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Liquid-Phase Hot-Pressing and WC-Particle Reinforcement of SiC–Si Composites

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Abstract

The sintering behaviour of SiC–Si and SiC–Si–WC composites hot-pressed via silicon liquid phase has been studied. The results show that the increase of silicon makes the sintered materials densify at lower temperature and the grain growth is restricted due to the effect of the liquid. The strength of SiC–25 vol.% Si samples is higher than the strength obtained for SiC–5 vol.% Si with the same hot-pressing conditions. The addition of WC particles improves relative densities and strengths of SiC–Si ceramics.

Das Sinterverhalten von heißgepreßten SiC–Si- und SiC–Si–WC-Verbunden wurde studiert. Die Ergebnisse zeigen eine Erniedrigung der Verdichtungstemperatur bei Erhöhung des Siliziumanteils im Sintermaterial, während das Kornwachstum aufgrund des Flüssigkeiteffekts eingeschränkt war. Unter denselben Heißpreßbedingungen zeigen SiC–25 vol.% Si Proben eine höhere Festigkeit als SiC–5 vol.% Si Proben. Die Zugabe von WC Partikeln verbessert die relative Dichte und Festigkeit der SiC–Si-Keramiken.

Le frittage des composites SiC–Si et SiC–Si–WC pressés à chaud par préparation en phase liquide de silicium a été étudié. Les résultats montrent que l'adjonction de silicium favorise la densification à basse température et que le silicium liquide limite la croissance des grains. Pour des conditions de préssion à chaud identiques, la résistance à la rupture des échantillons SiC–25 vol.% Si est plus élevée que celle des échantillons SiC–5 vol.% Si. Enfin l'addition de particules de WC améliore les densitées relatives et la résistance à la rupture des céramiques SiC–Si.

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1 Introduction

SiC–Si ceramics are usually manufactured by the reaction-bonded sintering method.[1–5] Silicon carbide is a very promising candidate material for heat engine and other engineering applications, because it possesses unique high-temperature strength, good resistance to corrosion and oxidation, very high hardness and wearability, high thermal-shock resistance and low thermal expansion coefficient.[6] However, owing to its highly refractory nature and covalent bonding, it is very difficult to sinter and densify with traditional sintering methods. Some special techniques have been developed and several secondary substances have been introduced to promote sintering in order to obtain densified materials with high thermomechanical performance; reaction-bonded sintering and hot-pressing (HP) are two of frequently used sintering methods. The reaction-bonded sintering of silicon carbide (RBSC) is based on the infiltration of liquid silicon into the previous shaped bodies where the reaction of the liquid silicon–solid carbon system occurs to produce secondary silicon carbide. Contemporaneously, the molten silicon infiltrates into the pores and makes the bodies densified. Commercially available RBSC exhibits room-temperature flexural strength of 200 to 600 MPa,[7] whereas laboratory materials prepared by Hucke[8] have resulted in the highest reported strength for RBSC: about 700 MPa. There is normally a substantial volume fraction (5% to >30%) of free silicon that is present to facilitate infiltration processing (these materials are also commonly referred to as siliconized silicon carbide). The thickness of articles prepared by this method would be limited to some extent, in fact, if silicon reacts with carbon prior to infiltrating inside, the silicon carbide generated on the surface blocks the entry for further liquid silicon, resulting in the samples not siliconized and not entirely

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densified, and also with a lower strength. In addition, the amount of free silicon in final RBSC bodies depends on the initial carbon content in the green compacts and on the extent of reaction.

On the other hand, in its pure form, SiC powder will not sinter to a fully dense material. With small additions of sintering aids, such as efficient elemental C and a B- or Al-containing phase,[9–11] and/or with application of high pressures,[12,13] sintering has been performed at high temperatures (2300 to 2400 K). One commercial example is an α-SiC[10] (Hexoloy, SOHIO Engineering Materials Co., Niagara Falls, NY) produced by conventional sintering of submicrometer SiC particles at about 2373 K, containing 0·5 wt% free C and 0·42 wt% B in the sintered state. Dense SiC can be obtained by hot-pressing[14–16] with the previously mentioned sintering aids, but SiC has never been hot-pressed by >95% of theoretical density below 1900°C.[12]

The present work is focused on another sintering method: liquid-phase hot-pressing. SiC and silicon powders are mixed together, ball-milled to distribute the silicon phase homogeneously in the silicon carbide matrix, and hot-pressed at a temperature range higher than the silicon melting point (producing liquid phase) but lower than the frequently used temperatures (e.g. 1900°C). In addition, in previous work[17] it has been shown that the addition of WC particles can enhance the densification of SiC and improve its mechanical properties. Thus, finally, some quantities of WC particles were introduced into the SiC–Si system with the purpose of studying their effects on hot-pressing behaviour and as a reinforcement.

2 Experimental Procedures

2.1 Batching

α-SiC powder (TLW5, α-SiC > 97 wt%; Wuxi Grinding Wheel Works, People's Republic of China) consisting mainly of 6H polytype phase and having particle diameter range of 2·5–5 μm was treated to remove SiO_2 and mixed with silicon powder (200 mesh, analytically pure, Shanghai Chemical Product Co., People's Republic of China) and WC particles (2–4 μm, North Tool Work, People's Republic of China) in proportion as follows: SiC + 5 vol.% Si; SiC + 25 vol.% Si and SiC + 25 vol.% Si + 10 vol. % WC. Very small quantities of Al and C have been used as sintering aids because of their beneficial effects on sintering and densification on silicon carbide.[12,13,18,19]

2.2 Ball-milling and drying

The mixing and ball-milling were carried out in a carbide ball mill with ethanol. Subsequently, the mixed slurry was dried under vacuum and then the powder mixture was sieved under nitrogen atmosphere.

2.3 Hot-pressing

The green compacts shaped in a steel die were hot-pressed with the graphite mould in hydrogen atmosphere within a temperature range of 1750 to 1900°C, with the pressure of 30 MPa.

2.4 Measurements

The samples were cut from the hot-pressed discs and machined into rectangular bars of 3 × 4 × 30 mm and surface polished. Fracture strength was measured at room temperature by the three-point bending method with a support distance of 20 mm. Mean flexural strength values were calculated for 4–6 samples. Fracture toughness was determined for some samples from direct measurements of the crack traces on the indented surfaces, following the indentation fracture theory[20–22] and using the following equation:

$$K_{IC} = A(E/H)^{1/2}Pc^{-3/2}$$

where $A = 0{\cdot}016 \pm 0{\cdot}004$; the hardness $H = 0{\cdot}4636P/a^2$; E is Young's modulus; P is the applied load on the indenter; a is the half-diagonal length of indentation; and c is the half crack length induced by indentation, as a straightforward basis for calculation. A 3 kg Knoop indenter load was used for examining elastic modulus to hardness ratios, whereas the indenter Vickers load was 5 kg in order to make the indentation with the requirement of $c \geq 2a$.[20] MicroVickers hardness (Hv) was also tested for selected samples with the indenter load of 1 kg. In the measurements of fracture toughness and hardness, for each sample, 3–5 indentations were made. Moreover, Vickers indentation (20 kg) was done for a SiC–Si–WC sample to observe the crack propagation. Densities of the samples were measured according to the Archimedian principle. The size of powder mixture, fracture surfaces and etched surfaces (etched in 10% HF for three days) of samples were examined by scanning electron microscopy (SEM). X-Ray diffraction (XRD) was used for the identification of the phase.

3 Results

3.1 Morphology and particle size of the powder mixture

By intensive ball-milling, powder mixture with the average particle diameter of about 0·4 μm was obtained. Figure 1 shows the SEM micrograph of SiC–Si–WC powders, most of them with spherical morphology. The morphology and particle diameter

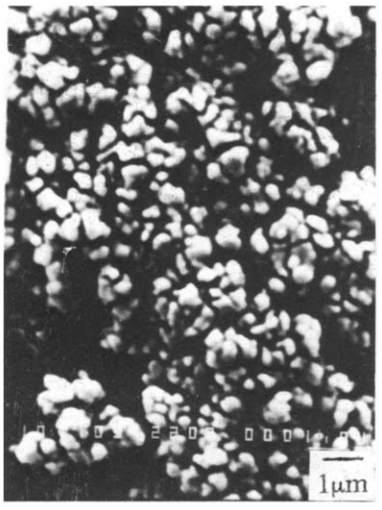

Fig. 1. SEM micrograph of SiC–Si–WC powder mixture.

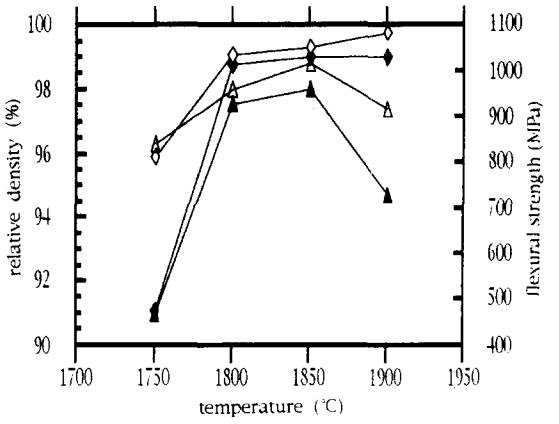

Fig. 2. Relative densities (◆, ◇; %) and flexural strengths (▲, Δ; MPa) of SiC–25 vol.% Si (◆, ▲) and SiC–Si–WC (◇, Δ) composites prepared at different hot-pressing temperatures.

for the SiC–Si powder mixture are similar to that shown in Fig. 1.

3.2 Relative densities and flexural strength of SiC–Si samples

Two groups of SiC–Si materials were prepared to study the influence of different initial silicon contents on the densification, hot-pressing behaviour and the physical–mechanical properties. Table 1 shows the relative densities and the flexural strengths of samples prepared under different hot-pressing conditions.

3.3 Hot-pressing behaviour and properties of the SiC–Si–WC (SSW) composite

The SiC–25 vol.% Si–10 vol.% WC composite was prepared. The variation of relative densities and flexural strengths with the hot-pressing temperature is shown in Table 2 (with constant pressure of 30 MPa and time 15 min).

Figure 2 shows the relative densities and the average flexural strengths of SiC–25 vol.% Si and SiC–Si–WC composites, as functions of the hot-pressing temperatures, from which can be seen the hot-pressing behaviour of the SiC–Si–WC composite and the densifying and reinforcing effect of WC particles on SiC–Si ceramics.

Two SSW3 (see Table 2) samples and one SiC–25 vol.% Si sample, with the highest relative density and flexural strength in each group, were selected to measure their fracture toughness and hardness. The MicroVickers hardness *Hv* (1 kg) of SSW3 is 23·20 GPa. The average fracture toughness (direct indentation measurements) of SSW3 is 4·65 MPa $m^{1/2}$, while the toughness value of SiC–Si obtained by the same measurement method and calculation equation is only 3·85 MPa $m^{1/2}$.

3.4 Microstructures

Scanning electron micrographs of SiC–25 vol.% Si and SiC–5 vol.% Si samples are shown in Figs 3 and 4 respectively, hot-pressed at the same temperature (1850°C). It is clearly shown that the grains of the former (in Fig. 3) are smaller, with the average grain size of 1 μm and with well-distributed particles. In Fig. 4, the average grain size is approximately 2 μm. Figure 5 shows the backscattering micrograph of the SiC–Si–WC sample hot-pressed at 1850°C, from which it can be observed that the WC particles (white region) are distributed in the SiC–Si matrix (black region) as a dispersed phase. Figure 6 shows a crack propagation in the SiC–Si–WC sample. The grain morphology of the SSW composite, with some needle-like grains, can be observed clearly from Fig. 7, on one etched

Table 1. The hot-pressing conditions and properties of SiC–Si

Composition (vol. %)		*Hot-pressing conditions*			*Relative density (%)*	*Flexural strength (MPa)*
SiC	*Si*	*T (°C)*	*P (MPa)*	*t (min)*		
95	5	1750	30	30	88·50	395
		1800	30	30	94·11	432
		1850	30	30	98·29	613
		1900	30	30	99·01	636
75	25	1750	30	15	91·08	468
		1800	30	15	98·77	930
		1850	30	15	99·01	961
		1900	30	15	99·01	730

Table 2. The relative densities and flexural strengths of SiC–Si–WC composite at different hot-pressing temperatures

Samples	*Hot-pressing temperature (°C)*	*Relative density (%)*	*Flexural strength (MPa)*
SSW1	1750	95·92	843
SSW2	1800	99·09	961
SSW3	1850	99·32	1017
SSW4	1900	99·77	918

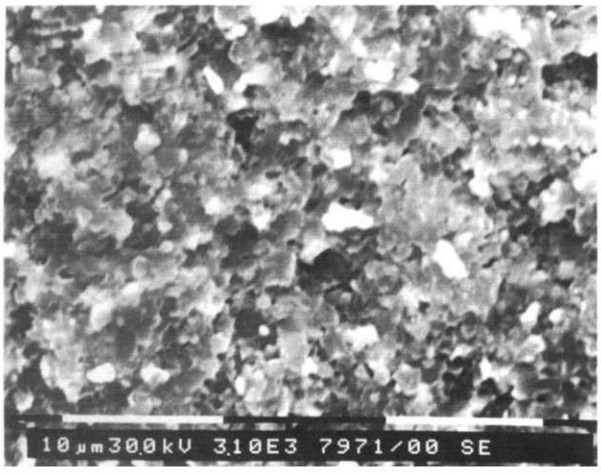

Fig. 3. SEM micrograph showing the fracture surface of a SiC–25 vol.% Si sample.

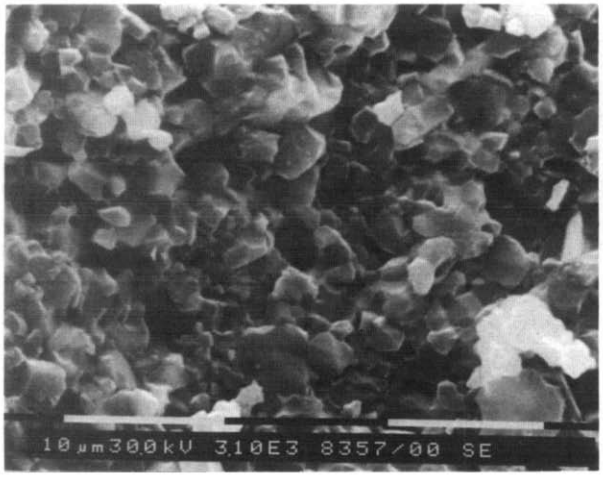

Fig. 4. SEM micrograph showing the fracture surface of a SiC–5 vol.% Si sample.

sample. The average grain sizes of SiC crystals and WC particles in the SSW3 sample are about 1 μm and 2 μm respectively. In contrast, no needle-like but mainly spherical grains were found in the etched SiC–Si sample. X-Ray diffraction patterns, however, do not show any variation of crystalline types of silicon carbide in SiC–Si and SSW composites at the present HP temperatures, which are still mainly 6H type.

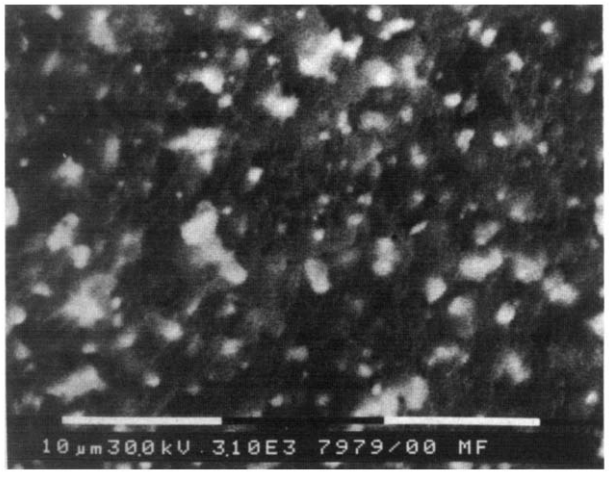

Fig. 5. The backscattering micrograph of SSW3 sample.

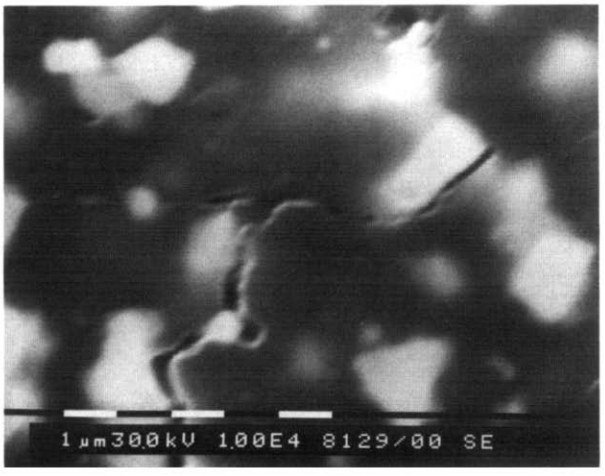

Fig. 6. SEM micrograph showing a crack propagation in SiC–Si–WC sample.

Fig. 7. SEM micrograph of SSW3 sample, etched in 10% HF for three days.

4 Discussion

4.1 Influence of different initial silicon contents on microstructure and properties of the SiC–Si composite

From the results shown in Table 1, it can be seen that, under the same hot-pressing temperature and pressure, each group of samples which contains 25 vol.% Si with a soaking time of 15 min shows higher relative densities than that containing 5 vol.% Si with a soaking time of 30 min. Actually, the relative density of the former, hot-pressed at 1850°C for 15 min, being over 99%, is similar with that of the latter hot-pressed at 1900°C for 30 min. This illustrates that the increase of initial silicon content makes the densification easier and the sintering faster. Furthermore, the flexural strength of the composite which contains 25 vol.% Si, under the hot-pressing conditions shown in Table 1, is also improved by increasing the relative density. The

flexural strength of samples hot-pressed at 1800°C and 1850°C respectively are both over 900 MPa, very much higher than the values of reaction-bonded sintering samples.[7,8]

A microstructural comparison can be made between the hot-pressed SiC–25 vol.% Si sample and the RBSC reference sample which contains 33 vol.% silicon.[23] In this RBSC material, some regions of the microstructure had a higher than average concentration of silicon, whereas others had a higher concentration of silicon carbide. High concentrations of silicon carbide were particularly evident within agglomerates that were dispersed throughout the structure. Furthermore, irregularly shaped 'pockets' of Si and 'stacking faults' of α-SiC were found by transmission electron microscopy observations. This inhomogeneous distribution of irregularly shaped SiC grains and intergranular Si is supposed to be a consequence of the manufacturing method of the material.[23] On the other hand, the hot-pressed SiC–25 vol.% Si sample shown in Fig. 3 has a microstructure with a small grain size of silicon carbide; no coarse α-SiC plates were observed. From the SEM observation on the etched SiC–Si sample (micrograph not shown in this paper), it can be supposed that the silicon carbide grains were surrounded by silicon, but no apparent concentration of silicon was found. Such homogeneous microstructure shown in the hot-pressed SiC–Si sample results not only from the previous uniform distribution of silicon in the SiC matrix, but also from the shorter sintering time, due to the application of the HP technique by which the irregular growth of SiC grains is restricted. The sharp decrease of the flexural strength for the SiC–25 vol.% Si samples prepared at 1900°C may be due to the large grain size, because this mechanical property is affected not only by the relative density but also by the average grain size.

Compared with the microstructure of the SiC–25 vol.% Si sample, the sample containing 5 vol.% Si shows a larger grain size, Figs 3 and 4 giving an obvious comparison. From this behaviour, it can be supposed that, in the composite containing more silicon, the grain growth has been decreased by effects of wetting, adhesion and wrapping of liquid-phase on the solid-state silicon carbide. Therefore, the SiC–Si composite with a higher silicon content, hot-pressed at a lower temperature and a shorter soaking time, also possesses higher relative density and flexural strength.

However, it should be mentioned that the value of flexural strength of SiC–5 vol.% Si samples hot-pressed at 1900°C for 30 min is still raised. This shows that the present hot-pressing conditions are not suitable for SiC containing such small quantities of silicon. The hot-pressing temperature must be raised or the soaking time prolonged.

4.2 Reinforcement effect of WC particles on SiC–Si ceramics

The plot-shaped results, summarizing Tables 1 and 2, are shown for a best comparison in Fig. 2. The SSW composite possesses higher relative density and higher flexural strength than SiC–Si samples hot-pressed under the same conditions. This illustrates that the addition of WC particles not only accelerates the densification of SiC–Si ceramics but also improves their strength. In particular, SSW3 (see Table 2) hot-pressed at 1850°C, 30 MPa and 15 min has the highest value of flexural strength, 1017 MPa.

WC particles dispersed in the SiC–Si matrix (white region in Fig. 5), have played a crack-deflection role. This can be clearly observed in Fig. 6 which shows the spread of a simulated crack. Crack deflection is a commonly observed fracture characteristic in brittle materials, associated either with crack attraction or repulsion by the second phase (due to residual strains) or with the presence of low-resistance interfaces.[24] In the present study, internal stresses could be produced at the particle/matrix interface because of both the thermal expansion mismatch[20,25,26] and the forced densification process of hot-pressing.[19] Consequently, these residual internal stresses divert the crack propagating along the WC particle/matrix interface for some distance. According to the theory of crack deflection processes,[24,25] when a crack front interacts with second-phase particles, the crack is deflected; this alters the crack-tip stress intensity, increasing toughness. Indeed, the fracture toughness of SSW3 (4·65 MPa $m^{1/2}$) is higher than that of SiC–Si (3·85 MPa $m^{1/2}$). The grain size and morphology of the WC dispersion in SSW3 can be observed in Figs 5 and 6. Some WC particles are rod-like, being the most effective morphology[25] for deflecting propagating cracks.

Although the grain size of SiC in SSW3 is similar to that in the SiC–Si sample obtained under the same hot-pressing conditions, the morphology of the former is different from the latter. Some needle-like SiC grains, which are supposed to benefit the toughning of samples, are evidently observed in Fig. 7. The growth of β-SiC needles from silicon melt has been reported,[27] but in the present study, it is difficult to explain this difference from the XRD analysis. Finally, SSW4 sintered at 1900°C has higher relative density than SSW3, but lower flexural strength, probably because of the grain growth of sintered bodies at higher temperature.

The SSW3 composite possesses not only higher flexural strength, but also higher fracture toughness than SiC–Si samples. In addition, its Vickers

hardness *Hv* (1 kg) is 23·20 GPa; the Rockwell hardness is over 92 (HRA) and the thermal expension coefficient is $4{\cdot}66 \times 10^{-6}$/°C.[28]

5 Conclusion

(1) The hot-pressed SiC–Si ceramics possess higher flexural strength than those prepared by the reaction-bonded sintering method, because of more homogeneous distributions of silicon and silicon carbide phases and grain growth reduction of SiC due to the application of the hot-pressing technique.

(2) The presence of liquid silicon reduces the hot-pressing temperature for silicon carbide. The increase of the initial silicon content accelerates the densification rate, slows down the grain growth and improves the strength by the effect of wetting, adhesion and wrapping of liquid-phase on silicon carbide.

(3) The best hot-pressing conditions suitable for SiC–Si–WC composites have been found by studying the sintering behaviour at different temperatures. The samples were densified at 1800°C. They possess higher relative density and higher flexural strength than SiC–Si obtained under the same hot-pressing conditions. The addition of WC particles not only accelerates the densification rate but also improves the flexural strength and fracture toughness of SiC–Si ceramics. WC particles dispersed in the SiC–Si matrix play a role in deflecting crack propagation.

(4) The SiC–Si–WC composite hot-pressed at a temperature of 1850°C, a pressure of 30 MPa and a soaking time of 15 min possesses excellent properties: relative density, 99·32%; flexural strength, 1017 MPa; fracture toughness, 4·65 MPa $m^{1/2}$; Vickers hardness *Hv* (1 kg), 23·20 GPa; Rockwell hardness, over 92 HRA, and thermal expansion coefficient, $4{\cdot}66 \times 10^{-6}$/°C.

According to these good properties, the SSW composite may be used as the material for dies and tools or for some heat engine components. Its thermomechanical properties remain to be analysed at high temperature.

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