate and the Al filler, the electrical conductivity of Ti_2AlC joints at different brazing parameters was measured using the four-probe technique. The brazing mechanisms were analyzed by observing the morphologies and characterizing the phases in the joints. The morphology of the joints were investigated using a Sigma 500, Zeiss scanning electron microscope (SEM), which is equipped with an energy-dispersive spectrometer (EDS) to determine the

elemental composition of the phases observed in the joints. In addition, the crystallographic structures of the bulk Ti_2AlC substrate, as well as the phases in the joints, were characterized using an X'pert pro MPD, Panalytical X-ray diffraction (XRD) using the $\text{CuK}\alpha$ radiation and steps of 0.05° at a scanning rate of $5^\circ \cdot \text{min}^{-1}$.

3. Results and discussion

3.1 Shear strength test and electrical conductivity measurement

Fig. 2(a) shows the shear strength of the Ti_2AlC joints brazed at different temperatures. It can be observed from the diagram that the room temperature shear strength significantly improves with the rising processing temperature, from 46 MPa at 1023 K to 85 MPa at 1073 K, and further increases up to 95 MPa at 1123 K. This improvement might be attributed to the reinforced interaction effect between the Ti_2AlC substrate and the pure Al brazing filler: with the increasing brazing temperature, the wetting process, as well as the inter-diffusion or reaction process is promoted. However, when the brazing temperature reaches 1173 K, the shear strength of the Ti_2AlC joints declines to 75 MPa. The decreasing of mechanical properties at high temperature might be attributed to many factors, including a severe transformation of the ceramic substrate due to the changing phase equilibrium, the formation of continuous reaction products and the consumption of brazing filler in the joints. Overall, the optimal brazing temperature of the Ti_2AlC joint is determined to be 1123 K in the present work. Fig. 2(b) shows the shear strength of the Ti_2AlC joints brazed at the optimal temperature, with holding time of 0 min, 10 min and 30 min, respectively. It can be concluded from the

figure that extending the holding time results in a similar tendency whatever the brazing temperature is. When the holding time prolongs from 0 min to 10 min, the shear strength of the Ti_2AlC joints increases from 53 MPa to 95 MPa, which may be attributed to the reinforced interaction effect as well; when the holding time further extends up to 30 min, however, the shear strength decreases to 30 MPa. Overall, the optimal holding time is determined to be 10 min in this work. The microstructure is considered to have a significant effect on the mechanical properties of the joints. Therefore, a detailed observation of the morphology has been performed in the following sections to further illustrate how the microstructure evolution affects the shear strength of the Ti_2AlC joints.

The electrical conductivity measurement results are also displayed in Fig. 2. Using the four-probe technique, the electrical conductivity of the Ti_2AlC substrate in this work is measured to be $2.38 \times 10^6 \text{ S} \cdot \text{m}^{-1}$, close to that reported in other works [17]. According to the diagrams, changing the brazing parameters has little effect on the electrical conductivity of the Ti_2AlC joints: with the rising brazing temperature, the electrical conductivity increases from $2.12 \times 10^6 \text{ S} \cdot \text{m}^{-1}$ at 1023 K, to $2.23 \times 10^6 \text{ S} \cdot \text{m}^{-1}$ and $2.21 \times 10^6 \text{ S} \cdot \text{m}^{-1}$ at 1073 K and 1123 K, respectively, then decreases to $2.13 \times 10^6 \text{ S} \cdot \text{m}^{-1}$ at 1173 K; with the extending holding time, the electrical conductivity changes from $2.17 \times 10^6 \text{ S} \cdot \text{m}^{-1}$ for 0 min to $2.21 \times 10^6 \text{ S} \cdot \text{m}^{-1}$ for 10 min, and finally declines to $2.10 \times 10^6 \text{ S} \cdot \text{m}^{-1}$ for 30 min. It is interesting to note that the electrical conductivity and shear strength change

similarly with the brazing parameters, suggesting that the two properties might be driven by the same microstructure characteristics of the Ti₂AlC joints.

In summary, the optimal brazing temperature and holding time in the present work have been determined to be 1123 K and 10 min, respectively. The maximum shear strength of the Ti₂AlC joints using the pure Al filler metal is measured to be 95 MPa, with the electrical conductivity of $2.21 \times 10^6 \text{ S} \cdot \text{m}^{-1}$, accounts for 43% and 93% of the Ti₂AlC substrate, respectively.

3.2 Microstructure observation and phase characterization

The morphologies of the Ti₂AlC joints brazed at different temperatures are displayed in Fig. 3. In the diagrams, two phases with different morphologies can be observed in the filler area. Firstly, a phase with a light grey contrast is labeled A in the figure. It has a rod-like shape and it generally originates from the interaction surface. Secondly, a dark grey phase labeled B in the figure, makes up the major part of the filler area. The EDS results suggest that the elemental composition of the phase A is Ti_{24.2}Al_{75.8} (in atomic percentage), and that of the phase B is Ti_{0.9}Al_{99.1} (in atomic percentage). Therefore, the light grey phase A is suggested to be the TiAl₃ intermetallic compound, while the dark grey phase B is supposed to correspond to the Al filler metal. It is worthwhile pointing that the formation of the TiAl₃ intermetallic compound has also been discovered in the Al alloy/Ti₂AlC composites produced by infiltration of sponge of MAX phase with molten Al metal (or alloy) [18], in spite of the short infiltration time of approximately 30 seconds. Therefore, it is reasonable to suggest that

the Ti_2AlC ceramics can readily react with molten Al during the brazing process. The formation of the TiAl_3 intermetallic compound can be explained by the equilibrium in the Al-C-Ti phase diagram. Indeed a transformation of the Ti_2AlC ceramics might occur under the presence of Al. The following reactions can provide a source of Ti atoms according to:

Both the two reactions are reasonable considering thermodynamic aspect, which has been verified by Witusiewicz *et al* [9], and the reaction products differ depending on the Al concentration and temperature. However, the other by-products of the reactions (1) and (2), either the Ti_3AlC_2 or the TiC_x compound, are not observed in the BSE micrographs. It is worthwhile pointing out that the close average atomic number between TiC compound ($\bar{Z}_{\text{TiC}}=14$) and Al metal ($\bar{Z}_{\text{Al}}=13$), as well as that between Ti_2AlC phase ($\bar{Z}_{\text{Ti}_2\text{AlC}}=15.75$) and Ti_3AlC_2 phase ($\bar{Z}_{\text{Ti}_3\text{AlC}_2}=15.17$) renders it difficult to discriminate these phases from BSE images. Therefore, the presence of these by-products should be investigated using other characterization techniques. Nevertheless, a microstructure evolution with the temperature can be evidenced from the micrographs. With the increasing temperature, an apparent rise in the content of the TiAl_3 compound can be observed in the joints, which, again, may be attributed to the heavier decomposition degree of the Ti_2AlC substrate. Therefore, the reinforced wetting and interaction process are considered to be the major factors contributing to the

improved mechanical property of the Ti_2AlC joints. In addition, the morphology of the TiAl_3 compound changes as well: at low brazing temperature, the TiAl_3 compound with low aspect ratio forms in the joints, originates from the wetting surface; while brazed at high temperature, the aspect ratio of the TiAl_3 compound increases significantly, and the grains even cross the whole joint at 1173 K. Since the TiAl_3 intermetallic compound possesses poorer deformation ability than the Al metal filler, cracks may easily be initiated from the TiAl_3 compound, which contributes to the declining shear strength of the Ti_2AlC joints at 1173 K. Furthermore, a measurement performed under the SEM suggests that the width of the Ti_2AlC joints brazed at 1023 K, 1073 K and 1123 K are very close to each other, as they are determined to be 31.8 μm , 34.4 μm and 29.6 μm , respectively. However, when the brazing temperature reaches up to 1173 K, the width remarkably reduces to 16.4 μm , which is considered to be another factor responsible for the poor mechanical property of the joints at high temperature.

Fig. 4 shows the morphology of the Ti_2AlC joints brazed at 1123 K, holding for 0 min and 30 min, respectively. It can be concluded from the figure that the extending holding time has similar effect on the microstructure evolution of the brazing joints with the increasing brazing temperature. When the holding time is 0 min, the wetting process, as well as the interaction process is not sufficient. Therefore, a small amount of TiAl_3 intermetallic compound with small aspect ratio forms on the interface. Afterwards, when the holding time prolongs, more TiAl_3 compound can be observed in the joints and the shear strength remarkable improves, which is attributed to the reinforced

interaction between Ti_2AlC substrate and pure Al metal filler. In addition, the holding time is supposed to have more significant effect on the width of the Ti_2AlC joints. It can be observed from the figures that the joints width remarkably decreases from $60.5\text{ }\mu\text{m}$ to $29.6\text{ }\mu\text{m}$ when the holding time is extended from 0 min (Fig. 4(a)) to 10 min (Fig. 3(c)), and then further declines to $20.9\text{ }\mu\text{m}$ for 30 min (Fig. 4(b)). The major factor responsible for the narrowing width of the Ti_2AlC joints is assumed to be the squeezed out liquid Al filler during the brazing process, due to the improved wettability and fluidity with extending holding time, which is verified by the macro morphology observation. According to the conclusion above, the decreasing width of the joints would lead to an increase of the relative fraction of the TiAl_3 intermetallic compound in the Ti_2AlC joints, which is considered to be unfavorable for the mechanical property. Correspondingly, the shear strength declines to a low level when the holding time is 30 min.

The pure Al metal filler adopted in the present work is considered to be favorable for restraining the outward-diffusion of Al atoms in the Ti_2AlC structure, thereby preventing the transformation of the ceramic substrate. However, a small amount of decomposition products can still be observed in the Ti_2AlC joint. In order to determine the reaction products in the joints, thereby revealing the decomposition mechanisms of the Ti_2AlC ceramic during the brazing process, some supplementary characterizations were performed. According to the reaction (1), the increasing Al content in the substrate would contribute to the transformation of the Ti_2AlC phase to the Ti_3AlC_2 phase, accompanied with the formation of the TiAl_3 intermetallic compound. Therefore, the

reaction product Ti_3AlC_2 might distribute in the Ti_2AlC substrate if the reaction proceeds following the equation (1). An EDS line scan was carried out across the joining interface, and the results are displayed in Fig. 5. Fig. 5(a) shows the magnified morphology of the interaction area in the Ti_2AlC brazing joints. It is worthwhile pointing out that the grain boundary of the Ti_2AlC ceramic can be apparently observed in the interaction area, which is mainly attributed to the diffusion of the Al filler into the ceramic substrate along the grain boundary. In addition, the yellow line in the figure illustrates the scanning path, and the corresponding EDS line scan result is shown in Fig. 5(b). It can be concluded from the figure that an inter-diffusion layer with a thickness of $2.6\text{ }\mu\text{m}$ forms in the interaction area (the corresponding position is labeled in the Fig. 5(a)), in which the Ti atoms diffuses into the brazing filler while the Al atoms diffuses into the ceramic substrate. The outward-diffusing Ti atoms can be rapidly consumed by reacting with the Al filler metal, and then forms the TiAl_3 intermetallic compound which nucleates on the interface because of the small temperature gradient. Meanwhile, the Al atoms diffuse into the Ti_2AlC substrate since the pure Al metal filler possesses higher Al concentration. However, the Al content of the Ti_2AlC substrate in the interaction area remains unchanged according to the EDS results. Considering the morphology observation in Fig. 5(a), it is reasonable to suppose that the inward-diffusion Al atoms mainly distribute on the grain boundary of the Ti_2AlC ceramic, and would not lead to the transformation from Ti_2AlC phase to Ti_3AlC_2 phase. Although it is difficult to characterize the phases in the inter-diffusion layer accurately

using the SEM and XRD techniques, the formation of such a thin layer would not be the major factor responsible for large quantities of the TiAl_3 compound in the joints. According to the reaction (2), part of the Ti_2AlC substrate would decompose during the brazing process, and then forms the TiAl_3 and TiC_x compounds. In general, the TiC_x phase with nanolaminate microstructure can be dissolved by the liquid filler, therefore, the reaction product TiC_x might be able to be found in the filler area if the reaction proceeds following the equation (2). Afterwards, an XRD analysis of the brazing joints was performed in the following way: the Ti_2AlC brazing couple was cut along one of the substrate sides and then cautiously ground until the joint was exposed. The corresponding XRD pattern is displayed in Fig. 5. It can be observed from the diagram that the Al filler metal is the major phase in the Ti_2AlC joints. In addition, the TiC_x compound, as well as the TiAl_3 intermetallic compound is detected, suggesting that the decomposition of Ti_2AlC substrate during the brazing process contributes to the formation of TiAl_3 and TiC_x compounds, following the reaction (2).

Conclusions

In the present work, joining of the Ti_2AlC ceramics has been successfully performed using the pure Al filler metal, at the brazing temperature of 1023 K, 1073 K, 1123 K and 1173 K, holding for 0 min, 10 min and 30min, respectively. The results lead to the following conclusions:

- (1). The maximum shear strength of the Ti_2AlC joints brazed using the pure Al filler metal is measured to be 95 MPa, together with an electrical conductivity of

$2.21 \times 10^6 \text{ S} \cdot \text{m}^{-1}$. This optimal characteristic is obtained at the brazing temperature of 1123 K and holding for 10 min. The shear strength of the brazing joints accounts for 43% of the shear strength of the Ti_2AlC substrate, while the electrical conductivity accounts for 93%.

(2). During the brazing process, an inter-diffusion layer forms in the Ti_2AlC substrate, in which the Ti atoms diffuses into the brazing filler while the Al atoms diffuses into the ceramic substrate. The outward-diffusing Ti atoms can be rapidly consumed by forming the TiAl_3 compound in the joints, while the inward-diffusing Al atoms, however, mainly distribute on the grain boundary of the Ti_2AlC substrate.

(3). Since the outward-diffusion of Al atoms in the Ti_2AlC structure has been restrained by utilizing the brazing filler with high Al content, only a small amount decomposition products, including the TiAl_3 and TiC_x compounds can still be observed in the Ti_2AlC joint. The weak interaction between Ti_2AlC ceramic and pure Al filler is considered to be favorable for restraining the thermal stability of the Ti_2AlC brazing joints.

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Fig. 1. Characterization of the raw Ti_2AlC ceramics: (a). The BSE morphology; (b). The XRD pattern.

Fig. 2. The shear strength and electrical conductivity of the Ti_2AlC joints: (a) achieved with different brazing temperature; (b) achieved with different holding time.

Fig. 3. The microstructure of the Ti₂AlC joints, brazed at (a). 1023 K; (b). 1073 K; (c). 1123 K and (d). 1173 K, respectively.

Fig. 4. The microstructure of the Ti₂AlC brazing joints holding for (a). 0 min and (b). 30 min, respectively.

Fig. 5. The EDS line scanning results across the interaction area: (a). The BSE morphology of the interaction area, together with the EDS line scanning path; (b). The EDS line scanning results.

Fig. 6. The XRD pattern collected in the filler area of the Ti₂AlC brazing joints.