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Engineering porosity in silicon carbide ceramics

**Young-Wook Kim · Yong-Jae Jin · Jung-Hye Eom ·
In-Hyuck Song · Hai-Doo Kim**

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Abstract Porosity and microstructure control of the strut are essential for tailoring the properties of porous SiC ceramics. This study examined four different strategies for engineering the porosity of SiC ceramics: adjusting the template content, processing parameters, filler content, and sintering additive content. The suggested strategies offer substantial flexibility for producing SiC ceramics with engineered porosity, whereby the total porosity can be controlled effectively from 35 to 95%. These results suggest that combinations of the proposed strategies will be useful for the manufacture of porous SiC ceramics with engineered porosity.

Introduction

Porous silicon carbide ceramics exhibit a unique set of characteristics, such as low density, controlled permeability, high thermal shock resistance, high specific strength, and excellent corrosion resistance at high temperatures. These materials are used in a wide range of applications, such as filters for molten metals, diesel particulate filters, gas burner media, vacuum chucks, preforms for metal-matrix composites, membrane supports for hydrogen separation, and lightweight structural materials [1–8].

The properties of porous SiC ceramics can be tailored by controlling the porosity and microstructure of the strut, which affect the mechanical, physical, thermal properties of the resulting ceramics. For example, the increase in porosity generally decreases the mechanical strength [9–12] and increases the permeability of porous SiC ceramics [13, 14]. Moreover, porous SiC ceramics with smaller pores showed better flexural strength than ceramics with larger pores at an equivalent porosity [15, 16]. A range of processing strategies for engineering the porosity have been suggested [17, 18] and Colombo well discussed the merits and drawbacks of the different approaches to foaming ceramics [19]. The strategies for engineering porosity in SiC ceramics include adjusting the sacrificial template content and sintering temperature, and employing different processing methods, such as replication, direct foaming, template, partial sintering, and reaction techniques. Each processing technique results in different range of porosity and pore morphologies. In the replica method, a porous structure is coated with a SiC suspension or precursor solution to produce a porous SiC ceramic with the same morphology as the original material, and can produce a total porosity (hereafter porosity denotes the total porosity if there is no other mention) ranging from 75 to 88% [5, 20–22]. The direct foaming method includes gel-casting and gas bubble formation method [23–28]. The latter employs preceramic polymers as a SiC precursor. The precursors are saturated with CO_2, foamed by inducing thermodynamic instability through a rapid pressure drop, and transformed into SiC ceramics by pyrolysis and optional sintering [24–28]. The porosity obtained ranged from 45 to 60%. In the sacrificial template technique, a composite of a matrix of SiC particles and dispersed sacrificial phase was prepared. The sacrificial phase was ultimately extracted to generate pores within the microstructure, resulting in a SiC ceramic with porosity ranging from 20 to 65% [16, 29, 30]. The partial sintering

Y.-W. Kim (✉) · Y.-J. Jin · J.-H. Eom
Department of Materials Science and Engineering,
The University of Seoul, Seoul 130-743, Korea
e-mail: ywkim@uos.ac.kr

I.-H. Song · H.-D. Kim
Powder Materials Research Division, Korea Institute
of Materials Science, Changwon 641-010, Republic of Korea

technique sinters the SiC compacts at lower or higher temperatures than the optimum, resulting in residual porosity ranging from 34 to 45% [4]. The reaction methods include siliciding techniques [31–34], gas-phase reaction techniques [35, 36], and carbothermal reduction techniques [37–45]. These reaction methods produce a porosity ranging from 30 to 82% [31–45].

This paper focuses on possible strategies for controlling the porosity to achieve better performance in a range of applications. Four different strategies for controlling the porosity of SiC ceramics are suggested.

Experimental

The following raw materials were used: polycarbosilane (PCS; type A, Nippon Carbon Co., Yokohama, Japan), polysiloxane (PS; YR3370, GE Toshiba Silicones Co. Ltd., Tokyo, Japan), carbon black (CB; Corax MAF, Korea Carbon Black Co. Ltd., Inchon, Korea), phenol resin (PR; TD739, Kangnam Chemical Co., Ltd., Seoul, Korea), SiC (Ultrafine grade, Betarundum, Ibiden Co. Ltd., Ogaki, Japan), poly(methyl methacrylate-co-ethylene glycol dimethacrylate) microbeads (Template A; ~8 μm, 1.190 g/cm^3, Sigma–Aldrich Inc., St. Louis, MO), hollow microspheres (Template B; ~20 μm, 461DE20, Expancel, Sundsvall, Sweden), expandable microspheres (Template C; ~13 μm, 091DU40, Expancel, Sundvall, Sweden), Al_2O_3 (AKP30, Sumitomo Chemical Co., Tokyo, Japan), Y_2O_3 (H.C. Starck GmbH & KG, Goslar, Germany), and MgO (High Purity Chemicals, Osaka, Japan).

Eighteen batches of powders were mixed (Table 1). Template and reaction methods were used to fabricate the specimens. All batches were mixed for 6 h in a polyethylene jar containing ethanol and SiC balls. The milled powder mixture was dried and pressed uniaxially under 28 MPa to produce the rectangular bars. The compacts formed were cross-linked by heating to 200 °C in air. The cross-linked samples prepared from the SC1–SC6 (batches for the template method) were pyrolyzed at 1500 °C for 1 h at a heating rate of 1 °C/min in argon. The SC3 samples were also pyrolyzed at 1100–1400 °C for 1 h at the same heating rate to examine the effect of the sintering temperature.

Table 1 Sample designation and batch composition of porous SiC ceramics

Sample	Batch composition (wt%)	Remarks
SC1	60% PCS[a] + 40% Template A[b]	38.1 vol% template
SC2	50% PCS + 50% Template A	48.0 vol% template
SC3	40% PCS + 60% Template A	58.1 vol% template
SC4	30% PCS + 70% Template A	68.3 vol% template
SC5	20% PCS + 80% Template A	78.7 vol% template
SC6	10% PCS + 90% Template A	89.3 vol% template
SC7	53.6% PS[c] + 8.4% CB[d] + 30% SiC[e] + 3% Template B[f] + 3% Al_2O_3 + 2% Y_2O_3	41.0 vol% template
SC8	51.9% PS + 8.1% CB + 30% SiC + 5% Template B + 3% Al_2O_3 + 2% Y_2O_3	54.0 vol% template
SC9	49.3% PS + 7.7% CB + 30% SiC + 8% Template B + 3% Al_2O_3 + 2% Y_2O_3	66.0 vol% template
SC10	47.5% PS + 7.5% CB + 30% SiC + 10% Template B + 3% Al_2O_3 + 2% Y_2O_3	72.0 vol% template
SC11	72.5% PS + 8.6% CB + 9% SiC + 10% Template C[g] + 3% Al_2O_3 + 2% Y_2O_3	80.0 vol% template
SC12	49.1% PS + 7.7% CB + 18.9% SiC + 19.0% Template A + 3.7% Al_2O_3 + 1.1% Y_2O_3 + 0.5% MgO	40 wt% filler
SC13	36.1% PS + 5.7% CB + 31.4% SiC + 21.0% Template A + 4.1% Al_2O_3 + 1.2% Y_2O_3 + 0.6% MgO	60 wt% filler
SC14	20.2% PS + 3.2% CB + 46.7% SiC + 23.5% Template A + 4.5% Al_2O_3 + 1.3% Y_2O_3 + 0.7% MgO	80 wt% filler
SC15	10.7% PS + 1.7% CB + 55.8% SiC + 24.9% Template A + 4.8% Al_2O_3 + 1.4% Y_2O_3 + 0.7% MgO	90 wt% filler
SC16	17% PS + 3% PR[h] + 80% Template A	No additive
SC17	17% PS + 3% PR + 80% Template A + 1.2% Al_2O_3 + 1.8% Y_2O_3	3 wt% additive
SC18	17% PS + 3% PR + 80% Template A + 2.0% Al_2O_3 + 3.0% Y_2O_3	5 wt% additive

[a] Polycarbosilane, Type A, Nippon Carbon Co., Ltd., Yokohama, Japan

[b] ~8 μm, poly(methyl methacrylate-co-ethylene glycol dimethacrylate), Sigma–Aldrich Inc, St. Louis, MO

[c] Polysiloxane, YR3370, GE Toshiba Silicones Co. Ltd., Tokyo, Japan

[d] Carbon black, Corax MAF, Korea Carbon Black Co., Ltd., Inchon, Korea

[e] SiC, Ultrafine, Betarundum, Ibiden co. Ltd., Ogaki, Japan

[f] ~20 μm, expanded microsphere, 461DE20, Expancel, Sundsvall, Sweden

[g] ~13 μm, expandable microsphere, 091DU40, Expancel, Sundsvall, Sweden

[h] Phenol resin, TD739, Kangnam Chemical Co., Ltd., Seoul, Korea

The samples prepared from SC7 to SC18 (batches for the reaction method) were pyrolyzed at 1100 °C for 1 h at a heating rate of 1 °C/min in argon. The pyrolyzed specimens were further heat-treated in argon to 1450 °C for 1 h at a heating rate of 10 °C/min and then sintered at various temperatures. The SC7–SC10 specimens for examining the effect of the template content were sintered at 1800 °C with a 1 h hold for liquid-phase sintering of SiC using Al_2O_3 and Y_2O_3. The SC7 samples were sintered at 1800, 1900, and 2000 °C to examine the effect of the sintering temperature. The SC11 specimens for investigating the effect of the sintering time were sintered at 1950 °C for 1, 3, and 6 h. The SC12–SC15 specimens for investigating the effect of the passive filler content were sintered at 1750 °C for 2 h. The SC14 samples were also sintered at 1800–1900 °C to examine the effect of the sintering temperature. The SC16–SC18 specimens used to evaluate the effect of the additive content were sintered at 1650 °C for 2 h.

The bulk density of the porous ceramics was calculated from the weight-to-volume ratio of the samples. The cell and grain morphology was observed by scanning electron microscopy. The total porosity was determined from the bulk density to true density ratio. The open porosity of some selected specimens was measured using a mercury porosimeter (Auto-Pore IV Series, USA). The flexural strength of some selected specimens with a size of $3 \times 4 \times 30$ mm^3 was measured using a three-point method with a span and cross-head speed of 20 mm and 0.5 mm/min, respectively.

Results and discussion

Effect of template content

The simplest method for adjusting the porosity is to adjust the sacrificial template content [4, 46–48]. Previous studies [42, 43, 48, 49] reported that the porosity of porous SiC ceramics generally increases with increasing template content. As shown in Fig. 1, the SC7–SC10 specimens sintered at 1800 °C followed the general trend between porosity and template content. However, the SC1–SC6 specimens showed a deviation from the general trend. The porosity increased from 62 to 88% with increasing template content from 38 to 79 vol%. However, further addition of template to 89 vol% decreased the porosity from 88 to 80%. Therefore, the total porosity does not always increase with increasing template content. It appears that when the template content exceeds a critical limit, the porosity decreases due to excessive shrinkage of the specimens during heat-treatment. Indeed, the linear shrinkage of the SC6 specimen was 45% and higher than that (32%) of the SC5 specimen after pyrolysis at 1500 °C.

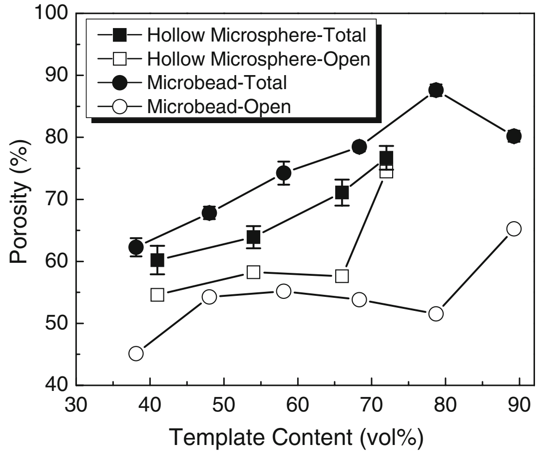

Fig. 1 Effect of the template content on the porosity of porous SiC ceramics. The total and open denote total porosity and open porosity, respectively

Figure 1 also shows open porosity of SC1–SC10 specimens as a function of template content. It can be seen that the open porosity increased slightly, then showed plateau, and increased again with increasing the template content for both set of specimens fabricated by the template and reaction methods. The difference between total porosity and open porosity, i.e., closed porosity generally increased with increasing the total porosity for both set of specimens. However, the specimens prepared by the reaction method (SC7–SC10) contained less closed porosity than the specimens processed by the template method (SC1–SC6) at the equivalent total porosity. The reaction method based on the carbothermal reduction of polysiloxane-derived SiOC evolves quite large amount of gaseous by-products [42, 43], which suppressed the formation of closed pores. In contrast, the porosity in SC1–SC6 specimens was formed by the decomposition of templates and subsequent pyrolysis of the polycarbosilane at low temperatures (<1000 °C). Thus, heat-treatment at 1500 °C increased the closed porosity by densifying the strut parts. The present results suggest that the reaction method is more favorable for increasing the open porosity whereas the template method by pyrolysis is beneficial for increasing the close porosity.

Figure 2 shows the typical fracture surfaces of the porous SiC ceramics with different template contents. A homogeneous microstructure with partially interconnected spherical cells was obtained for all specimens. The shape of the polymer microbeads was retained in those specimens up to its decomposition temperature. The coalescence of cells was also observed in the SC5 specimen and the occurrence of the coalescence phenomena increased with increasing polymer microbead content. There was no difference in cell morphology or cell size in each specimen when pyrolyzed at the same temperature.

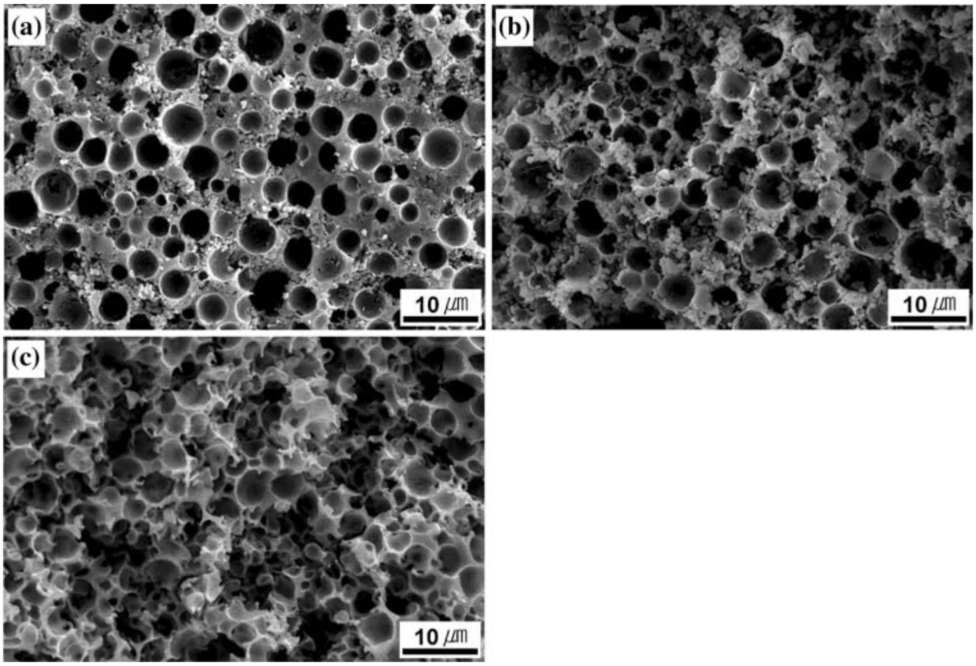

Fig. 2 Typical fracture surfaces of the porous SiC ceramics with various template contents: **a** 60% (SC3), **b** 70% (SC4), and **c** 80% (SC5)

However, there were differences in porosity, as shown in Fig. 1.

In this experiment, the polycarbosilane and polysiloxane were used as precursors for SiC. The polymeric materials acted as a binder for forming by uniaxial pressing. Therefore, up to 89 vol% of template (SC6) could be loaded. If powder processing is used, the maximum loading of the template is limited to approximately 60 vol% maximum [16] due to collapse of the specimens when the template is removed during sintering. In contrast, when polymeric precursors were used, the precursors transformed into amorphous phases during heat-treatment at temperatures between 700 and 1300 °C and acted as a transient binder to maintain the morphology of the pores and the shape of the specimens after removing the template [50]. These results suggest that an adjustment of the template content within a critical limit is a powerful tool for engineering the porosity. The critical limit would be dependent on the processing method and material system. Highly porous ceramics with a porosity >75% would be very difficult to fabricate if powder processing is used.

Effect of processing parameters

The most important processing parameters for engineering the porosity are the sintering temperature and sintering time because they mainly affect the sintered density and strut microstructure, i.e., porosity and pore morphology. To examine the effect of sintering temperature, the SC3, SC7, and SC14 specimens were sintered at various temperatures and the porosity obtained was plotted as a function of the normalized processing temperature (processing temperature divided by the maximum processing temperature). The SC3 specimen was prepared by pyrolysis at temperatures between 1100 and 1500 °C, and the SC7 and SC14 specimens were fabricated using a reaction method, i.e., carbothermal reduction and subsequent sintering process. SC7 was sintered at 1800–2000 °C and SC14 was sintered at 1750–1900 °C. As shown in Fig. 3, the SC3 specimens showed a minimum porosity (70.6%) at 1400 °C. There is an optimum sintering temperature leading to the maximum density in ceramic systems. When the temperature exceeds the optimum, the density decreases due to overfiring of the strut regions. The SC7 and SC14 specimens showed a decrease in porosity with increasing sintering temperature.

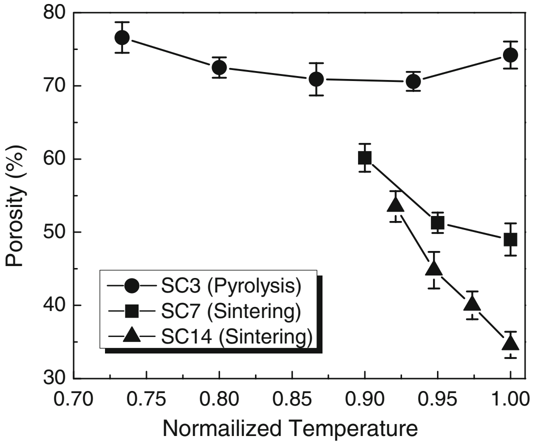

Fig. 3 Effect of processing temperature on porosity of porous SiC ceramics

The porosity of SC7 decreased from 60 to 49% with increasing sintering temperature from 1800 to 2000 °C. The porosity of the SC14 specimen decreased from 54 to 35% with increasing sintering temperature from 1750 to 1900 °C. The decrease in porosity was due to enhanced densification at higher temperatures. The SC7 specimen showed a decreased densification rate with increasing porosity whereas the SC14 specimen showed a rapid decrease in porosity with increasing temperature up to 1900 °C. The SC14 specimen contained 46.7 wt% (equivalent to 80 wt% in total SiC) of the passive filler, 23.5 wt% of the template, and a fairly large amount (6.5 wt%) of sintering additives. Those combinations led to continued densification in the specimens up to 1900 °C. However, it would be expected that the porosity would reach a minimum and increase again (similar to the SC3 specimen) with further increases in sintering temperature because there is an optimum sintering temperature for each system. Figure 4 shows the typical fracture surfaces of the SC3 and SC7 specimens sintered at different temperatures. The increase in processing temperature led to enhanced densification of the strut regions for both specimens resulting in decrease in porosity.

Figure 5 shows the effect of the sintering time on microstructure. The porosities of the 1-, 3-, and 6-h sintered SC11 specimens were 76, 74, and 76%, respectively. The porosity of the SC11 specimen reached a minimum at 3 h. In addition, there is an optimum sintering time leading to the maximum density in ceramic systems. When the sintering time exceeded the optimum, there was a decrease in density due to overfiring. However, the strut microstructure changed dramatically from a composite microstructure consisting of platelet grains and equiaxed grains for the 1-h sintered specimen to a microstructure consisting of platelet grains after prolonged sintering due to the $\beta \rightarrow \alpha$ phase transformation of SiC [51]. Therefore, the sintering time is not an effective parameter for engineering the porosity pf SiC ceramics. On the other hand, it is an important parameter for modifying the strut microstructure, which affects the mechanical properties and permeability of porous ceramics.

These results suggest that adjusting the sintering or pyrolysis temperature is an efficient way of controlling the porosity of SiC ceramics. However, a higher temperature and longer time than the optimum for densification generally leads to a decrease in porosity.

Effect of passive filler content

Figure 6 shows the porosity of the porous SiC ceramics fabricated with various passive filler contents (SC12–SC15). Depending on the passive filler content, the porosity of the porous SiC ceramics ranged from 50 to 64% when sintered at 1650 °C. Generally, the porosity decreased with increasing filler content. The density of the crosslinked specimens increased with increasing the passive filler content: 1.12, 1.27, and 1.32 g/cm^3 for SC12, SC14, and SC15, respectively. The more addition of a passive filler leads to a lower amount of a polysiloxane and carbon. The SiC yield from a mixture of polysiloxane and carbon was ~50% [39, 42], which indicates the evolution of less volatile species from the specimens containing higher amounts of passive filler. Therefore, the decrease in porosity with increasing filler content was attributed to the difference in both the

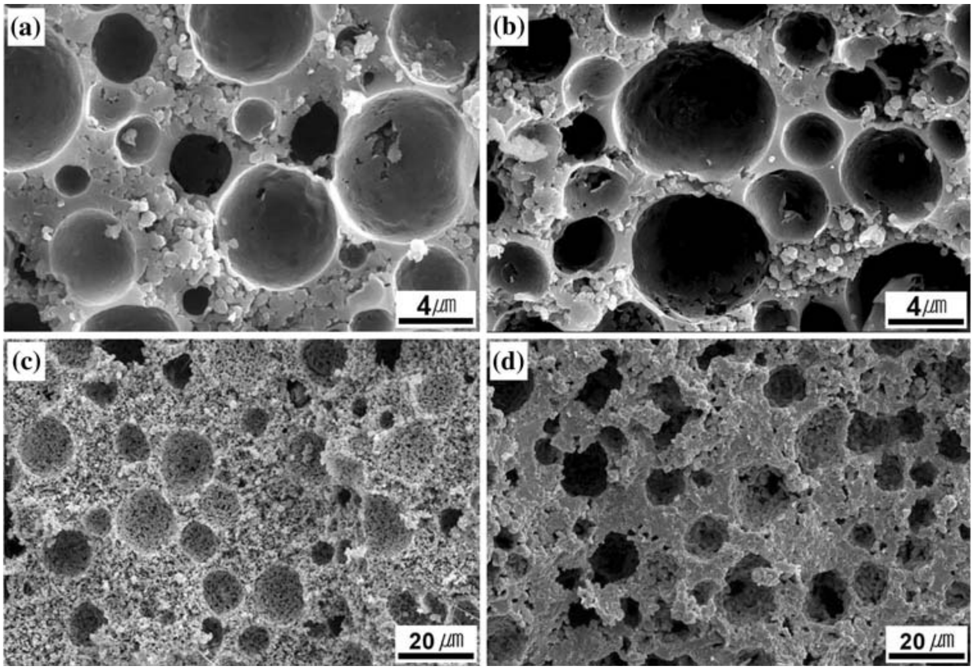

Fig. 4 Typical fracture surfaces of porous SiC ceramics processed at different temperatures: **a** 1200 °C (SC3), **b** 1400 °C (SC3), **c** 1800 °C (SC7), and **d** 1900 °C (SC7)

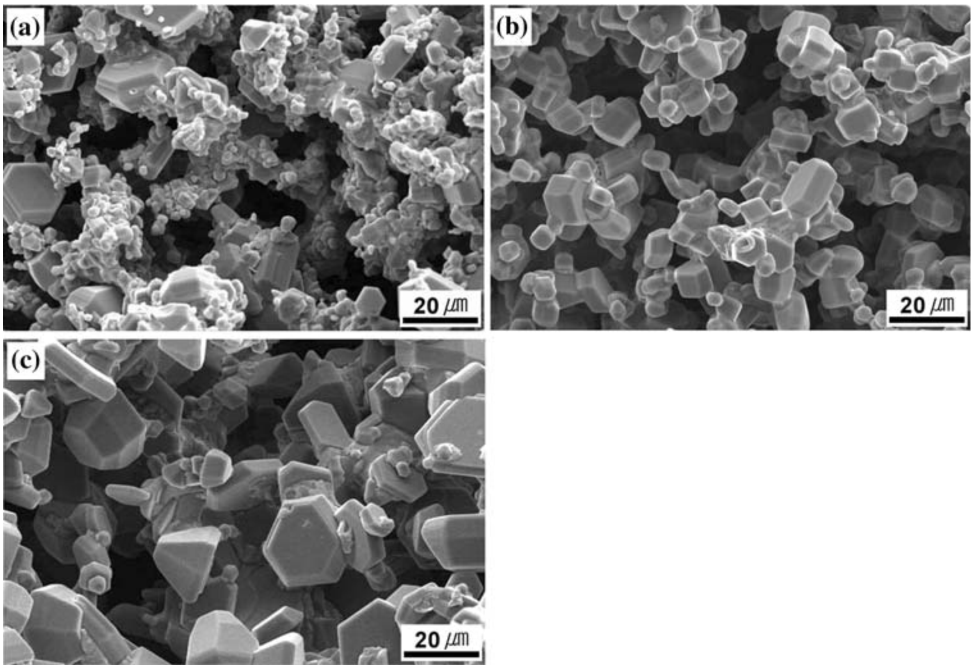

Fig. 5 Effect of the sintering time on the microstructure of porous SiC ceramics (SC11): **a** 1 h, **b** 3 h, and **c** 6 h at 1950 °C

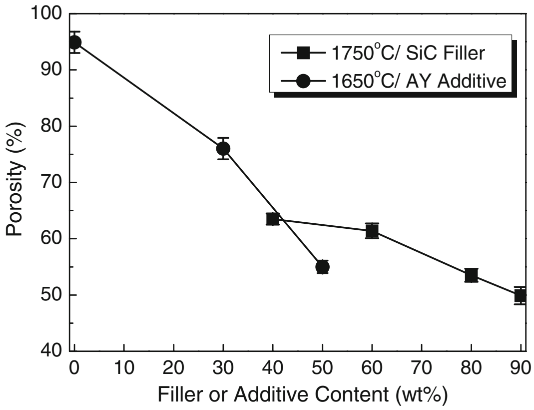

Fig. 6 Effects of the passive filler and additive contents (additive content ×10 times) on the porosity of porous SiC ceramics (SC12–SC18)

density of cross-linked specimens and the weight loss of each specimen during heat-treatment.

Figure 7 shows the typical microstructures of the SC12 (40% filler) and SC15 (90% filler) specimens. Both were sintered at 1750 °C for 2 h. There was no significant difference in the sizes of the pore and SiC grain but a difference in porosity: 64 and 50% porosities for the SC12 and SC15, respectively. This suggests that adjusting the passive filler content is an efficient way of tuning the porosity of porous SiC ceramics without affecting the pore size and grain size. A previous study [12] suggested that the addition of passive fillers was beneficial in improving the strength and dimensional control during the processing of SiC ceramics by a carbothermal reduction and subsequent sintering process.

Effect of additive content

The porosity of SiC ceramics can be engineered further by adjusting the additive content (Al_2O_3–Y_2O_3). Three batches were prepared to examine the effect of the additive content on porosity: SC16 with no additive, SC17 with 3 wt% additives, and SC18 with 5 wt% additives. As shown in Fig. 6, the porosity decreased dramatically with increasing additive content from 95% for SC16 to 55% for SC18. The addition of a higher additive content led to enhanced densification via liquid-phase sintering [52], resulting in lower porosity.

Figure 8 shows the typical microstructures of the SC16–SC18 specimens. The cell morphology was almost spherical and the windows in the struts were circular in SC16 and SC17. However, SC16 showed a more open cellular structure. In contrast, the addition of more additives (SC18) changed the cell morphology from spherical to irregular, and made the cell more closed due to enhanced densification of the strut region. Therefore, adjusting the additive content is a powerful tool for engineering the porosity of SiC ceramics but it is accompanied by a change in cell morphology and openness of the cells.

Mechanical property

The flexural strength of some selected specimens is shown in Fig. 9. The strength generally decreased with

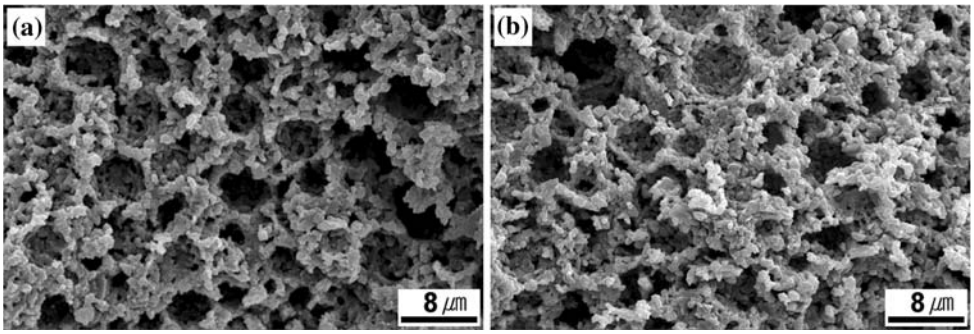

Fig. 7 Typical fracture surfaces of porous SiC with **a** 40% SiC filler (SC12) and **b** 90% SiC filler (SC15)

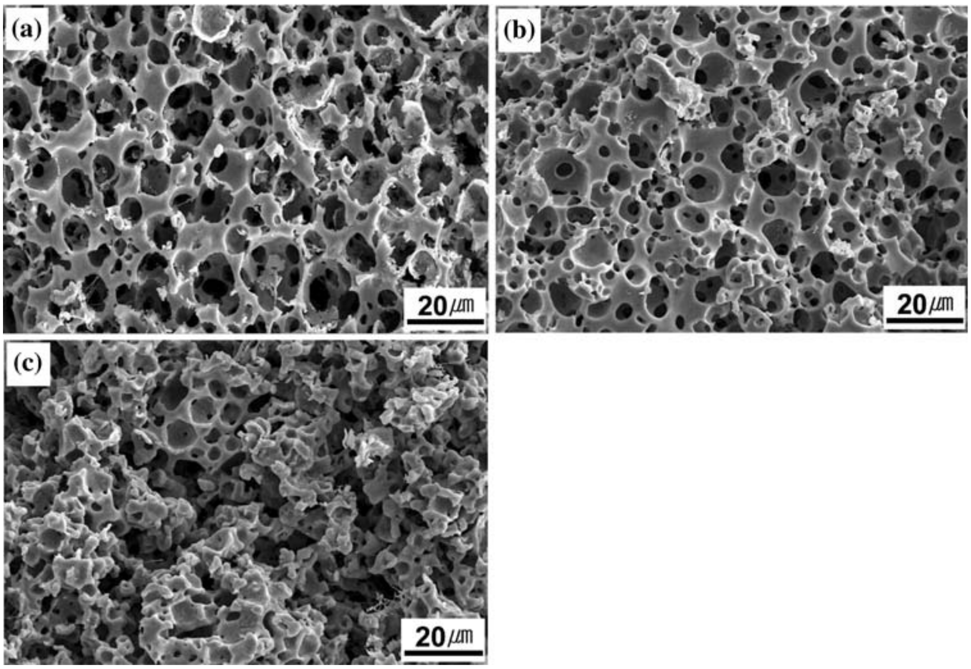

Fig. 8 Typical fracture surfaces of porous SiC ceramics sintered with various additive contents (Al_2O_3:Y_2O_3 in 2:3 weight ratio): **a** 0% (SC16), **b** 3% (SC17), and **c** 5% (SC18)

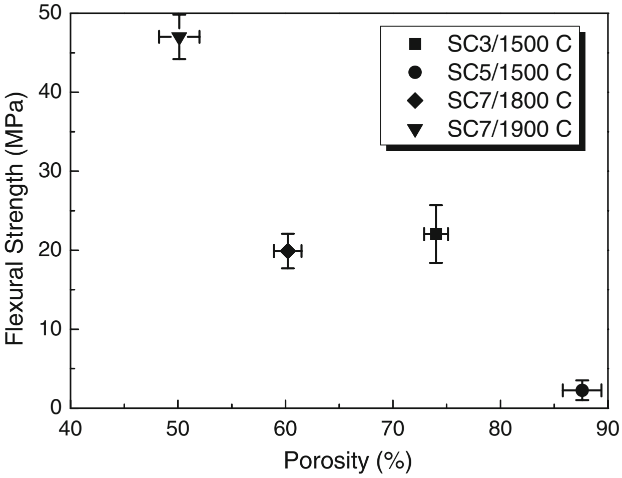

Fig. 9 Flexural strength of some selected specimens as a function of porosity

increasing porosity. The 1500 °C-sintered-SC3 specimen showed a strength value of 22.1 MPa at 74% porosity whereas the 1800 °C-sintered-SC7 specimen showed a strength value of 19.9 MPa at 60% porosity. Thus, the SC3 showed higher strength than the SC7 although it contained higher porosity. The comparison of Fig. 2a and Fig. 4c clearly shows that the better strength of SC3 was due to the finer pore size (6 µm vs. 15 µm) and denser strut of the specimen than 1800 °C-sintered-SC7. The 1900 °C-sintered-SC7 and 1500 °C-sintered-SC5 showed strength values of 47.0 MPa at 50% porosity and 2.3 MPa at 88% porosity, respectively. There are a few reports showing the mechanical properties of highly porous SiC ceramics. The flexural strength of porous SiC ceramics made by a replica technique and reaction method was 0.5–2.0 MPa at 80% porosity [21, 22] and 4 MPa at 73% porosity [45], respectively. The typical flexural strengths of the present porous SiC ceramics pyrolyzed at 1500 °C were 22 and 2.3 MPa at 74 and 88% porosity, respectively. The superior strength of the porous SiC ceramics fabricated from polycarbosilane by template method was attributed to the homogeneous microstructure (see Fig. 2a and c), smaller cell size (5–7 µm), and denser strut, compared to the porous SiC ceramics fabricated by reaction (see Fig. 4c and d) and replica method [21, 22].

Conclusions

The porosity in SiC ceramics was engineered using four different methods: adjusting the template content, processing parameters, filler content, and sintering additive content. Adjusting the template content is a powerful tool for engineering the porosity without altering the pore size and strut microstructure. Adjusting the sintering temperature affects the porosity and grain morphology, whereas adjusting the sintering time has little effect on porosity. Adjusting the passive filler content is also an efficient way of tuning the porosity of porous SiC ceramics without affecting both the pore size and grain size. The most dramatic change in porosity was obtained by controlling the additive content but the strategy accompanied a change in pore opening and pore morphology. The porosity of SiC ceramics was controlled from 35 to 95% using the different strategies. These results suggest that combinations of the proposed strategies would be useful for manufacturing porous SiC ceramics with engineered porosity and properties.

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