

Joining of Ti–Al–C ceramics by oxidation at low oxygen partial pressure

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Abstract

The joining of titanium aluminum carbides has been successfully performed at high temperature and low oxygen partial pressure. The mechanism of the bonding is attributed to the preferential oxidation of Al atoms in the titanium aluminum carbides at low oxygen partial pressure, which leads to the formation of an Al₂O₃ layer through the joint interface. The specimens joined at 1400 °C exhibit a high flexural strength of 315 ± 19.1 MPa for Ti₂AlC and 332 ± 2.83 MPa for Ti₃AlC₂, which is about 95% and 88% of the substrates, respectively, and the high flexural strength can be retained up to 1000 °C. The high mechanical performance of the joints is attributed to the similar density and thermal expansion coefficient values of Al₂O₃ to those of the Ti₂AlC and Ti₃AlC₂ substrates. It indicates that bonding via preferential oxidation at low oxygen partial pressure is a practical and efficient method for Ti₂AlC and Ti₃AlC₂.

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Keywords: Titanium aluminum carbides; Joining; Preferential oxidation; Al₂O₃ interfacial layer; Flexural strength

1. Introduction

The nanolaminated ternary ceramics M_{n+1}AX_n, where M is an early transitional metal, A is an A-group element, and X is carbon and/or nitrogen, have attracted extensive attention due to their combination of the excellent properties of both metals and ceramics.¹ Among these layered ternary compounds, the materials in the Ti–Al–C system, i.e., Ti₂AlC and Ti₃AlC₂, have promoted comprehensive research activity because of their extraordinary mechanical, physical and chemical properties. Other than the merits of high bulk modulus, good damage-tolerance and machinability commonly shared by the ternary carbides of the same family, the ternary carbides in the Ti–Al–C system have low density, excellent thermal-shock and oxidation resistance.^{1–21} Such unique properties make them promising candidates for high-temperature structural applications and oxidation-resistant coatings on alloys.¹² However, similar to other ceramics, it is difficult to synthesize bulk materials in

the Ti–Al–C system with complex shape and large dimensions, which greatly limits their widespread applications. A convenient and effective approach to solve this problem is the joining technology, which allows the manufacture of large, complex, multifunctional assemblies through the controlled integration of smaller, simpler, more easily manufactured parts. Joining is a critical enabling technology, being essential to the widespread application of ceramics in many fields.²² Therefore, studies on the joining of Ti–Al–C ceramics are significant for promoting their application. However, only a few papers on the joining of titanium aluminum carbides can be found in the literature. Yin et al.²³ bonded Ti₃AlC₂ ceramic via a Si interlayer based on the structural characteristics of Ti₃AlC₂ and the easy formation of a Ti₃Al_{1-x}Si_xC₂ solid solution. Firstly, a Si layer with a thickness of about 4 μm was deposited on the faying face of Ti₃AlC₂ specimens using a magnetron sputtering method, and then joining was performed at 1300–1400 °C for 120 min under 5 MPa. The mechanism of bonding is attributed to the inward diffusion of silicon into the Ti₃AlC₂ to form a Ti₃Al(Si)C₂ solid solution at the interface. A manufacturing process with pressure applied by program and magnetron sputtering goes against the widespread use of this process because of its high cost and complexity. Therefore, searching for a more practical and efficient joining method is significant for the widespread use of Ti–Al–C ceramics.

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The isothermal oxidation of Ti_2AlC and Ti_3AlC_2 in air has been extensively investigated.^{11–14} It was found that the scales formed on Ti_3AlC_2 substrate at 1000–1300 °C consisted of a continuous inner layer of Al_2O_3 and an outer layer of rutile TiO_2 or a mixture of Al_2TiO_5 and TiO_2 . The scales formed on a Ti_2AlC substrate over the 1000–1300 °C range consisted of a continuous inner layer of Al_2O_3 and a discontinuous outer layer of rutile TiO_2 . The scales formed on Ti_2AlC and Ti_3AlC_2 substrates at all temperatures were dense, adherent, and resistant to thermal cycling due to the negligible differences in the densities and thermal expansion coefficients between the Al_2O_3 scales and the Ti_2AlC or Ti_3AlC_2 substrates. The stability of the nanolaminated Ti_3AlC_2 upon heating at low oxygen partial pressure was also investigated.¹⁵ It was demonstrated that the selective oxidation of Al in Ti_3AlC_2 took place on the surface of the specimen, which resulted in the transformation of Ti_3AlC_2 into substoichiometric TiC_x and Al_2O_3 . Previous investigation into the oxidation behavior of Ti_2AlC and Ti_3AlC_2 demonstrated that a dense and crack-free Al_2O_3 layer rapidly formed on the surface of these materials at higher temperatures. The mechanism for the rapid formation of the Al_2O_3 layer was the weak bonding between Al and the adjacent Ti–C–Ti covalent bond chain.^{24,25} Also, Al has a higher affinity for oxygen than that of Ti and C because the Gibbs free energy for the reaction of Al with oxygen to form Al_2O_3 is more negative than that of Ti and C.^{15,26} Thus, it is thermodynamically favorable for the preferential oxidation of Al in Ti_2AlC and Ti_3AlC_2 . With the low oxygen content in argon, Ti_2AlC and Ti_3AlC_2 were selectively oxidized to Al_2O_3 . It is possible to bond Ti_2AlC and Ti_3AlC_2 via an Al_2O_3 interlayer formed at the interface through oxidation at low oxygen partial pressure.

The present work concentrates on the bonding through oxidation at low oxygen partial pressure to form an Al_2O_3 interlayer at the interface. The main purpose is to select proper bonding parameters to form joints without remarkable deterioration of the excellent properties of Ti_2AlC and Ti_3AlC_2 at both room and elevated temperatures.

2. Experimental procedures

Bulk Ti_2AlC and Ti_3AlC_2 used in this work were synthesized by in situ hot pressing/solid–liquid reaction method using Ti, Al and graphite powders as starting materials. Details on the synthesis and characteristics of Ti_2AlC and Ti_3AlC_2 have been described elsewhere.^{7,8} The physical and mechanical properties of Ti_2AlC and Ti_3AlC_2 are listed in Table 1. The measured density of bulk Ti_2AlC is 4.01 g/cm³ which is 98% of the theoretical density, while the density of Ti_3AlC_2 is 4.20 g/cm³ which is 99% of the theoretical density. Specimens with dimensions of 3 mm × 8 mm × 8 mm were cut from the as-synthesized bulk Ti_2AlC and Ti_3AlC_2 ceramics by an electrical-discharge method. The faying face of every specimen was ground down to 1200 grit SiC paper and polished using 0.5 μm diamond paste. Afterwards, the samples were degreased ultrasonically in acetone, cleaned by distilled water and then dried. The two samples were joined in stagger in order to find the interfacial region accurately after the bonding process. In order to have good contact

Table 1
Physical and mechanical properties of Ti_2AlC and Ti_3AlC_2 .

Properties	Values	
	Ti_3AlC_2	Ti_2AlC
Molecular weight (g/mol)	196.64	134.75
Space group	$P6_3/mmc^3$	$P6_3/mmc^2$
Lattice constants (nm)		
<i>a</i>	0.30753 ³	0.30579 ²⁹
<i>c</i>	1.8578 ³	1.36429 ²⁹
Density (g/cm ³)		
Theoretical	4.25	4.11
Measured	4.2 ⁶	4.1 ¹⁰
Vickers hardness (GPa)	2.7 ⁹	2.8 ⁷
Modulus (GPa)		
Young's modulus	298 ¹	275 ^{30,31}
Shear modulus	124 ¹	118 ³⁰
Poisson's ratio	0.20 ¹	0.17 ^{30,31}
Compressive strength (MPa)		
Room temperature	764 ⁹	763 ¹⁶
1200 °C	196 ⁹	103 ¹⁶
Flexural strength (MPa)	340 ⁹	275 ⁷
Fracture toughness	7.2 ⁹	6.5 ⁷

between two faying faces, a block was set on top of the two specimens. The pressure applied on the specimens can be calculated by measuring the weight of the block using the expression $P = mg/s$, where m is the weight of the block, s is the area of the specimens, and g is the acceleration of gravity. The calculated uniaxial pressure was $\sim 10^{-1}$ MPa. The bonding was conducted in a furnace under a flowing argon atmosphere. The joining temperature was from 1300 °C to 1400 °C, while the holding time was set as 120 min. The heating rate was 15 °C/min in all runs, and the joints were cooled in the furnace after bonding. The purity of the argon used in this study was 99.99%, and the oxygen partial pressure was about 1 Pa. The impurity contents claimed by the manufacturer are listed in Table 2.

To identify the change in phase compositions of Ti_2AlC and Ti_3AlC_2 after bonding, phase compositions before and after bonding at 1400 °C were analyzed by X-ray diffraction (XRD). The XRD data were collected by a step-scanning diffractometer with Cu K α radiation (Rigaku D/max-2400, Tokyo, Japan). The joint interface and fracture surface were investigated by a scanning electron microscope (SEM, LEO, Oberkochem, Germany) equipped with an energy-dispersive X-ray spectroscopy (EDS) system. After bonding, the sample was sliced in the direction perpendicular to the stagger joint and polished. The SEM investigation was carried out along the joint interface. The morphologies of the etched Ti_2AlC and Ti_3AlC_2 surfaces before and after bonding were also observed by SEM in order to evaluate the changes in grain size.

Table 2
Impurities and their contents in argon.

O ₂	N ₂	H ₂ O	H ₂	C _n H _n
<10 ppm	<70 ppm	<15 ppm	<5 ppm	<10 ppm

In order to determine the bending strength of joined samples, four-point bending tests were performed on a universal testing machine with an outer span of 30 mm and inner span of 10 mm at a crosshead speed of 0.5 mm/min at room temperature, 600, 800 and 1000 °C, respectively. Initially, two Ti_2AlC or Ti_3AlC_2 blocks with dimensions of 12 mm × 20 mm × 20 mm were

bonded. After joining, the bonded specimens were machined into dimensions of 3 mm × 4 mm × 40 mm and polished. The joint was set in the middle of the span. Three samples were tested for each testing condition. When tested at high temperatures, the samples were heated to the target temperature and held for 10 min to achieve temperature equilibrium. Because of the

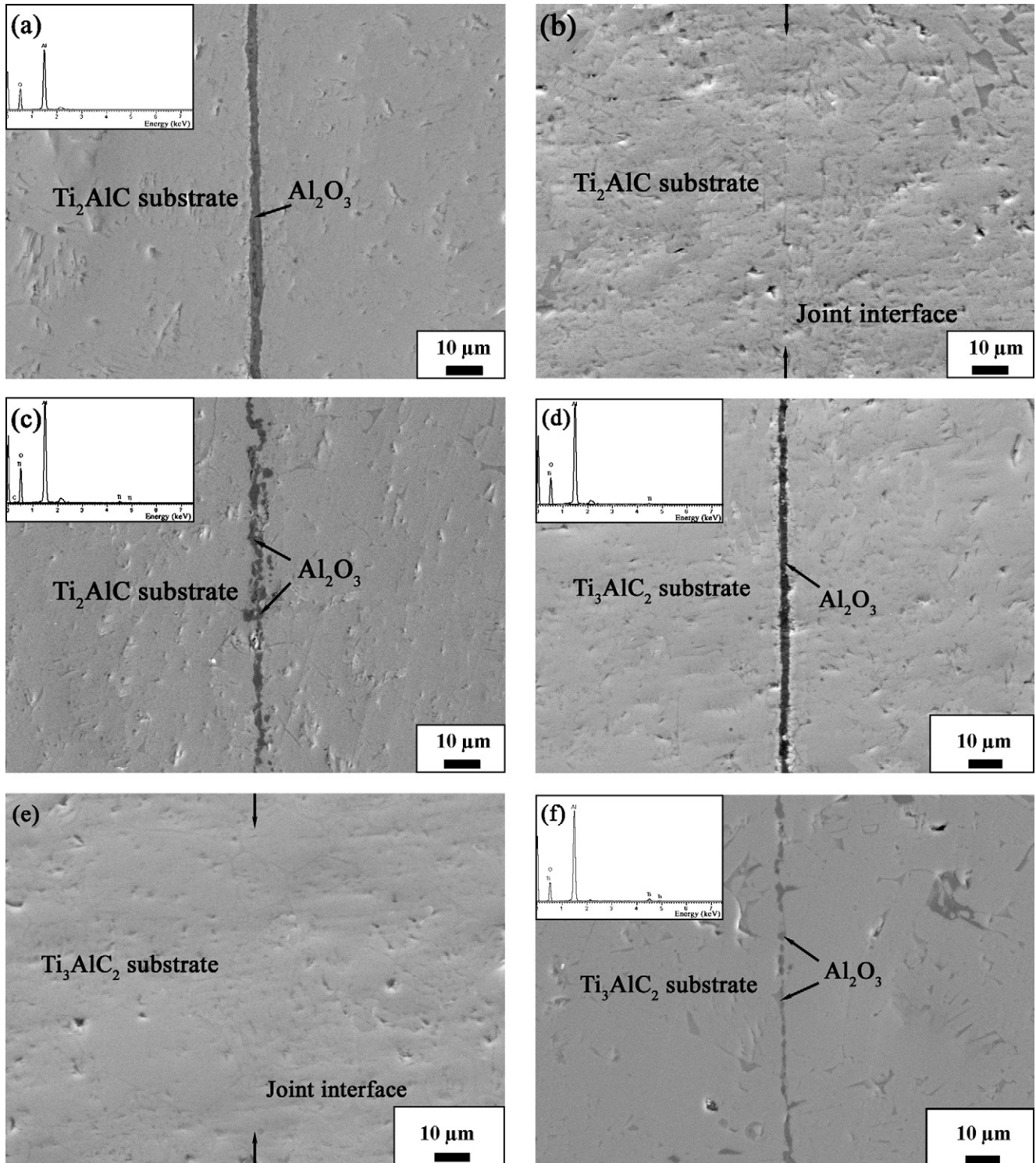


Fig. 1. SEM images of the interfacial structure of the Ti_2AlC couples bonded at (a and b) 1300 °C and (c) 1400 °C, and the Ti_3AlC_2 couples bonded at (d and e) 1300 °C and (f) 1400 °C for 120 min, respectively (the insets are the EDS maps of the interfacial layer).

excellent oxidation resistance of Ti_2AlC and Ti_3AlC_2 at temperatures below $1000^\circ C$ and the short annealing time, no attempt was made to avoid the slight oxidation of the samples during testing.

3. Results and discussion

Apparently, all of the Ti_2AlC and Ti_3AlC_2 couples could be joined together. Fig. 1 presents the cross-sectional views of typical microstructures of Ti_2AlC couples bonded at $1300^\circ C$ (Fig. 1a and b) and $1400^\circ C$ (Fig. 1c), and Ti_3AlC_2 couples bonded at $1300^\circ C$ (Fig. 1d and e) and $1400^\circ C$ (Fig. 1f) for 120 min, respectively. An oxidation layer formed in Fig. 1a, c, d and f, and the EDS dot maps (the inset images) proved that the oxidation layer was Al_2O_3 . Joints bonded at $1300^\circ C$ had two different morphologies. Fig. 1a for Ti_2AlC couple and Fig. 1d for Ti_3AlC_2 couple had continuous Al_2O_3 layer, while Fig. 1b for Ti_2AlC couple and Fig. 1e for Ti_3AlC_2 couple had hardly any Al_2O_3 layer. Joints bonded at $1400^\circ C$ had Al_2O_3 layer through the whole joint interface. Wang and Zhou¹³ revealed that the relative amount of the Al_2O_3 formed on the Ti_3AlC_2 samples increased with the increasing temperature through isothermal oxidation, originated from the rise in temperature promoted diffusion of Al from the matrix to the surface and enhances nucleation and growth of Al_2O_3 . This is in accordance with our results, i.e., at $1300^\circ C$ Al_2O_3 layer was discontinuous while at $1400^\circ C$ Al_2O_3 layer was through the whole joint interface.

Fig. 2a and b shows the XRD patterns of powders drilled from the substrate away from the interface of Ti_2AlC and Ti_3AlC_2 joints bonded at $1400^\circ C$ for 120 min, respectively. For comparison, XRD patterns of powders from Ti_2AlC and Ti_3AlC_2 substrates before bonding are also presented. As shown in Fig. 2, no impurity phases were detected in the Ti_2AlC and Ti_3AlC_2 substrates before bonding. After joining, no new reaction phase can be detected within the resolution of the X-ray diffractometer.

Fig. 3a shows the measured flexural strength of the Ti_2AlC joint bonded at $1400^\circ C$ for 120 min as a function of testing temperature. For comparison, the flexural strength of Ti_2AlC tested under the same conditions is also presented in Fig. 3a. In order to maintain consistency, the joint and substrate used for comparison are from the same bulk Ti_2AlC material. The flexural strength of the joint at room temperature is 315 ± 19.1 MPa, which is only slightly lower than that of the Ti_2AlC substrate (330 ± 63.1 MPa). At higher temperatures, the joint can still retain a strength of above 90% of the Ti_2AlC substrate. At $600^\circ C$, the joint has roughly the same flexural strength as the Ti_2AlC substrate. At $1000^\circ C$, the flexural strength of the joint is still as high as 262 ± 41.5 MPa. Fig. 3b shows the flexural strength of the Ti_3AlC_2 joint bonded at $1400^\circ C$ for 120 min. The flexural strength of Ti_3AlC_2 substrate from the same bulk material is also presented in Fig. 3b. The flexural strength of the joint at room temperature is 332 ± 2.83 MPa, which is about 88% of that of the Ti_3AlC_2 substrate, and the high strength can be retained up to $1000^\circ C$. At $1000^\circ C$, the flexural strength of the joint is still as high as 255 ± 2.12 MPa. The flexural strength of the joints bonded at $1300^\circ C$ for 120 min is 117 MPa for Ti_2AlC and 121 MPa for Ti_3AlC_2 , respectively. The flexural strength

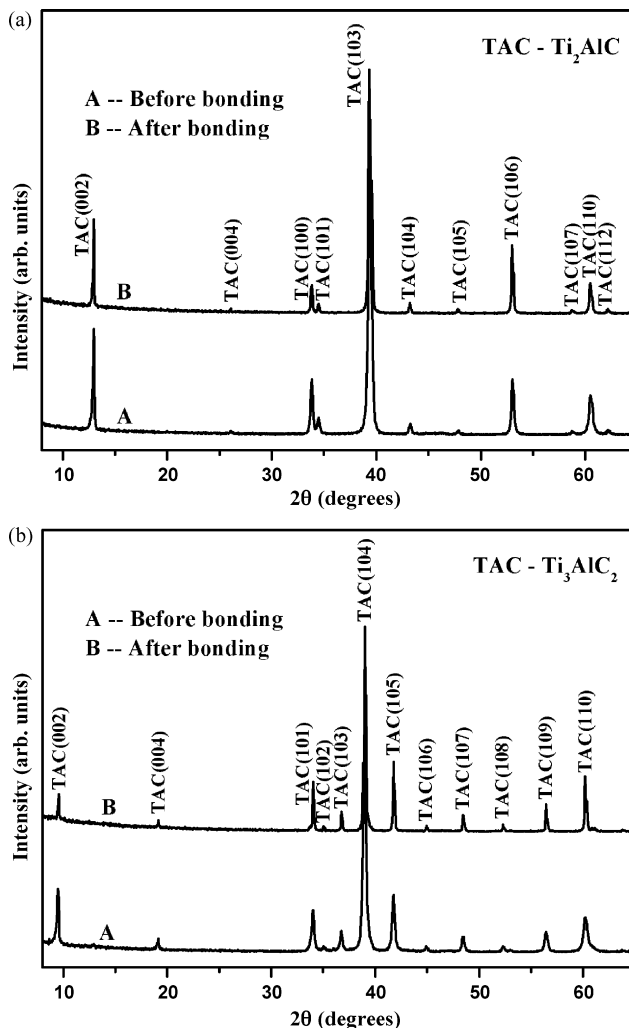


Fig. 2. XRD of the (a) Ti_2AlC and (b) Ti_3AlC_2 before and after bonding at $1400^\circ C$ in argon for 120 min.

of the joints bonded at $1300^\circ C$ is much lower than that of the joints bonded at $1400^\circ C$. This can be ascribed to the discontinuous Al_2O_3 oxidation layer formed at $1300^\circ C$ compared to the continuous Al_2O_3 layer through the whole interface at $1400^\circ C$. These results indicate that the Ti_2AlC and Ti_3AlC_2 joints bonded at $1400^\circ C$ with high mechanical performance can be achieved through oxidation bonding. Thus, they may meet the requirements for high-temperature applications. The room temperature flexural strength of joints in this work compared with those reported by Yin et al. is presented in Table 3. The advantages of the joining process in this work can be obviously seen, i.e., no silicon interlayer deposited by magnetron sputtering before joining, no high pressure applied during joining, and better mechanical performance. According to the work by Yin et al., the room temperature flexural strength of joints bonded at $1400^\circ C$ for 120 min under 5 MPa²³ is 285 ± 11 MPa, which is only 80% of that for the Ti_3AlC_2 substrate.

Fig. 4a and b shows the optical images of the fractured Ti_2AlC and Ti_3AlC_2 joints after room-temperature bending test. It was found that all the joints bonded at $1400^\circ C$ failed away from the joint interfaces, while the joints bonded at $1300^\circ C$ failed from

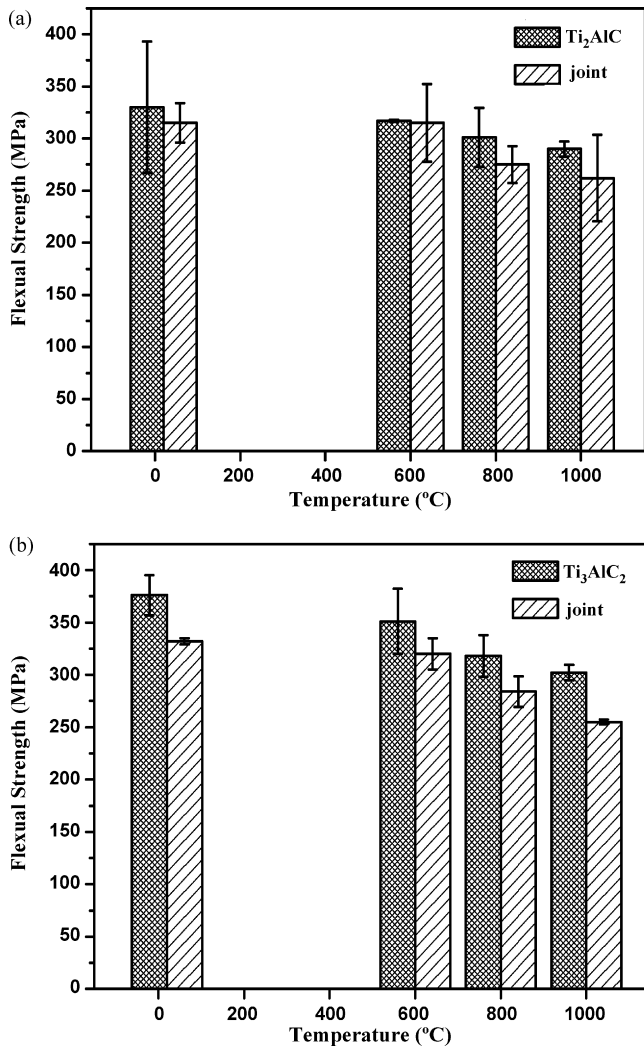


Fig. 3. Four-point bending strength of the joints bonded at 1400 °C compared with the substrate as a function of testing temperature: (a) Ti_2AlC and (b) Ti_3AlC_2 .

the interfacial layer, which also indicates the joints formed at 1400 °C are much stronger than those formed at 1300 °C.

Although joined samples bonded at 1400 °C fractured away from the joint interfaces, i.e., they fractured through the Ti_2AlC and Ti_3AlC_2 substrates, the strength of joined samples is still lower than that of the substrates at room temperature. This can be ascribed to the growth of Ti_2AlC and Ti_3AlC_2 grains through bonding at 1400 °C for 120 min in argon. Fig. 5 presents the morphologies of the Ti_2AlC and Ti_3AlC_2 grains before and after

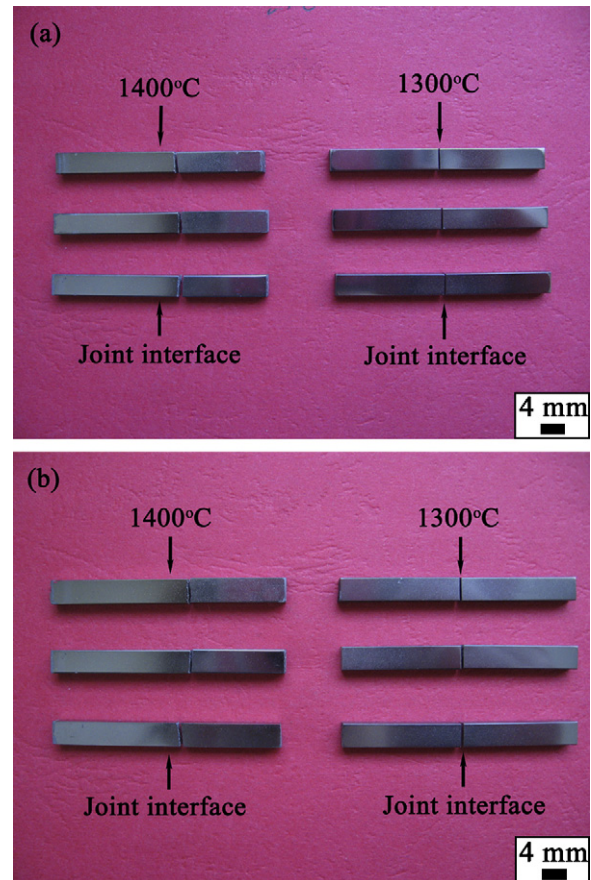


Fig. 4. Optical images of the fractured joints after room-temperature bending test: (a) Ti_2AlC and (b) Ti_3AlC_2 .

the bonding treatment. The grain size of Ti_2AlC before bonding is about $27 \pm 7 \mu\text{m}$ in length and $15 \pm 3 \mu\text{m}$ in width, which is smaller than that of the sample after bonding ($33 \pm 11 \mu\text{m}$ in length and $16 \pm 5 \mu\text{m}$ in width). The grain size of Ti_3AlC_2 before bonding is about $38 \pm 21 \mu\text{m}$ in length and $8 \pm 3 \mu\text{m}$ in width, while that after bonding is about $48 \pm 27 \mu\text{m}$ in length and $11 \pm 4 \mu\text{m}$ in width. So the decrease in strength of the joined samples is attributed to the growth of Ti_2AlC and Ti_3AlC_2 grains during the bonding treatment.

The formation of the Al_2O_3 oxidation layer through the whole joint interface leads to the joints to possess high strength. Lin et al.²⁷ evaluated the adhesive strength between the oxide scale and Ti–Al–C substrates. It was found that the adhesive strength was above 85 MPa. The oxidation scales are adherent to the substrates, which is attributed to the negligible difference between

Table 3

Room temperature mechanical strength of joints and substrates in this work compared with those reported by Yin et al. (superscripts denote the strength percentage of joints to substrates).

	Joining process	Bending strength (MPa)			
		Ti_3AlC_2 substrate	Ti_3AlC_2 joint	Ti_2AlC substrate	Ti_2AlC joint
Yin et al. ²³	1400 °C, 2 h in argon under 5 MPa with silicon interlayer	356	285 ^(80%)	–	–
This work	1400 °C, 2 h in argon under $\sim 10^{-1}$ MPa free of silicon interlayer	376	332 ^(88%)	330	315 ^(95%)

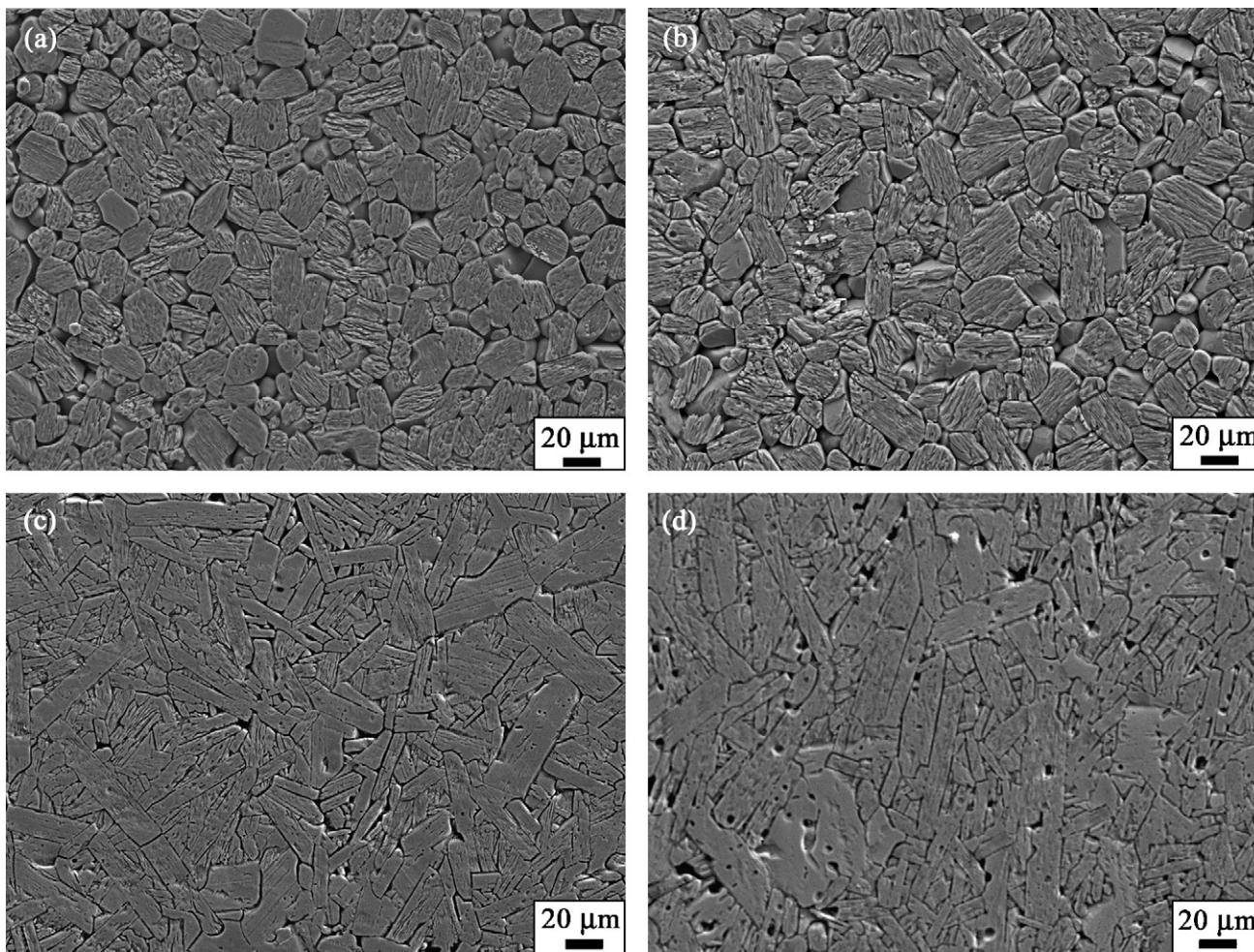


Fig. 5. SEM morphologies of (a and b) Ti_2AlC and (c and d) Ti_3AlC_2 grains before and after bonding treatment: (a and c) before bonding and (b and d) after bonding.

the density of the oxide layer formed and those of the substrates. The densities of Ti_2AlC and Ti_3AlC_2 are 4.1 and 4.2 g/cm^3 , respectively,^{6,10} while the density of Al_2O_3 is 3.98 g/cm^3 .²⁸ So there are only small volume changes as the scales form. Thus, the oxide scales have good adhesion to the substrates. The thermal expansion coefficients of Ti_2AlC and Ti_3AlC_2 are $8.8 \times 10^{-6} \text{ K}^{-1}$ and $9.0 \times 10^{-6} \text{ K}^{-1}$, respectively,^{1,6} while that of Al_2O_3 is $8.8 \times 10^{-6} \text{ K}^{-1}$ (parallel to the c -axis) and $9.0 \times 10^{-6} \text{ K}^{-1}$ (normal to the c -axis), respectively.²⁸ The small difference in thermal expansion coefficients between the substrates and Al_2O_3 accounts for the good adhesion of the Al_2O_3 layer to the substrates because the thermal stress generated during cooling after oxidation is proportional to the difference in thermal expansion coefficients between the substrates and the oxidation layer formed.¹² So bonding via the Al_2O_3 oxidation layer formed in situ through heat treatment is a practical and efficient method for joining titanium aluminum carbides.

4. Conclusions

Strong joints of titanium aluminum carbide ceramics can be achieved at 1400°C through preferential oxidation to form an Al_2O_3 oxidation layer through the whole joint interface. The

mechanism of bonding is attributed to the preferential oxidation of Al atoms in the Ti_2AlC and Ti_3AlC_2 substrates in argon with a low oxygen partial pressure. The formation of Al_2O_3 causes the joint to possess high strength, owing to the similar density and thermal expansion coefficient values of Al_2O_3 to those of the Ti_2AlC and Ti_3AlC_2 substrates. The room temperature flexural strength of the Ti_2AlC joint bonded at 1400°C for 120 min is $315 \pm 19.1 \text{ MPa}$, which is about 95% of that of the Ti_2AlC . At 1000°C , the flexural strength of the joint is still as high as $262 \pm 41.5 \text{ MPa}$. The room temperature flexural strength of the Ti_3AlC_2 joint bonded at 1400°C for 120 min is $332 \pm 2.83 \text{ MPa}$, which is about 88% of that of the Ti_3AlC_2 , and the high strength can be retained up to 1000°C . The high mechanical performance of the joints indicates that bonding through preferential oxidation to form an Al_2O_3 layer through the whole joint interface is an effective method for bonding the titanium aluminum carbide ceramics.

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