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Microstructural development of liquid-phase-sintered silicon carbide during annealing with uniaxial pressure

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Abstract

β-SiC powders containing 1.1 wt.% α-SiC particles as seeds were hot-pressed at 1800 °C and then annealed at 2000 °C under 25 MPa uniaxial pressure to enhance grain growth. Microstructural development during annealing with pressure was investigated quantitatively and statistically using image analysis. The bimodal grain-thickness distribution in samples annealed with pressure was obtained due to abnormal grain growth of some grains. In situ-toughened microstructure has been developed after 3-h annealing. The grain-thickness and aspect ratio of large grains increase with annealing time, but grain growth comes mainly from increases in thickness after 3-h annealing, owing to the impingement of large gains. Typical flexural strength and fracture toughness of 4-h annealed sample were ~500 MPa and ~7.5 MPa $m^{1/2}$, respectively.

Keywords: Aspect ratio; Grain growth; Mechanical properties; Seeding; SiC

1. Introduction

The general experimental experience with liquid-phase sintered SiC shows that elongated grains promote an increment of toughness by crack bridging and deflection.[1–6] Attempts to introduce the elongated SiC grains into the microstructure can be summarized as the following two strategies: (i) taking advantage of the β→α phase transformation at high temperatures, which usually accelerates the grain growth,[1–5] and (ii) use of a solution–reprecipitation process.[7–10] The latter efforts include the seeding for favoring preferential grain growth without phase transformation[7,8] and the use of fine α-SiC starting powders for triggering abnormal grain growth.[10] A fracture toughness of 8 MPa $m^{1/2}$ has been reported in toughened SiC ceramics with yttrium aluminum garnet ($Y_3Al_5O_{12}$, YAG) as a grain boundary phase[1] and a higher fracture toughness of ~9 MPa $m^{1/2}$ in toughened SiC ceramics with Al–B–C.[3]

Although there are many reports on microstructure of liquid-phase sintered SiC, the information on microstructural development during annealing with pressure is quite limited. Recently, Zhan et al.[11] reported that low annealing pressure (25 MPa) played an important role in the microstructural development and greatly retarded phase transformation from β to α polytypes. In the present work, the microstructures developed during annealing with uniaxial pressure were characterized by using image analysis on the polished and etched surfaces and compared with that from the annealed without pressure.[12] The analysis is based on the thickness, the aspect ratio, and the total volume fraction of key grains,[12] which play a key role regarding the toughening behavior of SiC ceramics.

2. Experimental procedure

Commercially available β-SiC (Ultrafine, Ibiden Co., Ltd., Nagoya, Japan), α-SiC (A-1 grade, Showa Denko, Tokyo, Japan), Al_2O_3 (99.9% pure, Sumitomo Chemical Co., Tokyo, Japan), Y_2O_3 (99.9% pure, Shin-Etsu Chemical Co., Tokyo, Japan), and CaO (>99.9% pure, High Purity Chemicals, Japan) were used as the starting powders. The mean particle sizes of the α and β powders were 0.45 and 0.27 μm, respectively. A combination of 89 wt.% β-SiC and 1 wt.% α-SiC mixed with 5.7 wt.%

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Al_2O_3, 3.3 wt.% Y_2O_3, and 1 wt.% CaO was milled in ethanol for 24 h using SiC media. The powder mixture was dried, subsequently sieved through a 60 mesh screen, and hot-pressed at 1800 °C for 1 h under a pressure of 25 MPa in an argon atmosphere. The hot-pressed samples were further annealed at 2000 °C for 1–8 h under a presence of 25 MPa in Ar atmosphere to enhance grain growth. The heating rate was 20 °C/min, and the cooling rate was ~70 °C/min from 2000 to 1200 °C.

The relative densities of the annealed samples were determined by the Archimedes method using deionized water as an immersion medium. The theoretical density of the samples, 3.284 g/cm^3, was calculated according to the rule of mixtures. The hot-pressed and annealed samples were cut, polished, and then etched by a plasma of CF_4 containing 7.8% O_2. The microstructures were observed by scanning electron microscopy (SEM). SEM micrographs were quantitatively analyzed by image analysis, according to a procedure shown in the previous study.[12] The three-dimensional morphology of SiC grain was a hexagonal platelet when we observed it in annealed specimens, where the intergranular phases were leached by a molten salt mixture. The thickness of each grain (t) was determined directly from the smallest grain dimension in its two-dimensional image; the apparent length of each grain (L) was obtained from the largest dimension. The mean value of the observed aspect ratio (L/t) considered to be an average aspect ratio. Grains deviating from the normal grain-thickness distribution are defined as large grains in the present work; the other grains belong to the normal grain-thickness distribution are defined as matrix grains. A total of 900–1200 grains was used for statistical analysis of each specimen.

Grains considering to be an effective contributor to the toughening are defined as key grains.[12] From the previous observation,[12] key grains are classified into two principal categories: (i) grains with a length (L) >2 μm, an aspect ratio (ar) >4, and a thickness (t) <3 μm (designated as KG1), which are frequently observed at crack deflection sites; (ii) grains with a length $L > 2$ μm and a thickness of 1 μm $< t < 3$ μm (designated as KG2), which are frequently observed at crack bridging sites. The volume fractions (V_{KG1} and V_{KG2}) of key grains were calculated from the results using an image analyzer. The total volume fraction (V_T) of key grains is calculated as follows:[12]

$$V_T = V_{KG1} + V_{KG2} - V_{\text{common grains of KG1 and KG2}} \quad (1)$$

X-ray diffractometry (XRD), using CuK_α radiation, was performed on the ground powders. The bar samples (2.5×3×25 mm) were machined to a 1 μm finish for flexural testing. Bend tests were performed at room temperature on five specimens at each condition, using a four-point method with inner and outer spans of 10 and 20 mm, respectively, and a crosshead speed of 0.5 mm/min. Microstructure–crack interactions were investigated (i) for cracks introduced by a Vickers indentor at a load of 196 N on the polished and etched surfaces and (ii) on the fracture surfaces of the samples. The fracture toughness was estimated by measuring the lengths of cracks that were generated by a Vickers indenter.[13] The variation of fracture toughness with indentation load (R-curve-like behavior) was estimated by changing the indentation load over a range of 49–294 N, and the toughness values that were measured in the steady-state region were reported in this study.

3. Results

3.1. Microstructure

Relative densities of ⩾98% were achieved by hot-pressing and subsequent annealing with pressure for all samples (Table 1). All the β-SiC phases were transformed into α-SiC after annealing for 1 h at 2000 °C. No secondary phase was detected by XRD, indicating the presence of amorphous grain boundary phase in this system. The microstructures of the hot-pressed and annealed samples are shown in Fig. 1. The microstructure of 1-h annealed sample consisted of fine, equiaxed SiC grains and relatively large, elongated grains that originated from abnormal grain growth. In contrast samples annealed under pressure for 3-, 4- and 8-h consisted of small elongated grains and large elongated grains.

The grain-thickness distributions are shown in Fig. 2. Bimodal grain-thickness distributions are clearly shown in all samples. The separation points between the large grains and the matrix grains, determined from the grain-thickness distributions, were 3.16 μm for the 1-h annealed sample, 5.01 μm for the 4-h annealed sample, and 7.94 μm for the 8-h annealed sample. The relative area of the matrix grains and large grains in a bimodal microstructure is directly related to the volume fraction of a certain range of grain thickness. The increase in annealing time shifts the grain-thickness distribution to larger grain size region while maintaining the bimodal distribution. The experimental observations confirm the previous results[10] that final microstructure is decisively controlled by the grain size distribution of hot-pressed materials, if there is no β→α phase transformation during annealing.

The quantitative image analysis for grain thickness and aspect ratio are summarized in Table 1, and their changes with annealing time are shown in Fig. 3. The average thickness and aspect ratio of both matrix and large grains increase at an early stage of annealing. However, after 3-h annealing, the growth in aspect ratio of matrix grains nearly stops and approaches ~3.2 and

Table 1
Annealing conditions, relative densities, and microstructural characteristics of the annealed samples

Sample[a]	Annealing conditions	Relative density[b] (%)	Microstructural parameters					
			Matrix grains		Large grains		Key grains[c] (vol.%)	
			Thickness (μm)	Aspect ratio	Thickness (μm)	Aspect ratio	Matrix gains	Large grains
1-h annealed	2000 °C/1 h/25 MPa	98.0	0.99	2.07	3.37	1.46	40.7	0
3-h annealed	2000 °C/3 h/25 MPa	98.2	2.07	3.11	5.43	2.82	58.5	0
4-h annealed	2000 °C/4 h/25 MPa	98.3	2.19	3.15	5.60	3.36	62.3	0
8-h annealed	2000 °C/8 h/25 MPa	98.1	2.63	3.16	8.66	3.15	50.1	0

[a] Hot-pressing conditions: 1800 °C/1 h/25 MPa.
[b] Theoretical density = 3.284 g/cm^3.
[c] Total volume fraction of key grains ($V_T - V_{KG1} + V_{KG2} - V_{\text{common grains of KG1 and KG2}}$).[12]

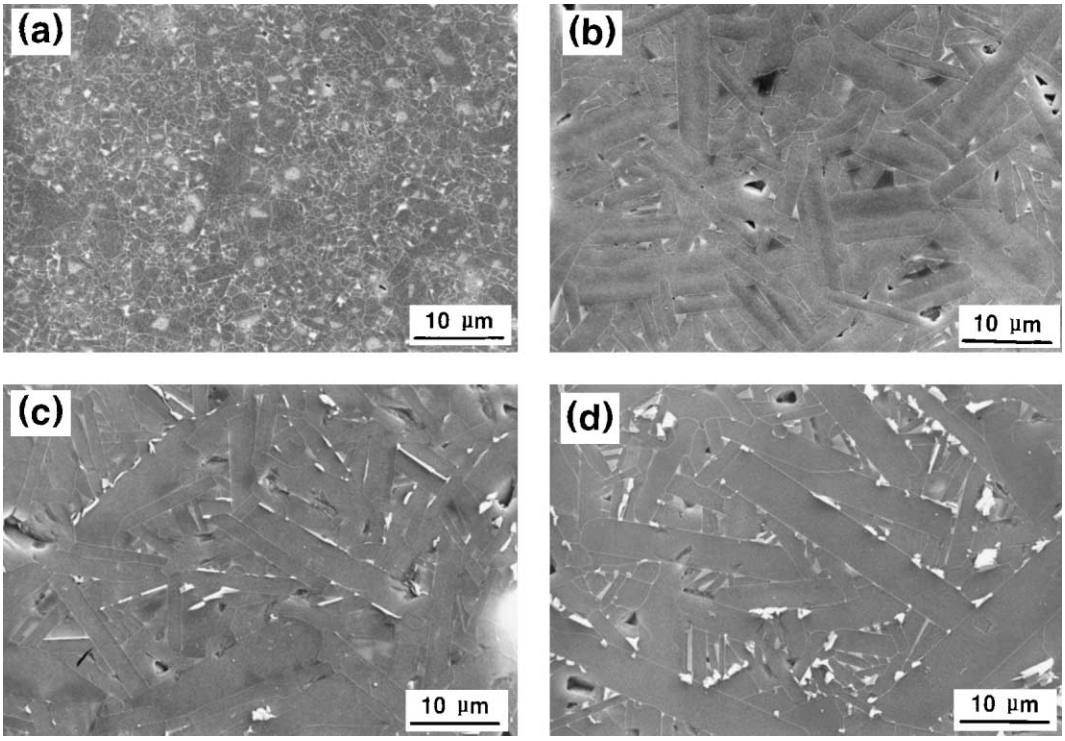

Fig. 1. Typical microstructures of annealed samples: (a) 1-h annealed, (b) 3-h annealed, (c) 4-h annealed, and (d) 8-h annealed samples.

aspect ratio of large grains decreases after 4-h annealing because of an increased impingement of elongated grains. In contrast, the average thickness increased gradually as the annealing time increased up to 8-h annealing. The increase in annealing time from 1 to 8 h increases the average thickness of large grains from 3.37 to 8.66 μm. These results indicate that the grain growth after 3-h annealing in the samples annealed with pressure is due mainly to an increase in grain thickness but not in aspect ratio.

Previous results[12] suggested that the total volume fraction of key grains, which take part in toughening mechanisms such as crack bridging and crack deflection, was a predominant factor regarding the toughening behavior of liquid-phase sintered SiC ceramics. The total volume fractions of 1-, 3-, 4-h, and 8-h annealed samples were 0.407, 0.585, 0.623, and 0.501, respectively.

3.2. Mechanical properties

Fig. 4 shows the variation of fracture toughness and strength as a function of annealing time. The 1-h annealed sample, which was composed of relatively equiaxed grains with bimodal distribution, had a fracture toughness of 4.6 MPa m$^{1/2}$ and a flexural strength of 495 MPa. In contrast, 4-h annealed sample, which was composed of relatively large, elongated grains with bimodal distribution, had a fracture toughness of 7.5

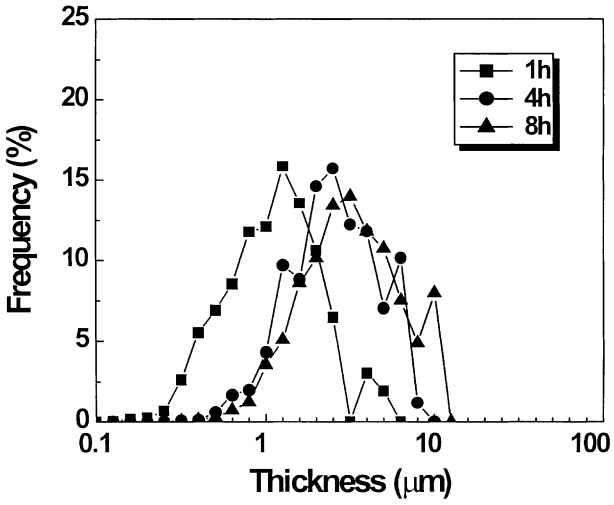

Fig. 2. Grain-thickness distribution revealed by the relation between grain thickness and areal frequency for 1-h annealed, 4-h annealed, and 8-h annealed samples.

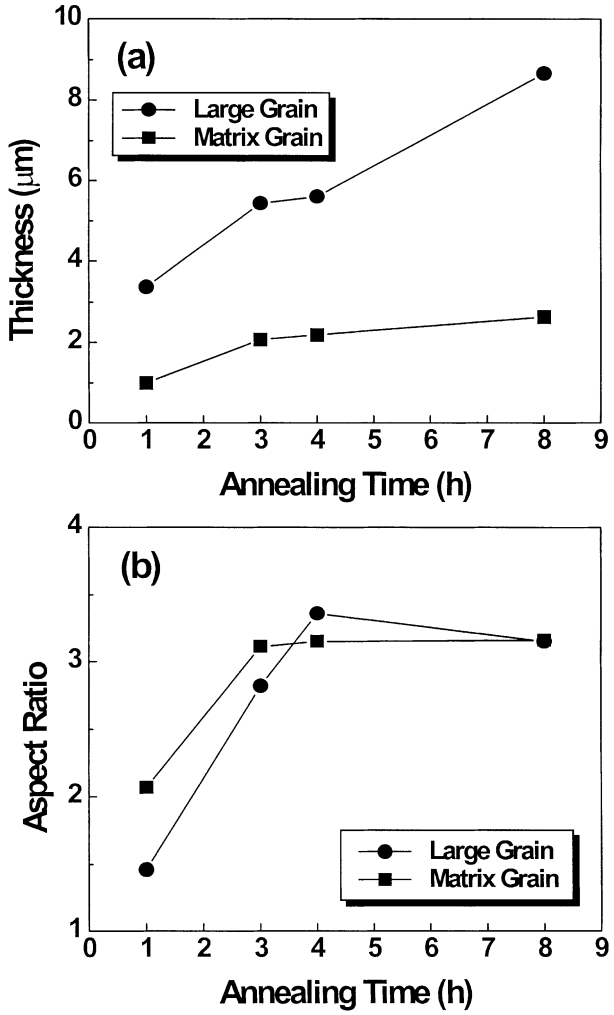

Fig. 3. Change of (a) grain thickness and (b) aspect ratio of large grains and matrix grains for liquid-phase sintered SiC samples annealed with uniaxial pressure, as a function of annealing time at 2000 °C.

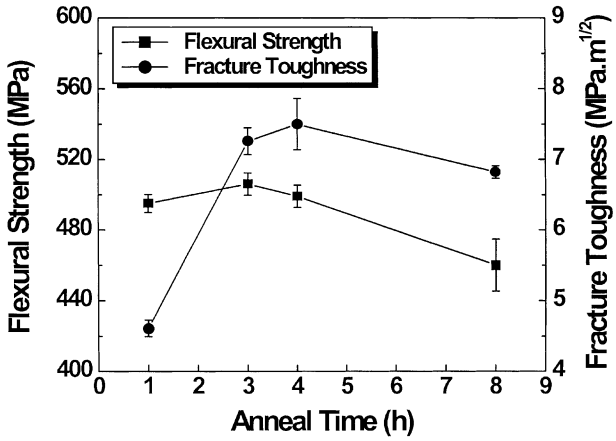

Fig. 4. Flexural strength and fracture toughness of the liquid-phase-sintered SiC samples annealed with pressure as a function of annealing time.

MPa $m^{1/2}$ and a flexural strength of 500 MPa. The growth of elongated α-SiC grains, i.e. development of toughened microstructure, produced the improvement in fracture toughness and maintained the strength of the 1-h annealed sample without degradation. However, further annealing up to 8 h led to the degradation in both toughness and strength, compared to the 4-h annealed sample.

The 4-h annealed sample showed a tortuous crack path and demonstrated significant crack bridging and deflection by elongated grains, as shown in Fig. 5(a). Typical fracture surface of the 4-h annealed sample is shown in Fig. 5(b). The fracture mode of 4-h annealed sample was a mixture of transgranular and intergranular. The increase in annealing time leads to an increased tendency of transgranular fracture because of relatively large grain size.

4. Discussion

When hot-pressed samples were annealed without pressure, a slight decrease of relative densities was commonly observed in liquid-phase sintered SiC.[1,14] Comparison of the present (Table 1) and the previous results[11,14] indicated that the samples annealed with pressure (relative density $\geqslant 98\%$) have higher densities than the samples annealed without pressure (relative density $\sim 97\%$). It suggests that annealing pressure is beneficial for preventing decrease in density during annealing.

Fig. 6 presents normalized grain-thickness (t/t_{avg}, where t_{avg} is an average grain thickness for overall grains) distributions of samples isothermally annealed with pressure at 2000 °C for various times. When normal grain growth occurs, the microstructure varies little with annealing time, and the microstructure development may be described by a simple law but, when abnormal grain growth occurs, a duplex grain structure

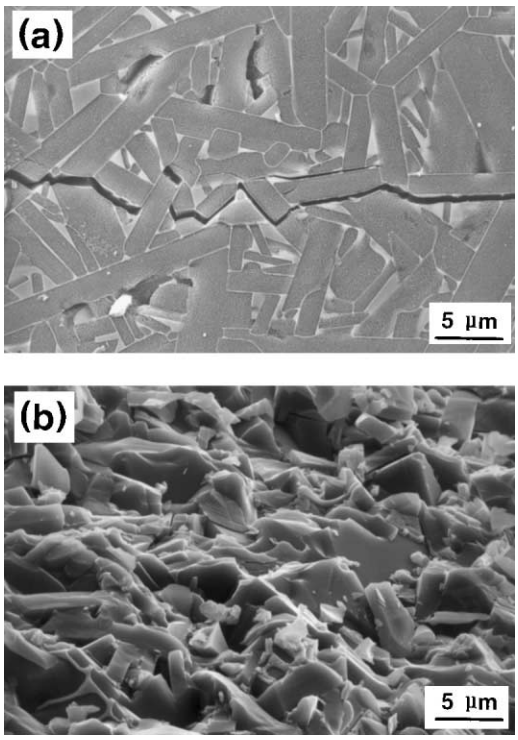

Fig. 5. Scanning electron micrographs of (a) a crack profile in 4-h annealed sample: crack bridging and deflection by elongated grains were observed; (b) typical fracture surface of 4-h annealed sample.

with a bimodal grain size distribution results; then no simple kinetic law can describe the microstructural development. As shown in Fig. 6, the thicknesses of the largest grains in each samples are larger than twice the respective average grain thicknesses (Log 2 ~0.301) and the distributions varied much with annealing time, indicating the occurrence of the abnormal grain growth. As the annealing time progresses, the distributions become flatten and the volume fraction of abnormally grown grains increases. The formation of a duplex microstructure in SiC ceramics, a result of abnormal grain growth, with some large α-SiC grains has been found to depend on the annealing temperature,[11] the β→α phase transformation,[1,3] initial grain size,[10] and initial grain size distribution.[7] Previous results[11] showed that the annealed samples with pressure showed bimodal grain thickness distribution, while the annealed samples without pressure showed the unimodal distribution. It is consistent with the present results. In this study, the starting β-SiC powder contained 1.1 wt.% α-SiC as seeds. During hot-pressing and annealing, the preexisting α-particles can act as a preferential nucleation site for grain growth. Then, the α-SiC grains grown on preexisting α-SiC particles must be larger than α-SiC particles nucleated within the liquid matrix via the β→α phase transformation of SiC. Thus, at the beginning of annealing at 2000 °C, the grain size distribution is likely to be bimodal and, therefore, abnormal grain growth can be taken place in 1-h annealed sample.

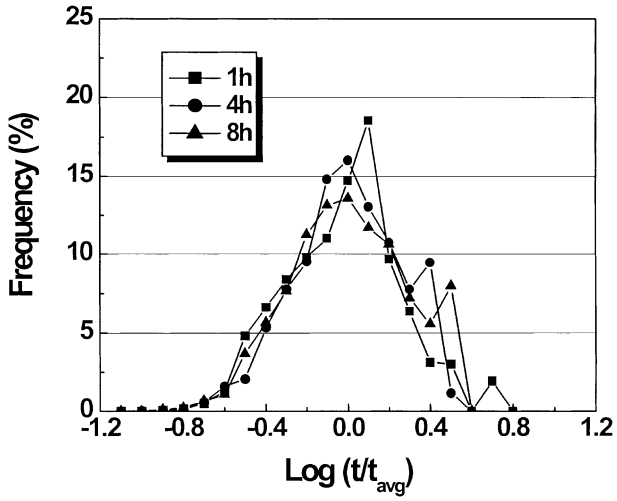

Fig. 6. Normalized grain-thickness distribution of liquid-phase sintered SiC samples annealed with uniaxial pressure.

For the microstructural development of 3-, 4- and 8-h annealed samples, only large α-SiC grains [as shown in the 1-h annealed sample, Fig. 1(a)] acted as nuclei for the growth of large, elongated grains, because the β→α phase transformation has already been finished after 1-h annealing and the growth of SiC grains proceeds by Ostwald ripening.[9,15] It is well documented that the faceted crystals can abnormally grow by two-dimensional nucleation and growth, for which the growth rate (R) is described by the following equation:[16]

$$R = A_1 \exp(-A_2/\Delta G) \quad (2)$$

where A_1 and A_2 are constants and ΔG is the driving force for the grain growth. The growth rate will thus remain negligible and increase sharply when ΔG exceeds a certain critical limit.[16,17] Thus, bimodal microstructure of 3, 4, and 8-h annealed samples resulted from the growth of large faceted grains existed in 1-h annealed sample [see Fig. 1(a)] via two-dimensional nucleation and growth. Present results are consistent with previous data[10] that the abnormal grain growth of faceted α-SiC grains during annealing is critically dependent on the average grain size of sintered materials.

Fig. 3 shows that the average thickness and aspect ratio of both matrix and large grains increase at an early stage of annealing. After 3-h annealing, the average thickness of both matrix and large grains grow continuously, but the growth in aspect ratio of matrix grains nearly stops and approaches ~3.2 and the aspect ratio of large grains decreases after 4-h annealing, owing to an increased impingement of elongated grains. These results suggest that growth in thickness has a stronger tendency than that in aspect ratio. In comparison to a previous result for grain growth in the annealed sample without pressure,[12] the average thickness of the 4-h annealed sample with pressure (2.33 μm for overall

grains) was larger than that of the 4-h annealed sample without pressure (1.02 μm); the latter has a unimodal grain size distribution, whereas the average aspect ratios of both samples for overall grains were approximately the same (3.36 for annealed sample without pressure and 3.18 for annealed sample with pressure). According to a local compressive stress calculation on any inclined boundary by Chen and Hwang,[18] the applied uniaxial stress during annealing places 2/3 of overall grain boundaries under compression. The compressive stress increases the solubility of the grains in compression[19] and, thus, accelerates the grain growth of some grains because the grain growth of faceted α-SiC grains is controlled by interface reaction.[20] In a previous work,[11] the applied pressure retarded the phase transformation of SiC, and the retardation of phase transformation depressed the abnormal grain growth. In contrast, the phase transformation has been taken place completely in the present study because of higher annealing temperature (~2000 °C). After the completion of the phase transformation, the grain growth of α-SiC grains is controlled by interface reaction.[20] Thus, the applied pressure accelerated the grain growth of faceted α-SiC grains.

As shown in Fig. 5(b), the fracture mode in the 4-h annealed sample was a mixture of transgranular and intergranular, which was a result of a weak interface created by the difference between the coefficients of thermal expansion of the liquid and the SiC grains after annealing.[21] Faceting of the fracture surface was observed in Fig. 5(b), indicating increased crack deflection. Tortuous fracture surface and transgranular fracture of some grains were also observed, indicating the occurrence of crack bridging mechanism [Fig. 5(b)].

Fracture toughness as a function of the volume fraction of key grains (V_T) is shown in Fig. 7. Previous data[12] obtained from the samples annealed without pressure were also included in Fig. 7. The fracture toughness increases linearly with increasing the volume fraction of key grains (V_T). The results confirm the linear relationship between the fracture toughness and the volume fraction of key grains, which was observed and suggested in toughened SiC samples annealed without pressure.[12] It should be noted that all the key grains (Table 1) in each samples belong to the matrix grains in all samples, as revealed by the separation points (>3 μm) between the large grains and the matrix grains. Previous result[12] suggested that the occurrence of elastic bridging was strongly limited to elongated grains with $t < 3$ μm. This restriction can be explained as follows. At the moment when the crack tip is just behind the toughening grain, the grain is stressed by bending. Since thick grains expose a lower flexibility than thin ones, larger α-SiC grains with $t > 3$ μm may not act as elastic bridges and fail during the period of bending, as observed in previous work.[12] Present results support the

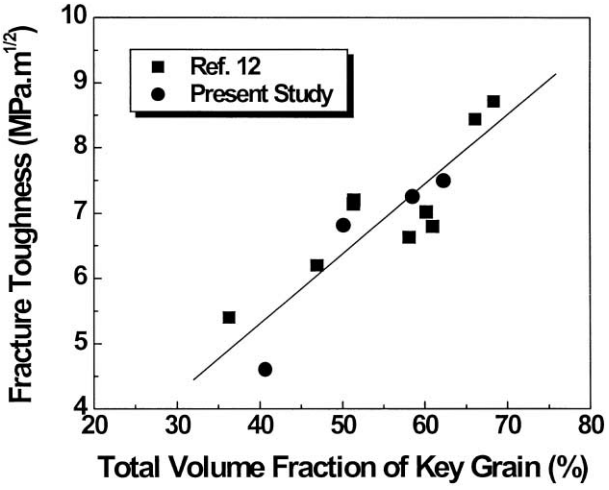

Fig. 7. Relation between fracture toughness and the total volume fraction of key grains. Previous data[12] obtained in samples annealed without pressure were included.

previous suggestion[12] that a bimodal microstructure itself is not important for toughening. The total volume fraction of key grains is a predominant factor regarding the toughening behavior of liquid-phase sintered SiC ceramics. Observations of Fig. 5 and the results of Fig. 7 suggest that both crack bridging and deflection by the key grains contribute to the increased toughness of the annealed samples.

5. Conclusions

In situ-toughened microstructures with bimodal grain-size distributions were developed during annealing with uniaxial pressure of liquid-phase sintered SiC. The bimodal grain-size distribution was related to abnormal grain growth of a small number of grains. Both the broad grain-size distribution of α-SiC nuclei, originating from the α-SiC seeds added and from the β→α phase transformation, and the two-dimensional nucleation and growth of faceted α-SiC grains after the completion of the phase transformation were responsible to the abnormal grain growth in liquid-phase sintered SiC ceramics annealed with uniaxial pressure. The average thickness and aspect ratio increase at an early stage of annealing. After 3-h annealing, grain growth in thickness direction has a stronger tendency than that in aspect ratio, owing to an increased impingement of elongated grains. The relationship between fracture toughness and volume fraction of key grains in annealed material with uniaxial pressure followed the linear law approximation, as suggested in toughened SiC ceramics annealed without pressure.[12] Typical flexural strength and fracture toughness of the 4-h annealed sample were ~500 MPa and ~7.5 MPa $m^{1/2}$, respectively.

Acknowledgements

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