



# Fast joining SiC ceramics with Ti<sub>3</sub>SiC<sub>2</sub> tape film by electric field-assisted sintering technology



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## ABSTRACT

Commercial SiC ceramics were joined by electric field-assisted sintering technology using a Ti<sub>3</sub>SiC<sub>2</sub> (TSC) tape film. A SiC/TSC/SiC joining sample with bend strength of 80.4 MPa was obtained at a low joining temperature of 1300 °C within a total time of 15 min. Three simple failure mechanism models were established, and the failure mechanisms of the joints joined at different temperatures were then revealed. The element diffusion and phase transition behaviour in the joining interface were investigated.

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## 1. Introduction

Silicon carbide (SiC) and SiC matrix ceramic composites have attracted considerable attention for use as high temperature structure materials, refractory materials, and abrasives [1–3]. The most important potential application of SiC matrix ceramic composites is to be next generation cladding material in pressure water reactor and flow channel insert material in thermonuclear fusion reactors because of its low atomic number, low cross-section neutron absorption and extremely low neutron activation, as well as its excellent thermomechanical properties and high melting points [4–6]. On the other hand, for the application of SiC ceramics and composites, the ability of joining SiC to itself or other materials is still a critical technology to develop because the manufacture of these materials with a complex shape or large size is quite difficult. Therefore improving the joining process and searching for proper joining materials has attracted increasing attention in recent years.

Many promising methods have already been considered for joining SiC to SiC [7], such as metallic braze-based joining [8], solid

state diffusion bonding [9], transient eutectic-phase joining [10], glass-ceramic joining [11], Si–C reaction joining [12], and MAX-phase joining [13–15]. On the other hand, most of these methods were based on hot pressing sintering technology, and normally require high temperatures and relatively long dwelling times (30–150 min). Recently, the electric current field-assisted sintering technology (FAST; sometimes referred to the spark plasma sintering, SPS) has been employed successfully to join Zr-alloy cladding tubes [16], ZrB<sub>2</sub>–SiC composites [17], MAX phase [18], and SiC and C/SiC composites within a 3–10 min holding time [19]. S. Grasso et al. [20] successfully joined β-SiC with or without Ti foil by SPS. The bend strength reached 126 ± 16 MPa for the sample joined with Ti foil at 1700 °C under a pressure of 60 MPa. They suggested pressure and surface polishing played a crucial role. During FAST, joule heating is generated by the higher electrical resistance at the contact interface within tens of milliseconds when an electrical current passes through. Both the rapid heating and short processing time allow a highly controllable reaction of the interlayer formed between the joined materials. At the same time, the electric field can accelerate self-diffusion, and promote the migration of ions through the joining interface [21]. FAST is not only a promising joining technique for ultra-high temperature ceramics, but also has been making nuclear fuel [22–24].

Moreover, a proper joining layer is another critical issue in the case of SiC applications in nuclear reactors. Ti<sub>3</sub>SiC<sub>2</sub> (TSC) has been considered a promising candidate structural material in nuclear

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and other high-temperature applications, because of its excellent mechanical properties, high melting pointing, good oxidation resistance, moderate irradiation tolerance, and corrosion resistance properties [25–27]. H. Dong et al. [28] used TSC powders to successfully join two pieces of SiC ceramic and C<sub>f</sub>/SiC composites by a hot-pressing method. The joining strength reached a maximum value of 110.4 MPa for SiC joints at a joining temperature of 1600 °C. To the best of the authors' knowledge, however, there has been little research done on the joining of SiC ceramics with a Ti<sub>3</sub>SiC<sub>2</sub> tape film using electric field-assisted sintering technology.

In the present work, commercial SiC ceramic was rapid joined by FAST using a Ti<sub>3</sub>SiC<sub>2</sub> tape film at low temperature. The failure mechanism, element diffusion and phase transition behavior in the joining interface were investigated.

## 2. Experimental procedure

The starting material was commercial pressure-less sintered 6H-SiC (98.5% purity, and density of 3.15 g/cm<sup>3</sup>). The commercial TSC powders (300 mesh), with a purity of 98.5 wt% according to the provider (Beijing Jinhezhi Materials Co., Ltd., Beijing, China) were used to prepare the tape film (thickness, 30 μm), and the TSC tape film was then applied as the joining layer. The SiC ceramics were machined into φ 20 mm × 20 mm cylinders. The surfaces to be joined were sanded and polished to 0.1 μm diamond slurry. Before joining, all surfaces were cleaned ultrasonically with ethanol. The SiC ceramic cylindrical bar, TSC tape film and the other SiC ceramic cylindrical bars were loaded into a graphite die, as shown in (Fig. 1), and then placed into a FAST furnace (FCT, HP D25, FCT Systeme GmbH, Germany). All samples were heated with the assistance of a pulsed electric current field at a heating rate of 100 °C/min. The joining temperature was controlled by the pyrometer placed on the top, which tested the inner bottom surface of the top graphite punch; the graphite punch was designed as a tubular type (Fig. 1). To obtain the difference phases of the joining interface and difference failure models, four temperatures (1300 °C, 1400 °C, 1500 °C, and 1600 °C) were used for joining. A contact pressure of 50 MPa and a dwelling time of 5 min were maintained for all samples, as detailed in Table 1.

After joining, all samples were cut into 3–5 rectangle bars (3 × 4 × 40 mm<sup>3</sup>, Fig. 1), and the joints strengths were then determined by a four-point bend method using a laboratory made assembly, as shown in Fig. 1. The rectangular bars were polished to

0.1 μm by diamond polishing slurry. The polished surface was then used to analyze the microstructure and the phase transition between the matrix and joining layer by scanning electron microscopy (SEM, Hitachi S-4800, Japan). The edges on the pressure load side were chamfered. All bend strength tests were carried out using a universal electro mechanical testing system (CMT5105, MTS, USA) equipped with a precision load cell calibrated to 1/3000 KN force was used to apply the load. The load speed for all the measurements was 0.5 mm/min (Fig. 1). The bend strength was calculated by using the standard formula of four-point bend:

$$\sigma = \frac{3P(L_2 - L_1)}{4ab^2} \quad (2-1)$$

where P is the applied load in N, and a and b are width and thickness of the specimen in mm, respectively; L<sub>1</sub> (15 mm) and L<sub>2</sub> (30 mm) are the inner and outer span, respectively. Three samples at each joining temperature were tested, and their mean and maximum deviations were calculated.

## 3. Results and discussion

Table 1 lists the joining conditions and mechanical properties of the SiC joined with Ti<sub>3</sub>SiC<sub>2</sub> tape films by FAST. According to S. Grasso et al. [20], pressure and surface polishing play a crucial role in SiC joining by SPS. Therefore, in the present work, all matrix surfaces of SiC were polished to 0.1 μm diamond slurry, and the pressure was maintained at 50 MPa. Fig. 2 presents backscattered electron (BSE) images of SiC joined at different temperatures by FAST. Regardless of whether the joining temperature is equal to or higher than 1300 °C, all joining layers were high density. It is more important to note that the interfaces were continuous. Although the thermal expansion coefficients (TEC) of SiC (−4.4 × 10<sup>−6</sup> K<sup>−1</sup>) [6] is just half of Ti<sub>3</sub>SiC<sub>2</sub> (−9.1 × 10<sup>−6</sup> K<sup>−1</sup>) [29], there were no distinguishing transverse cracks or micro cracks in the joining interface. This was attributed mainly to the unique layered structure and high Young's modulus (322 GPa) of the Ti<sub>3</sub>SiC<sub>2</sub> ceramic [29]. But for the samples joined at temperatures higher than 1300 °C, some cracks were observed in the inner areas of the joining layer. This might have occurred by the decomposition of TSC at higher temperatures and the stress concentration during the cooling process. In addition, because the TSC tape film is soft, and this ternary ceramic has reasonable toughness, the TSC ceramics can infiltrate

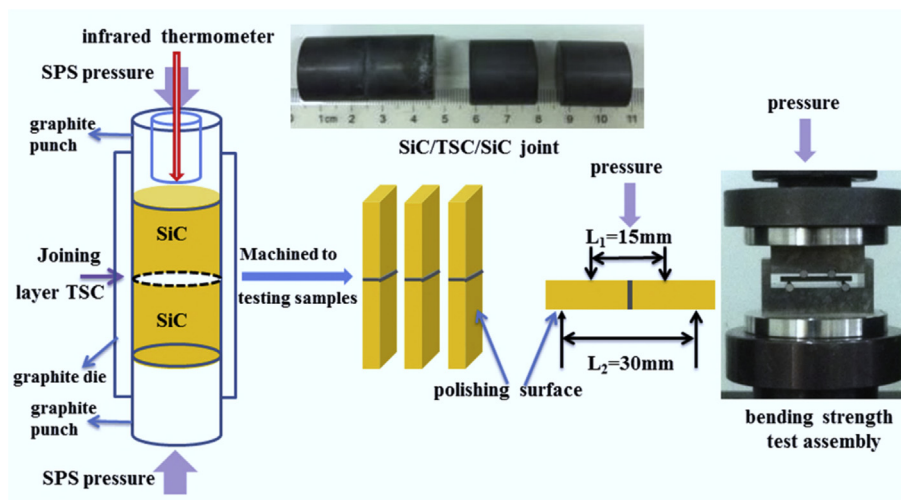
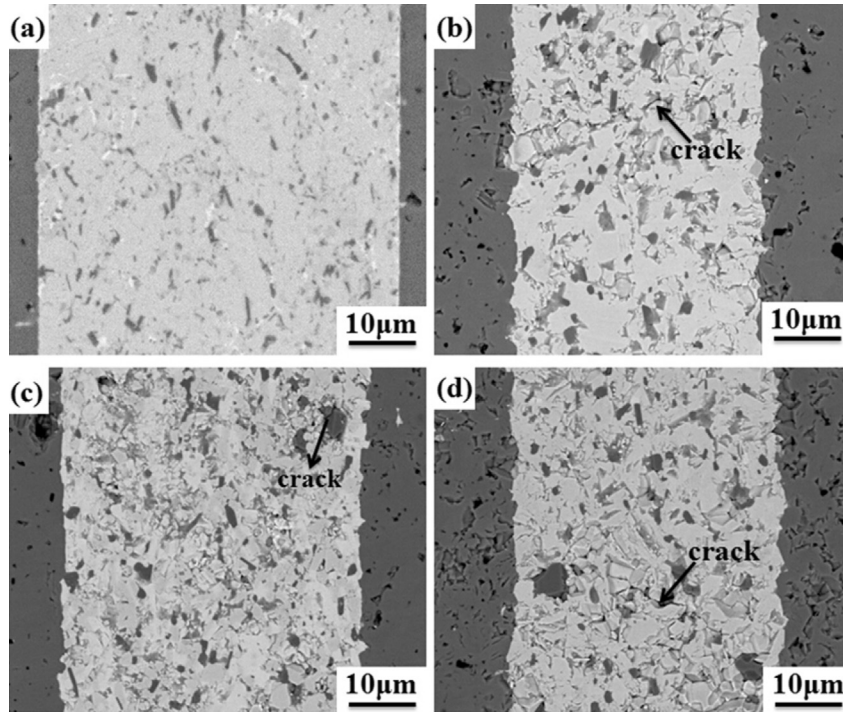


Fig. 1. Scheme of the FAST joining setup and bend strength samples preparation.

**Table 1**  
Joining parameters and mechanical properties of the SiC joined with the  $\text{Ti}_3\text{SiC}_2$  tape films by FAST.

No.	Temp. ( $^{\circ}\text{C}$ )	Joining layer thickness ( $\mu\text{m}$ )	Pressure (MPa)	$\sigma$ (MPa)	Failure location <sup>a</sup>
1	1300	60	50	80.4	M
2	1400	60	50	88.5	J + I + M
3	1500	60	50	99.1	I + M
4	1600	60	50	79.3	I

<sup>a</sup> J/I/M means failure occurred at the joining layer/interface/matrix.

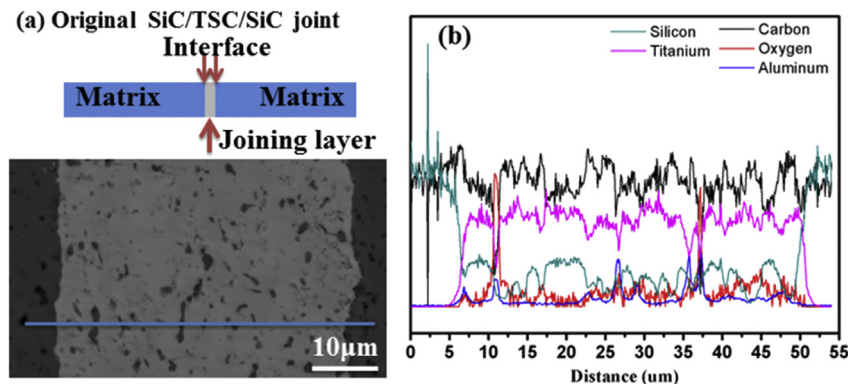


**Fig. 2.** Backscattered electron (BSE) images of SiC joined at various temperatures: (a) 1300  $^{\circ}\text{C}$ ; (b) 1400  $^{\circ}\text{C}$ ; (c) 1500  $^{\circ}\text{C}$ ; (d) 1600  $^{\circ}\text{C}$ .

into the surface pores and micro cracks of the SiC ceramics during FAST joining via capillary action. Therefore, the infiltrated area acts like a “nail” that is embedded into the matrix of the SiC ceramics (Fig. 2b, c, and d).

On the other hand, for a convenient explanation, Fig. 3a shows the typical structure of SiC/TSC/SiC joining sample. The original SiC/TSC/SiC joint can be named with three parts (Fig. 3a). The bulk of the SiC ceramic is the matrix (M); the filler of the TSC tape film is

the joining layer (J); the region between M and J is the interface (I). From the element distributions of the SiC joint joined at 1500  $^{\circ}\text{C}$  (Fig. 3b), at the interface nearby, there are some silicon enrichment areas (approximately 1–2  $\mu\text{m}$ ). Therefore, in the FAST joining process, when the temperature is higher than 1300  $^{\circ}\text{C}$ , the free silicon in the SiC matrix migrates to the interface. The enrichment of Si will improve the interface reaction between the joining layer and matrix. Therefore, the surface of the matrix appears to be etched by



**Fig. 3.** Microstructure and element distributions of SiC joint joined at 1500  $^{\circ}\text{C}$ : (a) BSE image and joint structure model; (b) element line-scanning result along the blue line in (a). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

mass diffusion or interface reactions at higher temperatures, high pressures and with the assistance of a large current density. Moreover, such nail-like morphology would benefit mechanical occlusions between the interlayer and matrix SiC ceramics, and improve the fracture toughness and shear strength along the bonding surface. In fact, J. Wang et al. [30] observed similar phenomena in the process of carbon/carbon composite joints using a Ti–Si–SiC–C filler, and suggested that the “nail effect” is favourable for improving the mechanical properties of the joints. On the other hand, the free Si is not beneficial to the application of cladding material in pressure water reactor, so in the future work we should improve the purity of SiC.

Fig. 4a shows the four points bend strength of SiC joined by FAST with a 2 layers TSC tape film (~60 μm) at different temperatures. Note that the bend strength of each of the three parallel samples has small variability. This means the experiment had good repeatability. The bend strength increased with the increasing joining temperature, and reached a maximum value of 99.1 MPa at a joining temperature of 1500 °C. Then the bend strength decreased to 79.3 MPa when the joining temperature was increased to 1600 °C. Table 1 provides the failure location details at different joining temperatures.

After the bend strength testing, the phase structure of the fracture surface of the SiC joints at different joining temperatures was examined by XRD. Fig. 4b shows the XRD patterns of the fracture surfaces of the SiC joints joined at various temperatures. The XRD peak at 21° was assigned to the clay used for fixing the XRD samples of rectangle testing bars. At joining temperature of 1300 °C, only α-SiC (6H) was identified because the failure location is in the matrix (Fig. 4b). That means the bend strength of this sample reflect the bend strength value of matrix instead of joining layer. At a joining temperature of 1400 °C (Fig. 4b), the main phases of the fracture surfaces were SiC, Ti<sub>3</sub>SiC<sub>2</sub>, TiC, and TiSi<sub>2</sub>. This suggests that the joining layer begins to decompose, which corresponds to the fracture location of “J + I + M”. Furthermore, compared to the sample joined at 1400 °C, the contents of the Ti<sub>3</sub>SiC<sub>2</sub> decreased with increasing joining temperature (Fig. 4b).

The difference in bend strength corresponded to the difference in failure mechanism. To examine the failure mechanism further, three typical simple failure models were established, as shown in Fig. 5a–c. First, for the sample joined at 1300 °C, the failure position occurred at the matrix (Fig. 5a). This means that both the strength of the joining layer and the interfaces are higher than the matrix. In the other words, in this failure mechanism, the SiC/TSC/SiC joining sample bend strength is decided by the strength of the matrix. The second model is that the failure position occurred in the joining layer (Fig. 5b). Therefore, in this mechanism, the mechanical

properties of the joining layer determined the joint bend strength. The last mechanism is the interface failure model (Fig. 5c); the mechanical properties of the joint are determined by the strength of the interface. As shown in Fig. 5c, when the joining temperature was 1600 °C, failure occurred at the interface; hence, if the temperature is too high, then it is not beneficial for obtaining a higher strength joint.

Normally, the bend strength or shear strength of the matrix is higher than that of the joining layer; the interface is the weakest link of these three models. In the present work, however, the matrix is the commercial pressure-less sintering 6H–SiC (98.5% purity, and density of 3.15 g/cm<sup>3</sup>). Some pores were observed in the matrix (Fig. 5d), and perhaps the applied pressure (50 MPa) at 1300 °C also damages the matrix, so the bend strength of the matrix SiC was lower than the typical value (200–500 MPa) [31]. SEM images of the fracture surface (Fig. 5d) showed that the fracture surface appears smooth. In other words, under this joining condition, higher bend strength of the joining layer and interface can be obtained. Moreover, when the strength of the matrix, interface and joining layer are at an equal level, the failure mechanism generally exhibits a complex model, such as the samples joined at 1400 °C (J + I + M, Fig. 5b) and 1500 °C (I + M, not shown in here). In the complex failure mechanism of J + I + M, the crack propagation path is along the interface and within the joining layer, and the SEM image of the fracture surface of this sample (Fig. 5e) revealed a honeycombed structure, which is a typical crack deflection mechanism. Therefore, more energy will be lost or absorbed in crack propagation, which leads directly to a higher bend strength than the simple failure mechanism.

Two factors exist that can lead to a high joining strength. One is the good plasticity of the filler, which can relax the thermal stress arising from the different TECs between the filler and the base material. Another is the chemical bonding resulting from a chemical reaction at the interface between the filler and the base material. H. Dong et al. [28] examined the interface reaction between SiC and TSC in the hot-press joining process at different joining temperatures. They indicated that the chemical reactions took place during the joining process at the temperature range, 1400–1600 °C. In addition, they suggested that the interface reaction is beneficial for achieving a higher joining strength. Barsoum [32] and Emmerlich [33] also reported that the TSC would decompose into TiCx following Si out-diffusion and evaporation in the graphite-rich environment at temperatures above 1350 °C. In the present work, the joining temperature, graphite die, electrical current and joining pressure may be the main influencing factors for the interface chemical reactions during the FAST joining process. Compared to the hot-pressing joining, the existence of a

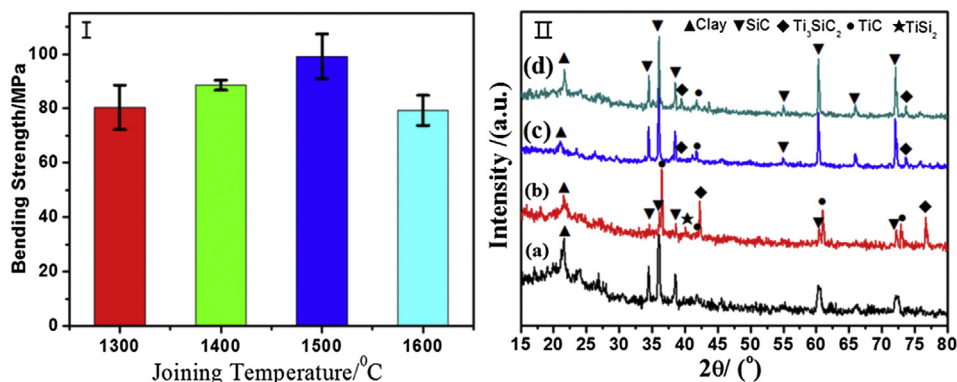
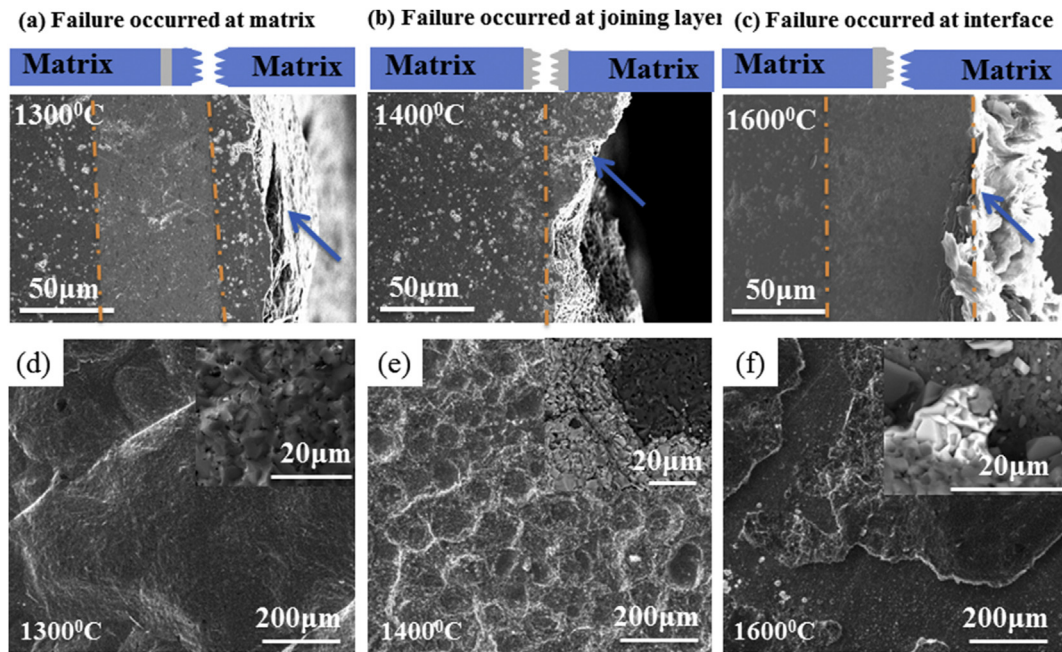


Fig. 4. (a) Bend strength of the SiC joined with TSC at different temperatures, three samples at each joining temperature were tested, and their mean and maximum deviations were calculated; (b) XRD patterns of the fracture surfaces of the SiC joints made at various temperatures.

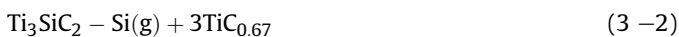


**Fig. 5.** Failure mechanism models occurred at specific areas (a) matrix, 1300 °C; (b) joining layer, 1400 °C; (c) interface, 1600 °C; Fracture surface SEM images of the samples joined at different temperatures: (d) low and high magnification (insert image) of 1300 °C; (e) low and high magnification (insert image) of 1400 °C; (f) low and high magnification (insert image) of 1600 °C; The dotted yellow lines represent the interface, the blue arrow represents the fracture position. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

strong electric current (0.6 KA) can enhance the diffusivity via an electro-migration phenomena at high temperature [21]; thus a Si-enriched environment can form easily at the interface of TSC and SiC, and promoted the migration of ions. The possible reaction is as follows [34].



The organic binding agent introduced in the preparation of the TSC tape film and graphite die provided a graphite-rich environment that also results in the decomposition of TSC, as follows [35]:



EDS (Fig. 3) and XRD (Fig. 4) also showed that there are some  $\text{TiC}_x$  and  $\text{Ti}_x\text{Si}_y$  in the interlayer. In addition, the chemical reactions took place during the FAST joining process. With increasing joining temperature, the reaction would be stronger and the interface product would increase. On the other hand, the joining layer density is also higher with the temperature increasing which can offset the brittle phase negative effects on the bend strength. Previous work [36,37] reported that the TSC/TiC and TSC/SiC composites have better mechanical properties than monolithic TSC. Therefore, an appropriate higher joining temperature is beneficial for obtaining a higher bend strength, which corresponds well with the bend strength testing. However, if the joining temperature is too high (1600 °C), the TSC will be decomposed to some  $\text{Ti}_x\text{Si}_y$  brittle phases in the joining interface. Even though because of some matrixes of SiC residual in the fracture surface, the  $\text{Ti}_x\text{Si}_y$  phases can't be detected by XRD (Fig. 4b), EDS analyses (not shown in here) indicated that there are  $\text{Ti}_x\text{Si}_y$  phases in the joining interface, which can lead to failure in the interface and decrease the joining layer bend strength. So, if the joining temperature is too high, it is not beneficial to get high quality joining layer.

For the SiC jointed by hot-pressing technology, the joint joined at 1300 °C has insufficient strength, even though the joining temperature was increased to 1500 °C, and the flexural strength is

approximately 60 MPa [28]. However, in the present work, a bend strength of 80.4 MPa can be obtained at 1300 °C with a dwelling time of just 5 min. Therefore, the existence of an electrical current field is beneficial improving diffusion and reaction between TSC and SiC. Akira Suzuki et al. [38] reported that the carrier concentrations of SiC increase rather rapidly with temperature, so the electric conductivity increases with temperature. Moreover, the electric conductivity of TSC is  $9.6 \times 10^6 \Omega^{-1} \text{m}^{-1}$ . Therefore, at high temperatures, during the FAST joining process, it is possible that the current might pass through the SiC and joining layer. Therefore, localized heating could be generated at the joining layer by joule heating. Hence, both the rapid, localized heating and a short processing time make a strong contribution to the interlayer formed between joined materials.

#### 4. Conclusion

Commercial SiC ceramics were joined successfully with a  $\text{Ti}_3\text{SiC}_2$  tape film by FAST. Compare to hot-pressing joining technology, the joint with a higher bend strength of 80.4 MPa can be obtained at a lower temperature of 1300 °C, and a shorter dwelling time of 5 min. This suggests that the existence of an electrical current field is beneficial to improving diffusion and the reaction between TSC and SiC. An appropriate higher joining temperature (1500 °C) is beneficial for obtaining a higher bend strength (99.1 MPa), because of a chemical reaction occurring at the joining interface and the partially decomposition of TSC. The failure mechanisms of the joints joined at different temperatures were revealed.

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