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# Deformation and fracture behavior of ternary compound $Ti_3SiC_2$ at 25–1300 °C

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**Abstract**

The present paper reports the bending deformation and fracture behavior of polycrystalline $Ti_3SiC_2$ sample synthesized from 2Ti/2Si/3TiC powder mixture through pulse discharge sintering (PDS) technique at 1300 °C for 15 min. The four-point bending tests were conducted on the $Ti_3SiC_2$ specimens in the temperature range of 25–1300 °C. The results showed that the $Ti_3SiC_2$ specimens displayed good plasticity at temperature higher than 1200 °C, which is consistent with those synthesized from Ti/SiC/C powder mixture through hot-isostatic pressing (HIP) technique. The fractography observations reveal that the damage and fracture processes of the $Ti_3SiC_2$ specimens mainly include the formation of sliding bands, cleavage steps and grain boundary cracking. The present test confirms that the PDS technique can also be used to synthesize $Ti_3SiC_2$ products with high purity and good performance.


*Keywords:* $Ti_3SiC_2$; Pulse discharge sintering (PDS); Bending deformation; Fracture

## 1. Introduction

Recently, ternary compound $Ti_3SiC_2$ has received increasing attention due to its excellent properties in strength, electric and thermal conductivity and oxidation resistance [1]. In general, bulk $Ti_3SiC_2$ products can be synthesized through sintering technique at high temperature. Barsoum et al. [2,3] successfully synthesized this material with high purity (about 98 vol.%) through hot-isostatic pressing (HIP) technique from Ti/SiC/C mixtures. Besides, there are some other successful syntheses of $Ti_3SiC_2$ samples from Ti/Si/C powder mixtures through the HIP technique or other methods [4–8]. Therefore, up to now, the corresponding properties of the $Ti_3SiC_2$ samples were mainly obtained from those sintered specimens from the two kinds of powder mixtures [5–12]. However, it should be noted that the sintering processes above were often performed at relatively high temperature (1400–1600 °C) for long time. Recently, we developed a rapid synthesis process of $Ti_3SiC_2$ at a temperature near 1300 °C from different powder mixtures through the pulse discharge sintering (PDS) technique [13–16]. However, the knowledge concerning the synthesized $Ti_3SiC_2$ samples by the PDS technique is rare. The main purpose of the

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present research is to study the high-temperature deformation and fracture behavior of the $Ti_3SiC_2$ samples fabricated by the PDS technique through a four-point bending test.

## 2. Experimental procedure

Commercially available Ti, Si and TiC powders with the sizes and purity of 10 μm and 99.9% (Ti), 10 μm and 99.9% (Si), 2–5 μm and 99% (TiC) were selected in the present investigation. Before sintering, the Ti/Si/TiC powder with the molar ratio of 2:2:3 was mixed in a Tubular shaker mixer in Ar atmosphere for 24 h. Then, the powder mixture was filled in a cylindrical graphite mould with an inner diameter of 50 mm. The chamber was evacuated to a residual pressure of $10^{-3}$ Pa before starting the sintering process. The heating rate was in the range of 50–60 °C/min and the sintering temperature was controlled at 1300 °C for 15 min. During sintering, the applied pressure was maintained constant at 50 MPa. After sintering, the surfaces of samples were ground to remove the graphite layer and analysed by X-ray diffractometry (XRD) with CuKα radiation at 30 kV and 40 mA to determine the purity of $Ti_3SiC_2$ by means of standard additive method. The samples were mechanically polished and etched by a solution of $H_2O/HNO_3/HF$ (2:1:1) to expose the $Ti_3SiC_2$ grains. The density of the synthesized samples was measured by means of the Archimedes' method. Four-point bending tests were performed on the polished $Ti_3SiC_2$ specimens with the size of $30 \times 4 \times 2$ mm with an Instron 8562 universal testing machine at crosshead speed of 0.05 mm/min. The bending tests were conducted from room temperature to 1300 °C in vacuum ($10^{-3}$ Pa). The flexural stresses of the specimens were calculated by the equation [10]:

$$\sigma = 3P\Delta L/2BW^2,$$

where $P$ is the load applied on the specimen, $\Delta L$ is the inner span, $B$ and $W$ are the width and thickness of the specimen. The microstructures and fracture surfaces of the specimens were observed by scanning electron microscope (SEM).

## 3. Results and discussion

In general, TiC is the second phase in the synthesized products and the three main peaks of $Ti_3SiC_2$ and TiC appear in the range of $2\theta = 36–42°$ [2–6]. Therefore, the scanning was mainly conducted in the range of $2\theta = 32–44°$ at a slow scanning rate of 0.02°/s. Fig. 1 shows the XRD patterns of the $Ti_3SiC_2$ sample with the diameter of 50 mm synthesized from the 2Ti/2Si/3TiC powder mixture through the PDS technique at 1300 °C for 15 min. It can be seen that the main peak of TiC at $2\theta = 41.8°$ (200) and the second peak at $2\theta = 36°$ (111) became too weak to be identified in the XRD pattern. Besides, all the other diffraction peaks correspond to $Ti_3SiC_2$ phase. It indicates that the content of the second phase TiC has been decreased to a very low value under the present sintering conditions. By the standard addition method, the purity of the $Ti_3SiC_2$ phase was calculated to be 98.6 vol.%, which is a substantially high value in comparison with those synthesized by other techniques [2–6]. Fig. 2 shows the microstructure of the etched $Ti_3SiC_2$ sample sintered under the same condition. Most of the $Ti_3SiC_2$ grains have a plate-like shape with 20–30 μm in length and 5–10 μm in width, which is identical with those sintered from HIP process [2,3]. At local site, there are only few TiC particles, as indicated by the arrow, indicating high purity of the $Ti_3SiC_2$ phase in the product. The middle-sized $Ti_3SiC_2$ grains can be attributed to the low-temperature rapid sintering process. Besides, the density of the $Ti_3SiC_2$ product was measured to be 4.51 g/cm$^3$, which is quite close to the theoretical

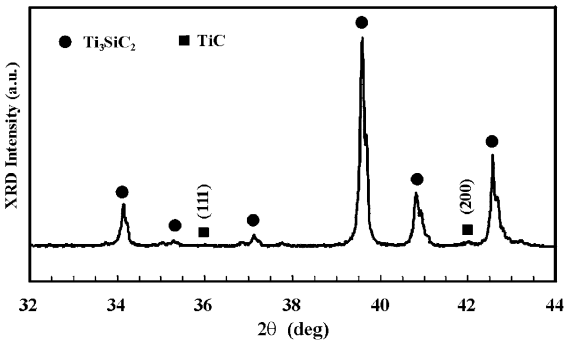


Fig. 1. X-ray diffraction patterns of the sample sintered at 1300 °C for 15 min from 2Ti/2Si/3TiC powder mixture.

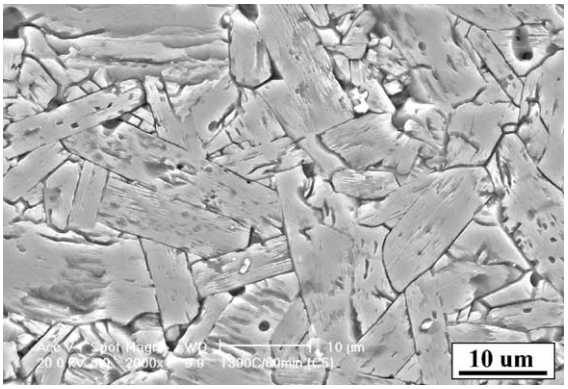


Fig. 2. Microstructure of $Ti_3SiC_2$ sample synthesized from Ti/Si/TiC at 1300 °C for 15 min.

density 4.53 g/cm$^3$ of the pure $Ti_3SiC_2$. It indicates that the $Ti_3SiC_2$ sample synthesized by the PDS technique at relatively low temperature for short time has a high purity and good densification.

Fig. 3(a) shows the four-point bending deformation curves of the $Ti_3SiC_2$ specimens at different temperatures. At room temperature and 1100 °C, the specimens did not exhibit obvious plastic deformation and fractured abruptly, showing a brittle fracture behavior. With increasing temperature to 1150 and 1200 °C, obvious plastic deformation can be seen from its deformation curves. The deformed specimens can be seen in Fig. 3(b), as an inset in Fig. 3(a). It is apparent that the specimen deformed at 1200 °C begins to bend. It implies that the brittle–ductile transition temperature corresponds to 1200 °C, which is identical with the $Ti_3SiC_2$ specimens deformed under tensile and bending loads [7,11]. However, it was found that the present $Ti_3SiC_2$ samples began to display obvious plastic deformation at a temperature above 900 °C under compressive load [16]. At higher temperature, the $Ti_3SiC_2$ specimen can display larger plasticity and did not fracture. As shown in Fig. 3(b), the three specimens E, F and G can be bended to be like a bow without fracture after severe plastic deformation, indicating that the $Ti_3SiC_2$ specimens can carry out larger plasticity at temperatures above 1200 °C. Therefore, it should be concluded that the $Ti_3SiC_2$ specimens synthesized by the PDS technique possess a good plasticity, which is identical with the $Ti_3SiC_2$ samples fabricated by the HIP technique [7,9–12].

Fig. 4 shows the fractography and surface deformation morphologies of $Ti_3SiC_2$ specimens at different temperatures. We have observed all the fractured $Ti_3SiC_2$ specimens. However, no obvious difference was found in the fracture mechanism at different temperatures. Typical fractography can be seen in

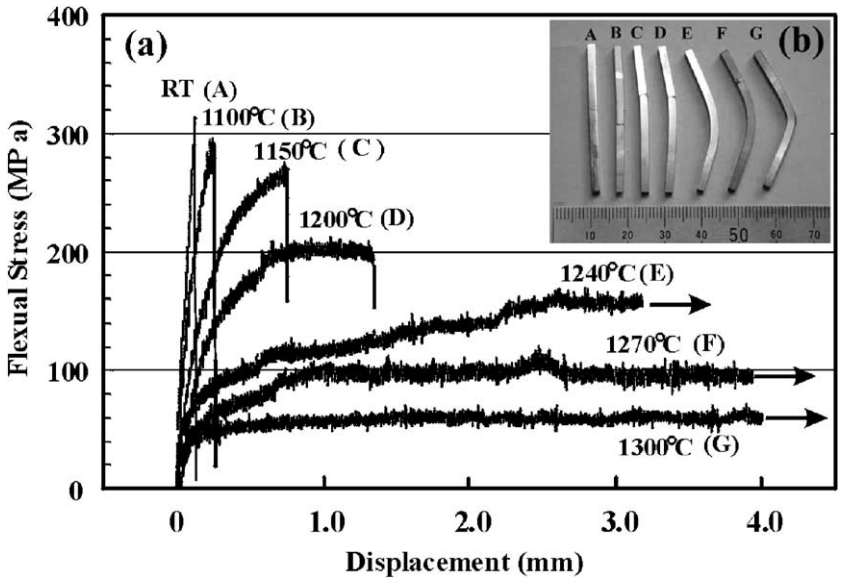


Fig. 3. Four-point bending deformation curves (a) and the deformed $Ti_3SiC_2$ samples (b) at different temperatures.

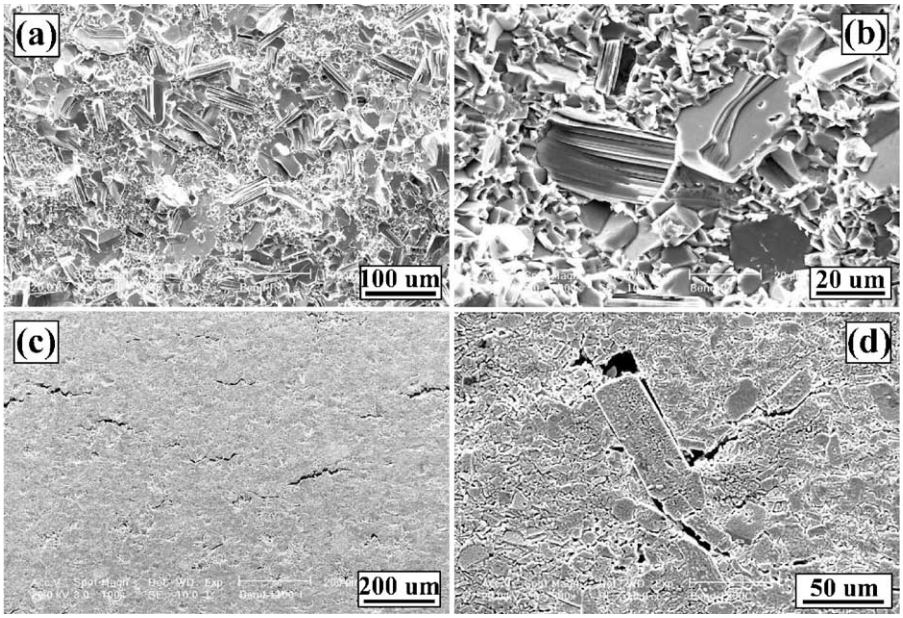


Fig. 4. (a, b) Fractography morphologies of the $Ti_3SiC_2$ specimens at 1100 °C; (c, d) surface deformation morphologies of $Ti_3SiC_2$ specimens at 1300 °C.

Fig. 4(a) and (b), which shows an obvious layered structure or cleavage. It indicates that the fracture mechanism of $Ti_3SiC_2$ in the temperature below 1200 °C is the same, i.e. typical brittle fracture mode. Fig. 4(b) clearly shows the cleavage feature and the layered nature of the $Ti_3SiC_2$ grains. Meanwhile, it can be noted that certain sliding along the basal planes of some $Ti_3SiC_2$ grains took place before fracture of the specimen. In our other series of test, it was observed that the $Ti_3SiC_2$ specimens can display certain plasticity under compressive load at lower temperature below 900 °C by basal plane sliding or interlayer sliding, intergranular cracking, grain buckling and kink band formation [16], which is consistent with the other observations [7,9,11]. At higher temperature (above 1200 °C), the fractography was not observed because all the specimens did not fracture. The surface observations show that there are many micro-cracks; however, those micro-cracks did not propagate or connect to a long crack, as shown in Fig. 4(c). It indicates that the $Ti_3SiC_2$ specimen can accommodate more micro-cracks and display large plasticity at higher temperature, correspondingly, the deformation and fracture mechanism must be changed. At local site, it was noted that micro-cracks nucleated along the surrounding grain boundary, leading to the intergranular cracking and grain separation, as shown in Fig. 4(d). The present observations confirm that the fracture mechanism of $Ti_3SiC_2$ is identical with those synthesized by other techniques [5,7,9–12]. However, it is desirable to further identify the high-temperature (over 1200 °C) deformation and fracture mechanism to explain the large plasticity of $Ti_3SiC_2$.

## 4. Conclusion

Ternary compound $Ti_3SiC_2$ with high purity can be rapidly synthesized from the Ti/Si/TiC powder mixture through pulse discharge sintering (PDS) technique at relatively low temperature of 1300 °C. The synthesized $Ti_3SiC_2$ samples have a good densification at the applied sintering conditions. Under four-point bending loading, the brittle–ductile transition temperature of the present $Ti_3SiC_2$ specimen occurs at 1200 °C. Over this temperature, the $Ti_3SiC_2$ specimen can display larger plastic deformation without fracture. The mechanical properties of the present $Ti_3SiC_2$ specimens confirm that the PDS technique is appli-

cable for the rapid synthesis of the $Ti_3SiC_2$ products with high purity and good performance.

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