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# Effect of Al<sub>2</sub>O<sub>3</sub> on mechanical properties of Ti<sub>3</sub>SiC<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> composite

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#### Abstract

The effect of  $Al_2O_3$  on mechanical properties of  $Ti_3SiC_2/Al_2O_3$  composite fabricated by SPS was studied systematically. The results show that the hardness of the  $Ti_3SiC_2/Al_2O_3$  composite can reach 10.28 GPa, 50% higher than that of pure  $Ti_3SiC_2$ . However, slight decrease in the other mechanical properties was observed with  $Al_2O_3$  addition higher than 5–10 vol.%, which is believed to be due to the agglomeration of  $Al_2O_3$  in the composite.

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# 1. Introduction

Ti<sub>3</sub>SiC<sub>2</sub> 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, etc. [1–5]. It is expected, therefore, to apply Ti<sub>3</sub>SiC<sub>2</sub> to various tough components such as commutating brushes for motors, armor, bearing and turbine blades. However, its relatively low hardness (4–5 Gpa) [6,7], leads to poor wear resistance, which limits its application as a structural component.

It has been shown that the addition of  $Al_2O_3$  can increase the hardness of some silicon-based composites. This motivated the study in this paper to fabricate the  $Ti_3SiC_2/Al_2O_3$  composite directly from raw powders of  $Ti/TiC/Si/Al_2O_3$  using the spark plasma sintering (SPS) technique. The mechanical properties of the composites with different  $Al_2O_3$  content were compared, assisted with the microstrutural observation on the fracture surface.

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# 2. Experimental procedure

High-purity powders of titanium (45  $\mu$ m, Grade TSPT-350, Sumitomo Sitix of Amagasaki, Inc, Japan), TiC (1.36  $\mu$ m, New metal Co, Ltd, Japan), Si (purity>99.99%, 200 mesh, High purity Chemical Co, Ltd, Japan) and Al<sub>2</sub>O<sub>3</sub> (AKP-30, Sumitomo Chemical Co. Japan) were mixed to yield a final composition with the appropriate stoichiometry, as shown in Table 1.

The powder mixtures were pressed in a graphite die with 30 mm inner diameter. Then the green compacts were hot pressed in the SPS system at 40 MPa in vacuum. The sintering temperature was  $1200-1400~^{\circ}$ C, with soaking time ranging from 5 to 15 min and a heating rate of 100~K/min.

The surfaces of the sintered samples were machined to remove the layer contaminated by the carbon sheet, using a fine grit, high speed diamond wheel.

X-ray diffraction (XRD) was used to determine the phase composition and relative Ti<sub>3</sub>SiC<sub>2</sub> content in the composite. The sample density and porosity were measured by Archimedes' method. Samples for Vickers hardness measurement were polished with diamond paste. An Ultra-Micro Indentation System (UMIS-2000) was used for the measurement with 15 s holding time at a load of 98 N.

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Table 1
The volume content of Al<sub>2</sub>O<sub>3</sub> in the designed compositions

No.	Ti <sub>3</sub> SiC <sub>2</sub> (vol.%)	Al <sub>2</sub> O <sub>3</sub> (vol.%)
1	95	5
2	90	10
3	80	20

Specimens for flexural strength and  $K_{\rm IC}$  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. The fracture toughness measurement was conducted using the single-edge notched beam (SENB) method with the specimen dimensions of  $3\times4\times20$  mm. The pre-crack was introduced by indentation in the middle of the specimen on an Instron 1195 machine. The microstructure and fracture surface of selected samples were observed by SEM (Jeol, made in Japan) and EPMA (JXA-8600, made in Japan).

#### 3. Results and discussion

# 3.1. Effect of $Al_2O_3$ on properties of $Ti_3SiC_2/Al_2O_3$

Fig. 1 shows the density and hardness of the Ti<sub>3</sub>SiC<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> composites as a function of the Al<sub>2</sub>O<sub>3</sub> volume content. All the samples were sintered at 1300 °C for 10 min. The density does not show much variance for Al<sub>2</sub>O<sub>3</sub> content between 0 and 20 vol.%. However, a significant increase in hardness is observed when the Al<sub>2</sub>O<sub>3</sub> content exceeds 10%. The Ti<sub>3</sub>SiC<sub>2</sub>/20 vol. % Al<sub>2</sub>O<sub>3</sub> composite shows a hardness value of 10.29 Gpa, which is 50% higher than that of the pure Ti<sub>3</sub>SiC<sub>2</sub> (6.69 GPa). Fig. 2 shows the Ti<sub>3</sub>SiC<sub>2</sub> phase content as a function of Al<sub>2</sub>O<sub>3</sub> content sintered at 1300°c for 10 min. When the Al<sub>2</sub>O<sub>3</sub> content increases from 0 to 10 vol.%, the

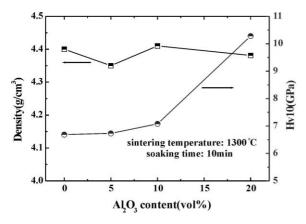


Fig. 1. The effect of  $Al_2O_3$  on the density and hardness of  $Ti_3SiC_2/Al_2O_3$  composite.

volume content of  $Ti_3SiC_2$  also increases from 92 to 96%. The content of  $Ti_3SiC_2$  gradually decreases when the  $Al_2O_3$  content exceeds 10 vol.%.

Fig. 3 shows the effect of  $Al_2O_3$  content on the flexural strength and fracture toughness of the composite sintered at 1300 °C for 10 min under 40 Mpa pressure. A gradual increase in  $\sigma_b$  and  $K_{IC}$  is observed for  $Al_2O_3$  lower than 5–10 vol.%. Further increase of  $Al_2O_3$  content slightly decreases the composite strength, yet the toughness almost remains constant.

## 3.2. Microstructure observation

Fig. 4 shows the microstructure of some selected  ${\rm Ti_3SiC_2/Al_2O_3}$  specimens sintered at 1200 °C for 5 min. The  ${\rm Al_2O_3}$  grains are uniformly distributed in the grain boundary [Fig. 4(e),(f)]. The pure  ${\rm Ti_3SiC_2}$  sample shows an average grain size of 10 µm. With increasing  ${\rm Al_2O_3}$  content, a finer microstructure is obtained with decreasing grain size. For the  ${\rm Ti_3SiC_2/10\%Al_2O_3}$  composite, the average grain size is only 1 µm, which could

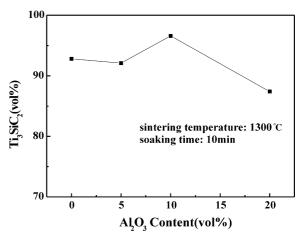


Fig. 2. The effect of  $Al_2O_3$  on the  $Ti_3SiC_2$  content of  $Ti_3SiC_2/Al_2O_3$  composite.

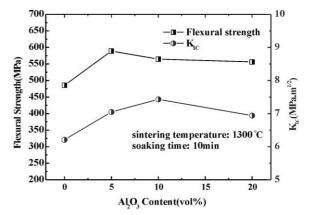


Fig. 3. The effect of  $Al_2O_3$  on the flexural strength and fracture toughness of  $Ti_3SiC_2/Al_2O_3$  composite.

be responsible for the slight increase in  $\sigma_b$  and  $K_{IC}$ . Such microstructural evolution can be understood if it is postulated that the  $Al_2O_3$  grains inhibit grain growth and the grain-boundary mobilities. But when the  $Al_2O_3$  content exceeds 10vol%, the agglomeration of the  $Al_2O_3$  grains is observed [Fig. 4(f)], lead to the decrease in  $\sigma_b$  and  $K_{IC}$ .

Fig. 5 shows the microstructure evolution of Ti<sub>3</sub>SiC<sub>2</sub>/10 vol.%Al<sub>2</sub>O<sub>3</sub> sintered at 1400 °C for 5 min. Compared with Fig. 4(c), higher sintering temperatures results in coarser-grained microstructure. Fig. 5(b) shows the

dispersion of  $Al_2O_3$  in the composite. Compared with Fig. 4(e), the agglomeration of  $Al_2O_3$  can be seen more obviously in the 1400 °C sintered sample, which consists of partially sintered  $Al_2O_3$ . When the temperature reaches 1400 °C, the grain size becomes larger, which may be the reason for the decrease of  $\sigma_b$ . But because of the card-frame structure is unaffected at this temperature, the fracture toughness does not change obviously [8].

In conclusion, this study has shown the improvement of the hardness of Ti<sub>3</sub>SiC<sub>2</sub> material with addition of

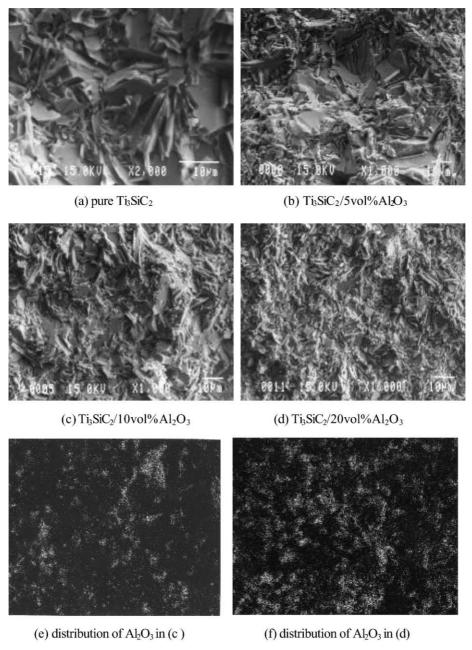


Fig. 4. Fracture surface and distribution of Al<sub>2</sub>O<sub>3</sub>.

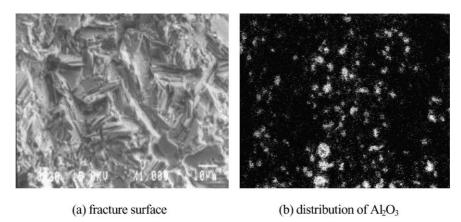


Fig. 5. Fracture surface and distribution of Al<sub>2</sub>O<sub>3</sub> of Ti<sub>3</sub>SiC<sub>2</sub>/10 vol.%Al<sub>2</sub>O<sub>3</sub> sintered at 1400 °C.

 $Al_2O_3$ . Suitable  $Al_2O_3$  content is about 10–20 vol.%. The sintering temperature should not exceed  $1350^{\circ}C$ ; otherwise the agglomeration of  $Al_2O_3$  occurs and decreases the composite strength and toughness.

## 4. Conclusion

- 1. Al<sub>2</sub>O<sub>3</sub> is an effective dopant to increase the hardness of Ti<sub>3</sub>SiC<sub>2</sub>. The Ti<sub>3</sub>SiC<sub>2</sub>/20vol%Al<sub>2</sub>O<sub>3</sub> composite shows hardness of about 10.28 GPa, which is 50% higher than that of pure Ti<sub>3</sub>SiC<sub>2</sub>.
- 2. The addition of Al<sub>2</sub>O<sub>3</sub> slightly decreases the other properties, such as Ti<sub>3</sub>SiC<sub>2</sub> content, strength and fracture toughness, of composite.
- 3. It is viable to add some Al<sub>2</sub>O<sub>3</sub> to Ti<sub>3</sub>SiC<sub>2</sub> in order to increase its wearability, and the appropriate content is about 10–20 vol.%.

#### References

- E.R. Tamer, M.W. Barsoum, Processing and mechanical properties of Ti<sub>3</sub>SiC<sub>2</sub>: I, reaction path and microstructure evolution, J. Am. Ceram. Soc. 82 (10) (1999) 2849–2854.
- [2] E.R. Tamer, M.W. Barsoum, et al., Processing and mechanical properties of Ti<sub>3</sub>SiC<sub>2</sub>: II, effect of grain size and deformation temperature, J. Am. Ceram. Soc. 82 (10) (1999) 2855–2860.
- [3] M. Radoric, M.W. Barsoum, et al., Tensile properties of  $Ti_3SiC_2$  in the 25–1300 °C temperature range, Acta Mater. 48 (2000) 457–459.
- [4] M.W. Barsoum, T. EI-Raghy, Synthesis and characterization of a remarkable ceramic: Ti<sub>3</sub>SiC<sub>2</sub>, J. Am. Ceram. Soc. 79 (7) (1996) 1953–1956.
- [5] M.W. Barsoum, T. EI-Raghy, et al., Oxidation of Ti<sub>3</sub>SiC<sub>2</sub> in air, J. Electrochem. Soc. 144 (1997) 2508–2516.
- [6] I.M. Low, Vickers contact damage of micro-layered Ti<sub>3</sub>SiC<sub>2</sub>, J. Eur. Ceram. Soc. 18 (1998) 709–713.
- [7] T. EI-Raghy, A. Zaraliangos, et al., Damage mechanisms around hardness indentations in Ti<sub>3</sub>SiC<sub>2</sub>, J. Am. Ceram. Soc. 80 (3) (1997) 513–516.
- [8] H.J. Wang, Z.H. Jin, Y. Miyamato. Ti<sub>3</sub>SiC<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> composite prepared by SPS, J. Eur. Ceram. Soc. (submitted for publication).