Effect of TiC_{0.4} Addition on Microstructures and Properties of Ti₃SiC₂ Matrix Composites

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Research Article

Keywords: Ti₃SiC₂ matrix composite, TiC_{0.4}, Microstructure, Mechanical properties, Tribology

Posted Date: February 3rd, 2022

DOI: <https://doi.org/10.21203/rs.3.rs-1292035/v1>

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Abstract

TiC_{0.4}-Ti₃SiC₂ composites were manufactured using spark plasma sintering (SPS) at 1400°C for 10 min, with an applied pressure of 40 MPa. The effect of TiC_{0.4} additions on microstructures and properties of Ti₃SiC₂ matrix composites was investigated. The addition of TiC_{0.4} induced the formation of TiC_x ($0.4 \leq x \leq 1$) and promoted the decomposition of Ti₃SiC₂, yielding TiC_x and Ti₅Si₃. The mechanical and tribological properties of the TiC_{0.4}-Ti₃SiC₂ composite improved with the TiC_{0.4} content due to C vacancy present in TiC_{0.4}, although the friction distance in the initial stage of friction test was increased. The 30 vol.% TiC_{0.4}-Ti₃SiC₂ composites exhibited the highest hardness and fracture toughness of 14.87 GPa and 5.45 MPa·m^{1/2}. At the same conditions, the wear rate at room temperature reached a minimum value of $4.32 \times 10^{-7} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$, while the friction coefficient was 0.74. The friction distance in the initial friction stage of 30 vol.% TiC_{0.4}-Ti₃SiC₂ was about 38 m, and the wear mechanism of the composite at room temperature was mainly adhesive wear.

Highlights

1. The effect of C vacancy in TiC_{0.4} can not only promote the decomposition of Ti₃SiC₂ to form hard phases of TiC_x and Ti₅Si₃, but also improve the interface bonding between Ti₃SiC₂ and TiC_x.
2. The addition of TiC_{0.4} forms the hard phase of TiC_x and decreases the Ti₃SiC₂ grain size.
3. The value of bulk density, relative density, hardness and fracture toughness has the same trend of continuous climbing up with the increase of TiC_{0.4} adding amount, while that of wear rate exhibits a declining trend. The value of the friction coefficient remains stable initially and then increases.
4. With the increase of TiC_{0.4} adding amount, the content of TiC_x particles in the detached particle increases, resulting in the difficulty of forming the complete lubricating film on the friction surface of TiC_{0.4}-Ti₃SiC₂ composite, and then increases the initial friction distance.
5. The integrity of the lubricating film is improved by the pinning effect of TiC_x particle, resulting in the decrease of friction coefficient and wear rate.

1 Introduction

Ti₃SiC₂ ternary ceramic embodies the advantages of both ceramics and metals, such as high Young's modulus (343 GPa) [1], high fracture toughness (7.20 MPa·m^{1/2}) [2], high corrosion resistance[3], good thermal and electrical conductivity (43 W/m·K and $9.6 \cdot 10^6 \Omega^{-1} \text{ m}^{-1}$) [4], thermal shock resistance [5], etc. Its layered crystal structure is comparable to that of well-known graphite and MoS₂ solid lubricants, implying that it may be an excellent solid lubricant material with a low friction coefficient [6]. Thus, Ti₃SiC₂ could find potential applications in bearings, turbines, cutting tools, electrical contacts, heat exchangers [7–11], etc. However, this is severely hampered by low hardness (4.54 GPa) and wear resistance ($0.21\text{--}1.87 \times 10^{-3} \text{ mm}^3/\text{N}\cdot\text{m}$) of Ti₃SiC₂ ceramics [12]. Many reinforcing phases, including

Al_2O_3 , SiC , TiC-TiS_2 , and TiC-TiB_2 , were applied to improve the hardness and wear resistance of Ti_3SiC_2 due to the nailing of the softer phase by the hard phase [13–15]. However, we still lack the solution for the weak interfacial bonding between these reinforcing phases and the Ti_3SiC_2 matrix, limiting the reinforcing phases' enhancement effect. $\text{TiC}_{0.4}$ is a representative of nonstoichiometric TiC_x ($0.3 < x < 0.9$) compounds, exhibiting a NaCl-type crystal structure [16–18]. It possesses high hardness (24.1–24.8 GPa), and the existence of vacancies improves the interfacial bonding between the reinforcing phase and the matrix [19–21]. Recently, TiC_x was used to improve the mechanical properties of the Al or Cu matrix composite, showing that the hardness and bending strength were increased by 84.7–116.3 and 80.7–122.4%, respectively, compared to the Al or Cu matrix composite without TiC_x [21, 22].

In this study, we prepared the $\text{TiC}_{0.4}$ (10, 20, and 30 vol.%) particles-reinforced Ti_3SiC_2 matrix composites using SPS. Meanwhile, the effect of $\text{TiC}_{0.4}$ content on the microstructure, mechanical properties, and friction and wear behaviour of the composites was systematically investigated, and the related mechanism was discussed.

2 Experimental Details

$\text{TiC}_{0.4}$ powder was synthesized via mechanical alloying (MA) using the production method described in Ref. 19. $\text{TiC}_{0.4}$ (purity 99.6%, size 180–200 nm) and Ti_3SiC_2 (purity 99%, size 30 μm) powders were used as raw materials. The $\text{TiC}_{0.4}$ volume fraction was fixed at 10, 20, and 30 vol.% in the whole raw material, and the corresponding sintered samples were denoted as TC0 (10 vol.% $\text{TiC}_{0.4}$), TC1 (20 vol.% $\text{TiC}_{0.4}$), and TC2 (30 vol.% $\text{TiC}_{0.4}$). These raw materials were well dispersed using the high-energy ball-milling method, and then the mixed powders were sintered using an SPS system (LABOXTM-110, Japan) at a sintering temperature of 1400°C and a holding time of 10 min under a pressure of 40 MPa in a 30 Pa vacuum. The heating rate was set to 100°C/min.

The bulk density of the $\text{TiC}_{0.4}$ - Ti_3SiC_2 composite was measured by Archimedes' method. The theoretical density of TC0, TC1, and TC2 was 3.78, 4.39, and 4.64 g/cm³, as calculated using the method described in Ref. 23. The hardness of the Ti_3SiC_2 matrix composite was measured by a Vicker's hardness instrument (FM-ARS9000, China) with a dwell time of 15 s at a load of 500 gf. Fracture toughness (K_{IC}) was determined by the indentation method proposed in Ref. 24 with a load of 5000 g and a holding time of 15 s. Friction and wear tests were carried out using a ball-on-disc system (TRB, Switzerland). A ball sliding on a linear reciprocating athletic flat specimen (5 mm × 15 mm × 5 mm) was adopted. The specimens were cut by electrical discharge method and the test surface was mechanically polished down to 2000# SiC paper. The Si_3N_4 balls were used as the friction counterparts with a diameter of 4 mm and a hardness of 15 GPa. Disc-specimens and Si_3N_4 balls were cleaned by acetone solution before frictional wear tests. All tests were carried out under dry sliding conditions at room temperature (25°C) in the air. The wear track diameter was typically 11–13 mm. The tribological tests were performed at an applied load of 5 N, at a sliding speed of 0.1 m/s, and a testing distance of 100 m. The friction coefficient was

continuously measured and automatically recorded by the computer system of the friction tester, while the wear rate was calculated using Archard's equation [25]:

$$W = V/P \cdot L$$

where $V(\text{mm}^3)$ is the wear volume obtained by measuring the weight loss in a microbalance after ultrasonic cleaning and drying and the measured density, $P(\text{N})$ is the applied load, and $L(\text{m})$ is the total sliding distance.

3 Results Ad Discussion

The $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composite is composed of Ti_3SiC_2 (Fig. 1a, marked as A, D, G, and J in Figs. 1c-f), TiC_x (Fig. 1a, marked as B, E, and H in Figs. 1c-e) and Ti_5Si_3 (Fig. 1a, marked as C, F, and I in Figs. 1c-e). Ti_3SiC_2 , TiC_x , and Ti_5Si_3 particles exhibit lamellar shape, equiaxed grains, and irregular shape, respectively. The relative content of each phase in the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composite is semi-quantitatively calculated using the method described in Ref. 26. The relative content of Ti_3SiC_2 decreases, while the relative contents of TiC_x and Ti_5Si_3 increase (Fig. 1b) with the increase of the $\text{TiC}_{0.4}$ amount. The Ti-Si bonds in Ti_3SiC_2 are weaker than Ti-C bonds [27], so that $\text{TiC}_{0.4}$ can promote the Ti_3SiC_2 decomposition to form TiC_x and Ti_5Si_3 in the environment of C vacancies generated by the $\text{TiC}_{0.4}$ additions [28]. At the same time, $\text{TiC}_{0.4}$ absorbs C atoms from Ti_3SiC_2 to form TiC_x , which is more stable than $\text{TiC}_{0.4}$, resulting in the small diffraction peak broadening of TiC_x , compared with that of $\text{TiC}_{0.4}$ (Fig. 1a). The Ti_3SiC_2 decomposition and TiC_x generation reactions can be described as follows [29]:

The Ti_3SiC_2 grain size decreases from 3.3-6.1 μm (mean of 5.2 μm) to 1.4-2.8 μm (mean of 2.1 μm) (Figs. 1c-e) because TiC_x is prone to concentrate around the Ti_3SiC_2 grain boundaries, hindering the Ti_3SiC_2 grain growth during the sintering process (Figs. 1c-e). The porosity in the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composite gradually decreases with the increase of the $\text{TiC}_{0.4}$ amount (Figs. 1c-e). In addition, the fracture morphology of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composite includes transgranular and intergranular fractures (Figs. 1c-e). The crack propagates at the Ti_3SiC_2 particles of TC2, indicating that the interfacial bonding between Ti_3SiC_2 and TiC_x is stronger than the Ti_3SiC_2 fracture strength (see Fig. 1f). Thus, the interfacial bonding between Ti_3SiC_2 and TiC_x is good. The good interfacial bonding between Ti_3SiC_2 and TiC_x is attributed to the atomic diffusion of the elements. The reaction layer is about 65-80 μm in the layered sintered $\text{TiC}_{0.4}$ and Ti_3SiC_2 composite, indicating a good wettability between $\text{TiC}_{0.4}$ and Ti_3SiC_2 (Fig. 2). The Si content in the reaction layer is higher than in the Ti_3SiC_2 region, while the C content is lower than in the Ti_3SiC_2

and $\text{TiC}_{0.4}$ regions. According to the phase composition of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composite (Fig. 1a), Ti_5Si_3 and TiC_x are formed in the reaction layer. The C vacancies can improve the wettability between $\text{TiC}_{0.4}$ and Ti_3SiC_2 by enhancing the atomic diffusion ability of the elements, yielding the formation of TiC_x and Ti_5Si_3 by the Ti_3SiC_2 decomposition. This can change the interfacial structure between $\text{TiC}_{0.4}$ and Ti_3SiC_2 and promote interfacial bonding, enhancing the mechanical and tribological properties of the composite [21, 22].

The bulk density, relative density, hardness, fracture toughness, friction coefficient, and wear rate of $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites are in the range of 3.7-4.63 g/cm³, 98.1-99.7%, 4-14.87 GPa, 3.6-5.45 MPa·m^{1/2}, 0.63-0.74, and (4.32-6.49)×10⁻⁷ mm³N⁻¹m⁻¹, respectively (Figs. 3a, b, and f). The indentations of hardness and fracture cover the whole phases of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites at the optical microscope so that the values of hardness and fracture toughness of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites measured by indentation method are accurate (Figs. 3c and d). The values of bulk density, relative density, hardness, and fracture toughness exhibit the same trend of a continuous increase with the $\text{TiC}_{0.4}$ amount, while the wear rate exhibits a declining trend. The value of the friction coefficient remains stable initially and then increases. With a 30 vol.% $\text{TiC}_{0.4}$ addition, the bulk density, relative density, hardness and fracture toughness of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites reach maximum values of 4.63 g/cm³, 99.7%, 14.87 GPa, and 5.45 MPa·m^{1/2}, respectively, while the wear rate reaches a minimum value of 4.32×10⁻⁷ mm³N⁻¹m⁻¹. With a 20 wt.% $\text{TiC}_{0.4}$ addition, the friction coefficient of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites reaches a minimum value of 0.63. The behaviour of bulk density, hardness, and fracture toughness values with the $\text{TiC}_{0.4}$ amount originates from the contribution of three factors. Firstly, the relative contents of TiC_x (4.46-4.86 g/cm³ and 18-32 GPa [20, 30]) and Ti_5Si_3 (4.32 g/cm³ and 9.5 GPa) phases increase with the increase of the $\text{TiC}_{0.4}$ amount, while the relative content of the Ti_3SiC_2 matrix (4.53 g/cm³ and 4 GPa) decrease, yielding a higher bulk density and hardness of $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites (Figs. 1a-b). Secondly, the $\text{TiC}_{0.4}$ addition can improve the interfacial bonding of Ti_3SiC_2 and TiC_x so that it is conducive to the stress transfer from the Ti_3SiC_2 matrix to TiC_x at high pressure and impeding the grain boundary sliding, which increases the resistance of dislocation movement (Figs. 1c-f). It yields an improvement in density, hardness and fracture toughness of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites. Finally, hardness and fracture toughness are directly related to the grain size [31]. The Ti_3SiC_2 particle size decreases with increase with the increase of the $\text{TiC}_{0.4}$ amount, causing an increase in the grain boundaries of $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites and resulting in higher hardness and fracture toughness (Figs. 1c-e) [32, 33]. Although the fracture toughness of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites is close to the mean value reported in the previous studies [7, 14, 30, 34–39], the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites exhibit an unprecedentedly high hardness of ~14.87 GPa (Fig. 3e). TiC_x particles can effectively pin the Ti_3SiC_2 matrix to prevent the Ti_3SiC_2 detaching during the friction process due to the high hardness and strong interfacial bonding between TiC_x and Ti_3SiC_2 (Fig. 1) [14]. In addition, the decrease of grain size lowers the wear rate due to the mitigation of multiple risks caused by plastic deformation, fracture,

fragmentation, and oxidation of grains [40]. The detached particles partly form a lubricating film on the friction surface of $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composite, while others yield wear debris, which is later removed [32]. As the $\text{TiC}_{0.4}$ addition increases from 20 to 30 vol.%, the friction coefficient increases although the wear rate decreases, implying that the excessive TiC_x particles destroy the continuity of the lubrication film formed by the Ti_3SiC_2 matrix (Fig. 4b-d). It is worth noting that $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites show a wear rate as low as $4.32\text{-}6.49 \times 10^{-7} \text{ mm}^3 \text{ N m}^{-1}$, which is much lower than the values reported for pure Ti_3SiC_2 and other Ti_3SiC_2 -based ceramics [14, 37, 41–46].

The relationship between friction coefficient and friction distance of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites can be divided into two stages, i.e., initial friction and stable friction (Fig. 4a). The friction distance of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites in the stable friction stage is from the end of the initial friction stage to the end of the friction process (Fig. 4a). The friction distance of TC0, TC1, and TC2 is about 4, 26, and 38 m in the initial friction stage. Detached particles gradually form the lubricating film on the friction surface of $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites during the friction process in the initial friction stage [38]. Since the more lubricating film is formed than lost through the wear debris formation, the friction coefficient gradually increases and fluctuates significantly in the initial friction stage. As the friction distance increases, the formed lubricating film covers the complete friction surface of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composite, yielding the stage of stable friction. Consequently, the lubricating film production of $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites is in dynamic balance with its loss during the friction process, and the friction coefficient curve becomes smooth [48]. The content of TiC_x particles in the detached particles increases with the increase of the $\text{TiC}_{0.4}$ amount, hampering the formation of the complete lubricating film on the friction surface of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composite so that the friction distance of the initial friction increases. The friction surface of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites mainly exhibits abrasive debris (Figs. 4b-d), indicating that the wear mechanism of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites is adhesive wear. The abrasive debris on the friction surface decreases with the increase of the $\text{TiC}_{0.4}$ amount. The C content in the smooth regions (L, N, and O points in Figs. 4b-d and Table 1) is lower than in the abrasive areas (K and M points in Figs. 4b-c and Table 1), unlike the O content. In addition, the O content in the smooth regions decreases with the increase of the $\text{TiC}_{0.4}$ amount, while the C content increases in the smooth regions. Conclusively, the formation of abrasive debris is caused by the shedding of the lubricating film, which is inhibited by the $\text{TiC}_{0.4}$ amount, decreasing the wear rate.

Table 1
The EDS elemental analysis (wt.%) of the regions marked in Figs. 4b-d

EDS measured points	Ti	Si	O	Fe	C
K	33.23	12.58	35.11	1.08	18
L	37.34	14.15	26	1.23	21.28
M	32.78	14.34	24.29	0.85	25.74
N	35.91	15.01	20	1.97	27.11
O	31.06	13.33	22.11	1.14	32.36

The C vacancies in $\text{TiC}_{0.4}$ promote the Ti_3SiC_2 decomposition, and thus, the formation of TiC_x and Ti_5Si_3 hard phases (Figs. 1a-b) and also improve the interfacial bonding between Ti_3SiC_2 and TiC_x (Figs. 1c-f and Fig. 2). In addition, the $\text{TiC}_{0.4}$ addition causes the formation of the hard TiC_x phase around the Ti_3SiC_2 grain boundaries, decreasing the Ti_3SiC_2 grain size (Figs. 1c-e). The synergistic effect of these factors improves the mechanical and tribological properties of the $\text{TiC}_{0.4}$ - Ti_3SiC_2 composites (Figs. 3). However, it affects the tribological properties mainly through the formation of the lubricating film on the surface of the $\text{TiC}_{0.4}$ - Ti_3SiC_2 composites. As the formation of the lubricating film is in a dynamic balance with its loss during the friction process, the lubricating film is formed on the entire friction surface of the $\text{TiC}_{0.4}$ - Ti_3SiC_2 composites, stabilizing the friction coefficient (Fig. 4 and Table 1). The lubricating film integrity is improved by the pinning effect of TiC particles, decreasing the friction coefficient and wear rate. Still, the pinning effect of TiC particles is limited by the continuity of the lubricating film. If TiC_x particles in the $\text{TiC}_{0.4}$ - Ti_3SiC_2 composites are present in excess, the continuity of lubricating film will decrease although the pinning effect of TiC particles is improved, yielding an increase of friction coefficient (Fig. 3d, Fig. 4, and Table 1).

4. Conclusions

$\text{TiC}_{0.4}$ - Ti_3SiC_2 composites composed of Ti_3SiC_2 , Ti_5Si_3 , and TiC_x were manufactured using SPS at 1400°C for 10 min. The relative content of Ti_3SiC_2 decreased, while the relative contents of TiC_x and Ti_5Si_3 increased with the increase of the $\text{TiC}_{0.4}$ amount. TiC_x was concentrated around the grain boundaries of Ti_3SiC_2 . The $\text{TiC}_{0.4}$ addition improved the mechanical and tribological properties of the $\text{TiC}_{0.4}$ - Ti_3SiC_2 composites due to the presence of C vacancies in $\text{TiC}_{0.4}$. The Ti_3SiC_2 composite with 30 vol.% $\text{TiC}_{0.4}$ exhibited better comprehensive properties than other composites. The bulk density, relative density, hardness, fracture toughness, friction coefficient, and wear rate of the 30 vol. % $\text{TiC}_{0.4}$ - Ti_3SiC_2 composite were 4.63 g/cm^3 , 99.7%, 14.87 GPa, $5.45 \text{ MPa}\cdot\text{m}^{1/2}$, 0.74, and $4.32\times 10^{-7} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$, respectively.

This work provides a theoretical basis for studying other materials enhanced by the addition of nonstoichiometric compounds and broadens the application spectra of the Ti_3SiC_2 matrix composites under severe environments.

Declarations

Acknowledgments

This work was supported by the Key Program of Scientific Research of Higher Education of Hebei Province (Grant No. ZD2021099).

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Figures

Figure 1

(a) XRD patterns of $\text{TiC}_{0.4}$ and the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites; (b) relative content of each phase in the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites; FESEM micrographs of the cross-sectional microstructures of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites: (c) 10 vol.% $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$, (d) 20 vol.% $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$, and (e) 30 vol.% $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$; (f) Crack propagation in the 30 vol.% $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composite.

Figure 2

FESEM micrograph and the EDS line-scan analysis of the layered interface between $\text{TiC}_{0.4}$ and Ti_3SiC_2 sintered at 1400 $^{\circ}\text{C}$.

Figure 3

Mechanical and tribological properties of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites. (a) density, (b) hardness and fracture toughness, (c) an optical micrograph of the Vickers indentation at load of 500gf, (d) an optical micrograph of the Fractures indentation at a load of 5000gf, (e) friction coefficient and wear rate; (f) the comparison of the hardness and fracture toughness values between the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites and other Ti_3SiC_2 matrix composites reported in the literature.

Figure 4

Tribological properties of the $\text{TiC}_{0.4}\text{-Ti}_3\text{SiC}_2$ composites. (a) the relationship of friction coefficient and friction distance; FESEM micrographs of the friction surface of (b) TC0, (c) TC1 and (d) TC2.