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Fabrication and characterization of Ti_3SiC_2–SiC nanocomposite by *in situ* reaction synthesis of TiC/Si/Al powders

BAOYAN LIANG[†], MINGZHI WANG*, XIAOPU LI and YUNCHAO MU[†]

Key Laboratory of Metastable Materials Science and Technology, Yanshan University, Qinhuangdao 066004, China
[†]Materials and Chemical Engineering School, Zhongyuan University of Technology, Zhengzhou 450007, China

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Abstract. **The microstructure and mechanical properties of Ti_3SiC_2–SiC nanocomposite fabricated by *in situ* hot pressing (HP) synthesis process were studied. The results show that dense Ti_3SiC_2–SiC composite contained minor $TiSi_2$ obtained by hot sintering at 1350°C for 1 h. The average grain size of Ti_3SiC_2 was 4 μm in length, and the size of SiC grains is about 100 nm. With its fine microstructure, the Ti_3SiC_2–SiC nanocomposite shows good mechanical properties.**

Keywords. **Ti_3SiC_2–SiC composites; hot pressing; reaction synthesis.**

1. Introduction

Ti_3SiC_2 is a layered ternary carbide compound. It has the excellent characteristics of both ceramic and metal (Barsoum and El-Raghy 1997; Li *et al* 1999). It is very refractory with high melting point, good oxidation resistance and high temperature strength. Like metal, it shows good thermal and electric conductivities, and it is not susceptible to thermal shock. Therefore, this compound has attracted much attention.

However, because of its low hardness, Ti_3SiC_2 is not wear resistant. As for reinforcing the phase in composite, SiC has many desirable properties, such as high hardness, high temperature stability and excellent high temperature oxidation. Addition of SiC improves mechanical properties and the oxidation stability of Ti_3SiC_2-based materials at high temperature. Application of Ti_3SiC_2–SiC composites in aerospace engine components is very promising.

Ti_3SiC_2–SiC composite had been fabricated by many methods, such as hot iso-static pressing (HIP) (Linh *et al* 2003), hot pressing (HP) (Tong *et al* 1995; Radhakrishnan *et al* 1996; Lia *et al* 2003; Li *et al* 2004; Lin *et al* 2005; Wan *et al* 2006) and spark plasma sintering (SPS) (Zhang *et al* 2007). Linh *et al* (2003) fabricated a Ti_3SiC_2–30 vol% SiC composites at 1500°C and 1600°C for 8 h by the HIP method. Tong *et al* (1995) fabricated a Ti_3SiC_2–20 vol% SiC composite at 1500°C or 1600°C for 0·5 h by the HP method. Ti_3SiC_2–SiC composites were fabricated by the HP method using displacement reactions of TiC and Si (Tong *et al* 1995; Radhakrishnan *et al* 1996; Lia *et al* 2003; Li *et al* 2004). They first kept the samples at below 1400°C for 2 h to avoid Si melting, and then kept them at 1500°C for 1 or 2 h for densification.

The literatures (Linh *et al* 2003; Wan *et al* 2006) have pointed that the SiC added, affects adversely the strength of the SiC–Ti_3SiC_2 composites for the great thermal expansion coefficients (TECs) mismatch between the Ti_3SiC_2 ($9{\cdot}1 \times 10^{-6}$ K^{-1}) and the SiC ($5{\cdot}12 \times 10^{-6}$ K^{-1}). The finer SiC particles will be less detrimental (Zhang *et al* 2007), which has positive effect on the elevating of the flexural strength of Ti_3SiC_2/SiC composites. Recently, the literature (Zhang *et al* 2007) fabricated SiC–Ti_3SiC_2 nanocomposites by advanced spark plasma sintering (SPS), expressing good mechanical strength. However, to date, there was no report of fabricating SiC–Ti_3SiC_2 nanocomposites by HP.

The purpose of the present study is to fabricate SiC–Ti_3SiC_2 composite by *in situ* synthesis of TiC/Si powders by HP. In order to erase the TiC impurity, appropriate Al was used as aids.

2. Experimental procedure

TiC powder (99·5% pure, 3 μm), Si powder (99·6% pure, 50 μm), Al powder (99·6% pure, 20 μm) were mixed in a mole ratio calculated from 3TiC/2·2Si/0·2Al. After being dry-mixed for 2 h, the mixtures were put in a 40 mm graphite mould and subsequently hot pressed at 30 MPa in a flowing Ar at 1350°C for 1 h.

The as-sintered sample were ground and analysed by X-ray diffraction (XRD). XRD analysis was carried out using a rotating anode X-ray diffractometer (Model D/MAX-2500PC) with CuKα radiation. The Rietveld method (Young 1993) was used to calculate the phase contents in the sample. In the Rietveld method, crystal structure and

*Author for correspondence (wmzw@ysu.edu.cn)

peak profile parameters are refined in several stages. The complete profile of the powder diffraction pattern is refined by employing a DBWS code (Young 1993) in Cerius computational program for materials research (Molecular Simulation Inc., USA). The intensity is represented by

$$I_{\text{Rietveld}}(2\theta) = b(2\theta) + S\sum_{K} L_{\text{K}}|F_{\text{K}}|^2\phi(2\theta_i - 2\theta_{\text{K}})P_{\text{K}}A_{\text{K}}, \quad (1)$$

where $b(2\theta)$ is the background intensity; S, the scale factor; L_{K} contains the Lorentz polarization and multiplicity factors; F_{K}, the structure factor; ϕ, the profile function; P_{K}, the preferred orientation function and A_{K}, the absorption factor. The index K represents Miller indices for the Bragg reflections. The mass fraction of a phase q, Wq, is given by Wiles and Young (1981)

$$W_{\text{q}} = \frac{S_{\text{q}}M_{\text{q}}V_{\text{q}}}{\sum(S_iM_iV_i)}, \quad (2)$$

where S is the Rietveld scale factor for the phase q; M the molar mass and V, the volume of the unit cell. Archimedes method was adopted to identify the degree of densification. The microstructure analyses of the samples were conducted using field emission scanning electron microscopy (FESEM) with energy-dispersive spectroscopy (EDS).

Vickers hardness (Hv) of the polished samples was measured by the indentation technique. The indentation parameters were made using a 1 kg load with a dwell of 15 s. The sample was machined and polished to 5 mm × 7 mm × 27 mm bars for measuring the three-point bending strength with a span of 20 mm.

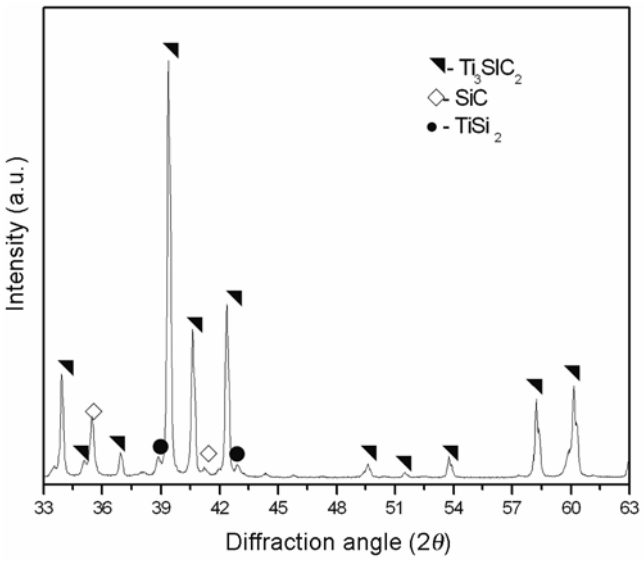

Figure 1. XRD patterns of Ti_3SiC_2–SiC composite.

3. Results and discussion

3.1 *Phase compositions and microstructure*

Figure 1 shows that the XRD of the as-sintered sample. The strong Ti_3SiC_2 and SiC peaks existed in the XRD patterns. In addition, very faint $TiSi_2$ peaks were observed in the patterns. According to the Rietveld method, SiC, Ti_3SiC_2 and $TiSi_2$ content of this sample were determined to 22 vol%, 74 vol% and 4 vol%, respectively.

Figure 2a shows fractured morphology of the sample. A little porosity can be found on the fracture surface. The sample was near fully dense, with the measured density (4·092 g/cm^3) and the calculated theoretical one (4·13 g/cm^3). Its relative density reached 99·1%. The synthesized Ti_3SiC_2 grains are very fine and plate-like. As shown in figure 2b, its grain size is about 4 μm. Besides, there were many fine equiaxed grains with the size of 50–100 nm. By EDS, they were determined as SiC.

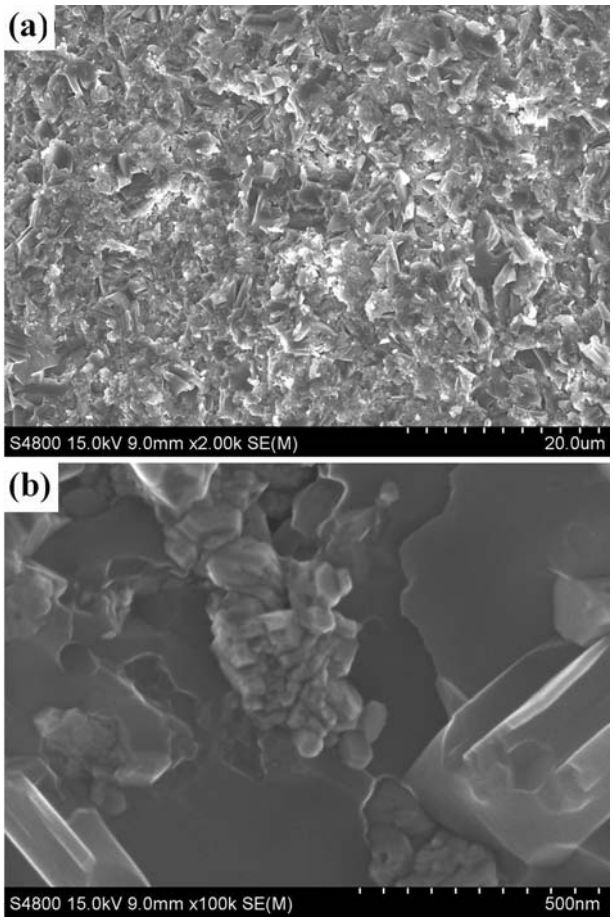

Figure 2. (**a**) Lower magnification and (**b**) higher magnification of FESEM of fracture morphology of the as-sintered sample.

Table 1. Summary of grain sizes and mechanical properties of the Ti_3SiC_2–SiC composites.

		Grain size				
Material	SiC content (vol%)	Ti_3SiC_2 (length)	SiC	Hv (GPa)	δ_f (MPa)	Literature
1	15·9	4	0·1	8·96	548	This work
2	0	~ 100		4	260 ± 2	1
3	0	~ 10		5·77	360 ± 10	2
4	30 (S1500)	10·5 ± 9	9 ± 7	~ 12	315 ± 5	3
	30 (S1600)	13 ± 12	14 ± 8	~ 16	218 ± 9	
5	14·2	~ 10	1–5	8·9		7
6	20	11·1 ± 6·5	17 ± 9	~ 9·9	~ 360	9
7	20	5	0·1	9·2	~ 500	10

The results showed that dense Ti_3SiC_2–SiC nanocomposite with fine microstructure was obtained at 1350°C for 1 h. The sintered temperature of the Ti_3SiC_2–SiC nanocomposite decreased by 150–250°C compared with those literatures (Tong *et al* 1995; Radhakrishnan *et al* 1996; Lia *et al* 2003; Linh *et al* 2003; Li *et al* 2004; Lin *et al* 2005; Wan *et al* 2006) for fabrication of Ti_3SiC_2–SiC composites using HP and HIP methods. Meanwhile, since the sintering temperature was reduced greatly, the finer microstructure was obtained (table 1).

3.2 *Mechanical properties*

Table 1 indicates that SiC content, the grain sizes, Vickers hardness and bending strength of Ti_3SiC_2–SiC composites fabricated in this work and in the reported literatures (Young 1993; Tong *et al* 1995; Radhakrishnan *et al* 1996; Linh *et al* 2003; Wan *et al* 2006). As shown in table 1, the Vickers hardness and bend strength of No. 1 was apparently higher than monoclinic Ti_3SiC_2 (Nos. 2 and 3). The reason for increasing the hardness was attributed to the existence of SiC. It is known that reducing of grain size should, in principle, significantly improve the strength. Among these composites, No. 1 had the finer microstructure. Therefore, the strength value of No. 1 was higher than that of Nos. 4–7.

The present study shows that this composite possesses good mechanical properties. In the future, we will fabricate the SiC–Ti_3SiC_2 composites with different ratio by changing the ratio of the raw materials.

4. Conclusions

Dense and Ti_3SiC_2–SiC nanocomposite was synthesized by *in situ* hot pressing at 1350°C for 1 h using 3TiC/2·2Si/0·2Al powders as raw materials. This composite possessed a fine microstructure. The size of Ti_3SiC_2 grains was about 4 μm, and SiC grains was 50–100 nm. The flexural strength of the Ti_3SiC_2–SiC composite reached 548 MPa.

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