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Ceramics International 29 (2003) 539–542

CERAMICS INTERNATIONAL

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# $Ti_3SiC_2/Al_2O_3$ composites prepared by SPS

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Received 8 May 2002; received in revised form 7 August 2002; accepted 10 September 2002

**Abstract**

The $Ti_3SiC_2/Al_2O_3$ composite has been prepared successfully by spark plasma sintering (SPS). The effects of sintering temperature and soaking time on the properties of $Ti_3SiC_2/Al_2O_3$ composite were studied. The results showed that the suitable sintering temperature was 1300 °C, and the soaking time was not the main factor which effected the properties of $Ti_3SiC_2/Al_2O_3$ composite. The highest flexural strength is about 600 MPa, the highest fracture toughness is about 7.4 MPa·$m^{1/2}$.


*Keywords:* C. Mechanical properties; $Ti_3SiC_2/Al_2O_3$ composite; SPS

## 1. Introduction

$Ti_3SiC_2$ is a remarkable material for its combination of properties such as high toughness, high fatigue-crack growth threshold, high Young's modulus, moderate flexural strength, plasticity at high temperature, good electrical conductivity, excellent chemical resistance, high thermal shock resistance, easy machinability and so on [1–5]. It is expected, therefore, to apply $Ti_3SiC_2$ to various tough components such as commutating brushes for motors, armor, bearings and turbine blades. However, its hardness is very low [6,7], which limits the application of this kind of material. So it is necessary to improve the wear resistance.

The inclusion of $Al_2O_3$ in $Ti_3SiC_2$ may enhance the hardness, wear resistance and mechanical performance of $Ti_3SiC_2$. The purpose of this paper is to synthesize the $Ti_3SiC_2/Al_2O_3$ composite directly from the raw powders of $Ti/TiC/Si/Al_2O_3$ by spark plasma sintering.

## 2. Experimental procedure

This work was conducted using high-purity powder mixtures of titanium (45 μm, Grade TSPT-350, Sumitomo Sitix of Amagasaki, Inc, Japan), TiC (1.36 μm, New metal Co, LTD, Japan), Si (purity > 99.99%, 200 mesh, High purity Chemical Co,LTD, Japan), $Al_2O_3$ (AKP-30, Sumitomo Chemical Co. Japan) to yield a final composition with the appropriate stoichiometry. In this experiment, 10 vol.%$Al_2O_3$/90 vol.% $Ti_3SiC_2$ (corresponding to 22.5 wt.%Ti, 56.2 wt.%TiC, 13.1 wt.%Si and 8.2 wt.% $Al_2O_3$) was selected.

The powders were carefully weighted and poured into a graphite die (the inner diameter is 30 mm). Then the green samples were hot pressed in the spark plasma sintering system at 40 MPa in vacuum. The pulse duration was 3.3 ms, and one pulse sequence contained 12 pulses, while the interval between pulse sequence was 6.6 ms. The pulse-sequence current averaged 5000 A. The heating rate is 100 K/min, and the sintering temperature was 1200–1400 °C. The soaking time was changed from 5 to 15 min.

After sintering, the surfaces of the samples were machined to remove the layer contained by the carbon sheet, using a fine grit, high speed diamond wheel.

The sintered products were characterized by X-ray diffraction (D/MAX-γA) to determine the phase compositions and relative-$Ti_3SiC_2$ content. Density and porosity were measured by the Archimedes method.

Vickers hardness was measured at a load of 98 N for 15 s. The surface of a sintered body was polished with diamond paste. The ultra-micro Vickers hardness data were controlled by UMIS (Ultra-Micro Indentation System)-2000 system.

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0272-8842/03/$30.00 
PII: S0272-8842(02)00199-2

Specimens for flexural strength and $K_{IC}$ tests were cut and ground. The three-point bending flexural strength was measured on Instron-1195 machine with cross head speed of 0.5 mm/min and span of 18 mm. Fracture toughness measurement was conducted by the single-edge notched beam (SENB) method with the specimen dimensions of 3×4×20 mm. The pre-crack was introduced by indentation in the middle of the specimen, using an Instron 1195 machine. The microstructure and fracture surface were observed by using EPMA (Jeol, JXA-8600).

## 3. Results and discussion

Fig. 1 shows the effects of sintering temperature on the density and the hardness of samples soaked for 5 min. It can be seen that the good densification is obtained at ~1300–1350 °C. But the hardness reaches the maximum value (8.18 GPa) at 1300 °C and then decreases somewhat as the sintering temperature increases. These results reveal that the spark plasma-sintering system can sinter the $Ti_3SiC_2/Al_2O_3$ composite very fast. The effects of sintering temperature on the flexural strength and $K_{IC}$ are presented in Fig. 2. The results show that the $K_{IC}$ is not changed largely when the sintering temperature is higher than 1300 °C, but the flexural strength is decreased when the temperature increased beyond 1300 °C.

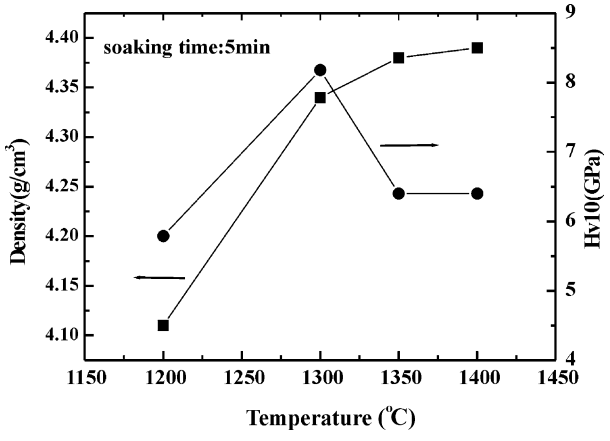


Fig. 1. The dependences of the density and the hardness on sintering temperature.

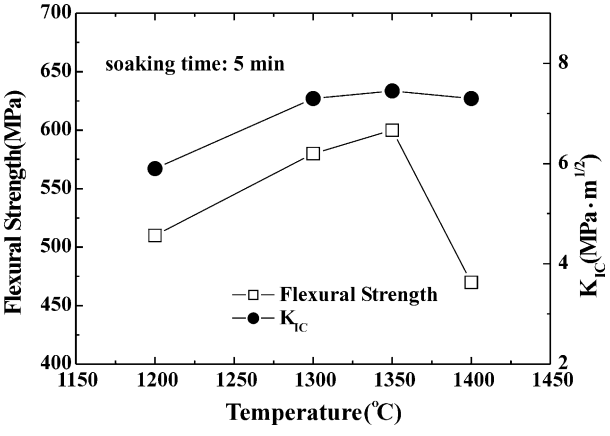


Fig. 2. The dependences of the flexural strength and $K_{IC}$ on sintering temperature.

X-ray diffraction patterns of the spark plasma sintered $Ti_3SiC_2/Al_2O_3$ composite are shown in Fig. 3. Three different phases of $Ti_3SiC_2$,$TiC_x$ and $Al_2O_3$ are identified. For samples sintered at 1350 °C and 40 MPa for 5min, the main peak intensity of $TiC_x$ is the weakest compared with the peak intensity of $Ti_3SiC_2$. When the sintering temperature is higher than 1350 °C, the volume content of $Ti_3SiC_2$ drops substantially, which maybe caused by the decomposition of $Ti_3SiC_2$ at higher temperatures. And at the same time, the open porosity increase, which caused the strength decrease at high temperature.

From the fracture surface (Fig. 4), the $Ti_3SiC_2$ grains, which shape is like plate, can be distinguished easily [Fig. 4(a) and 4(b)]. The grain size of $Ti_3SiC_2$ grains increases with the temperature increasing. Fig. 4(c) and (d) are Al distribution images which indicated the dispersion of $Al_2O_3$ in the composite, corresponds with Fig. 4(a) and (b). From Fig. 4(c) and 4(d), the accumu-

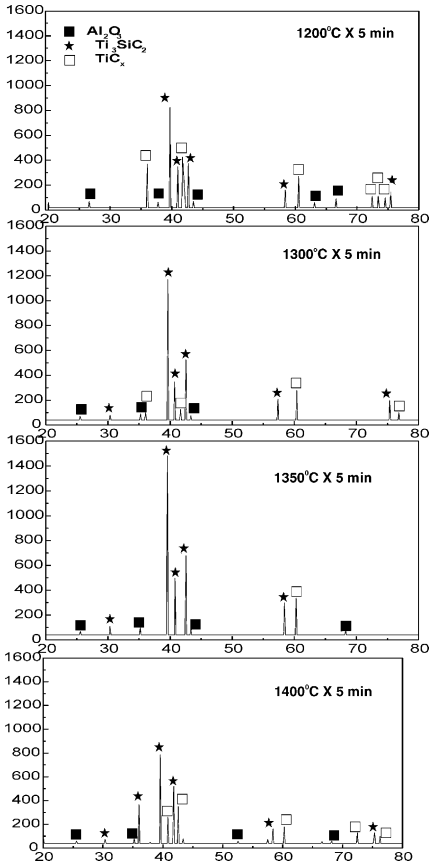


Fig. 3. X-ray diffraction patterns of the $Ti_3SiC_2/Al_2O_3$ composite.

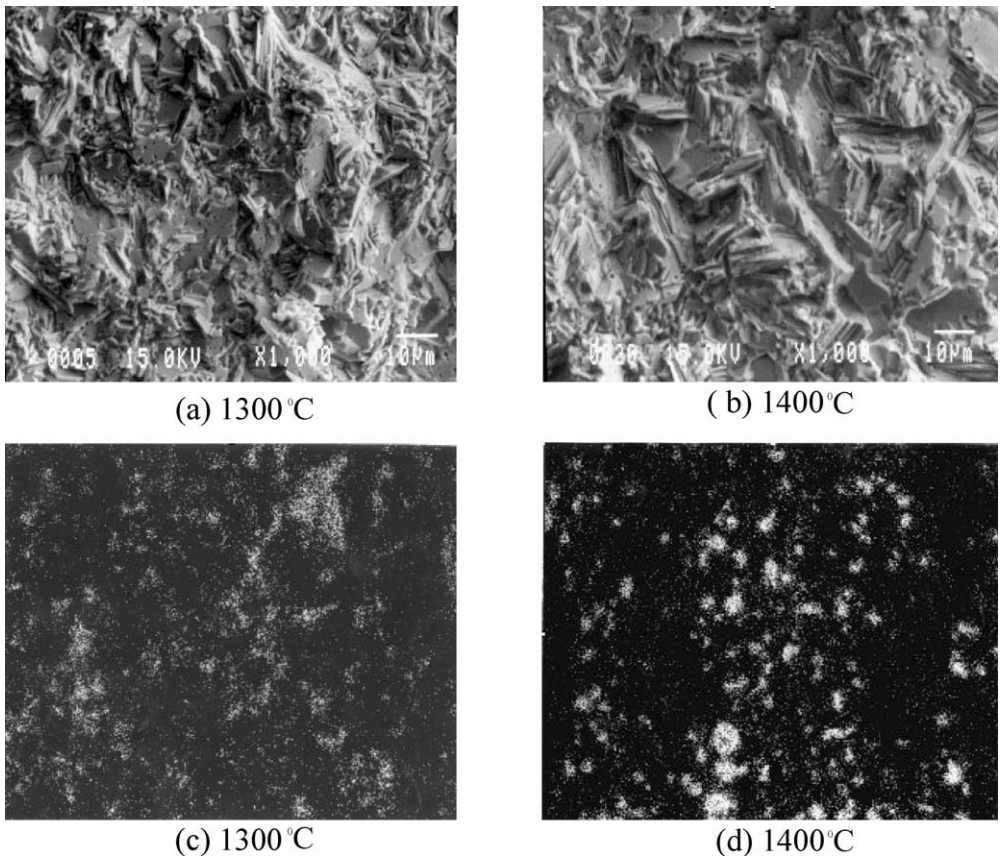


Fig. 4. Fracture surface and distribution of $Al_2O_3$ particles.

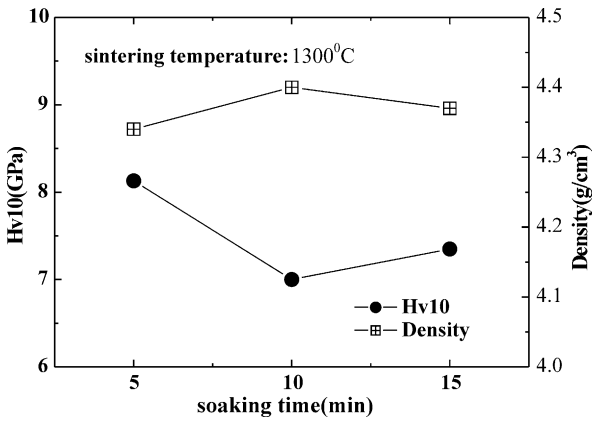


Fig. 5. The effects of the soaking time on the density and the hardness.

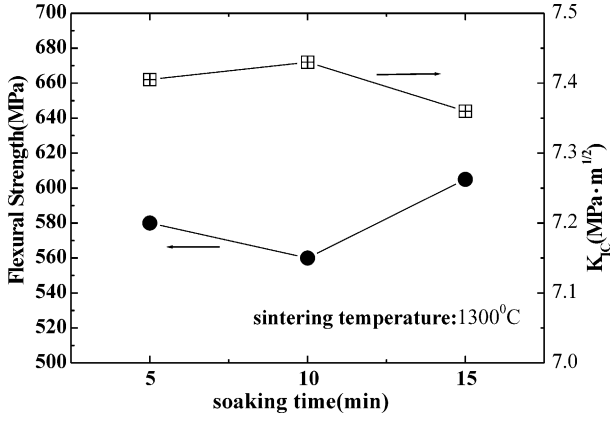


Fig. 6. The effects of the soaking time on the flexural strength and the fracture toughness.

lation of $Al_2O_3$ can be seen in samples sintered at 1400 °C which is caused by the partly sintering of $Al_2O_3$, and the larger $Ti_3SiC_2$ grains were seen at 1400 °C. So the flexural strength decreases. But the $K_{IC}$ is not changed obviously because of the existence of the plate-like $Ti_3SiC_2$ grains.

Fig. 5 shows the effects of the soaking time on the density and the hardness of samples sintered at 1300 °C. The density reaches the highest value when the soaking time is 10 min, and then decreases as the soaking time increases. However, the hardness decreases to the lowest when the soaking time is longer than 10 min. According to the literature [7], the hardness of $Ti_3SiC_2$ is lower than that of TiC or $Ti_5Si_3$. So the lowest value of the hardness of composites is obtained as it is soaked for 10 min because the content of $Ti_3SiC_2$ reaches the highest.

The effects of the soaking time on the flexural strength and the fracture toughness are shown in Fig. 6. The values of strength and $K_{IC}$ are not changed obviously. It can be concluded that the soaking time is not the main factor which effects the properties of $Ti_3SiC_2/Al_2O_3$ composites.

## 4. Conclusions

1. The dense $Ti_3SiC_2/Al_2O_3$ composite can be fabricated by spark plasma-sintering system successfully.
2. The suitable sintering temperature is $\sim$1300 °C, at which point, the highest flexural strength is about 600 MPa, and the highest fracture toughness is about 7.4 $MPa \cdot m^{1/2}$.
3. The soaking time is not the main factor which effects the properties of $Ti_3SiC_2/Al_2O_3$ composites.

## References

[1] E.R. Tamer, M.W. Barsoum, Processing and mechanical properties of $Ti_3SiC_2$: I, Reaction path and microstructure evolution, J. Am. Ceram. Soc. 82 (10) (1999) 2849–2854.
[2] E.R. Tamer, M.W. Barsoum, T. El-Raghy, Processing and mechanical properties of $Ti_3SiC_2$: II, Effect of grain size and deformation temperature, J. Am. Ceram. Soc. 82 (10) (1999) 2855–2860.
[3] M. Radoric, M.W. Barsoum, T. El-Raghy, J. Seidensticker, Tensile properties of $Ti_3SiC_2$ in the 25–1300 °C temperature range, Acta Mater. 48 (2000) 453–459.
[4] M.W. Barsoum, T. El-Raghy, Synthesis and characterization of a remarkable ceramic: $Ti_3SiC_2$, J. Am. Ceram. Soc. 79 (7) (1996) 1953–1956.
[5] M.W. Barsoum, T. El-Raghy, M. Radoric, Oxidation of $Ti_3SiC_2$ in air, J Electrochem. Soc. 144 (1997) 2508–2516.
[6] I.M. Low, Vickers contact damage of micro-layered $Ti_3SiC_2$, J. Eur. Ceram. Soc. 18 (1998) 709–713.
[7] T. El-Raghy, A. Zaraliangos, Damage mechanisms around hardness indentations in $Ti_3SiC_2$, J. Am. Ceram. Soc. 80 (3) (1997) 513–516.