

Ternary Compound Ti_3SiC_2 : Part II. Deformation and Fracture Behavior at Different Temperatures ^{*1}

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Compressive tests were conducted in vacuum at temperatures up to 1203 K, on the Ti_3SiC_2 samples synthesized from 2Ti/2Si/3TiC powder mixture through pulse discharge sintering technique. The compressive strength of Ti_3SiC_2 compound showed a monotonic decrease with increasing testing temperature. The Ti_3SiC_2 samples displayed obvious brittle fracture behavior at temperatures below 1073 K. When the testing temperature was above 1123 K, obvious pseudo-plastic deformation behavior was observed in the stress-strain curves. At these temperatures, after an initial pseudo-strain-hardening, a decrease in stress follows with further increasing strain. The pseudo-plastic behavior of Ti_3SiC_2 at temperature above 1123 K could be attributed to the basal plane slip, kink-band formation, delamination of layers at local sites, and intergranular cracking. Further increase in strain causes the growth and linkage of microcracks, which gives rise to the reduction of the real area of the cross section of specimen and hence lead to the "strain-softening" phenomenon.

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1. Introduction

Ternary compound Ti_3SiC_2 is a layered hexagonal material in which almost close-packed Ti planes are separated from each other by hexagonal nets of Si; every fourth layer is a Si layer. The C atoms occupy the octahedral sites between the Ti layers. This compound displays a unique combination of properties. It is a better thermal and electrical conductor than titanium metal, relatively lightweight (4.53 Mg/m^3), and oxidation resistant,¹⁻³⁾ as machinable as graphite.⁴⁾ This compound is very damage tolerant at ambient temperatures, not susceptible to either mechanical or thermal shock.⁴⁾ Apart from the various unique mechanical and physical properties, it has been reported recently that the thermopower of single-phase Ti_3SiC_2 was found to be essentially zero over an extended temperature range (300–850 K), which allows the thermopower of other substances to be determined directly at high temperatures.⁵⁾ The unique combination of the properties makes Ti_3SiC_2 a promising candidate material in many diverse high temperature applications, and therefore it is very important to understand the mechanical behavior of Ti_3SiC_2 at room temperature and also at elevated temperatures.

As described in Part I of this paper, synthesis of Ti_3SiC_2 compound with very high Ti_3SiC_2 phase purity was achieved by using the pulse discharge sintering technique.^{6,7)} As a second part of this study, the mechanical behavior of deformation and fracture under compressive loading at various temperatures were investigated. It is one of the objectives of the present study to understand the difference of the mechanical properties of the Ti_3SiC_2 compound fabricated with this low-temperature short-time sintering process from those synthesized by the conventional method.^{1-3,8-11)}

2. Experimental Procedures

Commercially available Ti (10 μm and 99.9 mass%), Si (10 μm and 99.9 mass%) and TiC (2–5 μm and 99 mass%) powders with a molar ratio of 2:2:3 were selected in the present test and processed as described in Part I of this paper. The powder mixture was sintered at 1573 K for 3.6 Ks by using the pulse discharge sintering technique. Specimens for compression tests were cut and mechanically polished to the size of $2 \times 2 \times 6 \text{ mm}^3$. The compression tests were performed on an Instron 8562 universal testing machine under a constant strain rate of $5.6 \times 10^{-4} \text{ s}^{-1}$ from room temperature to 1203 K in vacuum with a pressure lower than 10^{-3} Pa in the chamber. After the compression test, the deformation morphology on the specimen surfaces and the fracture surfaces were observed by SEM.

3. Results and Discussion

The Ti_3SiC_2 compound used in this study possesses a duplex microstructure, consisting of plate-shaped large grains of 20–50 μm and equiaxed small grains of around 5 μm , as can be found in Fig. 4 in Part I of this paper. The Ti_3SiC_2 phase content in this sintered compact was calculated from the XRD data to be 99.2 mass%. Figure 1 shows the stress-strain curves of the Ti_3SiC_2 compound under compression at various testing temperatures. At temperatures lower than 1073 K, the stress-strain relationship shows almost linearity until failure. Compared with this brittle fracture behavior, when tested at a temperature higher than 1123 K, the compound demonstrates plastic deformation, as shown by the distinct non-linearity in the stress-strain curves. After the linear part of the stress-strain curves at high temperatures, an increase in stress with increasing strain was observed, similar to the strain hardening behavior of metals. Further increase in strain, however, causes a decrease in stress.

Barsoum¹⁾ has pointed out that the transition between the elastic and inelastic deformation is not a yield point and hence

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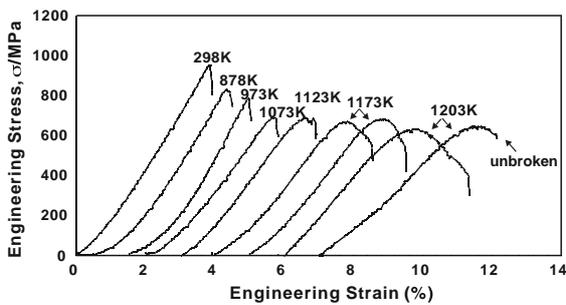


Fig. 1 Stress-strain curves of Ti_3SiC_2 in compression at various temperatures.

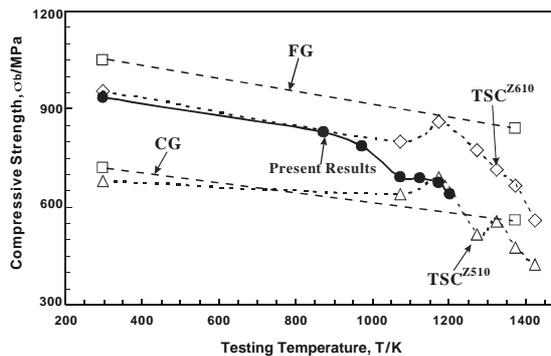


Fig. 2 Variation of compressive strength of Ti_3SiC_2 with testing temperature. Some literature data also plotted, in open symbols and broken lines.

referred to as the inelastic deformation stress or IDS. Since yielding occurred only when at least five independent slip systems are available, while in Ti_3SiC_2 slip has so far not been observed except on the basal plane.^{12,13} Besides, there are quite a few experimental phenomena which could not be explained when the IDS is a yield point: (1) Large asymmetry exists in the IDS under compression and tension;^{3,14} (2) Extremely high “strain-hardening” rate was observed after IDS,³ as can also be seen in Fig. 1; (3) The IDS point and the stress-strain shape are highly strain rate-dependent,¹³ and (4) The mechanical properties of Ti_3SiC_2 are identical to those of ice.¹⁵ The pseudo-strain-hardening and softening observed in Fig. 1 at elevated temperatures higher than 1123 K is then attributed to, rather than yielding, the formation of shear bands along the basal planes, kink-band-formation, microcrack formation and so forth, as discussed in the following sections.

The compressive strength is plotted in Fig. 2 as a function of testing temperature, which shows a monotonic decrease with increasing testing temperature below the transition temperature for the compound from brittle to ductile deformation, and a drastic decrease in strength with temperature was observed when tested at a temperature higher than this transition temperature. Except the data of the present investigation, some literature data were also plotted in the figure for comparison. The data marked with FG (FG: a few microns) and CG (CG: 100–200 μm) are the materials fabricated by reactive HIPing titanium, SiC and graphite powders at temperatures of higher than 1700 K for a few hours.^{2,3} It can be seen that the strength of the Ti_3SiC_2 compound fabricated using the present process is located between the strength values of coarse and fine grain compounds. The microstructure used in the present study is a duplex structure consisting of coarse

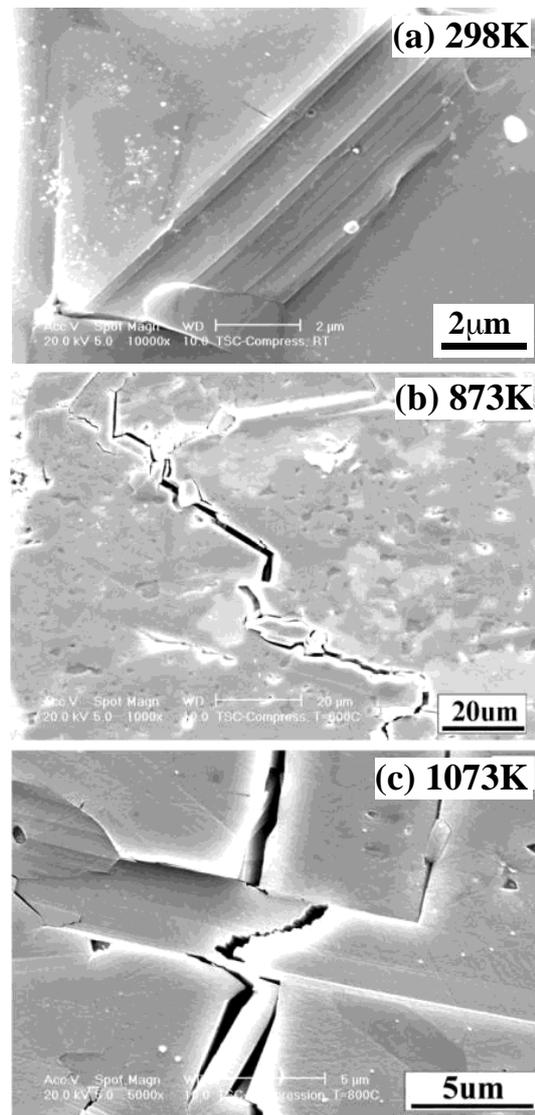


Fig. 3 Scanning electron micrographs of the surface morphology of Ti_3SiC_2 specimens after deformation at different temperatures of (a) 298 K, (b) 873 K and (c) 1073 K.

plate shaped grains and fine equiaxed grains. It is in good consistent that the strength of the compound, fabricated with this pulse discharge sintering technique at a lower temperature and for a shorter sintering time, demonstrates comparable values in the testing temperature range. Also plotted in the figure are data marked with TSC^{Z610} and TSC^{Z510}, fabricated by *in-situ* solid-liquid reaction process¹⁶ with Ti_3SiC_2 content of 93 mass% and 87%, respectively. Compared with the data of the literature, it has been found that the ductile-brittle transition temperature for the compound obtained in this study is about 200–300 K lower than that of the samples sintered from Ti/Si/C and Ti/SiC/C powders. This could be due to the low content of the second phase of TiC in the material used in this study.

The surface morphologies of the specimens after compressive deformation at temperatures below 1073 K are shown in Fig. 3 (Fig. 3(a): 298 K, Fig. 3(b): 873 K, and Fig. 3(c): 1073 K). Except some basal plane slip traces in isolated grains as shown in Fig. 3(a), generally a main crack, as can be seen in Fig. 3(b), can be found in the deformed surface and the prop-

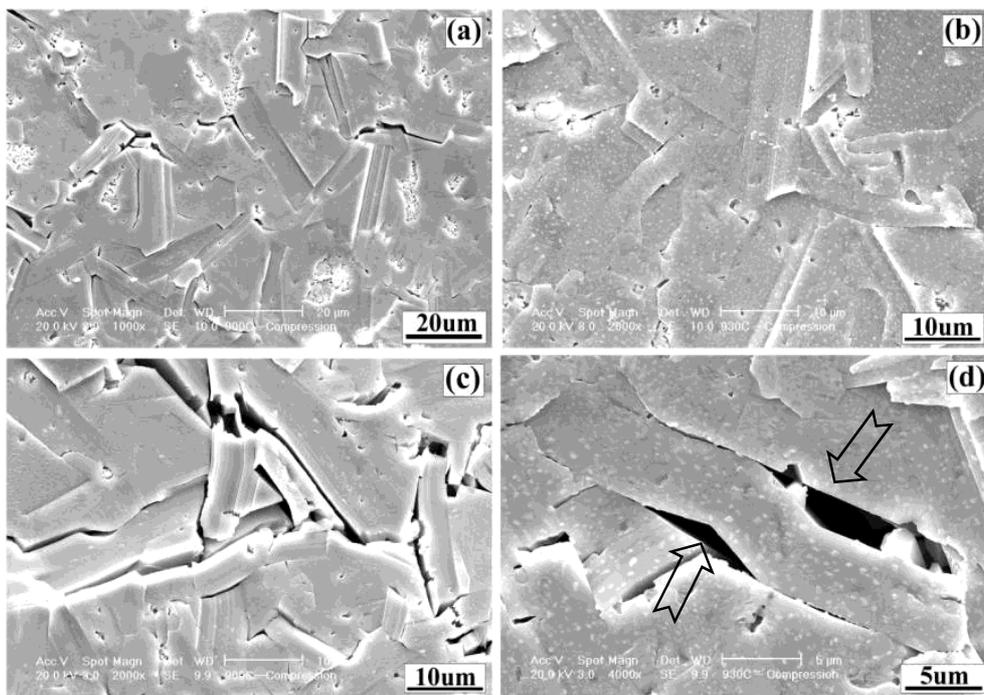


Fig. 4 Scanning electron micrographs of the surface morphology of the Ti_3SiC_2 specimens after deformation at 1203 K.

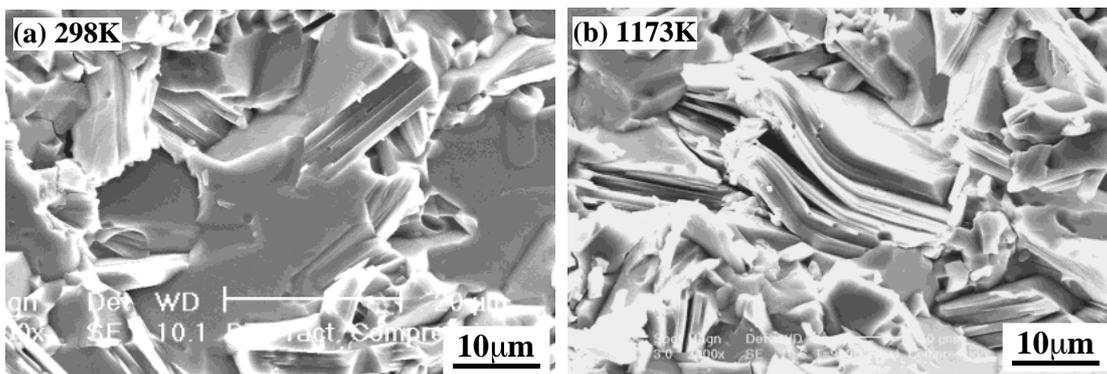


Fig. 5 Scanning electron micrographs showing the fracture surface morphology of the Ti_3SiC_2 specimens after compression test at different temperatures of (a) 298 K, (b) 1173 K.

agation of the main crack will finally lead to a catastrophic failure of the specimen. Figure 3(c) shows the main crack propagating across a plate-shaped grain, causing the breakage of the grain and giving rise to the pullout of the grains. The formation of cracks and their propagation of a main crack accounts for the brittle fracture of the material when compressed at a temperature lower than 1073 K.

When tested at higher testing temperatures, at which plastic deformation was observed on the stress-strain curves, the surface morphology is quite different as shown in Fig. 4. The most prominent difference in the surface morphology from that tested at low temperature is that large amount of microcracks were observed. As can be seen in the low magnification micrographs of Figs. 4(a) and (b), the microcracks are mainly formed at the grain boundaries. Beside basal plane slipping or interlayer sliding, intergranular cracking, grain buckling, and kink band formation¹⁷⁾ was found to be common deformation phenomena.

The fracture surfaces of the specimens after compression tests at 298 K and 1173 K are shown in Figs. 5(a) and (b),

respectively. On the room temperature fracture surface, the cleavage facets and the fractured layer structure can be clearly seen, indicating typical brittle fracture characteristics. On the other hand, when tested at high temperature the fracture surface is considerably different (Fig. 5(b)), where the buckled layered-grain is clearly shown, likely caused by kink-band formation. In this photograph, microcracks can also be found in between the layers and also on the grain boundaries, consistent with the surface observation result as shown in Fig. 4. In the buckled grain, the delamination of the layered grains can also be clearly seen.

The experimental results in this study suggest that Ti_3SiC_2 deforms in a multiple mechanism as illustrated schematically in Fig. 6. Figure 6(a) shows the basal-plane slipping in one grain and the formation of a microcrack at the grain boundary, due to the difficulty in accommodating the deformation because of the lack of slip system in the crystal structure (Cf. Fig. 3(a)). Figure 6(b) shows a grain buckling or bending under compression and the formation of microcracks at the grain boundaries. This also gives rise to the formation of kink

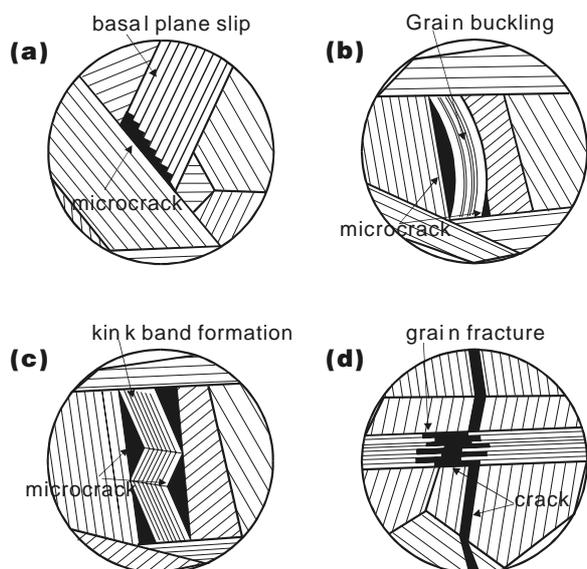


Fig. 6 Schematic illustration showing the deformation and crack formation in Ti_3SiC_2 compound under compression loading. (a) basal plane slip and the microcrack formation at the grain boundary, (b) grain buckling and the induced microcracks, (c) kink band formation and the associated cracking, (d) grain fracture due to crack propagation and the formation of grain pullout.

band when further deformation is undergone, as can be seen in Fig. 6(c). Kinking is predicted in hexagonal metals and alloys having an axial c/a ratio greater than 1.732. With a c/a ratio of 5.76, it is not surprising that Ti_3SiC_2 is susceptible to kinking.¹⁷ Especially when tested at high testing temperatures, kink band formation is a common deformation mechanism and gives rise to microcracks in the adjacent boundaries as indicated in Fig. 6(c). While the micro-deformation and microcracks give rise to a pseudo-plastic deformation and pseudo-strain-hardening after the linear deformation, further increase in strain causes the linkage of microcracks and void formation, and the specimen softens due to the decrease in the real cross section. As illustrated in Fig. 6(d), when a propagating crack encounters a grain with the basal plane perpendicular to the crack, usually a grain pullout, and also lamella pullout will be resulted after the grain fracture.

4. Summary

The fracture and deformation behavior of a Ti_3SiC_2 compound synthesized with pulse discharge sintering technique from 2Ti/2Si/3TiC powders was investigated by compression test and the main results can be summarized as follows.

- (1) The fracture stress of Ti_3SiC_2 decreases monotonically with increasing testing temperature.
- (2) Under compressive loading, Ti_3SiC_2 exhibits brittle fracture at the testing temperature below 1073 K. However, obvious plastic deformation was observed in the stress-strain curves at the temperature above 1123 K, where a pseudo-strain-hardening was observed followed by a strain softening.
- (3) At temperature below 1073 K, fracture of Ti_3SiC_2 mainly originates from one main crack and its propagation, resulting in catastrophic failure. When tested at temperature above 1123 K, many microcracks forms homogenously on the sample surfaces during compression.
- (4) The micro-deformation mechanisms of Ti_3SiC_2 subjected to compressive deformation at different temperatures consist of basal plane slipping, kink band formation, grain buckling, fracture of layers and delamination of the layered structure. It was observed that kink band formation is a common deformation mode of Ti_3SiC_2 , especially at high temperatures.

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