

C_{0.3}N_{0.7}Ti-SiC toughed silicon nitride hybrids with non-oxide additives Ti₃SiC₂

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Abstract: *In-situ* grown C_{0.3}N_{0.7}Ti and SiC, which derived from non-oxide additives Ti₃SiC₂, are proposed to densify silicon nitride (Si₃N₄) ceramics with enhanced mechanical performance. Remarkable increase of density from 79.20% to 95.48% could be achieved for Si₃N₄ ceramics with 5vol% Ti₃SiC₂. The capillarity of decomposed Si from Ti₃SiC₂, and *in-situ* reaction between nonstoichiometric TiC_x and Si₃N₄ were believed to be responsible for densification of Si₃N₄ ceramics. An obvious enhancement of flexural strength and fracture toughness for Ti₃SiC₂ doped Si₃N₄ ceramics was observed. The maximum flexural strength of 795 MPa for Si₃N₄ composites with 5vol% Ti₃SiC₂ and maximum fracture toughness of 6.97 MPa·m^{1/2} for Si₃N₄ composites with 20vol% Ti₃SiC₂ are achieved when mixed powders are hot-press sintered at 1700 °C. Pull out of elongated Si₃N₄ grains, crack bridging, crack branching and crack deflection were demonstrated to dominate enhance fracture toughness of Si₃N₄ composites.

Keywords: Ti₃SiC₂; Si₃N₄; mechanical properties; fracture toughness

1. Introduction

Although a number of alternatives of structural ceramics have been proposed, silicon nitride (Si₃N₄)-based ceramics remain competitive due to their superior properties, involving high strength and hardness at elevated temperatures, high resistance to oxidation and chemical attack, low coefficient of tribological friction and thermal expansion, and low dielectric permittivity, etc. [1-9]. As important multifunctional materials, Si₃N₄ ceramics have found wide range of successful application towards gas turbine engine components [10-13], cutting tools [10, 14], radomes [2], and even integrated circuit [15, 16], optical devices [17, 18], etc.

However, due to the high degree of covalent bonding, Si₃N₄-based ceramics are very difficult to densify through the solid-state sintering process. Therefore, effective approaches to ensure rapid consolidation and high mechanical performance of Si₃N₄-based ceramics are actively being explored, including gas pressure sintering (GPS)^[10], hot-pressing sintering (HPS)^[19-25], hot isostatic pressing sintering (HIP)^[26], spark plasma sintering (SPS)^[25, 27, 28], and microwave sintering [1, 29], etc. However, considering the requirement of high gas pressures for gas pressure sintering and extra current devices for SPS with a significantly higher furnace costs, HP sintering allows the dense and complex-shaped parts with medium cost. Previous considerable efforts have demonstrated that fully dense Si₃N₄ ceramics with superior strength could be achieved through liquid phase sintering by addition of rare-earth oxides to promote mass transport and accelerate the rate of $\alpha - \beta$ transformation, most notably the rare-earth oxides involving Y₂O₃ [4, 30, 31]. A combination of various rare-earth oxides

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and other metallic oxides, such as Y_2O_3 , La_2O_3 , Nd_2O_3 , Sm_2O_3 , Yb_2O_3 , Lu_2O_3 , Al_2O_3 and MgO , also are effective sintering aids to densify Si_3N_4 [24, 29, 31-34].

Nevertheless, these oxides additives crystallized to intergranular glassy phase in the cooling stage [30], which deteriorate the high-temperature performance of the ceramics such as creep and high-temperature strength due to the relative low eutectic temperature [31, 35]. As a result of the early interest in hot-pressed Si_3N_4 ceramics as a high-temperature gas-turbine material, attention was directed to high-temperature strength and creep resistance. Therefore, it is quite essential to explore novel heat-resistant sintering aids for high-performance Si_3N_4 ceramics from new view point. The last two decades have been witness to the dramatic development on MAX phase cermets with the hexagonal symmetry due to their unique combination of characteristics of both ceramics and metals (M is an early transition metal, A is a group A element, X is either carbon and/or nitrogen), especially the layered ternary carbide titanium aluminum carbide (Ti_3AlC_2) and titanium silicon carbide (Ti_3SiC_2) [36-38]. The crystal structure of these MAX cermets can be described by alternately stacking of TiC_6 and Al/Si atomic planes. The unique combination of excellent properties of Ti_3AlC_2 or Ti_3SiC_2 , including high melting point, high hardness, high elastic modulus, good thermal and electrical conductivity, and considerable chemical stability, make them to be fascinating candidates for various application. Moreover, elemental metal powder-derived MAX materials have demonstrated to be effective reinforcement in TiB_2 [39-41], Al_2O_3 [42-44] composites with enhanced mechanical properties by *in situ* reaction. More recently, as explicated in our previous work [45-49], titanium aluminum carbide (Ti_3AlC_2) was chosen as an effective sintering aid to effectively densify B_4C ceramics with enhanced sintering ability and mechanical performance simultaneously. High hardness and toughness values of 28.5GPa and $7.02 MPa \cdot m^{1/2}$ respectively were achieved for B_4C composites sintered with 20vol% Ti_3AlC_2 at $1900^\circ C$. The mechanisms of the enhanced sinterability of high-performance ceramics in previous works could be classified into two aspects: Firstly, the decomposed metals from Ti_3SiC_2 or Ti_3AlC_2 at high temperature can form liquid phase which promote sintering effectively. Secondly, *in situ* reaction sintering between matrix and titanium carbon compound would also promote densification and mechanical performance. The main competitive advantage of MAX aids is considered to be formation of reaction bonding between Si_3N_4 matrix and aids, rather than intergranular glassy phase. Motivated by such an idea, these non-oxides cermets are highly expected to play a multifunctional role in densification and enhancement of mechanical properties of Si_3N_4 ceramics.

It is also noteworthy that Al decomposed from Ti_3AlC_2 and residual O originated from raw Si_3N_4 powders would be dissolved into Si_3N_4 grains during high-temperature sintering procedure, which is harmful to the purity and thermal performance of Si_3N_4 -based ceramics. As Y. Zhou illustrated [50], a tendency of decreasing fracture toughness with increasing Al dopant could be observed. Moreover, even the 0.4wt% concentration of Al would lead to a drastically reduce of thermal conductivity by 36.9% (from $91.9W \cdot m^{-1} \cdot K^{-1}$ to $58.0W \cdot m^{-1} \cdot K^{-1}$) for Si_3N_4 ceramics. Therefore in this work, Ti_3SiC_2 were introduced to densify Si_3N_4 ceramics in order to demonstrate that it provided any advantages over the rare-earth oxide system. Besides, the effect of

Ti₃SiC₂ volume fraction on the microstructure, hardness, flexural strength and fracture toughness was also studied. It is believed that Ti₃SiC₂ or other members of MAX family would lead to new scientific and technological data providing new insight into functionalization of Si₃N₄ ceramics.

2. Experimental procedure

2.1 Preparation of samples

Commercially available α -Si₃N₄ powder (purity>93%, d₅₀=0.7 μ m, Jinshenghao New Materials Co. Ltd., China) was used as a starting material. As a novel sintering aid, Ti₃SiC₂ powders (d₅₀=5 μ m, purity>98%) were kindly provided by Forsman Scientific Co., Ltd., Beijing, China. In order to investigate the effect of Ti₃SiC₂ content on the mechanical properties, experiments were conducted with various amounts of Ti₃SiC₂ powders (1 to 20 vol.%) embedded in α -Si₃N₄ powders. To ensure the homogeneity of the mixed powders, α -Si₃N₄-x vol.% Ti₃SiC₂ powders (x=1~20) were wet ball-milled for 10h by using ethanol as ball-milling media. The substance was dried at 80°C, and sieved with a filter with a mesh size of 63 μ m, then placed in a graphite die coated with BN powder to avoid reaction between the powder and graphite die. Hot-press (HP) sintering was performed with ramp of 10°C/min 1600°C and 1700°C for 90min in flowing nitrogen under 30MPa uniaxial pressure during the whole cycle. After natural cooling to room temperature inside furnace, samples were polished and ultrasonic cleaned before characterization. For comparison, α -Si₃N₄ powders with 2wt.% Alumina (Al₂O₃, AR, Sinopharm Chemical Reagent Co., Ltd., China) and 5wt.% yttria (Y₂O₃, AR, Sinopharm Chemical Reagent Co., Ltd., China) were hot-pressed at the same sintering condition.

2.2 Characterizations

The bulk density of each sample was determined according to the Archimedes principle in distilled water. XRD patterns were recorded on X'pert PRO (PANalytical B.V., Netherlands) for phase identification. The microstructures of polished surfaces and fracture surfaces were observed using scanning electron microscopy (SEM, Nova NanoSEM230) with an energy-dispersive X-ray (EDX) analyzer. The Vickers hardness was performed on micro hardness tester (VTD 512) under load of 9.8 N with a dwell time of 10 seconds, and determined by the Vickers diamond indentation method using the following equation:

$$H_v = 0.102 \frac{F}{S} = 0.102 \frac{2F \sin \frac{136^\circ}{2}}{d^2} = 0.1891 \frac{F}{d^2} \quad (1)$$

where P is the indentation load on the polished surface and d is the average diagonal length of the Vickers indentation. For accuracy, 11 Vickers indentations on each specimen were applied. After indentation, the microstructures were immediately observed by optical microscopy (ECLISPE LV150N, Japan). As a simple way of estimating toughness, indentation techniques were applied from observed corner cracks and calculated Vickers hardness using the Anstis equation:

$$K_{IC} = 0.016 \left(\frac{E}{H_v} \right)^{1/2} \left(\frac{P}{c^{3/2}} \right) \quad (2)$$

where E is the Young's modulus and c is the half-length of cracks formed by the indentation. Three-point flexural strength of specimens with size of 3mm×4mm×36mm

was performed on the mechanical testing machine (Instron3369, USA) at a cross head speed of 0.5mm/min.

3. Results and discussion

Fig. 1 illustrates the density of Ti_3SiC_2 filled Si_3N_4 ceramics as a function of Ti_3SiC_2 volume fraction. For Si_3N_4 ceramics which HP sintered at 1600°C without aids, the density is only 2.58 g cm^{-3} . Partial densification may be attributed to the residual SiO_2 liquid phase during firing at high temperature which always present on Si_3N_4 powder particles. A remarkable increase to 3.11 g cm^{-3} could be observed for Si_3N_4 ceramics filled with only 5vol% Ti_3SiC_2 when sintered at the same temperature. The enhanced density is even more noticeable than the Si_3N_4 ceramics with 7wt% $\text{Y}_2\text{O}_3\text{-Al}_2\text{O}_3$ aids. These observed results demonstrate Ti_3SiC_2 to be a effective sintering aid to densify Si_3N_4 ceramics. However, further increase in Ti_3SiC_2 content dose not bring any appreciable consolidation.

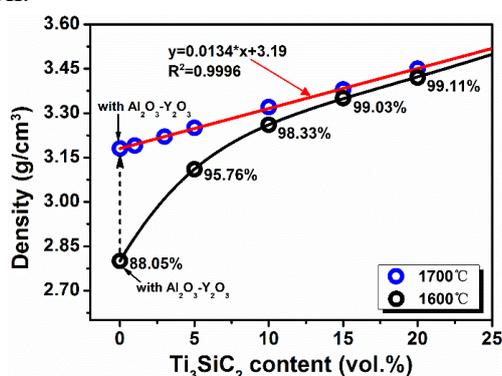


Fig. 1 Density of Si_3N_4 ceramics as a function of Ti_3SiC_2 content

As expected, higher sintering temperature 1700°C could further promote densification of Si_3N_4 ceramics filled with Ti_3SiC_2 . Besides, experimental points are inclined to distribute on a straight line with coefficient of determination (R^2) above 0.99, which is accordant with mix law. Due to the complex reaction between Si_3N_4 and Ti_3SiC_2 which will discuss later, it is difficult to determine the theoretical density of the $\text{Ti}_3\text{SiC}_2\text{-Si}_3\text{N}_4$ composites accurately. However, an approximation method could be applied by assuming a nearly full densification for 20vol% $\text{Ti}_3\text{SiC}_2\text{-Si}_3\text{N}_4$ ceramics sintered at 1700°C according to this linear behavior.

Fig. 2 shows the XRD patterns of Si_3N_4 ceramics filled with different volume fraction of Ti_3SiC_2 sintered at 1600°C and 1700°C , as well as 7wt% ($\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3$) densified Si_3N_4 ceramics. As seen in Fig. 2(a), both α and β phase of Si_3N_4 could be detected when sintering temperature is 1600°C , which suggests only a partial transformation of α phase to the more stable β phase. In contrast, when further improving sintering temperature to 1700°C , all diffraction peaks of $\alpha\text{-Si}_3\text{N}_4$ phase disappear (see in Fig. 2(b)). This completely transformation of α to $\beta\text{-Si}_3\text{N}_4$ phase is believed to be essential to the enhancement of densification and mechanical performance.

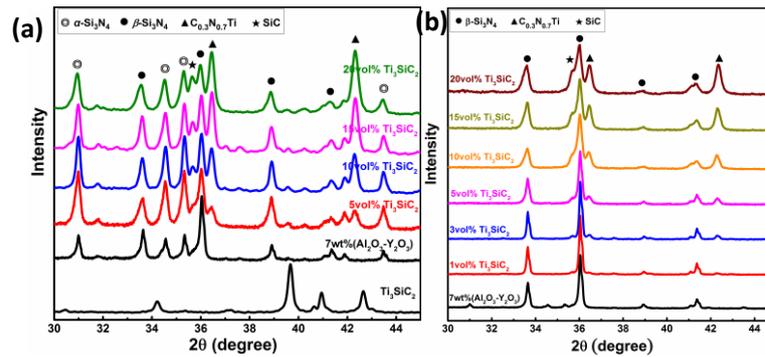


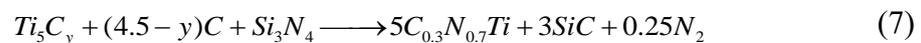
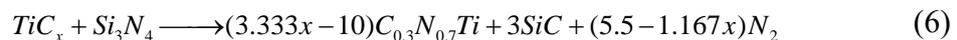
Fig. 2 XRD patterns of Ti_3SiC_2 doped Si_3N_4 ceramics sintered at (a) $1600^\circ C$ and (b) $1700^\circ C$. Another important feature should be noted here is that the characteristic diffraction peaks of the raw Ti_3SiC_2 powder nearly disappear completely after sintering. This could be ascribed to the fact that Ti_3SiC_2 powder is thermal stable up to $\sim 800^\circ C$, and the following reaction can be responsible for the decomposition of Ti_3SiC_2 [51]:



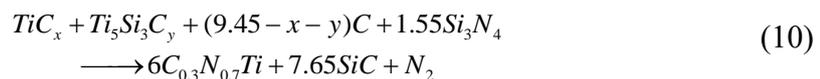
where the value of x ranges from 0.6 to 0.8 and $y \leq 1$. Besides, the TiC_x phase appears to result in more rapid deterioration of the Ti_3SiC_2 phase. Also noted that the decomposition usually accomplished with decomposition of Ti_3SiC_2 to form nonstoichiometric TiC_x and gaseous Si, as demonstrated previously [52]:



The Si is believed to be act as lubricating phase between Si_3N_4 grains to promote densification of Si_3N_4 ceramics through capillarity. Meanwhile, the residual Si would further react with nitrogen to form Si_3N_4 . On the other hand, further heating during insulation stage will result in the likely loss of gaseous silicon. Furthermore, the decomposition products of Ti_3SiC_2 would further react with Si_3N_4 through diffusion of C and N according to the following reactions:



or



It is reasonable to claim that the *in situ* reaction is responsible for the additional characteristic diffraction peaks of $C_{0.3}N_{0.7}Ti$ and SiC in XRD patterns.

Fig.3 shows the micro-morphology of polished surface of Si_3N_4 with different volume fractions of Ti_3SiC_2 sintered at $1700^\circ C$. Due to the lack of sufficient sintering aids, lots of pores could be observed, and grain growth of β - Si_3N_4 is not complete for monolithic Si_3N_4 ceramic (see Fig.3(a)). However, the microstructures of Ti_3SiC_2 - Si_3N_4 ceramics (see Fig.3(b) to Fig.3(g)) exhibit much more close-grain

structure, and consist of randomly oriented elongated Si_3N_4 grains which is accordant with XRD results in Fig.2. The average diameters of grains present slight increasing trend from $0.6771 \mu\text{m}$ to $0.9802 \mu\text{m}$ by quantitative image analysis as the amount of Ti_3SiC_2 increased. Besides, the bright contrasted phase which uniformly embedded in Si_3N_4 matrix could be observed, and are inclined to aggregate especially when Ti_3SiC_2 content exceeds 15vol%. Furthermore, as shown in Table 1, energy dispersive spectrometer (EDS) at spot A in Fig. 3(e) suggests dominant phase of Si_3N_4 and SiC , which is associated with reaction described by Eq.(10). Additional O element may be originated from surface of raw $\alpha\text{-Si}_3\text{N}_4$ powders. Meanwhile, the bright region at spot B is proved to be enriched by Ti according to the EDS results in Table 1. Combined with the results of XRD analysis, it is reasonable to claim that the dispersive bright regions consist of $\text{C}_{0.3}\text{N}_{0.7}\text{Ti}$ and SiC , which are believed to affect the mechanical performance of reaction bonded Si_3N_4 ceramics.

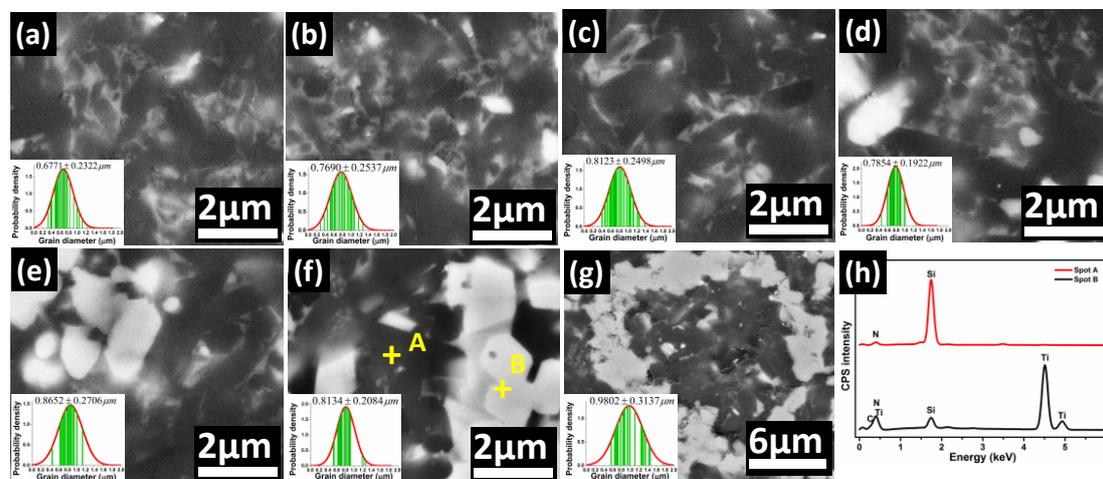


Fig. 3 Polished surface of Si_3N_4 ceramics with different content of Ti_3SiC_2 sintered at 1700°C : (a) 5wt% Y_2O_3 -2wt% Al_2O_3 , (b) 1vol% Ti_3SiC_2 , (c) 3vol% Ti_3SiC_2 , (d) 5vol% Ti_3SiC_2 , (e) 10vol% Ti_3SiC_2 , (f) 15vol% Ti_3SiC_2 , (g) 20vol% Ti_3SiC_2 , (h) EDS spectra at spot A and B
Table 1 EDS chemical analysis (at.%) at different positions in Fig. 3(g)

	Si	N	O	Ti	C	Possible phases
Spot A	52.21	35.43	2.04	1.29	9.03	Si_3N_4 , SiC
Spot B	5.10	27.18	-	50.13	17.59	$\text{C}_{0.3}\text{N}_{0.7}\text{Ti}$, SiC

The mechanical properties, including Vickers hardness, flexural strength and fracture toughness, of dense Si_3N_4 ceramics with different Ti_3SiC_2 content sintered at 1700°C are illustrated in Fig. 4. Clearly, the Vickers hardness of Si_3N_4 ceramics has been upgraded after modification of Ti_3SiC_2 , and presents slight increase compared with that of Si_3N_4 ceramics containing conventional oxides aids. Besides, an obvious enhancement of flexural strength and fracture toughness could be observed. A maximum flexural strength of 795 MPa could be achieved for 5vol% Ti_3SiC_2 doped Si_3N_4 composites, which is almost twice that of 7wt% ($\text{Y}_2\text{O}_3\text{-Al}_2\text{O}_3$)- Si_3N_4 ceramics prepared at the same condition. This enhancement of flexural strength could be attributed to the $\text{C}_{0.3}\text{N}_{0.7}\text{Ti}$ and SiC which originated from reaction bonding between Ti_3SiC_2 and Si_3N_4 . However, further increment of Ti_3SiC_2 content reduces the flexural strength of Si_3N_4 ceramics which may be ascribed to the enhanced residual stresses around grain boundary. Note that this residual stress is believed to result in

microcracks and intergranular fracture mode which will be discussed later. Moreover, the fracture toughness of Si_3N_4 composites is also effectively boosted after Ti_3SiC_2 decoration, and reaches maximum value of $6.97 \text{ MPa}\cdot\text{m}^{1/2}$ for 20vol% Ti_3SiC_2 - Si_3N_4 ceramics which is 37% higher than that of 7wt% (Y_2O_3 - Al_2O_3)- Si_3N_4 ceramics.

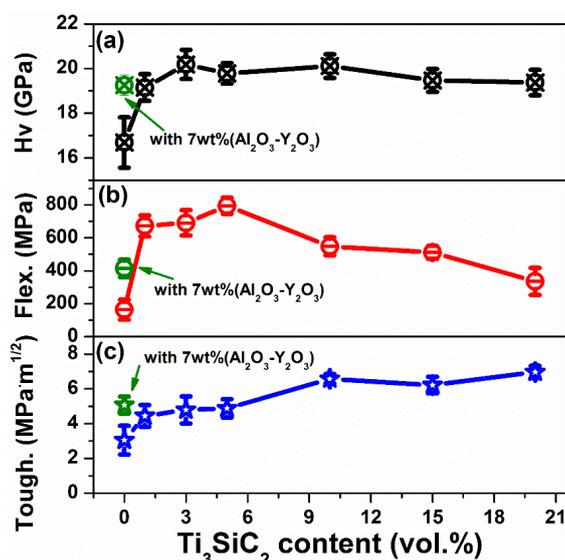


Fig. 4 Mechanical properties of Ti_3SiC_2 doped Si_3N_4 ceramics: (a) Vickers hardness, (b) flexural strength and fracture toughness

Fig. 5 illustrates the typical optical micrographs of the Vickers hardness indents and the induced cracks of Si_3N_4 ceramics with different Ti_3SiC_2 contents, as well as 7wt% (Y_2O_3 - Al_2O_3). Clearly, the polished surfaces of Ti_3SiC_2 doped Si_3N_4 ceramics become much smoother than the monolithic Si_3N_4 ceramic which HP sintered at 1700°C , corresponding to the enhancement of densification. Besides, it can be seen that the area of indentation presents no obvious change for Ti_3SiC_2 doped Si_3N_4 ceramics, which is consistent with the stable Vickers hardness. However, the cracks obviously become shorter especially when the Ti_3SiC_2 contents exceed 10vol%, which is responsible for the enhancement of fracture toughness.

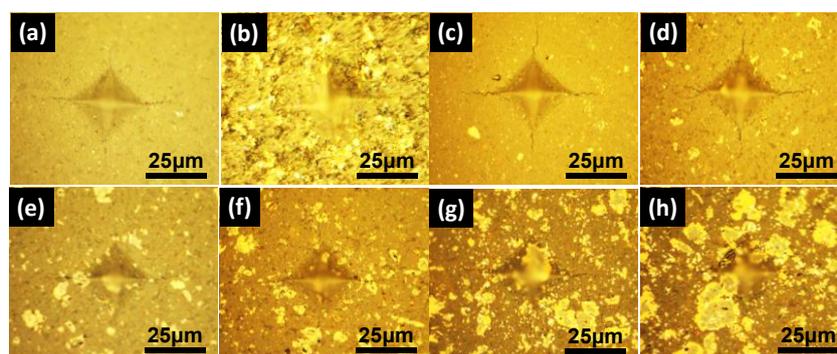


Fig. 5 Optical micrographs of the Vickers hardness indents and the induced cracks in (a) Si_3N_4 -7wt% (Al_2O_3 - Y_2O_3), (b) Si_3N_4 -3vol% Ti_3SiC_2 , (c) Si_3N_4 -5vol% Ti_3SiC_2 , (d) Si_3N_4 -10vol% Ti_3SiC_2 , (e) Si_3N_4 -15vol% Ti_3SiC_2 , (f) Si_3N_4 -20vol% Ti_3SiC_2

To illustrate the fracture behaviors and activated toughening mechanisms, micromorphology and crack paths are investigated on cross-sectional fracture surfaces and polished surfaces, respectively. Comparison of typical fracture surfaces between

Si_3N_4 doped with $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3$ and Ti_3SiC_2 is illustrated in Fig. 6. As can be seen from Fig. 6(a), a small amount of pores occur in the $\text{Si}_3\text{N}_4\text{-7wt}\%$ ($\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3$) composites, which is harmful for the mechanical performances. In contrast, the $\text{Si}_3\text{N}_4\text{-Ti}_3\text{SiC}_2$ specimen presents a much more close-grain fracture surface owing to the higher density. As marked by red arrows in Fig. 6(b), large amounts of dimples corresponding to the transgranular fracture could be observed. And this fracture mode is considered to make a dominate contribution to the superior flexural strength of $\text{Si}_3\text{N}_4\text{-Ti}_3\text{SiC}_2$ composites. Besides, as marked by yellow arrows, lots of interface debonding between the Si_3N_4 grains and grain boundary phase could be observed. This intergranular fracture mode may result from the pullout of elongated $\beta\text{-Si}_3\text{N}_4$ grains, which is believed to make a contribution to the enhancement of overall fracture toughness.

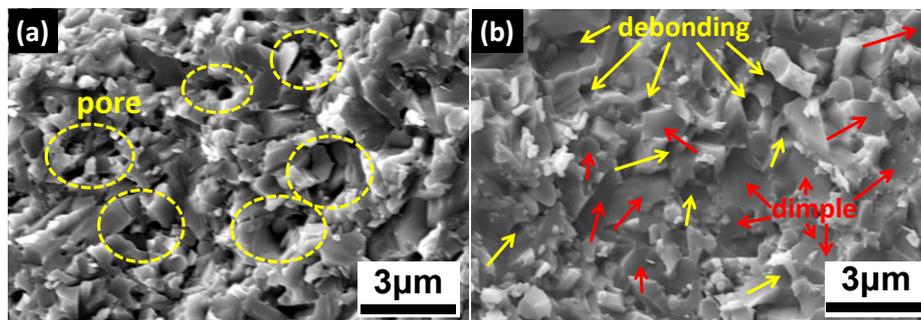


Fig. 6 Typical fracture surfaces of Si_3N_4 with (a) 7wt% ($\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3$), (b) 10vol% Ti_3SiC_2

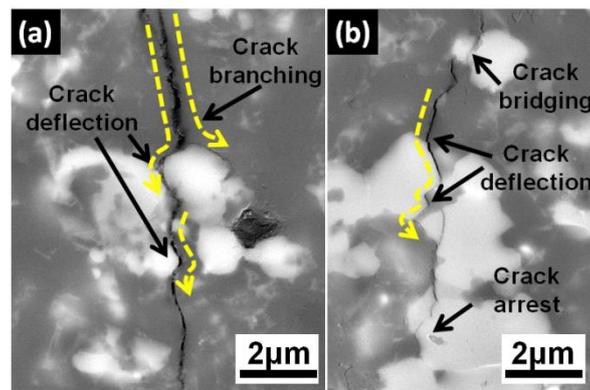


Fig. 7 Typical SEM images of crack deflection in Ti_3SiC_2 densified Si_3N_4 composites

Another mechanism of the enhanced fracture toughness of $\text{Si}_3\text{N}_4\text{-Ti}_3\text{SiC}_2$ composites could be ascribed to the crack branching, deflection and grain bridging by *in-situ* derived $\text{C}_{0.3}\text{N}_{0.7}\text{Ti}$ and SiC grains embedded in Si_3N_4 matrix, which illustrated in Fig. 7. Due to the superior hardness of $\text{C}_{0.3}\text{N}_{0.7}\text{Ti}$ and the thermal mismatch between Si_3N_4 and $\text{C}_{0.3}\text{N}_{0.7}\text{Ti}$, there exists residual stress around $\text{C}_{0.3}\text{N}_{0.7}\text{Ti}$ grains during cooling, giving rise to the microcracks inside composites. When subjected to the external mechanical stress, these microcracks tend to be activated and the propagation path of cracks tends to be splitted by $\text{C}_{0.3}\text{N}_{0.7}\text{Ti}$ hard-phase and deflected along the interface. Such mechanisms consumed more fracture energy during the crack propagation which leads to crack arrest.

A comparison of mechanical properties of Si_3N_4 -based ceramics obtained in the present work and selected previous works with conventional oxide aids is shown in Table 2.

Clearly, the Vickers, flexural strength and toughness of Ti_3SiC_2 doped Si_3N_4 ceramics present the same level or even better compared with Si_3N_4 ceramics sintered with oxide aids. Moreover, due to the superior mechanical and thermal properties of *in situ* formed $\text{C}_{0.3}\text{N}_{0.7}\text{Ti}$ and SiC , Si_3N_4 ceramics obtained in this work are believed to have a significant competitive advantage and to promote the development of Si_3N_4 -based ceramics at high temperatures.

Table 2 Selected results on mechanical properties of Si_3N_4 ceramics by pressure-assisted sintering

Composition	Sintering conditions	Vickers hardness (GPa)	Flexural strength (MPa)	Fracture toughness (MPa.m ^{1/2})	Ref.
α - Si_3N_4 +4wt% Al_2O_3 +6wt% Y_2O_3	Hot isostatic pressing at 1700 °C, 20MPa, 3h	16.4	730	6.5	[26]
α - Si_3N_4 +5wt% Al_2O_3 +5wt% Y_2O_3	Hot press at 1800 °C, 30MPa, 1.5h	16.1	-	5.2	[19]
α - Si_3N_4 +4wt% Al_2O_3 +6wt% Y_2O_3	Hot press at 1700 °C, 50MPa, 1.5h	17.01	-	-	[20]
α - Si_3N_4 +30vol% β - Si_3N_4 whiskers+5wt% Al_2O_3 +5wt% Y_2O_3 +5wt% CeO_2	Hot press at 1700 °C, 30MPa, 30min	19.0	794	8.6	[21]
α - Si_3N_4 +4wt% Al_2O_3 +4wt% Y_2O_3 +15vol% SiC whiskers	Hot press at 1800 °C, 30MPa, 30min	16	680	6.1	[53]
α - Si_3N_4 +5wt% Al_2O_3 +4wt% Y_2O_3 +3wt% TiC	Gas pressure sintering at 1750 °C, 2MPa	16.4	475	7.6	[10]
α - Si_3N_4 +5vol% Ti_3SiC_2	Hot-pressed at 1700 °C for 90min	19.78	795	4.88	This work
α - Si_3N_4 +20vol% Ti_3SiC_2	Hot-pressed at 1700 °C for 90min	20.11	549	6.58	This work

4. Conclusions

In summary, we proposed non-oxide Ti_3SiC_2 (one of typical MAX cermets) as a novel sintering aid to densify Si_3N_4 ceramics with enhanced mechanical properties. A remarkable relative density increment of 20.5% (from 2.58 to 3.11 g·cm⁻³) could be observed for 1600 °C hot-press sintered Si_3N_4 ceramics doped with only 5vol% Ti_3SiC_2 compared with Si_3N_4 ceramics without aids. Further increase in sintering temperature to 1700 °C brought appreciable consolidation of nearly full dense Ti_3SiC_2 - Si_3N_4 ceramics. XRD and EDS investigations demonstrated the formation of $\text{C}_{0.3}\text{N}_{0.7}\text{Ti}$ and SiC which resulted from *in-situ* reaction between Ti_3SiC_2 and Si_3N_4 through diffusion of C and N. The Vickers hardness of Ti_3SiC_2 doped Si_3N_4 ceramics increased slightly compared with that of Si_3N_4 ceramics containing conventional oxides aids. Nevertheless, an obvious enhancement of flexural strength and fracture toughness could be observed. A maximum flexural strength of 795 MPa could be obtained for 5vol% Ti_3SiC_2 doped Si_3N_4 composites. Moreover, the fracture toughness of Ti_3SiC_2 densified Si_3N_4 composites exhibited a remarkable increase with increasing in volume fraction, and reached maximum value of 6.97 MPa·m^{1/2} for 20vol% Ti_3SiC_2 - Si_3N_4 ceramics. Pull out of elongated Si_3N_4 grains, crack bridging and deflection were demonstrated to promote fracture toughness of Ti_3SiC_2 densified Si_3N_4 composites. With these successes, MAX phase densified Si_3N_4 ceramics with enhanced strength and toughness will be necessary to meet demands of potential future markets for

advanced ceramics. Further efforts are encouraged to be devoted to thermal properties investigations of MAX enabled Si₃N₄ composites.

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