

Possible Strategies for Microstructure Control of Liquid-Phase-Sintered Silicon Carbide Ceramics

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ABSTRACT

Keys to the attainment of tailored properties in SiC ceramics are microstructure control and judicious selection of the sintering additives. In this study, three different strategies for controlling microstructure of liquid-phase-sintered SiC ceramics (LPS-SiC) have been suggested: control of the initial α -SiC content in the starting powder, a seeding technique, and a post-sintering heat treatment. The strategies suggested offer substantial flexibility for producing toughened SiC ceramics whereby grain size, grain size distribution, and aspect ratio can be effectively controlled. The present results suggest that the proposed strategies are suitable for the manufacture of toughened SiC ceramics with improved toughness.

Key words : Silicon carbide, Microstructure, Liquid-phase-sintering, Processing

1. Introduction

Silicon carbide is an important structural ceramic because of its unique properties, including high temperature strength and resistance to wear, corrosion, and thermal shock.^{1,3)} Because of the highly covalent nature of the Si-C bond and low self-diffusion coefficients, SiC must be densified with sintering additives. Densification is achieved in solid-state sintering by using B and C additives,⁴⁾ and in liquid-phase sintering by using metal oxides additives.⁵⁻⁷⁾ Interest in LPS-SiC has expanded continually in recent years, because these materials are easier to process and they appear to offer superior mechanical properties over solid-state sintered SiC.⁸⁻¹⁰⁾

Tailored properties in LPS-SiC ceramics can be attained by microstructure control^{11,12)} and judicious selection of the sintering additive composition, which influence the chemistry of the glassy phase in the resulting ceramics.^{7,13,14)} Clear examples are SiC nanoceramics for superplasticity^{15,16)} and toughened SiC ceramics for improved toughness.^{17,18)} Various kinds of liquid-forming additives, such as Al_2O_3 ,¹⁹⁾ $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3$,^{1,5)} $\text{Al}_2\text{O}_3\text{-Gd}_2\text{O}_3$,²⁰⁾ $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3\text{-CaO}$,^{2,11)} $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3\text{-MgO}$,²¹⁾ $\text{Y}_2\text{O}_3\text{-AlN}$,²²⁾ $\text{Yb}_2\text{O}_3\text{-AlN}$,²³⁾ $\text{Er}_2\text{O}_3\text{-AlN}$,²⁴⁾ $\text{Lu}_2\text{O}_3\text{-AlN}$,²⁶⁾ $\text{Sc}_2\text{O}_3\text{-AlN}$,²⁷⁾ and Y-Mg-Si-Al-O-N oxynitride glass,²⁸⁾ and their effects on mechanical properties, have already been explored with respect to the liquid-phase sintering of SiC.

The control of microstructure is also one of the primary

concerns in LPS-SiC, because toughened SiC ceramics with a microstructure containing anisotropically grown, plate-shaped SiC grains are known to exhibit high fracture toughness.^{1-3,11)} Recently, several attempts to control the microstructure of SiC ceramics have been reported, including seeding for preferential grain growth¹¹⁾ and the incorporation of α -SiC platelets.²⁹⁾ However, strategies for microstructure control of LPS-SiC are not well established. In this study, SiC ceramics sintered with $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3$, $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3\text{-CaO}$, or Y-Mg-Si-Al-O-N oxynitride glass have been prepared and their microstructures were observed by scanning electron microscopy (SEM) and subsequently characterized using an image analyzer. This paper focuses on possible strategies for controlling microstructure so as to achieve toughened SiC ceramics. Three different strategies for controlling the microstructure of LPS-SiC are suggested.

2. Experimental Procedure

The characteristics of SiC starting powders are listed in Table 1. Al_2O_3 (99.9% pure, Sumitomo Chemical Co., Tokyo, Japan), Y_2O_3 (99.9% pure, Shin-Etsu Chemical Co., Tokyo, Japan), CaO (99.9% pure, High Purity Chemicals, Japan), SiO_2 (Reagent Grade, Kanto Chemical Co., Inc., Tokyo, Japan), MgO (High-Purity Grade, Wako Pure Chemical Industries, Ltd., Osaka, Japan), and AlN (Grade F, Tokuyama Soda Co., Tokyo, Japan) powders were used as sintering additives. For the composition of SC4 and SC5, ultrafine β -SiC powder (Sumitomo-Osaka Cement Co., Tokyo, Japan) was oxidized at 600°C for 2 h under air to eliminate free carbon, and hydrofluoric acid-treated to remove SiO_2 . The particle size of the β -SiC powder (β -SiC-2

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Table 1. SiC Powder Characteristics and Designation

| Characteristic | Type of powder | | | |
|---|---------------------------|---------------------------|---|--------------------------------|
| | α -SiC (A-1) | β -SiC (B-1) | β -SiC (F) | β -SiC (Ultrafine) |
| Average particle size (μm) | 0.45 | 0.43 | 0.04 | 0.32 |
| Specific surface area (m^2/g) | 15 | 15 | 46.8 | 20.0 |
| Crystalline phase | α | β | β | β |
| Impurity (wt%) | 0.37 | 0.55 | 1.00 | 0.22 |
| Oxygen free carbon | 1.08 | 1.67 | 1.88 | 0.87 |
| Manufacturer | Showa Denko, Tokyo, Japan | Showa Denko, Tokyo, Japan | Sumitomo-Osaka Cement Co., Tokyo, Japan | Ibiden Co., Ltd., Ogaki, Japan |
| Designation | α -SiC | β -SiC-1 | β -SiC-2 | β -SiC-3 |

Table 2. Batch Composition of SiC Ceramics

| Material | Batch composition (wt%) |
|----------|--|
| SC1 | 83.8% β -SiC-1* + 7% Al_2O_3 + 9.2% Y_2O_3 |
| SC2 | 8.4% α -SiC** + 75.4% β -SiC-1 + 7% Al_2O_3 + 9.2% Y_2O_3 |
| SC3 | 83.8% α -SiC + 7% Al_2O_3 + 9.2% Y_2O_3 |
| SC4 | 85% β -SiC-2 [†] + 10% Oxynitride Glass [‡] |
| SC5 | 75% β -SiC-2 + 10% β -SiC whisker [‡] + 10% Oxynitride Glass [‡] |
| SC6 | 1.0 α -SiC + 89.0% α -SiC-3 [†] + 5.7% Al_2O_3 + 3.3% Y_2O_3 + 1.0% CaO |

* $\sim 0.43 \mu\text{m}$, B-1 grade, Showa Denko, Tokyo, Japan.

** $\sim 0.45 \mu\text{m}$, A-1 grade, Showa Denko, Tokyo, Japan.

† $\sim 30 \text{ nm}$, Sumitomo-Osaka Cement Co., Tokyo, Japan.

‡ The composition of oxynitride glass is $\text{Y}_{0.124}\text{Mg}_{0.160}\text{Si}_{0.414}\text{Al}_{0.302}\text{O}_{1.400}\text{N}_{0.151}$.

‡ The diameter and length are $0.1\text{--}1.0 \mu\text{m}$ and $10\text{--}30 \mu\text{m}$, respectively. Tokamax, Takai Carbon Co., Ltd., Tokyo, Japan.

† $\sim 0.32 \mu\text{m}$, Ultrafine grade, Betarundum, Ibiden Co., Ltd., Ogaki, Japan.

in Table 1) was $\sim 40 \text{ nm}$. A mixture of SiO_2 , MgO , Y_2O_3 , Al_2O_3 , and AlN powders was prepared to an oxynitride composition of $\text{Y}_{0.124}\text{Mg}_{0.160}\text{Si}_{0.414}\text{Al}_{0.302}\text{O}_{1.400}\text{N}_{0.151}$ by ball milling in hexane for 3 h using SiC media and a SiC container. The oxynitride composition had an appreciable SiC solubility at high temperatures and a good potential for crystallization control.³⁰⁾

Six batches of powders were mixed (Table 2). All the batches were milled separately in ethanol for 24 h using SiC grinding balls and a jar. The milled slurry was dried and sintered at temperatures between 1800°C and 1950°C with or without an applied pressure of 25 MPa. The heating rate was $20^\circ\text{C}/\text{min}$, and the cooling rate was $\sim 40^\circ\text{C}/\text{min}$ from 1800°C to 1200°C . Some sintered samples were further heated at 1900°C for 2–8 h under an applied pressure of 25 MPa to enhance grain growth.

The relative densities of the sintered and annealed specimens were determined by the Archimedes method. The theoretical densities of each specimen were calculated according to the rule of mixtures. The sintered and annealed specimens were cut, polished, and then etched by CF_4 plasma containing 7.8% O_2 . The microstructures were observed using SEM. The SEM micrographs were quantitatively analyzed by an image analysis, according to the procedure outlined in a previous study.¹⁰⁾ The thickness of each grain (t) was determined directly from the shortest grain dimension observed in two-dimensional images; the apparent length of each grain (l) was obtained from the largest

dimension. The mean value of the 10% highest observed aspect ratio (l/t) was considered to be the mean of the actual value. A total of 800 to 1200 grains were used for the statistical analysis of each specimen. X-Ray Diffraction (XRD) using $\text{CuK}\alpha$ radiation was performed on ground powders for the annealed specimens. The fracture toughness was determined by measuring the lengths of cracks generated by a Vickers indenter.³¹⁾

3. Results and Discussion

3.1. Microstructure Control by Controlling Initial α -SiC Content

Previous studies^{17,22)} showed that equiaxed grain morphology was obtained with high α -SiC powders, whereas elongated grain morphology resulted from β -SiC due to the $\beta \rightarrow \alpha$ phase transformation during sintering or annealing. In order to investigate the influence of the initial α -SiC content in the starting powder, three batches containing different polytypes of starting powders were prepared (SC1–SC3 in Table 2). The relative content of α -SiC powder in those batches was 0, 10, and 100 vol%. All specimens were sintered at 1950°C for 3 h. The microstructures of the sintered specimens are shown in Fig. 1. The microstructures of the specimens fabricated from 100% β -SiC (SC1) and 90% β /10% α powders (SC2) consisted of both elongated grains and equiaxed grains. In contrast, the microstructure of the specimen fabricated from α -SiC (SC3) consisted of mostly fine,

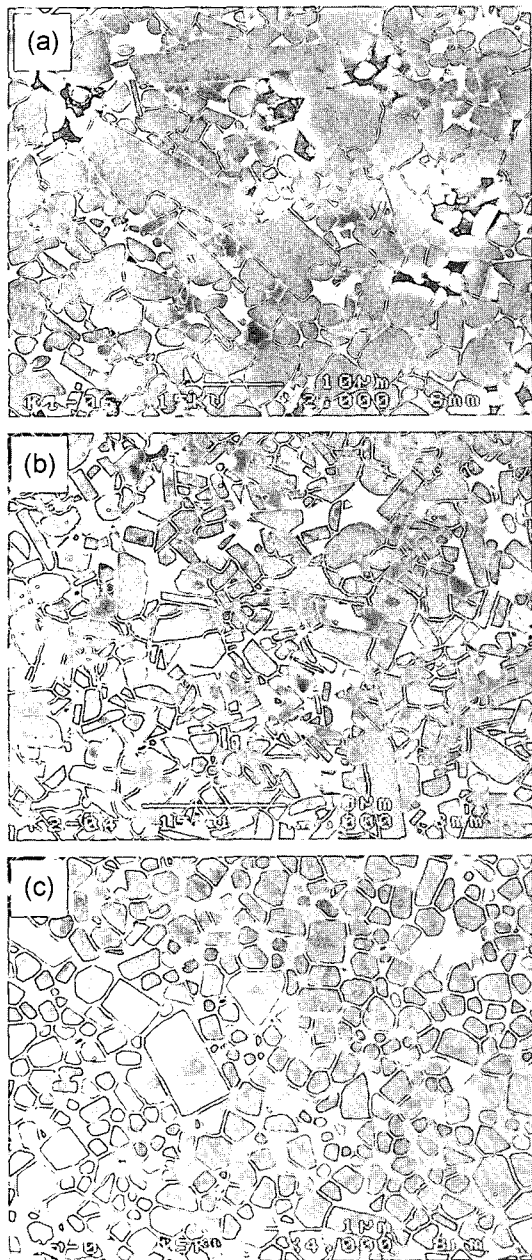


Fig. 1. Typical microstructures of SiC ceramics sintered at 1950°C for 3 h; (a) SC1, (b) SC2, and (c) SC3 (refer to Table 1).

equiaxed grains. The average thickness of SiC grains decreased from 2.7 μm for SC1 to 1.5 μm for SC2, and to 0.9 μm for SC1 with increasing α -content in the starting powder. Upon increase of the α -content, the shape of the grains changed from elongated to equiaxed and, as a consequence, the aspect ratio decreased from 3.9 for SC2 to 2.5 for SC3. A lower amount of initial α -phase in the starting powder resulted in a larger interparticle distance of α -grains and the grains could grow in the first stage of densification without hindrance. Therefore, the starting composition containing low ($\leq 10\%$) α -phase content developed a coarser microstructure with large, elongated grains. With

increasing initial α -phase content, steric hindrance of grain growth increases,²⁾ resulting in an increasingly equiaxed microstructure, as shown in Fig. 1. Thus, a large addition of α -SiC led to a decrease in grain length, resulting in fine, equiaxed microstructure. In contrast, from β powder or a mixture of α/β powders containing small ($\leq 10\text{ vol}\%$) amounts of α -content, a more elongated SiC grain structure could be obtained.

The fracture toughness of SC1, which was composed of both elongated and equiaxed grains, was $6.9 \pm 0.6 \text{ MPa} \cdot \text{m}^{1/2}$ whereas SC3, which was composed of relatively, small, equiaxed grains, displayed a fracture toughness of $4.4 \pm 0.5 \text{ MPa} \cdot \text{m}^{1/2}$. Thus, the introduction of elongated grains into the microstructure produced improved fracture toughness, as reported earlier.^{1-3,7,11)}

3.2. Microstructure Control by Seeding

Previous studies^{11,32)} showed that the introduction of larger seeds into fine-grained β -SiC accelerated the grain growth of elongated large grains during annealing via solution-precipitation, resulting in toughened microstructures. A material with large β -SiC seeds had a bimodal microstructure of small matrix grains and large elongated grains. In contrast, a material with large α -SiC seeds had a uniform microstructure consisting of elongated grains.¹¹⁾ In order to investigate the effect of seed shape on the microstructural development in LPS-SiC, β -SiC whiskers were added as seeds into fine-grained β -SiC. So as to simplify the analysis, the $\beta \rightarrow \alpha$ phase transformation was avoided completely by using an oxynitride glass ($\text{Y}_{0.124}\text{Mg}_{0.160}\text{Si}_{0.414}\text{Al}_{0.302}\text{O}_{1.4}\text{N}_{0.151}$) as a sintering additive, which enlarges the stability region of β -SiC up to 2000°C.³³⁾ SiC ceramics without seeds (SC4) and with seeds (β -SiC whiskers) (SC5) were hot-pressed at 1900°C for 1 h under a pressure of 25 MPa in a nitrogen atmosphere.

Relative densities of $>99.0\%$ were achieved by hot-pressing for both specimens. Typical microstructures of the hot-pressed specimens are shown in Fig. 2. The microstructure of SC4 consists of very fine, equiaxed grains (average size $\sim 190 \text{ nm}$). In contrast, the growth of large grains in small matrix grains, i.e., bimodal microstructure, is shown in materials with seeds (SC5). The microstructure of SC5 consisted of large, rod-like grains and fine matrix grains. Large grain size difference between seeds (whiskers) and matrix grains was a driving force for abnormal grain growth of the seeds via solution-precipitation.^{34,35)}

X-ray diffraction spectra for ground powders of the specimens without and with seeds showed the presence of β -SiC only as a crystalline phase, indicating the absence of phase transformation during sintering. This is consistent with previous results demonstrating that oxynitride glass enlarges the stability region of β -SiC.^{28,33)} Previous results^{10,11,32)} showed that the addition of large, equiaxed α -SiC seeds into a fine β -SiC matrix leads to elongation of some grains due to both the presence of α/β interfaces in grains and the occurrence of $\beta \rightarrow \alpha$ phase transformation. The present results suggest

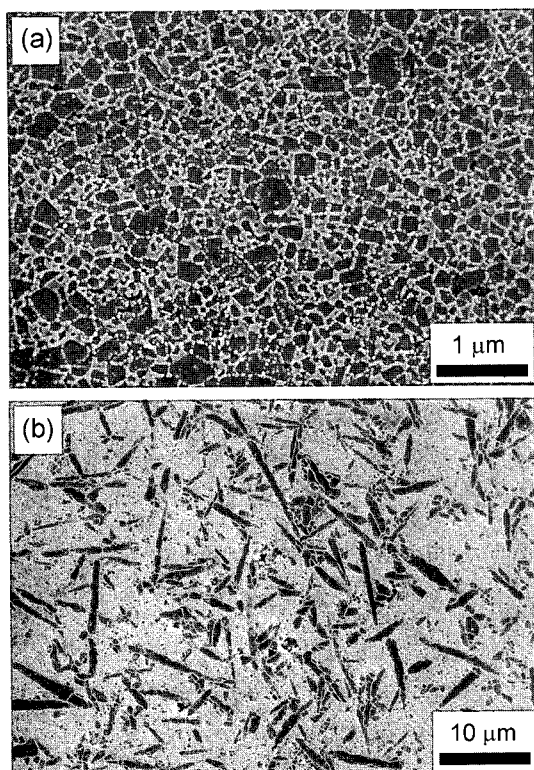


Fig. 2. Typical microstructures of SiC ceramics sintered at 1900°C for 1 h under 25 MPa in a nitrogen atmosphere; (a) SC4 and (b) SC5 (refer to Table 1).

that the grain morphology in LPS-SiC can be controlled by manipulating the seed shape, if there is no phase transformation during sintering and/or annealing.

The microstructure of SC5 is a new kind of “self-reinforced microstructure,” consisting of large, rod-like β -SiC grains and fine β -SiC matrix grains. Previously reported self-reinforced microstructures were composed of platelet-shaped α -SiC grains or platelet-shaped α/β composite grains.^{7,10,11} However, the present results demonstrate that self-reinforced β -SiC with rod-like β -SiC grains can be obtained by seeding SiC whiskers into a fine β -SiC matrix and sintering the specimen in a region where the β -SiC is stable. The fracture toughnesses of SC4 and SC5 were $2.7 \pm 0.4 \text{ MPa} \cdot \text{m}^{1/2}$ and $6.7 \pm 0.5 \text{ MPa} \cdot \text{m}^{1/2}$, respectively.

3.3. Microstructure Control by Heat-Treatment

Attempts to introduce elongated SiC grains into the SiC microstructure can be grouped into the following two strategies: (i) utilizing the $\beta \rightarrow \alpha$ phase transformation at high temperatures^{1,3,36} and (ii) use of a solution-precipitation.^{32,37,38} The latter efforts include the previously discussed approach of seeding for favoring preferential grain growth.^{11,32} One method to trigger the $\beta \rightarrow \alpha$ phase transformation of SiC is to anneal the sintered specimens at higher temperatures. In this study, SC6 specimens were first hot-pressed at 1800°C for 1 h under an applied pressure of 25 MPa in argon and subsequently annealed at 1900°C for

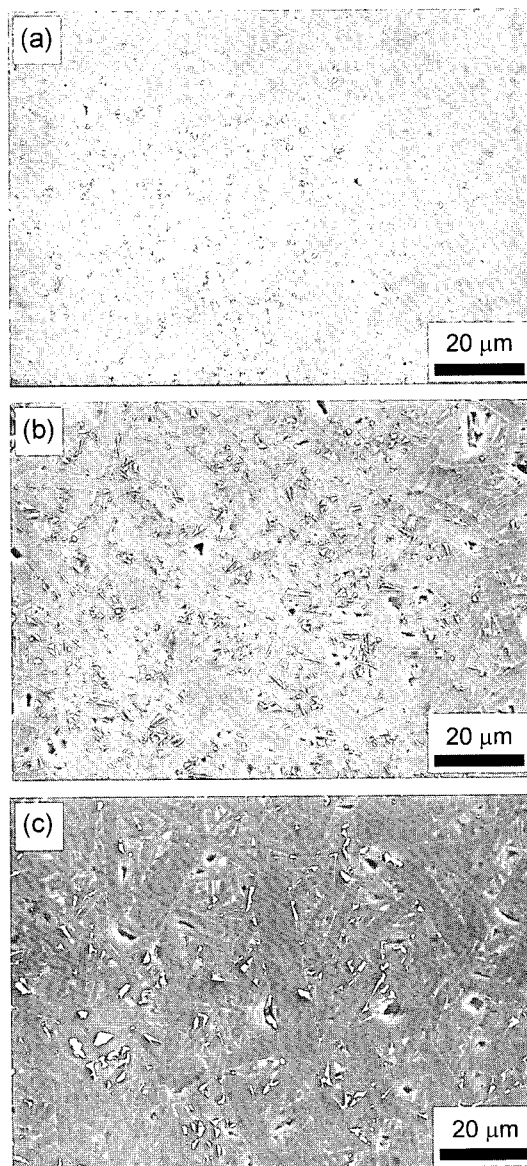


Fig. 3. Typical microstructures of SiC ceramics sintered at 1800°C for 1 h under 25 MPa in an argon atmosphere and subsequently annealed at 1900°C under 25 MPa in an argon atmosphere; (a) 2-h annealed specimen, (b) 4-h annealed specimen, and (c) 8-h annealed specimen.

2 – 8 h under 25 MPa in argon.

Relative densities of $\geq 98.5\%$ were achieved by hot-pressing and subsequent annealing with pressure for all specimens. All the β -SiC phases were transformed into α -SiC after annealing at 1900°C for 4 h. Typical microstructures of the hot-pressed and annealed specimens are shown in Fig. 3. The microstructure of the 2-h annealed specimen consisted of fine, equiaxed grains. In contrast, specimens annealed for 4-h and 8-h under pressure were comprised of small elongated grains and large elongated grains. Self-reinforced microstructures were obtained after 4-h annealing at 1900°C.

Table 3. Characteristics of Annealed Specimens (SC6)

| Specimen | Annealing conditions | Relative density (%) | Microstructural parameters | | | |
|--------------|----------------------|----------------------|-----------------------------|--------------|-----------------------------|--------------|
| | | | Matrix grain | | Large grain | |
| | | | Thickness (μm) | Aspect ratio | Thickness (μm) | Aspect ratio |
| 2-h annealed | 1900°C/2 h/25 MPa | 98.9 | 0.9 | 2.5 | 3.3 | 1.9 |
| 4-h annealed | 1900°C/4 h/25 MPa | 98.8 | 2.2 | 8.5 | 5.5 | 9.2 |
| 8-h annealed | 1900°C/8 h/25 MPa | 98.5 | 2.7 | 7.1 | 8.7 | 7.7 |

The quantitative image analysis results for grain thickness and aspect ratio are summarized in Table 3. The average thickness and aspect ratio of both matrix and large grains increased during annealing up to 4 h. However, after 4-h annealing, thickening of both matrix and large grains was observed. The increased thickness is attributed to increased impingement of elongated grains and the consequent decreased aspect ratio of both the matrix and large grains (Table 3). The present results suggest that the microstructure of SiC ceramics can be controlled by optimizing the annealing time at high temperatures. The fracture toughnesses of 4-h annealed and 8-h annealed specimens were $7.7 \pm 0.5 \text{ MPa} \cdot \text{m}^{1/2}$ and $6.8 \pm 0.5 \text{ MPa} \cdot \text{m}^{1/2}$, respectively.

4. Conclusions

Toughened microstructures in LPS-SiC ceramics were obtained by three different methods: control of initial α -SiC content in the starting powder, a seeding technique, and a post-sintering heat treatment. The strategies suggested in this study offer substantial flexibility for producing toughened SiC ceramics whereby grain size, grain size distribution, and aspect ratio can be effectively controlled. Further studies for adjusting and optimizing the processing parameters, initial α -SiC content, seed content and morphology, and heat-treatment conditions may enable microstructure control of LPS-SiC ceramics.

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