

## Reactions between Ti and $\text{Ti}_3\text{SiC}_2$ in temperature range of 1273–1573 K

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**Abstract:** The reactions of  $\text{Ti}_3\text{SiC}_2$  and Ti in the temperature range of 1273–1573 K under a pressure of 20 MPa were investigated. The results confirm that Ti reacts with  $\text{Ti}_3\text{SiC}_2$  above 1273 K and new phases like  $\text{TiC}_x$ ,  $\text{Ti}_5\text{Si}_3$  and  $\text{TiSi}_2$  are identified. The reactions are closely related to temperature and content of  $\text{Ti}_3\text{SiC}_2$  in Ti. During the reaction process,  $\text{Ti}_3\text{SiC}_2$  decomposes in two different modes. The first is caused by the de-intercalation of Si from it and the  $\text{TiC}_x$  is formed by the remained titanium and carbon; the second is that the carbon is separated from the  $\text{Ti}_3\text{SiC}_2$  and reacts with titanium furthermore. The diffusing of silicon is believed to be the determinant ingredient of the reaction.

**Key words:**  $\text{Ti}_3\text{SiC}_2$ ; Ti; high-temperature reaction

### 1 Introduction

Recently, the layered ternary carbide  $\text{Ti}_3\text{SiC}_2$  has attracted attention due to its unique properties such as high toughness, high fatigue-crack growth threshold and elastic modulus, plasticity at high temperature, excellent electrical and thermal conductivity, and easy machinability[1–6]. The unique combination of these properties makes  $\text{Ti}_3\text{SiC}_2$  as a promising structural material for high temperature applications. In addition, the high modulus, metallic conductivity and self-lubricated properties also make  $\text{Ti}_3\text{SiC}_2$  as a potential reinforcement for soft metals such as Al, Cu, Ti and Ni. The advantage of  $\text{Ti}_3\text{SiC}_2$  reinforced soft metals over other ceramics is that high strength will be achieved without loss of conductivity. During preparing  $\text{Ti}_3\text{SiC}_2$  reinforced metal matrix composites, reactions between the matrix and  $\text{Ti}_3\text{SiC}_2$  is one of the key factors controlling the microstructure and properties of the composites. The stability of  $\text{Ti}_3\text{SiC}_2$  in metal matrix is also important in selecting the preparation method and processing parameters. The knowledge of compounds formed and the diffusion path between the ceramic and

metal interface is necessary to control the interface structures, which essentially control the mechanical properties.

In practice, when used as a high-temperature structural material,  $\text{Ti}_3\text{SiC}_2$  often needs to be joined with structural metals to make a complex component. While as a reinforcement of soft metals,  $\text{Ti}_3\text{SiC}_2$  particulates are incorporated uniformly into metal matrix to make metal matrix composites with controlled microstructure. In both of these applications, understanding the interfacial chemical reactions and interfacial microstructure/chemistry is of vital importance in selecting processing parameters and optimizing properties.

However, very limited information of the interaction between  $\text{Ti}_3\text{SiC}_2$  and metals is available. Our previous works reported the reaction between  $\text{Ti}_3\text{SiC}_2$  and Al[7],  $\text{Ti}_3\text{SiC}_2$  and Cu[8], respectively. Recently, we are intrigued in investigating the interfacial reactions in Ti- $\text{Ti}_3\text{SiC}_2$  systems. The interests stem from the need in developing high temperature self-lubricated materials such as Ti/ $\text{Ti}_3\text{SiC}_2$  composite, as well as joining  $\text{Ti}_3\text{SiC}_2$  with Ti-based alloys for high-temperature applications. This paper reports the reaction between  $\text{Ti}_3\text{SiC}_2$  and Ti powder, which is useful in both selecting processing



conditions of Ti/Ti<sub>3</sub>SiC<sub>2</sub> composite and controlling the interfacial microstructure of Ti/Ti<sub>3</sub>SiC<sub>2</sub> joint.

## 2 Experimental

To investigate the possible reactions during processing of Ti/Ti<sub>3</sub>SiC<sub>2</sub> composites, Ti and Ti<sub>3</sub>SiC<sub>2</sub> powders were mixed and hot-pressed at 1 273–1 573 K. The average particle size of Ti powders and Ti<sub>3</sub>SiC<sub>2</sub> powders was 40 μm (from General Research Institute For Nonferrous Metals, China) and 10 μm (prepared at author's lab), respectively. The Ti<sub>3</sub>SiC<sub>2</sub> powder used in this work was fabricated by using the in-situ hot pressing solid-liquid reaction process, which was described in Ref.[9]. The as-prepared powders contained a small amount of TiC, which was described in the previous work[9].

According to the phase diagram of Ti-Si system [10], at low Si content (less than 1% Si, mole fraction) the solid solution of Ti(Si) forms; beyond the solubility there are five kinds of Ti-Si compounds, which are Ti<sub>3</sub>Si, Ti<sub>5</sub>Si<sub>3</sub>, Ti<sub>5</sub>Si<sub>4</sub>, TiSi and TiSi<sub>2</sub>. If Ti reacts with Si in Ti<sub>3</sub>SiC<sub>2</sub> when Ti<sub>3</sub>SiC<sub>2</sub> and Ti mix together at high temperatures, Ti<sub>3</sub>SiC<sub>2</sub> will not be stable, and the reaction products depend on the content of Si, i.e. the content of Ti<sub>3</sub>SiC<sub>2</sub>. Supposing that Ti reacts with Si from the decomposed Ti<sub>3</sub>SiC<sub>2</sub> and the Ti<sub>3</sub>Si, Ti<sub>5</sub>Si<sub>3</sub>, Ti<sub>5</sub>Si<sub>4</sub>, TiSi and TiSi<sub>2</sub> can be formed, respectively, their mass ratio of Ti to Ti<sub>3</sub>SiC<sub>2</sub> can be calculated as 3/4, 1.67/4, 1.25/4, 1/4 and 0.5/4, respectively. To confirm this hypothesis and

understand the high temperature reactions between Ti and Ti<sub>3</sub>SiC<sub>2</sub>, Ti/Ti<sub>3</sub>SiC<sub>2</sub> composites with above mass ratios were prepared in the temperature range of 1 273–1 573 K under a pressure of 20 MPa.

The phase compositions of the composites prepared under different conditions were identified by X-ray diffraction(XRD), wherein the data were collected by a step-scanning diffractometer (Rigaku D/max-2500PC, Japan) using Cu K<sub>α</sub> radiation. To understand the mechanism of the reaction and the interfacial microstructure, bulk couple of Ti/Ti<sub>3</sub>SiC<sub>2</sub> was hot pressed at 1 473 K with a pressure of 20 MPa for 60 min. The cross-section of the hot pressed Ti/Ti<sub>3</sub>SiC<sub>2</sub> couple was observed in an S-360 scanning electron microscope (Cambridge Instrument Ltd., UK) equipped with an energy dispersive spectroscopy(EDS) system. X-ray dot maps and concentration profiles were determined to identify the interfacial chemistry.

## 3 Results

To study the possible reactions between Ti<sub>3</sub>SiC<sub>2</sub> and Ti, we should know the thermal stability of Ti<sub>3</sub>SiC<sub>2</sub>. According to previous works, the dissociation temperature of Ti<sub>3</sub>SiC<sub>2</sub> was reported from 1 573 K to 2 073 K depending on the purity, atmosphere and sample form (bulk or powders)[11–14]. To determine the dissociation temperature of Ti<sub>3</sub>SiC<sub>2</sub> powders prepared by the fluctuation method, the as-prepared Ti<sub>3</sub>SiC<sub>2</sub> powders were annealed at different temperatures.

Fig.1 shows the X-ray diffraction patterns of the as-

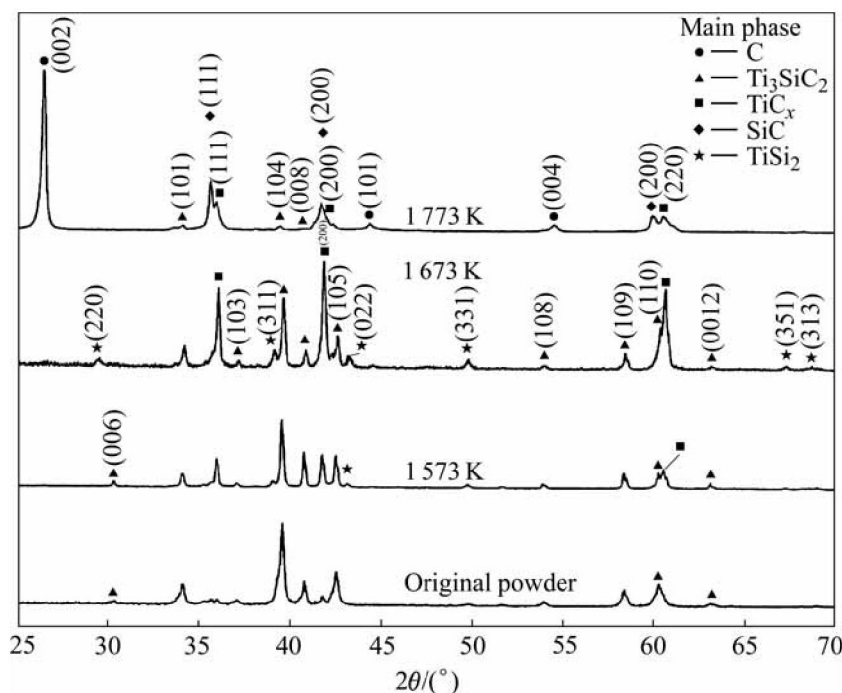


Fig.1 XRD patterns of original Ti<sub>3</sub>SiC<sub>2</sub> powders and those annealed at 1 573, 1 673 and 1 773 K for 30 min, respectively

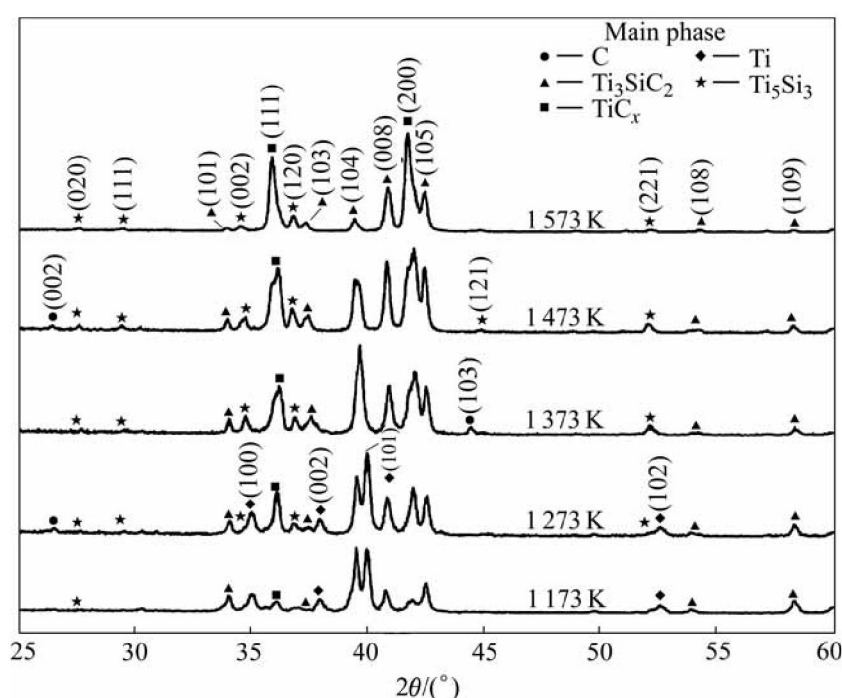


prepared  $\text{Ti}_3\text{SiC}_2$  powders and those annealed at 1 573, 1 673 and 1 773 K for 30 min, respectively. In the as-prepared  $\text{Ti}_3\text{SiC}_2$  powders there is a small amount of TiC and SiC, which is the main impurity phase in  $\text{Ti}_3\text{SiC}_2$ [9]. After annealing at 1 573 K, the intensity of reflections from  $\text{TiC}_x$  (Here C replaced by  $\text{C}_x$  is because that the atomic ratio of Ti and C is 3 to 2 in  $\text{Ti}_3\text{SiC}_2$ , the subscript  $x$  in the new-formed  $\text{TiC}_x$  is less than 1) increase and a new phase  $\text{TiSi}_2$  emerges. The increase in the intensity of  $\text{TiC}_x$  and the appearance of  $\text{TiSi}_2$  reveal that  $\text{Ti}_3\text{SiC}_2$  powder is decomposed obviously over the temperature of 1 573 K. At 1 673 K, the intensities of diffraction peaks of  $\text{TiC}_x$  are almost as high as that of  $\text{Ti}_3\text{SiC}_2$ , indicating that more  $\text{Ti}_3\text{SiC}_2$  powders are decomposed. At 1 773 K, the reflections from  $\text{Ti}_3\text{SiC}_2$  almost disappear but SiC and graphite emerge as decomposition products. The above results are similar to result of RACAULT et al[13] and different from reports of BARSOUM et al[11] and RADHAKRISHNAN et al[12] on the thermal stable of  $\text{Ti}_3\text{SiC}_2$ . The observed low decomposing temperature is attributed to the fact that the  $\text{Ti}_3\text{SiC}_2$  powders studied in our present work have large surface area and higher activity than bulk material used in previous works[11,12]. Owing to the decomposition of  $\text{Ti}_3\text{SiC}_2$  powder above 1 573 K, the temperature range for the reaction in Ti- $\text{Ti}_3\text{SiC}_2$  system is chosen below 1 573 K.

Fig.2 shows the X-ray diffraction patterns for samples of Ti/ $\text{Ti}_3\text{SiC}_2$  composites with a mass ratio of

3/4, which were hot pressed at 1 173, 1 273, 1 373, 1 473 and 1 573 K under a pressure of 20 MPa for 30 min, respectively. For the sample hot pressed at 1 173 K, there are very weak diffraction peaks from  $\text{TiC}_x$  and the main phases are Ti and  $\text{Ti}_3\text{SiC}_2$ , which implies that the reaction between Ti and  $\text{Ti}_3\text{SiC}_2$  almost can be neglected at the temperature of 1 173 K. For the sample hot pressed at 1 273 K, the intensities of reflections from  $\text{TiC}_x$  are higher than those at 1 173 K. In addition, very weak diffraction peaks from element carbon and titanium-silicon compound  $\text{Ti}_5\text{Si}_3$  can be identified, which indicates that the starting reaction should be in the range of 1 173–1 273 K. For the sample prepared at 1 373 K, the diffraction peaks of  $\text{TiC}_x$  become even higher and the reflections from Ti and C disappear, indicating that the amount of  $\text{TiC}_x$  increases with the reaction temperature increasing. The reflections from  $\text{Ti}_3\text{SiC}_2$  are weaker than those from TiC at 1 473 K and almost disappear at 1 573 K. All the above results demonstrate that Ti reacts with  $\text{Ti}_3\text{SiC}_2$  at temperatures above 1 273 K and the reaction extent is temperature dependent.

To further investigate the effect of  $\text{Ti}_3\text{SiC}_2$  content on the reaction products, Ti/ $\text{Ti}_3\text{SiC}_2$  powders with a mass ratio of 1.67/4 were hot pressed under a pressure of 20 MPa at 1 273, 1 373, 1 473 and 1 573 K for 30 min, respectively, and the X-ray diffraction patterns of these composites are shown in Fig.3. The difference between Figs.3 and 2 is that new reflection from  $\text{TiSi}_2$  is detected in Fig.3 besides  $\text{TiC}_x$  and  $\text{Ti}_5\text{Si}_3$ . Meanwhile, the reflec-



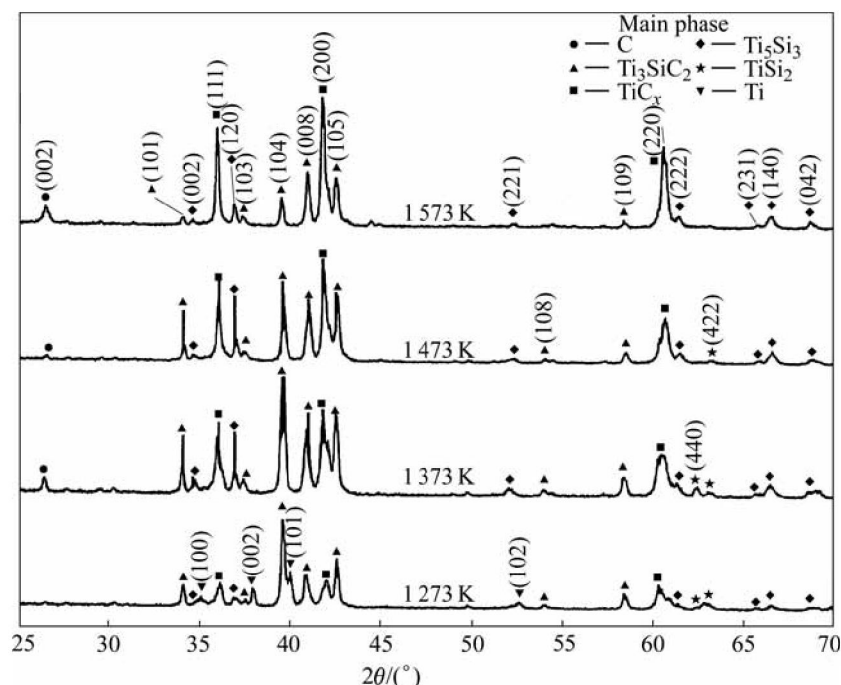
**Fig.2** XRD patterns for samples of Ti/ $\text{Ti}_3\text{SiC}_2$  composites with mass ratio of 3/4 hot pressed at 1 173, 1 273, 1 373, 1 473 and 1 573 K for 30 min, respectively



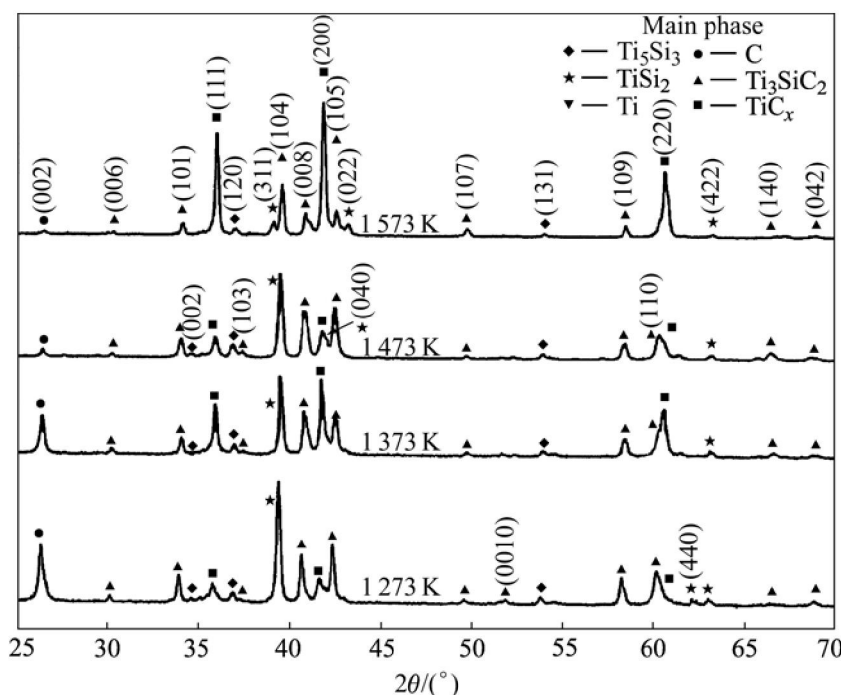
tions from graphite emerge again, which is similar to the situation in Fig.2. The emerging of the graphite (002) peak implies that  $\text{Ti}_3\text{SiC}_2$  is decomposed at 1 273 K and the element carbon is one of the products of the decomposition.

Fig.4 shows that the X-ray diffraction patterns for samples of  $\text{Ti}/\text{Ti}_3\text{SiC}_2$  composites with a mass ratio of

0.5/4, which were hot pressed under a pressure of 20 MPa at 1 273, 1 373, 1 473 and 1 573 K for 30 min, respectively. No new phase can be detected besides  $\text{TiSi}_2$  and  $\text{Ti}_5\text{Si}_3$ . It can be found that the diffraction peak (200) from  $\text{TiC}_x$  increases with the increasing temperature. In another hand, the diffraction peaks of graphite at 1 273 K and 1 373 K are higher than the same peak detected in



**Fig.3** XRD patterns for samples of  $\text{Ti}/\text{Ti}_3\text{SiC}_2$  composites with mass ratio of 1.67/4 hot pressed at 1 273, 1 373, 1 473 and 1 573 K for 30 min, respectively



**Fig.4** XRD patterns for samples of  $\text{Ti}/\text{Ti}_3\text{SiC}_2$  composites with mass ratio of 0.5/4 hot pressed at 1 273, 1 373, 1 473 and 1 573 K for 30 min, respectively



Fig.3. Combined with Fig.3, it is clear that the intensity of graphite's peak has a close relationship not only with the content of  $\text{Ti}_3\text{SiC}_2$  but also with the reaction temperature. Briefly, when the content of Ti is relatively low the diffraction peak of graphite is relatively high, meanwhile, for a certain mass ratio of Ti to  $\text{Ti}_3\text{SiC}_2$ , the diffraction peak of graphite is high at a low temperature.

As mentioned above, there are five compounds in Ti-Si system. However, the XRD results shown in Figs.2-4 indicate that only  $\text{Ti}_5\text{Si}_3$  and  $\text{TiSi}_2$  can be detected during the reaction process. The important question is whether other three compounds would be detected if the mass ratio is changed. In fact, there are marked disagreements concerning  $\text{Ti}_3\text{Si}$  and  $\text{Ti}_5\text{Si}_4$ . HANSEN et al[15] has not shown either of these compounds in their paper about the Ti-Si phase diagram. According to Ref.[10], this judgement is based on: 1) the probable purity of the alloys; 2) the fact that the phases of composition 25% and 45% (mole fraction) were observed by different authors, and 3) the present thermodynamic analysis of the system[10]. Based on these facts, it is acceptable that no  $\text{Ti}_3\text{Si}$  is detected in the product of reaction. On the other hand,  $\text{Ti}_5\text{Si}_4$  and  $\text{TiSi}$  will be formed above temperature of 1 843 and 1 753 K [10], respectively. It is impossible for these two compounds to be formed under 1 573 K. To conform this deduction, a sample of Ti/ $\text{Ti}_3\text{SiC}_2$  composites with a mass ratio of 1/4 (with the mass ratio the compound of

$\text{TiSi}$  should be formed) is hot pressed at 1 573 K for 30 min and the XRD result is shown in Fig.5. It is clear that all the diffraction peaks of Ti-Si compound are from  $\text{Ti}_5\text{Si}_3$  and  $\text{TiSi}_2$  and no  $\text{TiSi}$  can be detected. This result implies that only  $\text{Ti}_5\text{Si}_3$  and  $\text{TiSi}_2$  can be formed under the condition of our investigation.

Based on the experimental results described above, the reaction between  $\text{Ti}_3\text{SiC}_2$  and Ti can be described as the following equation:



The equation represents a typical reaction and maybe only one kind of Ti-Si compound can be detected for certain content as shown in Fig.2. The subscript  $x$  in  $\text{TiC}_x$  represents the atomic ratio of titanium and carbon.

## 4 Discussion

In the above section, we have known that during the high temperature processing of Ti- $\text{Ti}_3\text{SiC}_2$  composite, Ti reacts with  $\text{Ti}_3\text{SiC}_2$  and the reaction depends on both  $\text{Ti}_3\text{SiC}_2$  content and the temperature. To reveal the reaction process a bulk  $\text{Ti}_3\text{SiC}_2$  was polished and buried in powder of Ti, then hot pressed at 1 573 K with a pressure of 20 MPa for 60 min. The back-scattered SEM image and the chemical composition profile across the interface of the bonded Ti/ $\text{Ti}_3\text{SiC}_2$  are shown in Fig.6. The composition profiles(Fig.6(b)) detected along the

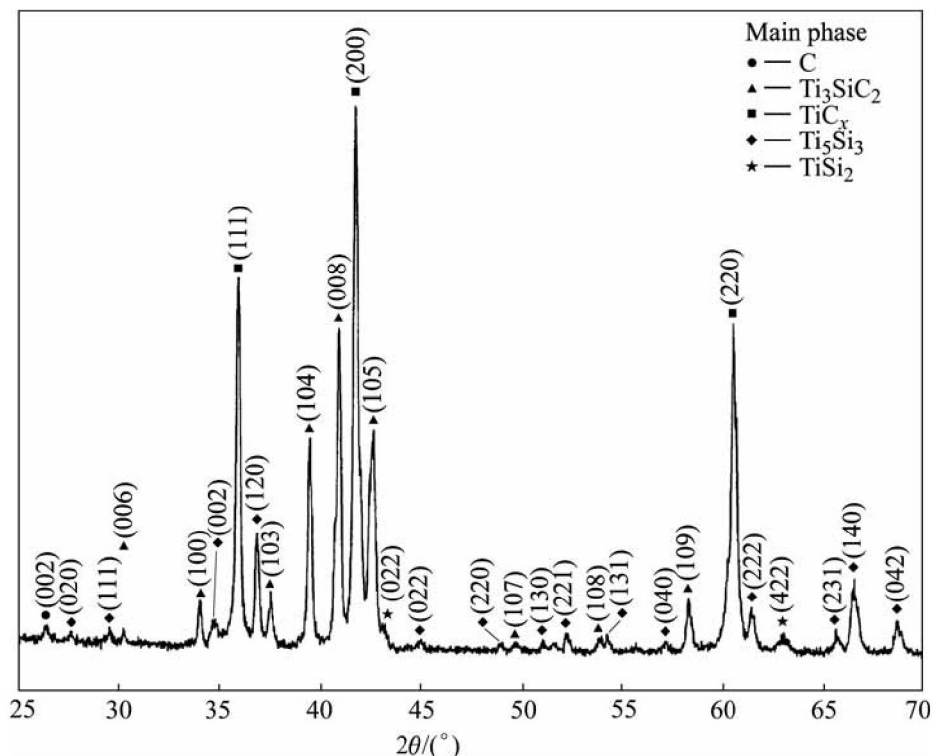
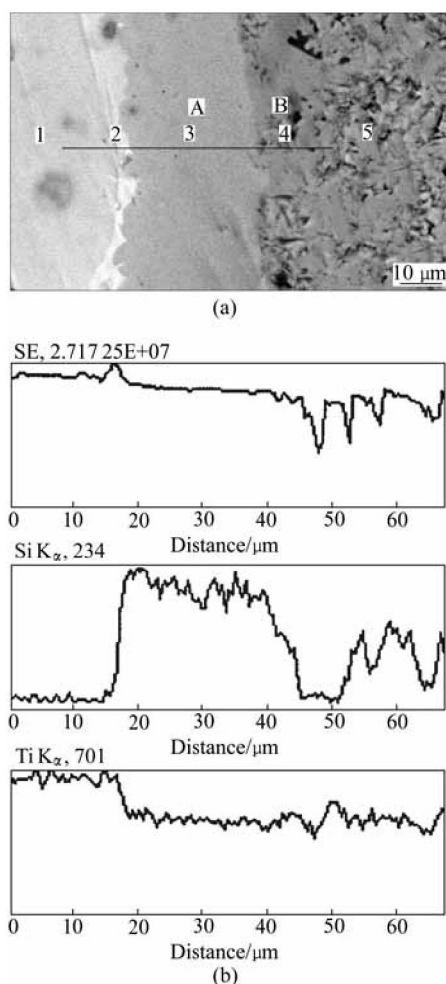


Fig.5 XRD patterns for samples of Ti/ $\text{Ti}_3\text{SiC}_2$  composites with mass ratio of 1/4 hot pressed at 1 573 K for 30 min





**Fig.6** Back-scattered SEM image and chemical composition profile across interface of bulk  $\text{Ti}_3\text{SiC}_2$  buried in powder of pure Ti hot pressed at 1 573 K with pressure of 20 MPa for 60 min

line as shown in Fig.6(a), indicate the diffusion of Si occurred on the boundary at high temperature. However, the diffusion of other elements can be neglected. On the other hand, Ti reacts with the Si in  $\text{Ti}_3\text{SiC}_2$  to form Ti-Si compound (dim part of interface marked 3 in Fig.6(a)), which results in the formation of  $\text{TiC}_x$  (dark part marked 4 in Fig.6(a)) near the interface. The EDS semi-quantitative analysis of Ti and Si at the five different points in Fig.6(a) is listed in Table 1, which implies that at layer A in Fig.6(a)  $\text{Ti}_5\text{Si}_3$  forms and at layer B in Fig.6(a)  $\text{TiC}_x$  forms. Fig.6(b) also reveals that the composition of Si declines with a steep gradient on the boundary of Ti and the layer A, which implies that the diffusion of Si from  $\text{Ti}_3\text{SiC}_2$  to Ti is limited by the forming of Ti-Si compound.

Previous study on the structure characteristics of  $\text{Ti}_3\text{SiC}_2$  revealed that the structure of  $\text{Ti}_3\text{SiC}_2$  can be described as a layer of Si intercalated into the  $\{111\}$  twin

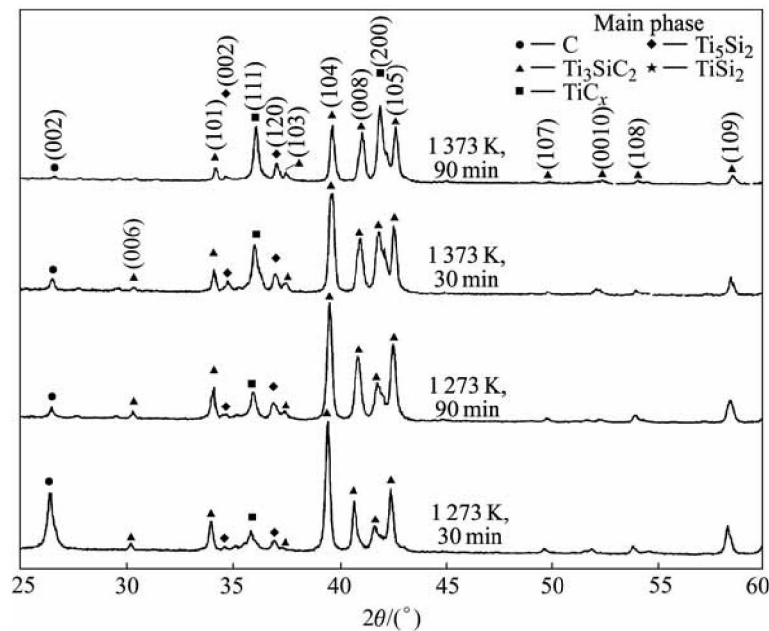
**Table 1** Mole fraction of elements in different regions in Fig.6(a) (%)

Region	Ti	Si
1	99	1
2	85	15
3	67	33
4	93	7
5	78	22

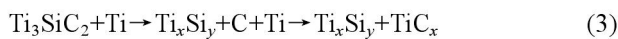
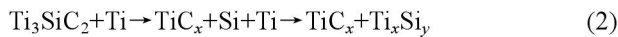
boundary of  $\text{TiC}$ , and the chemical bonding between Si and Ti is relatively weak compared to the strong Ti—C bonding[16–18]. And the decomposition of this layered ternary compound is usually caused by the de-intercalation of Si from it[16–18]. In other words, if Si forms solid solution or a stable compound with other elements,  $\text{Ti}_3\text{SiC}_2$  will decompose. In this work, the Si-Ti compound such as  $\text{Ti}_5\text{Si}_3$  and  $\text{TiSi}_2$  forms during the reaction process, which gives a direct evidence for the de-intercalation of Si from  $\text{Ti}_3\text{SiC}_2$ . In addition, the presence of elemental carbon in the Ti- $\text{Ti}_3\text{SiC}_2$  reaction product implies that the decomposition mode of  $\text{Ti}_3\text{SiC}_2$  in Ti may not solely follow the one described above. The experimental results shown in Figs.2–4 reveal that  $\text{Ti}_3\text{SiC}_2$  can be decomposed with other ways besides the de-intercalation of Si from  $\text{Ti}_3\text{SiC}_2$  and the formation of  $\text{TiC}_x$ .

Analyzing the graphite's peak shown in Figs.3 and 4 we would like to hypothesize that the graphite from the decomposed  $\text{Ti}_3\text{SiC}_2$  comes into existence at 1 273 K and then reacts with titanium to form  $\text{TiC}_x$  at higher temperature. If the titanium is insufficient or the temperature is low, the reaction will not be sufficient and the graphite will be left. To confirm this hypothesis the reaction time is extended to 90 min for sample Ti/ $\text{Ti}_3\text{SiC}_2$  composites with a ratio of 0.5/4 at temperatures of 1 273 and 1 373 K, respectively. The XRD results of the samples prepared in 90 and 30 min are compared and shown in Fig.7. It is clear that the graphite's diffraction peaks disappear for the sample with a longer reaction time. This result implies that the element carbon is an interim phase during the reaction process; the carbon from the decomposed  $\text{Ti}_3\text{SiC}_2$  will react with Ti furthermore if the reaction time is long enough. Combining with the above result,  $\text{Ti}_3\text{SiC}_2$  may be decomposed in two different modes. The first mode is caused by the de-intercalation of Si from it and the  $\text{TiC}_x$  is formed by the remained titanium and carbon; the second mode is that the carbon is separated from the  $\text{Ti}_3\text{SiC}_2$  and reacts with titanium furthermore. Thus, the reaction route for the high temperature reaction between  $\text{Ti}_3\text{SiC}_2$  and Ti can be described by the following procedures.





**Fig.7** XRD patterns for samples of Ti/Ti<sub>3</sub>SiC<sub>2</sub> composites with mass ratio of 0.5/4 hot pressed at 1 273 and 1 373 K for 30 and 90 min, respectively



The molecular formula  $\text{Ti}_x\text{Si}_y$  represents all the two sorts of titanium silicon compound detected in the reaction process. The subscript  $x$  in  $\text{TiC}_x$  represents the atomic ratio of titanium and carbon.

In Fig.2, as calculated above, the mass ratio of Ti and  $\text{Ti}_3\text{SiC}_2$  corresponds to the compound  $\text{Ti}_3\text{Si}$ . According to this mass ratio, the element Ti should be left after the reaction is finished, because the detected compound  $\text{Ti}_5\text{Si}_3$  has a relatively low Ti content compared to  $\text{Ti}_3\text{Si}$ . However, the experiment result indicates that no element Ti remains. This result implies that the value of subscript  $x$  in  $\text{TiC}_x$  may be less than 0.67, because more Ti will be consumed for a small value of  $x$ . On the other hand, the XRD result in Fig.4 shows that besides  $\text{TiSi}_2$ , the compound should appear for this content,  $\text{Ti}_5\text{Si}_3$  is detected as well. This result implies that the value of subscript  $x$  in  $\text{TiC}_x$  may be larger than 0.67. All these experiment results suggest that the subscript  $x$  in  $\text{TiC}_x$  will change in a large range. From Ti-C phase diagram[19] we know that the range of  $x$  will be changed from 0.5 to 1.

Considering the crystal structure of  $\text{TiC}_x$ , the change of  $x$  will bring the change of lattice spacing and the remove of the diffraction peak. As described above, the results in Figs.2–4 show that the diffraction peaks of  $\text{TiC}_x$  is low at 1 273 K and becomes higher and wider at

1 373 K and 1 473 K. This result implies the composite of  $\text{TiC}_x$  is uneven in the different region. The peak becomes very sharp at 1 573 K, which implies a higher temperature is in favor of the harmoniousness in composition.

Our work demonstrates that Ti reacts with  $\text{Ti}_3\text{SiC}_2$  at high temperatures and the reaction depends on the temperature and  $\text{Ti}_3\text{SiC}_2$  content. The result of this work is instructive for selecting the preparation method and for processing parameters of Ti-Ti<sub>3</sub>SiC<sub>2</sub> composite. Due to the reaction between Ti matrix and the  $\text{Ti}_3\text{SiC}_2$  reinforcement at temperatures above 1 273 K, it is desirable to make  $\text{Ti}_3\text{SiC}_2$  reinforced Ti below 1 273 K.

## 5 Conclusions

The chemical reactions between Ti and  $\text{Ti}_3\text{SiC}_2$  have been investigated during the preparation of Ti-Ti<sub>3</sub>SiC<sub>2</sub> composites in the temperature range of 1 273–1 573 K. The results demonstrate that  $\text{Ti}_3\text{SiC}_2$  reacts with Ti to form  $\text{TiC}_x$ ,  $\text{Ti}_5\text{Si}_3$  and  $\text{TiSi}_2$ . It is shown that the hot pressing temperature is the key factor that determines the extent of reactions. The relative proportion of Ti and  $\text{Ti}_3\text{SiC}_2$  has a relationship with the product of reaction. The Si diffused from  $\text{Ti}_3\text{SiC}_2$  to Ti forms stable compounds like  $\text{Ti}_5\text{Si}_3$  is believed to be the main mechanisms for the reaction. Because the reactions between Ti and  $\text{Ti}_3\text{SiC}_2$  occur at 1 273 K, which is lower than the melting point of Ti (1 943 K), it is difficult to synthesize composite with titanium and  $\text{Ti}_3\text{SiC}_2$ .



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