

Microstructure and mechanical strength of transient liquid phase bonded Ti_3SiC_2 joints using Al interlayer

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Abstract

Based on the structure characteristic of Ti_3SiC_2 and the easy formation of $\text{Ti}_3\text{Si}_{1-x}\text{Al}_x\text{C}_2$ solid solution, a transient liquid phase (TLP) bonding method was used for bonding layered ternary Ti_3SiC_2 ceramic via Al interlayer. Joining was performed at 1100–1500 °C for 120 min under a 5 MPa load in Ar atmosphere. SEM and XRD analyses revealed that $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution rather than intermetallic compounds formed at the interface. The mechanism of bonding is attributed to aluminum diffusing into the Ti_3SiC_2 . The strength of joints was evaluated by three point bending test. The maximum flexural strength reaches a value of 263 ± 16 MPa, which is about 65% of that of Ti_3SiC_2 ; for the sample prepared under the joining condition of 1500 °C for 120 min under 5 MPa. This flexural strength of the joint is sustained up to 1000 °C.

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1. Introduction

Joining is a critical enabling technology, essential to widespread use of ceramics in many applications. Specifically, it allows manufacture of large, complex, multifunctional assemblies through the controlled integrations of smaller, simple, more easily manufactured parts. Moreover, it can provide an effective method for repair of damaged structures through the replacement of defective components. This can extend the lifetime of assemblies, and permit the reuse of components that are not readily recycled.

Recently, the nanolaminate ternary ceramics $M_{n+1}AX_n$ (where M is an early transition metal, A is an A-group element, and X is either carbon or nitrogen) have attracted extensive attention due to their combination of the excellent properties of metals and ceramics. The salient properties of these ternaries include low density, high bulk modulus, good thermal and electrical conductivity, excellent thermal shock resistance and

oxidation resistance, good machinability, damage tolerance and microscale ductility at room temperature.¹ Among these layered ternary compounds, titanium silicon carbide (Ti_3SiC_2) has been extensively investigated because of its explicit industrial application prospects as a high temperature structural material, owing to its superior high temperature properties.^{1–8} However, up to now, its applications are very limited. Similar to other ceramics, the synthesis of bulk Ti_3SiC_2 with big dimensions is also difficult in practice. This limitation can be overcome through joining technology. Therefore, studies on joining of Ti_3SiC_2 with metals or ceramics are significant for promoting its applications. Gao and Miyamoto⁹ conducted the diffusion bonding of Ti_3SiC_2 with Ti-6Al-4V in the temperature range from 1200 to 1400 °C, and the bending strength of the joints was determined to be a quarter of that of Ti_3SiC_2 . Yin et al.¹⁰ conducted the diffusion bonding of Ti_3SiC_2 with Ni. Their results indicated that Ti_3SiC_2 could be bonded with nickel in the temperature range from 800 to 1100 °C, and the shear strength of the joint was close to that of Ti_3SiC_2 . Work on joining of Ti_3SiC_2 with other alloys or ceramics has not been reported.

Previous work by Li et al.¹¹ revealed that Al-La co-cementation on Ti_3SiC_2 improved the oxidation resistance of Ti_3SiC_2 dramatically. A part of Al was observed to diffuse into the whole Ti_3SiC_2 bulk sample to form $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solu-

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tion. Zhou et al.¹² synthesized $\text{Ti}_3\text{Si}_{1-x}\text{Al}_x\text{C}_2$ solid solutions by the substitution of a small amount of Si with Al. They demonstrated that the mechanical properties of $\text{Ti}_3\text{Si}_{1-x}\text{Al}_x\text{C}_2$ solid solutions were close to those of Ti_3SiC_2 . A similar approach of adding Al into Ti_3SiC_2 was also used by Zhu et al.^{13,14} for accelerating the synthesis process and fabricating the single phase bulk Ti_3SiC_2 . Since addition of Al in Ti_3SiC_2 is therefore favorable to the formation of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution, it should be possible to form $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution through diffusion bonding Ti_3SiC_2 via an Al interlayer. However, it has been demonstrated that $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution could not form through solid state reaction between Ti_3SiC_2 and Al when they existed as either bulk material¹⁵ or powders.¹⁶ So an alternative joining technique, transient liquid phase (TLP) bonding, has been considered.

The TLP bonding technique has the potential to join ceramics or complex alloys with minimum disruption to the parent material microstructure.¹⁷ This bonding technique involves placing a thin metal interlayer between the two bonding surfaces and heating them to a temperature above the melting point of the interlayer but below the melting point of the parent materials. The joint region is held at this bonding temperature until the joint solidifies isothermally due to interdiffusion. The TLP bonding technique has been used successfully in joining structural metals,^{17,18} intermetallics,^{19,20} oxide and non-oxide ceramics.^{21–24}

In this paper, joining of Ti_3SiC_2 ceramic via Al interlayer was conducted using TLP bonding method. The main purpose is to select proper bonding parameters to form joints of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution without deteriorating the excellent properties of Ti_3SiC_2 at both room and elevated temperatures.

2. Experimental procedures

The materials used in this work were bulk Ti_3SiC_2 with the dimension of $\varnothing 10 \text{ mm} \times 2 \text{ mm}$, and commercial pure aluminum foil with the thickness of $50 \mu\text{m}$. Bulk Ti_3SiC_2 containing a small amount of TiC impurity was synthesized from Ti, Si and graphite powders by in situ hot pressing/solid–liquid reaction. The measured density of bulk Ti_3SiC_2 is 4.41 g/cm^3 and its relative density is 97%. The microstructure of the Ti_3SiC_2 is shown in Fig. 1. Details for the synthesis and characteristics of Ti_3SiC_2 were described elsewhere.²⁵ The joining face of the Ti_3SiC_2 was polished to $1 \mu\text{m}$ diamond paste, and Al foil was ground to 1000 grade abrasive paper. All specimens were ultrasonically cleaned in acetone prior to joining. An Al foil was placed between a pair of Ti_3SiC_2 couples to form a sandwich. In order to find the interface region accurately after the bonding process, the Ti_3SiC_2 couples were staggered, as shown in Fig. 2. The sandwich was placed in a furnace and heated under flowing Ar atmosphere. The bonding was conducted in the temperature range from 1100 to $1500 \text{ }^\circ\text{C}$ for 120 min under 5 MPa pressure. The heating rate was $15 \text{ }^\circ\text{C}/\text{min}$ in all runs, and joints were cooled in the furnace after bonding.

The joints for 3-point bending tests were prepared by inserting an Al foil between two Ti_3SiC_2 blocks with the dimensions

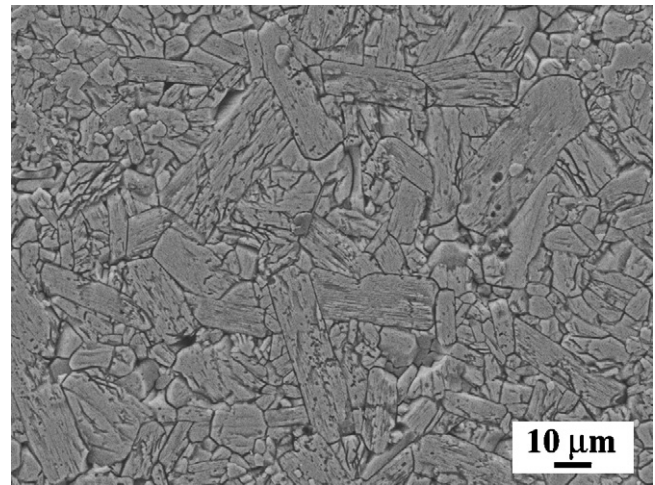


Fig. 1. Microstructure of the bulk Ti_3SiC_2 .

of $20 \text{ mm} \times 20 \text{ mm} \times 12 \text{ mm}$. After joining, test specimens of the dimensions of $3 \text{ mm} \times 4 \text{ mm} \times 40 \text{ mm}$ were machined out and polished. The bending tests were performed in a universal testing machine at room temperature, 600, 800 and $1000 \text{ }^\circ\text{C}$ respectively. The crosshead speed was $0.5 \text{ mm}/\text{min}$ and the span was 30 mm. The joint was set in the middle of the span. When tested at high temperatures in air, the samples were heated to target temperature and held for 15 min to get temperature equilibrium. Taking account of the excellent oxidation resistance of Ti_3SiC_2 at temperature below $1000 \text{ }^\circ\text{C}$ and the short annealing time, no attempt was made to avoid the slight oxidation of the samples.

The joint interface and fracture surface were investigated by a scanning electron microscope (SEM, Supra 35, LEO, Germany) equipped with energy-dispersive spectroscopy (EDS) system. After bonding, the sample was sliced in the direction perpendicular to the staggered joint and polished. SEM investigation was carried out along the staggered line. The phase compositions of the interface were analyzed by X-ray diffraction. The XRD data were collected by a step-scanning diffractometer with $\text{Cu K}\alpha$ radiation (Rigaku D/max-2400, Japan). To identify the change in lattice parameters of Ti_3SiC_2 after bonding, non-linear least square fitting was performed by using the structure models of Ti_3SiC_2 and TiC. The complete profile of the powder diffraction pattern was refined by Rietveld method employing the DBWS code in Cerius² computational program for materials research (Molecular Simulation Inc., USA). To measure peak angles accurately, each tested peak was scanned with a slow scanning step of 0.02 degree and a speed of 2 s/step.

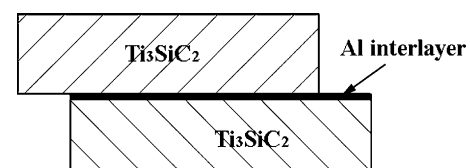


Fig. 2. Schematic illustration of the assembly for bonding.

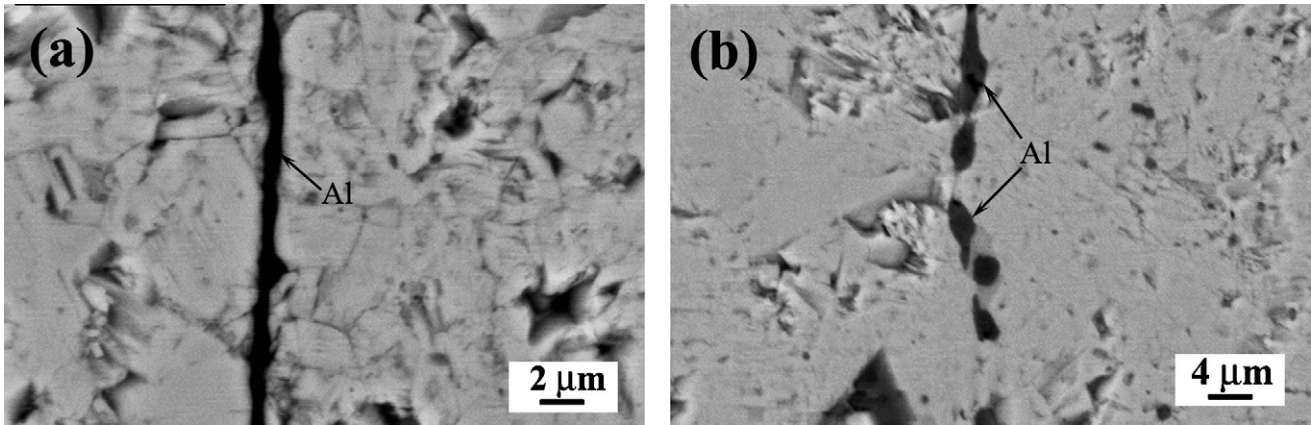


Fig. 3. Backscattered SEM images of the interfacial sections of the $\text{Ti}_3\text{SiC}_2/\text{Al}/\text{Ti}_3\text{SiC}_2$ couples treated at (a) 1100 °C and (b) 1400 °C for 120 min under 5 MPa.

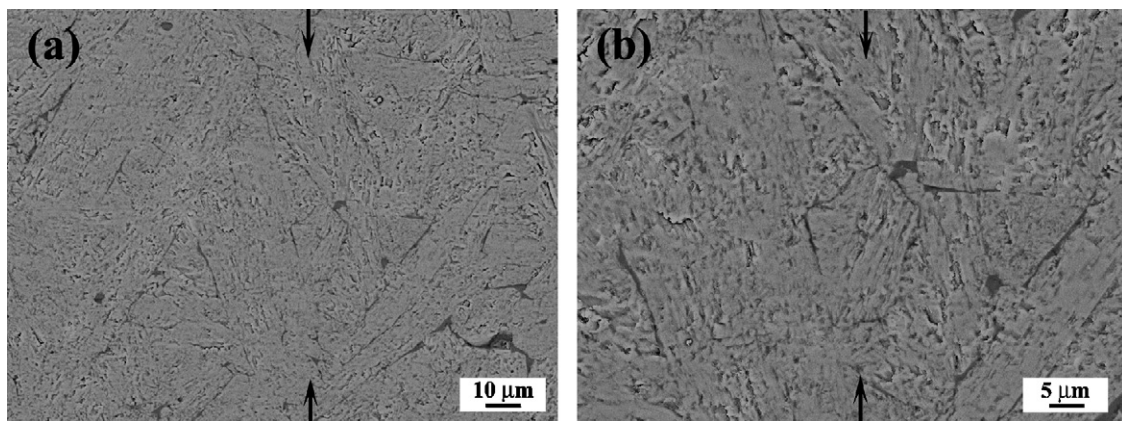


Fig. 4. Backscattered SEM images of the cross sections of the $\text{Ti}_3\text{SiC}_2/\text{Al}/\text{Ti}_3\text{SiC}_2$ couples treated at 1500 °C for 120 min under 5 MPa pressure at two different magnifications. Arrows denote the interface.

3. Results and discussions

At the bonding temperature, 1100–1500 °C, the Al interlayer melts, while Ti_3SiC_2 ceramic remains solid. As a result, a thin liquid layer forms between the Ti_3SiC_2 ceramic couples. With increasing time at the bonding temperature, the liquid aluminum diffuses into the Ti_3SiC_2 ceramic until the joint solidifies isothermally, and the bonding develops along the interface.

All of the Ti_3SiC_2 couples gave the appearance of being joined. However, when bonded at temperature lower than 1500 °C, an amount of residual aluminum was detected at the interface, as shown in Fig. 3. The residual aluminum would deteriorate the mechanical properties of the joints, especially at high temperatures. So, all joints were made thereafter at 1500 °C.

The cross sectional views of typical microstructures of $\text{Ti}_3\text{SiC}_2/\text{Al}/\text{Ti}_3\text{SiC}_2$ couple bonded at 1500 °C for 120 min under 5 MPa are shown in Fig. 4. It can be seen that the Al interlayer disappeared completely, and reaction zone, pores or original bond line were not observed at the interface, even in the large magnified micrograph (Fig. 4(b)).

In order to identify the phase composition of the interface, XRD analysis was carried out. The powders prepared for XRD analysis were obtained by drilling the joint interface. Fig. 5 shows the X-ray diffraction pattern of the joint bonded

at 1500 °C for 120 min under 5 MPa. For comparison, the XRD pattern of Ti_3SiC_2 is also presented in Fig. 5. As shown in the figure, the phase of the joint can be indexed using the structure of Ti_3SiC_2 and TiC, which is an impurity phase in the Ti_3SiC_2

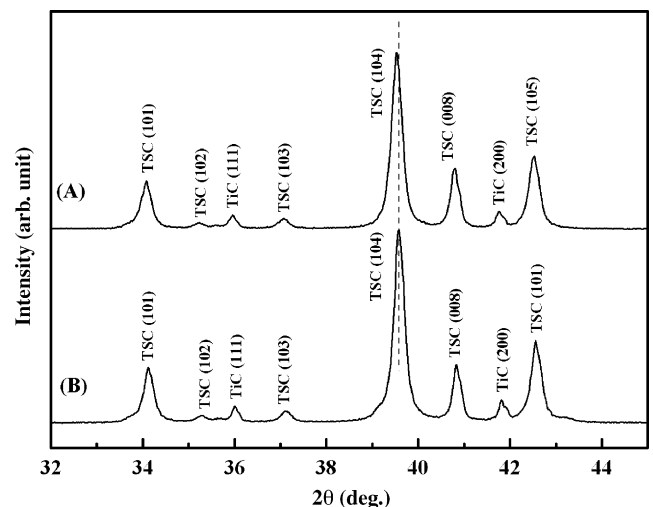


Fig. 5. X-ray diffraction patterns of powders from (A) the $\text{Ti}_3\text{SiC}_2/\text{Al}/\text{Ti}_3\text{SiC}_2$ joint bonded at 1500 °C for 120 min under 5 MPa and (B) Ti_3SiC_2 .

Table 1

Lattice parameters (in nm) and c/a ratio of Ti_3SiC_2 , $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ (joint), $\text{Ti}_3\text{Si}_{0.9}\text{Al}_{0.1}\text{C}_2$, and Ti_3AlC_2

	a	c	c/a	Remarks
Ti_3SiC_2	0.3067	1.7673	5.762	This work
	0.3067	1.767	5.761	Ref. 1
	0.3067	1.7671	5.762	Ref. 26
$\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ (Joint)	0.3071	1.7692	5.761	This work
$\text{Ti}_3\text{Si}_{0.9}\text{Al}_{0.1}\text{C}_2$	0.3078	1.7704	5.751	Ref. 12
	0.3069	1.7685	5.762	Ref. 27
Ti_3AlC_2	0.3075	1.858	6.042	Ref. 1

substrate. No new reaction phases can be detected within the resolution of the X-ray diffractometer. The bonding process therefore has almost no detectable influence on the present phases. However, by careful analysis of the XRD patterns in Fig. 5, it can be seen that the reflections corresponding to Ti_3SiC_2 shift to lower angles after the bonding process, which suggests that Al diffused into Ti_3SiC_2 to form $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution. To quantitatively determine the change of lattice parameters, Rietveld refinement of the diffraction patterns was performed using Ti_3SiC_2 and TiC as the structure models. In all refinement, the reliability factors, i.e., R-P and R-WP values, are less than 10%. The calculated lattice parameters a and c of the $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution and Ti_3SiC_2 are listed in Table 1. In our treatment, the lattice parameters of standard Ti_3SiC_2 sample are $a = 0.3067$ nm, $c = 1.7673$ nm. This result is in good agreement with the values from other previous measurements for Ti_3SiC_2 .^{1,26} The measured lattice parameters of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ are $a = 0.3071$ nm, $c = 1.7692$ nm, which are bigger than that of Ti_3SiC_2 and smaller than that of Ti_3AlC_2 . This trend is consistent with the results from previous X-ray diffraction analysis¹² and neutron diffraction measurements²⁷ for $\text{Ti}_3\text{Si}_{1-x}\text{Al}_x\text{C}_2$ solid solutions, and provides an important indication for the formation of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution at the interface. According to the equation²⁸:

$$c(x) = 1.8541 - (8.5674 \times 10^{-2})x \quad (1)$$

where x is the Si content, $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution can be described as $\text{Ti}_3\text{Si}_{0.99}\text{Al}_{0.01}\text{C}_2$.

Based on the results of SEM and XRD analysis, it can be concluded that aluminum diffused into Ti_3SiC_2 to form $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution at the interface during the bonding process. It has been found that continuous solid solution can be formed in Ti_3SiC_2 - Ti_3AlC_2 system.¹ Previous experiments also proved that addition of Al was favorable to the formation of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution.^{11,12} Yu et al.²⁹ found by HREM investigations that Si reduced the twin boundary energy of TiC, leading to the formation of thin Ti_3SiC_2 platelets. They also^{30,31} demonstrated that, compared to Si, Al was more effective in reducing the twin boundary energy of TiC and in the presence of Al, $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution formed easily. Wang and Zhou³² demonstrated that the $\text{Ti}_3\text{Si}_{0.75}\text{Al}_{0.25}\text{C}_2$ solid solution was stable by first-principles calculations. In addition, Ti_3SiC_2 crystallizes in a space group of $\text{P}6_3/\text{mmc}$ symmetry and its crystal structure can

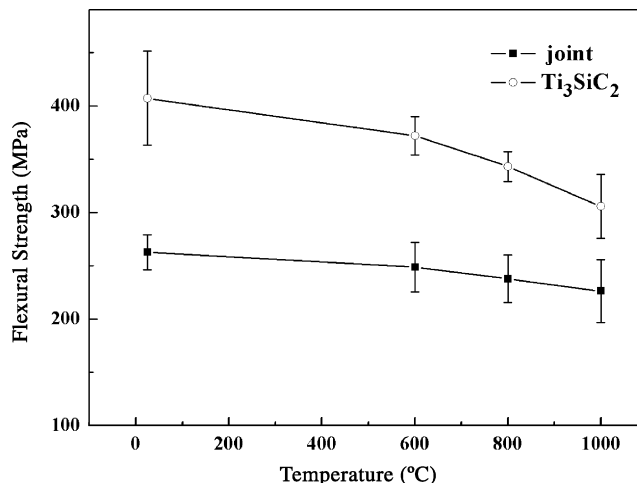


Fig. 6. Flexural strength of the joint and Ti_3SiC_2 as a function of testing temperatures.

be regarded as two edge-shared Ti_6C octahedral being weakly bonded with the interleaved planar closed-packed Si atomic layers.²⁶ This crystal structure is beneficial to intercalation of Al into Ti_3SiC_2 . Bulk Ti_3SiC_2 has a layered microstructure, and these interlamination supply a great number of rapid diffusion paths for Al.

Fig. 6 shows the measured flexural strength of the joint bonded at 1500 °C for 120 min as a function of testing temperature. For comparison, the flexural strength of Ti_3SiC_2 tested under the same condition as the joints is also presented in Fig. 6. The flexural strength of the joint tested at room temperature is 263 ± 16 MPa, which is about 65% of that of Ti_3SiC_2 substrate. Compared with Ti_3SiC_2 , the flexural strength of the joint decreases slightly with increasing testing temperature. At 1000 °C, the strength of the joint remains 226 ± 30 MPa, which is about 74% of that of Ti_3SiC_2 substrate. These results indicate that a joint with good mechanical strength can be achieved through TLP bonding and may meet the requirement of high temperature applications.

The fractured specimens were also examined by scanning electron microscope. It was found that samples failed along the joint, and no residual Al was detected on the fracture surface. Fig. 7 shows the morphology of the fractured surface of the joint treated at 1500 °C for 120 min under 5 MPa. The characteristic of the laminated Ti_3SiC_2 grain can be obviously seen from Fig. 7. These observations are consistent with the fact that Al diffuses into the Ti_3SiC_2 substrate.

Generally, if no defects exist at the interface, the strength of a joint is mainly related to its composition, interfacial microstructure and the residual stress resulting from mismatch of thermal expansion coefficient (TEC) during cooling from bonding temperature to ambient temperature. In the present work, due to the formation of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution, the joint has similar microstructure with the parent materials. Zhou et al.¹² have proved that the mechanical properties of $\text{Ti}_3\text{Si}_{1-x}\text{Al}_x\text{C}_2$ solid solution ($0.01 \leq x \leq 0.1$) are close to that of Ti_3SiC_2 . Though the values of thermal expansion coefficient of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ with different Al content has not been measured, taking account of

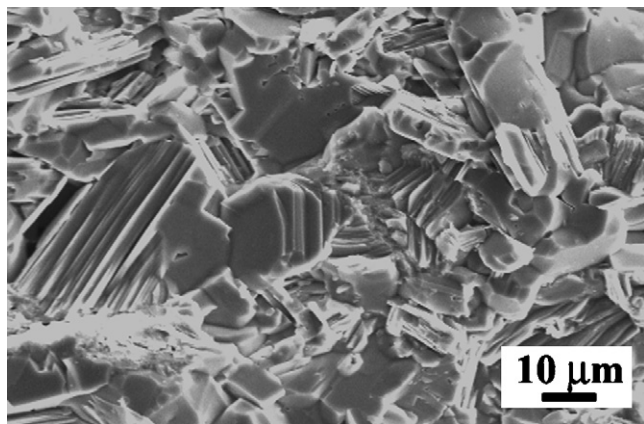


Fig. 7. Scanning electron micrograph of the fracture surface of the joint bonded at 1500 °C for 120 min under 5 MPa pressure.

the same crystal structure of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ and Ti_3SiC_2 , similar composition, and the mismatch of lattice parameters between $\text{Ti}_3\text{Si}(\text{Al})$ and Ti_3SiC_2 is less than 0.15%, it is reasonable to propose that both $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ and Ti_3SiC_2 may have a close value of TEC. Consequently, the residual stress in the joint is very small. In a word, owing to the formation of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution and the small TEC mismatch, the joint has a high flexural strength.

4. Conclusion

Joining of Ti_3SiC_2 ceramic via Al interlayer through transient liquid phase bonding method is studied and shown to be effective for the production of strong joints.

SEM and XRD analyses revealed that $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution rather than intermetallic compounds formed at the interface during bonding process. The mechanism of bonding is attributed to aluminum diffusing into the Ti_3SiC_2 . The formation of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution causes the joint to possess high strength, owing to the similar mechanical properties and TEC value of $\text{Ti}_3\text{Si}(\text{Al})\text{C}_2$ solid solution to Ti_3SiC_2 substrate. The maximum flexural strength with value of 263 ± 16 MPa, about 65% of that of Ti_3SiC_2 , has been obtained in the bonding condition at 1500 °C for 120 min under 5 MPa. At 1000 °C, the flexural strength of the joint was still as high as 226 ± 30 MPa, about 74% of that of Ti_3SiC_2 substrate.

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