Investigation on microstructure and mechanical properties of short-carbon-fiber/Ti$_3$SiC$_2$ composites

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Abstract

In this paper, short-carbon-fibers (Csf) reinforced Ti$_3$SiC$_2$ matrix composites (Csf/Ti$_3$SiC$_2$, the Csf content was 0, 2, 5 and 10 vol.%) were fabricated by spark-plasma-sintering (SPS) using Ti$_3$SiC$_2$ powders and Csf as starting materials at 1300 °C. The effects of Csf addition on the phase compositions, microstructures and mechanical properties (including hardness, flexural strength and fracture toughness) of Csf/Ti$_3$SiC$_2$ composites were investigated. The Csf, with a bi-layered transition layers, i.e. TiC and SiC layer, were homogeneously distributed in the as-prepared Csf/Ti$_3$SiC$_2$ composites. With the increase of Csf content, the fracture toughness of Csf/Ti$_3$SiC$_2$ composites increased, but the flexural...
strength decreased, while the Vickers hardness decreased initially then increased steadily when the C_{sf} content was higher than 2 vol.%. These changed performances could be attributed to the introduction of C_{sf} and the formation of much stronger interfacial phases.

**Keywords:** Ti$_3$SiC$_2$; short-carbon-fibers; spark-plasma-sintering; microstructure; mechanical properties

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1. Introduction

Ti$_3$SiC$_2$, a typical member of the MAX phase family, is a ternary layered compound. It has attracted extensive attention for its unique combination of metals and ceramics properties, such as low density, easy machinability, high melting point, high tensile strength and damage tolerance, excellent thermal shock resistance, good chemical stability and oxidation resistance (below 900 °C) [1-7]. Therefore, Ti$_3$SiC$_2$ is a potential structural material for the applications in the environments of high temperature, oxidation, corrosion and wear etc. [1, 2, 7, 8]. However, low fracture toughness and hardness limit its practical applications. The introduction of secondary phase as reinforcement into Ti$_3$SiC$_2$ has been proved to be an effective approach to overcome these disadvantages.
For example, numerous reinforcements, including TiB₂ [9], SiC [10], c-BN [11], TiC [12], Al₂O₃ [13] and ZrO₂ [14], have been introduced to improve the mechanical properties of Ti₃SiC₂. Commonly, the additions of ceramic particles can improve Vickers hardness and flexural strength of Ti₃SiC₂ matrix composites. For example, Górny et al. [9] reported that the addition of TiB₂ could effectively improve the Vickers hardness and elastic modulus of Ti₃SiC₂. Tian et al. [12] prepared Ti₃SiC₂-TiC composites and found that the Vickers hardness of the composite increased with increasing TiC content up to 90 vol.%, the flexural strength was enhanced by 64% when the content of TiC was 50 vol.%. Wang et al. [13] fabricated Ti₃SiC₂-xAl₂O₃ (x= 0-20 vol.%) composites by spark-plasma-sintering (SPS), found that Ti₃SiC₂-20 vol.% Al₂O₃ exhibited the highest Vickers hardness, while other mechanical properties deteriorated because of the aggregation of Al₂O₃ in the composite. On the other hand, the toughness of Ti₃SiC₂ matrix composites has hardly ever been strengthened due to intrinsic brittleness of these ceramic phases.

Carbon fibers, with numerous attractively comprehensive properties, such as low density, high strength and modulus, excellent chemical inertness and small thermal expansion coefficient, are considered to be the most promising reinforcing phase for many structural ceramics [15-17]. Especially, Short-carbon-fibers (Cₛ𝐟) have been applied extensively in the preparation of ceramic matrix composites by using SPS or hot-pressing (HP), due to their low cost, easy to add, and chemical inert at high temperatures. Interestingly, Cₛ𝐟 reinforced ceramic matrix composites usually possess relatively higher fracture toughness. For example, Wang et. al. [18] fabricated Cₛ𝐟 (up to 1 wt.%) reinforced
B₄C composites, and found that the composites had higher fracture toughness compared with monolithic B₄C due to the occurrence of crack deflection and bridging resulting from interface debonding between fiber and matrix. Similarly, when Cₐf was introduced in ZrB₂-SiC composites, the toughness of the composites was significantly improved, resulted from fiber debonding, pulling-out and bridging as well as crack deflection [19]. Unfortunately, to date, only Lagos et. al. prepared Ti₃SiC₂-Cf composites by SPS, and processing, microstructure and thermo-mechanical properties (thermal expansion coefficient and thermal conductivity) were investigated [20], while the mechanical properties of Cₐf reinforced Ti₃SiC₂ composites are not available.

In this work, Cₐf/Ti₃SiC₂ composites (the content of Cₐf was 0, 2, 5 and 10 vol.%) were fabricated by using SPS technique. The interfacial microstructure between Cₐf and Ti₃SiC₂ matrix and effects of Cₐf introduction on room-temperature mechanical properties of Cₐf/Ti₃SiC₂ composites were systematically investigated, and the role of Cₐf addition played in reinforcement effect was also discussed.

2. Experimental

2.1 Materials preparation

Ti₃SiC₂ powders (Forsman Technology Co., Beijing, China) and Cₐf (ZLXC, Co., Cangzhou, China) were used as raw materials. The purity and mean particle size of Ti₃SiC₂ powders was 98% and 3 ~ 5 µm, respectively, and the main impurity was Al₂O₃. The length and diameter of Cₐf was 3 mm and 7 µm, respectively. Four Cₐf/Ti₃SiC₂
composites with C_{sf} volume fractions of 0, 2, 5, 10% were fabricated by using SPS (SPS-30T-15-3, Chenhua (Shanghai) Technology Co., Ltd, Shanghai, China). For convenience, the composites were denoted as 2, 5 and 10 C_{sf}/Ti_{3}SiC_{2}, accordingly.

The preparation process of C_{sf}/Ti_{3}SiC_{2} composites is schematically illustrated in Fig. 1. Firstly, C_{sf} was dispersed into deionized water with carboxymethyl cellulose sodium (CMC-Na) as the dispersant. The mass ratio of CMC-Na and C_{sf} was 1:3. Then, Ti_{3}SiC_{2} powders were added into the as-prepared solution. After stirring with a magnetic stirrer, the slurry of Ti_{3}SiC_{2} powders and C_{sf} was obtained. Dried in an oven, the mixed material was packed into a cylindrical graphite die. Followed that, a green body with a diameter of Φ 40 mm was obtained by cold pressing under a load of 20 MPa. Subsequently, the sintering was conducted in SPS facility. After the vacuum of 10 Pa in the sintering chamber was acquired, the green body was heated to 1300 °C, under a constant pressure of 40 MPa soaked for 8 min, then cooled to room temperature in the sintering chamber. All composites were fabricated under such identical SPS condition.
Fig. 1 Schematic illustrations of the preparation process of $C_{sf}/Ti_3SiC_2$ composites by using SPS.

2.2 Characterizations of composition and microstructure of the composites

Determinations of actual densities of $C_{sf}/Ti_3SiC_2$ composites by Archimedes’ method. X-ray diffraction (XRD, Rigaku, Japan) with Cu Kα radiation was used to identified phase compositions of the samples. The microstructure, cross-section morphologies and fracture surfaces of the composites were performed by scanning electron microscope (SEM, Oberkochen, Germany) with energy dispersive spectrometer (EDS, Oxford Instruments, UK).

2.3 Determinations of mechanical properties

For mechanical tests, the samples were cut into a series of bars from the as-prepared composites by electrical discharge machining. Before testing, the surfaces of all samples were sanded to 2000# SiC sandpaper, polished to a mirror surface with 1.0 mm diamond paste, then ultrasonically cleaned in ethanol and distilled water, finally dried.

Vickers hardness test was carried out using Vickers indenter (432SVD, WOLPERT, USA) at a load of 9.8 N for 15 s. The parallel operations were conducted for nine times, finally the mean value was obtained for each sample.

Three-point bending tests were employed to determine flexural strength. The dimensions of rectangular samples were 3 mm × 4 mm × 34 mm. During tests, the
crosshead speed and support span were 0.5 mm/min and 30 mm, respectively. The flexural strength ($\sigma_f$) was calculated by the following Eq. (1):

$$\sigma_f = \frac{3F(N)L}{2BW^2}$$  \hspace{1cm} (1)

Where, F (N) is maximum load, L is support span, B and W are width and height of the rectangular sample, respectively.

Four-point bending tests were employed to determine fracture toughness. The dimensions of rectangular samples were 4 mm × 8 mm × 34 mm. The tested bars were machined as the single-edge-notched-beam (SENB) with the depth of 4 mm and the width of 0.2 mm. During tests, the crosshead speed was 0.05 mm/min, and the inner span and outer span were 10 mm and 30 mm, respectively. The fracture toughness ($K_{IC}$) was calculated by the following Eqs. (2) and (3)

$$K_{IC} = \frac{F(N)}{B} \cdot \frac{S}{W} \cdot f\left(\frac{C}{W}\right)$$ \hspace{1cm} (2)

$$f\left(\frac{C}{W}\right) = 2.9\left(\frac{C}{W}\right)^{1/2} - 4.6\left(\frac{C}{W}\right)^{3/2} + 21.8\left(\frac{C}{W}\right)^{5/2} - 37.6\left(\frac{C}{W}\right)^{3/2} + 38.7\left(\frac{C}{W}\right)^{9/2}$$ \hspace{1cm} (3)

Where, C is notch length, B and W are the width and height of the sample, respectively.

3. Results and discussion

3.1 Phase compositions of the composites

Fig. 2 shows XRD patterns of C$_{sf}$/Ti$_3$SiC$_2$ composites with various volume amounts of C$_{sf}$. For Ti$_3$SiC$_2$ and 2 C$_{sf}$/Ti$_3$SiC$_2$, main phase was identified as Ti$_3$SiC$_2$, and a small amount of Al$_2$O$_3$ impurity could be detected as well. However, when the content of C$_{sf}$ was increased to 5 and 10 vol.%, two impurities of TiC and SiC appeared. And, the peak
intensities of TiC and SiC increased simultaneously with an increase of C_{sf} content in C_{sf}/Ti_{3}SiC_{2} composites. In all three composites, C_{sf} was not identified, which may result from the low weight content of C_{sf} in the composites. The formation of TiC and SiC were caused mainly by the interface reaction between C_{sf} and Ti_{3}SiC_{2} \[21\], which will be discussed in the following section.

**Fig. 2** XRD patterns of C_{sf}/Ti_{3}SiC_{2} composites with the different contents of C_{sf}. The inset shows the enlarged views in the 2θ range of 33°-43°.

### 3.2 Microstructures of the composites

SEM micrographs of the polished surfaces of the as-prepared Ti_{3}SiC_{2} and C_{sf}/Ti_{3}SiC_{2} composites are presented in Fig. 3. Minor micro-pores existed in the Ti_{3}SiC_{2} sample (Fig. 3(a)). For the C_{sf}/Ti_{3}SiC_{2} composites (Fig. 3(b, c, d)), the black circle-like and stripe-like C_{sf} were uniformly dispersed in the gray Ti_{3}SiC_{2} matrix, and exhibited various orientations in a three-dimensional space. Additionally, the average length (100 μm) of C_{sf} in the Ti_{3}SiC_{2} matrix was apparently shorter than their original length (3-5 μm).
The reduction in Csf length is caused by the applied pressure during sintering process.[22] And, no pores were found in these composites, indicating that the well dispersed Csf could promote the densification of Ti₃SiC₂ matrix. The most conceivable reason is that the incorporation of Csf resulted in the existence of more phase boundaries, which is beneficial to the elimination of pores. The fact that Csf well dispersed in the Ti₃SiC₂ matrix confirmed the suitable fabrication approach of these composites at present.

**Fig. 3** SEM micrographs of the polished surfaces of (a) Ti₃SiC₂, (b) 2 Csf/Ti₃SiC₂, (c) 5 Csf/Ti₃SiC₂ and (d) 10 Csf/Ti₃SiC₂.

To understand the bonding of Csf and Ti₃SiC₂ well, the polished cross-section morphology of 10 Csf/Ti₃SiC₂ was observed by SEM, and elemental distribution was identified by EDS, the results are presented in Fig. 4. According to Fig. 4 (a) and (b), there were two carbon fibers, which orientation was perpendicular to the viewing plane. The
bonding of Csf and Ti₃SiC₂ matrix was very strong, no any pores or flaws could be observed in the interface zone. The chemical compositions of the selected zones 1-5 in Fig. 4 (b) were determined by EDS, as listed in Table 1. It was found that the interface phase with duplex structure was formed between Csf and Ti₃SiC₂ matrix. Based on EDS analysis results, the inner interface layer adjacent to Csf was identified as TiC, while the outer interface layer was SiC. Such result was quite different from the results of Ti₃SiC₂-Cr composites prepared by Lagos et al [20]. The main reason is that our experiments actively increased the dwell time from 5 min to 8 min to improve the relative densities of Csf/Ti₃SiC₂ composites, which is favorable to the occurrence of the reaction between Csf and Ti₃SiC₂.

Fig. 4 (a) SEM image of polished cross section of 10 Csf/Ti₃SiC₂; (b) element mappings in the white frame area in Fig. 4 (a); element distributions of (c) Ti, (d) Si, (e) Al, and (f) C.
Table 1 EDS results of the marked spots 1-5 in Fig. 4(b).

| Marked zone | Compositions (atomic %) | Corresponding phase |
|-------------|-------------------------|---------------------|
|             | Ti  | Si  | Al  | C   | O   |       |
| 1           | 6.81 | 2.36 | 0.40 | 90.42 |  -  | Csf   |
| 2           | 50.93 | 3.84 | 1.10 | 44.13 |  -  | TiC (rich in Ti) |
| 3           | 5.23  | 57.71 | 0.79 | 36.27 |  -  | SiC   |
| 4           | 45.56 | 15.34 | 0.59 | 38.51 |  -  | Ti3SiC2 |
| 5           | 2.67  | 1.36  | 26.21 | 23.68 | 46.09 | Al2O3 |

The interfacial reaction mechanism can be proposed tentatively based on the above SEM observations and EDS results. A literature reported show that Ti3SiC2 can maintain thermal stability at about 1300 °C [23]. Therefore, the above-mentioned phenomenon could be attributed to the environment-dependent decomposition behavior of Ti3SiC2, i.e., a carbon-rich environment will promote the decomposition of Ti3SiC2 and transform them into TiC0.67 and Si [23-25], as described by the reaction (4).

\[ \text{Ti}_3\text{SiC}_2 \rightarrow 3 \text{TiC}_{0.67} + \text{Si (g)} \quad (\Delta G (1600 k)=181 \text{ kJ/mol}) \quad (4) \]

Furthermore, for the composites of Csf/Ti3SiC2, due to the reaction of the added Csf could act as C reservoir and Si to form SiC (Eq. (5)) [23], the reaction (4) could be promoted by the consumption of Si.

\[ \text{C + Si (g)} \rightarrow \text{SiC} \quad (5) \]

The formation of the TiC phase is due to the diffusion of C into the carbon vacancies in TiC0.67, as shown in the reaction (6).

\[ \text{C + TiC}_{0.67} \rightarrow \text{TiC} \quad (6) \]

As a result, a double-layered interface phases are formed, which can serve as a
diffusion barrier and prevent Csf from providing a C source to inhibit the decomposition of Ti3SC2.

From the EDS result in Fig. 4(e), the bright agglomerates were enriched in Al. Meanwhile, the EDS analysis also confirmed the existence of minor well-dispersed Al2O3 particles with the size of 2-3 μm, which corresponds to the XRD results.

The actual densities of the as-prepared composites are listed in Table 2. With increasing the content of Csf, the density of the composite decreased. This is because that the density of Csf (1.78 g/cm³) was much lower than that of Ti3SiC2 (4.53 g/cm³). Based on the nominal ratio of the initial contents of Ti3SiC2 and Csf, the theoretical densities of the as-prepared Ti3SiC2 and Csf/Ti3SiC2 composites were calculated using the rule of mixtures, also listed in Table 2. Obviously, the relative densities of all materials prepared by SPS were higher than 98% in this work. In addition, under the condition of the same SPS processing, the relative densities of the as-prepared Csf/Ti3SiC2 composite increased with increasing Csf content. It should be noted that, during the calculation of the theoretical densities of the composites, only the nominal composition of the composites were considered, the formation of high density TiC (4.93 g/cm³ [1]) during the sintering of the bulk material was neglected. Therefore, correspondingly, the higher contents of Csf as well as TiC led to the greater deviation of calculated density.
Table 2 The determined and calculated densities of the as-prepared Ti₃SiC₂ and C₅/Ti₃SiC₂ composites.

| Nominal compositions (vol %) | Apparent density (g cm⁻³) | Theoretical density (g cm⁻³) | Relative density (%) |
|-----------------------------|---------------------------|-------------------------------|----------------------|
| Ti₃SiC₂                     | 4.45                      | 4.53                          | 98.23                |
| 2 C₅/Ti₃SiC₂                | 4.41                      | 4.47                          | 98.65                |
| 5 C₅/Ti₃SiC₂                | 4.35                      | 4.39                          | 99.09                |
| 10 C₅/Ti₃SiC₂               | 4.24                      | 4.25                          | 99.76                |

3.3 Mechanical properties of the composites

3.3.1 Vickers hardness

The dependence of Vickers hardness of Ti₃SiC₂ and C₅/Ti₃SiC₂ composites on the theoretical content of C₅ is shown in Fig. 5. The Vickers hardness of the pristine Ti₃SiC₂ sintered by SPS in the present work was 5.40 ± 0.06 GPa (measured at the indentation load of 9.8 N). It was reported previously that the hardness of monolithic Ti₃SiC₂ was about 4 GPa [26], lower than that of Ti₃SiC₂ synthesized by SPS in this work. From Fig. 5, the Vickers hardness of the composite decreased slightly for the case of 2 vol.% C₅, and then increased monotonously to 6.80 ± 0.87 GPa with increasing C₅ content up to 10 vol.%. For a small amount of C₅ (2 vol.%) addition, the slightly decline of the hardness of the composite was related to C₅ possessing lower hardness compared to Ti₃SiC₂ matrix. The formation of minor TiC and SiC interfacial phases with the higher hardness of about 28-30 GPa [27] and 22 GPa [28] compared to Ti₃SiC₂ matrix, respectively, was insufficient to compensate the hardness loss by the introduction of C₅. For the 5 C₅/Ti₃SiC₂, similar situation took place. Although the increased contents of TiC and SiC
gave rise to the increased hardness of the composite, which was still lower than that of the matrix Ti$_3$SiC$_2$. When the content of C$_{sf}$ was 10 vol.%, the hardness of the as-prepared composite was 6.80 GPa, which was larger than that of the Ti$_3$SiC$_2$. For the 10 C$_{sf}$/Ti$_3$SiC$_2$, the contents of formed TiC and SiC interfacial phases with higher hardness was sufficient to compensate the hardness loss by the introduction of C$_{sf}$. Zhang et al. [29] presented that the maximum Vickers hardness of Ti$_3$SiC$_2$-40 vol.% TiC composite was about 13 GPa. Accordingly, it is reasonable to believe that the contents of added C$_{sf}$ and TiC derived from the interfacial reaction between C$_{sf}$ and Ti$_3$SiC$_2$ simultaneously determine the hardness of C$_{sf}$/Ti$_3$SiC$_2$ composite.

![Graph](image)

**Fig. 5** The dependence of Vickers hardness of the as-prepared C$_{sf}$/Ti$_3$SiC$_2$ composites on the content of C$_{sf}$.

### 3.3.2 Flexural strength and fracture toughness

The dependence of the flexural strength and fracture toughness of Ti$_3$SiC$_2$ and C$_{sf}$/Ti$_3$SiC$_2$ composites on the theoretical content of C$_{sf}$ are illustrated in Fig. 6. When the
content of C_{sf} increased from 0 to 10 vol.%, the flexural strength decreased from 602 ± 34 MPa to 468 ± 34 MPa. The main reason for the deduction of flexural strength is that the modulus of C_{sf} (∼250 GPa) is lower than that of Ti₃SiC₂ matrix (∼326 GPa), and the presence of C_{sf} in the matrix resulted in the reduced load carrying capacity of the composites [1, 19]. Meanwhile, it was also related to the formation of TiC with a high content between the matrix and C_{sf}, because the flexural strength of pure TiC is as low as 240-390 MPa [30].

It also can be clearly seen from Fig. 6 that as the increase of C_{sf} content, the fracture toughness of C_{sf}/Ti₃SiC₂ composites increased, reaching a maximum value of 6.48 ± 0.29 MPa·m^{1/2} for 10 C_{sf}/Ti₃SiC₂. After adding C_{sf}, the fracture toughness of C_{sf}/Ti₃SiC₂ composites were enhanced, which was related to the activation of certain kinds of toughening mechanisms [31, 32], as described as follows.

**Fig. 6** The dependence of flexural strength and fracture toughness of the as-prepared C_{sf}/Ti₃SiC₂ composites on the content of C_{sf}.

The mechanical properties of fiber-reinforced composites rely on not only the intrinsic properties of fiber and matrix, but also the characteristics of fiber/matrix
interface interface [33, 34]. Interface delamination and the formation of a “weak” interphase are significantly vital to the comprehensive properties of fiber-reinforced ceramic matrix composites [35]. During the failure process of composites, their fracture toughness could be improved effectively by interface debonding, fiber bridging and fiber pull out caused by such characteristics mentioned above. To further understand the possible toughening mechanisms of the as-prepared Csf reinforced Ti3SiC2 composites, the fractured surfaces of the composites were observed by SEM after SENB test are shown in Fig. 7. Obviously, these composites exhibited a fully brittle fracture. The breakage of Csf and interface debonding could be found, but fiber pull-out did not appear, corresponding to a strong interface bonding between Csf and Ti3SiC2. During the preparation of Csf/Ti3SiC2 composites, original Csf did not undergo any surface treatment. As mentioned above, the formation of interfacial phases of TiC and SiC during heat-pressing sintering caused an enhanced interface bonding between Csf and Ti3SiC2 matrix, and such chemical bonding was much stronger than van der Waals’ force in the matrix [36, 37]. Therefore, the fiber bridging or fiber pulling-out became very difficult to occur due to the presence of strong interface between fiber reinforcement and matrix [34, 38]. On the other hand, under this condition, as the Csf content increases, more energy is consumed during fracture process due to interface debonding and Csf breakage, thereby resulting in the increased fracture toughness of the as-prepared composite.
Conclusions

Csf reinforced Ti3SiC2 composites were prepared by SPS process. The behavior of Csf in the as-prepared Csf/Ti3SiC2 composites, microstructures and mechanical properties of the as-prepared Csf/Ti3SiC2 composites were studied. The main results are as follows:

1. Dense Csf/Ti3SiC2 composites with 2, 5, 10 vol.% Csf were prepared by SPS at 1300 °C. The interfacial reaction layer with duplex structure of TiC inner layer and SiC outer layer was formed between Csf and Ti3SiC2 matrix.

2. Among the as-prepared Ti3SiC2 matrix composites, the one with 10 vol.% Csf exhibited the highest Vickers hardness (6.80 ± 0.87 GPa) and fracture toughness
(6.48 ± 0.29 MPa⋅m$^{1/2}$), which were increased by 36.7% and 17.8% compared with Ti$_3$SiC$_2$, respectively. Its flexural strength was 467.7 MPa, decreased by 22.3%.

(3) The contents of added C$_{sf}$ and TiC produced by the interfacial reaction between C$_{sf}$ and Ti$_3$SiC$_2$ matrix played a critical part in the mechanical properties of C$_{sf}$/Ti$_3$SiC$_2$ composites.

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Figure captions

**Fig. 1** Schematic illustrations of the preparation process of C$_{sf}$/Ti$_3$SiC$_2$ composites by using SPS.

**Fig. 2** XRD patterns of C$_{sf}$/Ti$_3$SiC$_2$ composites with the different contents of C$_{sf}$. The inset shows the enlarged views in the 2$\theta$ range of 33°-43°.

**Fig. 3** SEM micrographs of the polished surfaces of (a) Ti$_3$SiC$_2$, (b) 2 C$_{sf}$/Ti$_3$SiC$_2$, (c) 5 C$_{sf}$/Ti$_3$SiC$_2$ and (d) 10 C$_{sf}$/Ti$_3$SiC$_2$.

**Fig. 4** (a) SEM image of polished cross section of 10 C$_{sf}$/Ti$_3$SiC$_2$; (b) element mappings in the white frame area in Fig. 4 (a); element distributions of (c) Ti, (d) Si, (e) Al, and (f) C.

**Fig. 5** The dependence of Vickers hardness of the as-prepared C$_{sf}$/Ti$_3$SiC$_2$ composites on the content of C$_{sf}$.

**Fig. 6** The dependence of flexural strength and fracture toughness of the as-prepared C$_{sf}$/Ti$_3$SiC$_2$ composites on the content of C$_{sf}$.

**Fig. 7** SEM micrographs of the fracture surfaces of (a) Ti$_3$SiC$_2$, (b) 2 C$_{sf}$/Ti$_3$SiC$_2$, (c) 5 C$_{sf}$/Ti$_3$SiC$_2$ and (d) 10 C$_{sf}$/Ti$_3$SiC$_2$. 

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