

Porosity control of porous silicon carbide ceramics

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Abstract

Porous SiC ceramics were fabricated by the carbothermal reduction of polysiloxane-derived SiOC containing polymer microbeads followed by sintering. The effect of the SiC powder:polysiloxane-derived SiC (SiC:PDSiC) ratio on the porosity and flexural strength of the porous SiC ceramics were investigated. The porosity generally increased with decreasing SiC:PDSiC ratio when sintered at the same temperature. It was possible to control the porosity of porous SiC ceramics within a range of 32–64% by adjusting the sintering temperature and SiC:PDSiC ratio while keeping the sacrificial template content to 50 vol%.

The flexural strengths generally decreased with increasing porosity at the same SiC:PDSiC ratio. However, a SiC:PDSiC ratio of 9:1 and a sintering temperature of 1750 °C resulted in excellent strength of 57 MPa at 50% porosity. Judicious selection of the sintering temperature and SiC:PDSiC ratio is an efficient way of controlling the porosity and strength of porous SiC ceramics.

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1. Introduction

Porous silicon carbide (SiC) ceramics possess a unique set of characteristics: low density, high specific strength, high thermal shock resistance, chemical stability, and high temperature stability. These materials are used in a broad range of applications, such as filters for molten metal, gas burner media, diesel particulate filters, preforms for metal–matrix composites, and light-weight structural materials for high-temperature applications.^{1–9}

Different processing routes for porous SiC ceramics have been developed for specific applications in order to satisfy the associated requirements of porosity, pore size, and degree of interconnectivity.^{10–15} These manufacturing techniques are typically divided into three categories: replica techniques, sacrificial template techniques and reaction techniques. The replica method is based on the impregnation or coating of a cellular structure with a SiC suspension or precursor solution to produce a porous SiC ceramic with the same morphology as

the original cellular structure. Polyurethane foams¹⁶ have been used as templates to fabricate porous SiC ceramics using this technique. The sacrificial template technique usually involves the preparation of a composite consisting of a matrix of SiC particles and a dispersed sacrificial phase that is ultimately extracted to generate pores within the microstructure. Polymer microbeads¹⁷ and hollow microspheres¹⁵ have been used as templates to fabricate porous SiC ceramics using this technique. There are two main types of reaction methods: siliciding and carbothermal reduction techniques. Siliciding techniques involve a chemical reaction between wood-derived carbon foams and molten Si,^{12,18,19} while carbothermal reduction techniques involve the carbothermal reduction of SiO₂, SiO, or SiOC using carbon.^{11,13,14,20}

In addition to the above approaches, new techniques that permit the deliberate tuning of the porosity and pore size of porous SiC ceramics with pores homogeneously distributed within the microstructure are of particular interest for applications in new areas, such as vacuum chucks and hot gas filters. The simplest processing route for adjusting the porosity is to adjust the sacrificial template content^{15,17} and sintering temperature.^{7,21,22} This paper reports an alternative way of controlling the porosity of porous SiC ceramics deliberately by adjusting the SiC

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powder:polysiloxane-derived SiC ratio (hereafter referred to as the “SiC:PDSiC ratio”) in the starting composition.

2. Experimental procedure

The following raw materials were used: α -SiC (FCP 15C, Norton AS, Eydehavn, Norway), polysiloxane (YR3370, GE Toshiba Silicones Co., Ltd., Tokyo, Japan), carbon black (Corax MAF, Korea Carbon Black Co., Ltd., Inchon, Korea), Al_2O_3 (AKP30, Sumitomo Chemical Co., Tokyo, Japan), Y_2O_3 (H.C. Starck GmbH & KG, Goslar, Germany), MgO (High Purity Chemicals, Osaka, Japan), and poly(methyl methacrylate-co-ethylene glycol dimethacrylate) microbeads ($\sim 8 \mu\text{m}$, Sigma-Aldrich Inc., St. Louis, MO, hereafter “polymer microbeads”). The oxides were used as sintering additives. The polymer microbeads were used as the sacrificial templates.

Five batches of the powder were prepared. The microbead content was fixed to 50 vol% (Table 1). Each batch was mixed for 24 h in a polyethylene jar containing the SiC balls and ethanol. The ethanol was used as a solvent for dissolving polysiloxane. The dissolution of polysiloxane was beneficial for making the powder mixture homogeneous. The milled powder mixture was dried and pressed uniaxially under 50 MPa to produce rectangular bars. The compacts formed were cross-linked by heating them to 200 °C in air. The cross-linked samples were pyrolyzed at 1000 °C for 1 h at a heating rate of 2 °C/min in argon. The heat treatment resulted in the conversion of polysiloxane to silicon oxycarbide.^{23,24} The pyrolyzed specimens were further heated to 1450 °C and held at that temperature for 0.5 h in argon. This resulted in the carbothermal reduction of the polysiloxane-derived SiOC.¹⁴ The samples were then sintered at 1750–1950 °C for 2 h in argon in order to cause the liquid-phase sintering of SiC. A graphite furnace was used for both heat treatment and sintering at 1450 °C and 1750–1950 °C, respectively.

The bulk density of each porous ceramic was calculated from its weight-to-volume ratio. The cell and grain morphology was examined by scanning electron microscopy. X-ray diffraction (XRD) was performed on ground powders using Cu K α radiation. The porosity was determined from the ratio of the bulk density to the true density. For the flexural strength measurements, bar-shaped samples were cut and polished to a size of 4 mm \times 5 mm \times 30 mm. Bend tests were carried out at room

temperature using a four-point method with inner and outer spans of 10 and 20 mm, respectively.

3. Results and discussion

3.1. Porosity control

The general reaction for synthesizing SiC from a mixture of polysiloxane and carbon black is reported elsewhere.^{15,17,21} The reaction occurs in two steps: (1) the pyrolysis of polysiloxane at 1000 °C, which involves the loss of the organic material and also leads to the conversion of polysiloxane to an amorphous Si–O–C material (SiOC) and (2) the carbothermal reduction of SiOC and C at 1450 °C, which converts the mixture to a SiC ceramic with the evolution of gaseous CO. Further heat treatment of the specimens at 1750–1950 °C using oxide additives led to liquid-phase sintering of the added SiC powder and SiC synthesized by the carbothermal reduction. XRD showed that the specimens sintered at 1750–1850 °C consisted of β -SiC, indicating that the SiC synthesized by carbothermal reduction was β -SiC. In contrast, the specimens sintered at 1900–1950 °C consisted of α -SiC and β -SiC, indicating the $\beta \rightarrow \alpha$ phase transformation of SiC.

Fig. 1 shows the typical fracture surfaces of the S9 (SiC:PDSiC = 9:1) specimens sintered at various temperatures for 2 h. The micrographs provide evidence of fine and well-distributed open cells with porous struts in the cellular structure regardless of the sintering temperature. The morphology of the primary cells (cells replicated from polymer microbeads) was almost spherical, indicating that the shape of the polymer microbeads was retained in the SiC–polysiloxane–carbon black-additives compacts up to its decomposition temperature. The cell size decreased from $\sim 7 \mu\text{m}$ to $\sim 5 \mu\text{m}$ with increasing sintering temperature from 1750 °C to 1950 °C. There were no large voids in the bulk samples when 50 vol% microbeads were added and sintered at 1750–1950 °C. This suggests that the processing method adopted here can avoid the significant agglomeration of microbeads while at the same time, allow for the uniform distribution of polysiloxane and other raw materials throughout the microspheres. These results also suggest that the pore size can be adjusted to some extent by controlling the sintering temperature. The grain size of SiC in the struts ranged from 0.5 to 2 μm in the 1750 °C-sintered specimen and 1 to 5 μm

Table 1
Sample designation and batch composition of the porous SiC ceramics.

Sample	Batch composition (wt%)							SiC:PDSiC weight ratio
	SiC ^a	Polysiloxane ^b	Carbon black ^c	Al_2O_3	Y_2O_3	MgO	Polymer microbead ^d	
S10	66.08			5.14	1.47	0.73	26.58	10:0
S9	55.79	10.71	1.68	4.82	1.38	0.69	24.93	9:1
S8	46.69	20.18	3.16	4.54	1.30	0.65	23.48	8:2
S6	31.36	36.14	5.67	4.07	1.16	0.58	21.02	6:4
S4	18.93	49.08	7.70	3.68	1.05	0.52	19.04	4:6

^a α -SiC (FCP 15C, Norton AS, Eydehavn, Norway).

^b YR3370, GE Toshiba Silicones Co., Ltd., Tokyo, Japan.

^c Corax MAF, Korea Carbon Black Co., Ltd., Inchon, Korea.

^d Poly(methyl methacrylate-co-ethylene glycol dimethacrylate) microbeads, $\sim 8 \mu\text{m}$, Sigma-Aldrich Inc., St. Louis, MO.

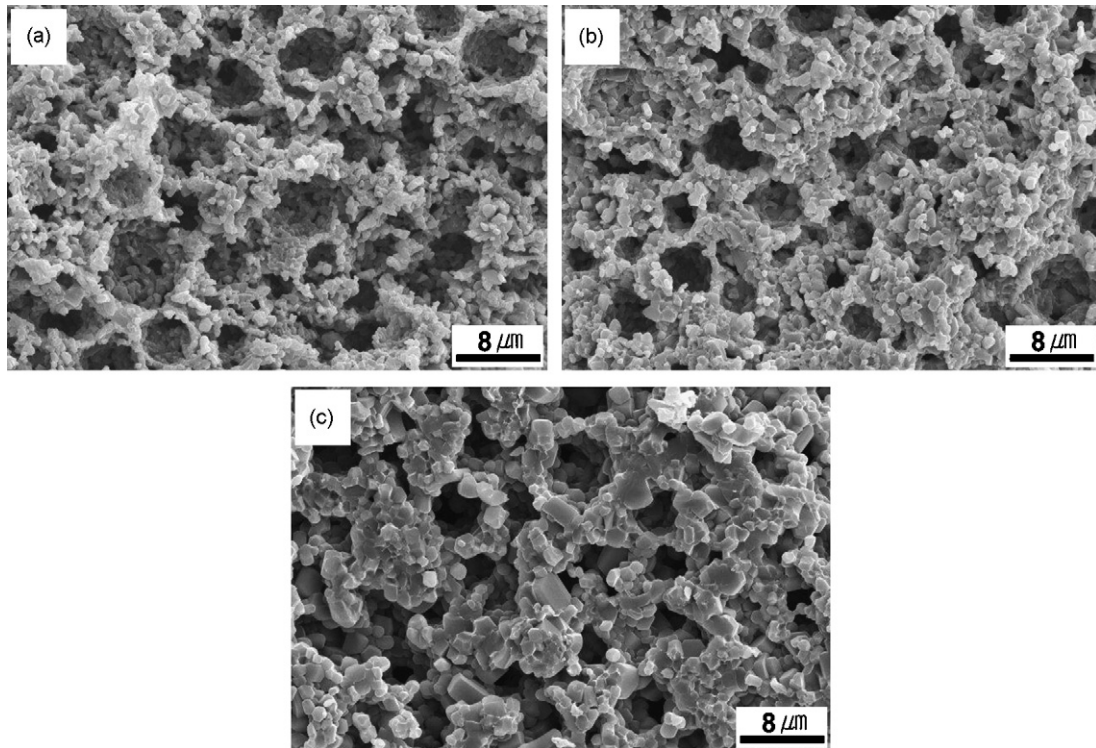


Fig. 1. Effect of the sintering temperature on the microstructure of the porous SiC ceramics (S9) sintered at various temperatures for 2 h in argon: (a) 1750 °C, (b) 1850 °C, and (c) 1950 °C (refer to Table 1).

in the 1950 °C-sintered specimen. Therefore, grain growth and pore size reduction occurred simultaneously when the sintering temperature was increased from 1750 °C to 1950 °C.

Fig. 2 shows the typical microstructures of the S10 (SiC:PDSiC ratio = 10:0) and S6 (SiC:PDSiC ratio = 6:4) specimens. Both were sintered at 1950 °C for 2 h. There was no significant difference in the sizes of the SiC grains and pores, regardless of the SiC:PDSiC ratio. This suggests that the microstructure of the porous SiC ceramics processed by the present method is insensitive to the SiC:PDSiC ratio.

Fig. 3 shows the porosity of the porous SiC ceramics fabricated at various SiC:PDSiC ratios and sintered at various temperatures. The porosity of the porous SiC ceramics ranged from 45% to 64% when sintered at 1750 °C, and from 32% to

49% when sintered at 1900 °C, depending on the SiC:PDSiC ratio. The 1900 °C-sintered S10 specimen showed the lowest porosity of 32%, whereas the 1750 °C-sintered S4 specimen showed the highest porosity of 64% among the compositions examined. The differences in porosity were attributed to the differences in the density of pressed bars after crosslinking and weight loss of each composition at each temperature. Generally, the porosity increased with increasing PDSiC content when sintered at the same temperature. The density of pressed bars after crosslinking decreased with increasing PDSiC content in the starting composition: 1.32 g/cm³ for SC10, 1.31 g/cm³ for SC9, 1.22 g/cm³ for SC6 and 1.12 g/cm³ for SC4. Thus, the porosity increase observed with increasing PDSiC content was attributed to the difference in the density of pressed bars after

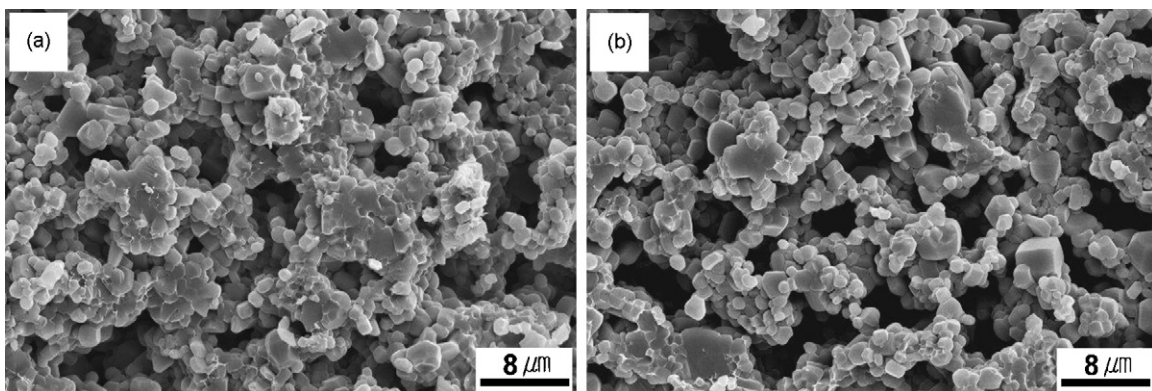


Fig. 2. Effect of the SiC:polysiloxane-derived SiC ratio on the microstructure of the porous SiC ceramics sintered at 1950 °C for 2 h in argon: (a) S10 and (b) S6 (refer to Table 1).

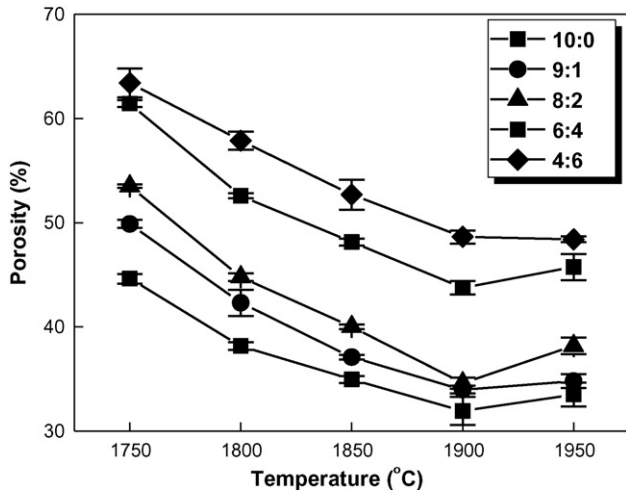
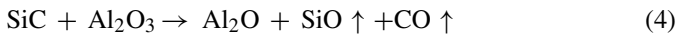
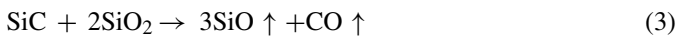


Fig. 3. Effect of the sintering temperature on the porosity of the porous SiC ceramics sintered for 2 h in argon.

crosslinking. The SiC yield from a mixture of polysiloxane and carbon was $\sim 50\%$, which indicates the evolution of more volatile species from the specimens containing a higher amount of the mixture resulting in higher porosity. The weight losses of the 1900 °C-sintered S10 and S4 specimens were $\sim 35\%$ and $\sim 66\%$, respectively. Therefore, the increase in porosity with increasing PDSiC content was attributed to the increased weight loss of the specimens. Some of possible reactions causing the weight loss are as follows^{7,17,25}:



When the same starting composition was used, the porosity decreased with increasing sintering temperature from 1750 °C to 1900 °C with an increase at 1950 °C. Hence, the decrease in porosity with increasing sintering temperature and the minimum porosity obtained at 1900 °C was caused mainly by further densification as the sintering temperature was increased. In contrast, the increase in porosity observed at 1950 °C was caused by the increased weight loss during sintering at 1950 °C. The weight losses of the S8 specimens sintered at 1900 °C and 1950 °C were $\sim 45\%$ and $\sim 49\%$, respectively. The occurrence of reactions (3) and (4) would be accelerated at higher temperatures,²⁵ leading to higher weight loss at 1950 °C than 1900 °C. The porosity of the S10 specimens ranged from 32% to 45% depending on the sintering temperature, whereas the porosity of the S4 specimens ranged from 48% to 64%. Therefore, porous SiC ceramics with higher porosity could be fabricated by adding polysiloxane–carbon mixture to the SiC powder.

It was possible to control the porosity of porous SiC ceramics from 32% to 64%, by adjusting both the SiC:PDSiC ratio and sintering temperature while fixing the polymer microbead content to 50%. These results also suggest that porosity control

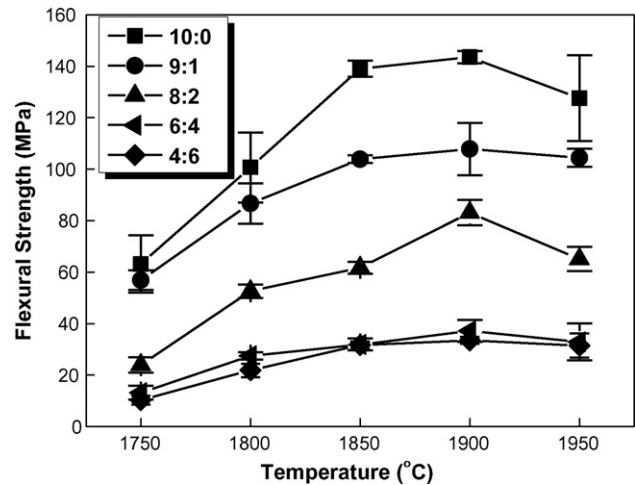


Fig. 4. Flexural strength of the porous SiC ceramics sintered for 2 h in argon as a function of the sintering temperature.

over wider ranges may be possible using the present processing technique by adjusting the polymer microbead content.

3.2. Flexural strength

Fig. 4 shows the flexural strength of the porous SiC ceramics as a function of the sintering temperature. The flexural strength of all specimens increased with increasing the temperature to 1900 °C, followed by a decrease in strength when sintered at 1950 °C. This tendency is in contrast to the change in porosity with sintering temperature (Fig. 3). Fig. 4 also shows that the strength increased with decreasing PDSiC content. The 1900 °C-sintered S10 specimen showed a maximal strength of 144 MPa at 32% porosity. In order to understand the relationship between the flexural strength and porosity, the flexural strengths of some selected specimens were plotted as a function of the porosity (Fig. 5). As shown, the flexural strength generally decreased with increasing porosity when the same starting composition was used. However, the 1850 °C-sintered specimens showed higher strength than the 1900 °C-sintered

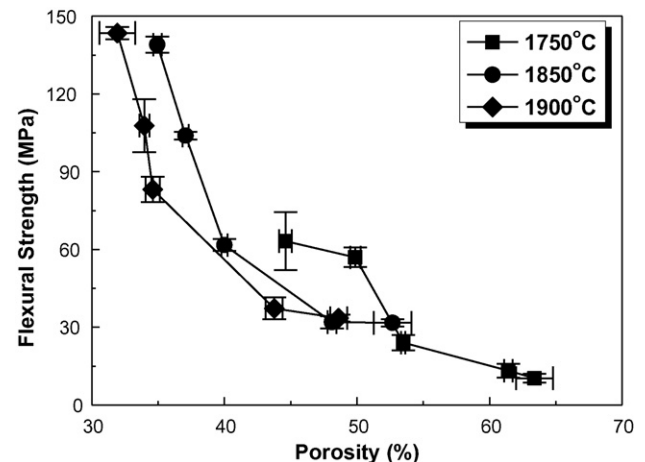


Fig. 5. Flexural strength of the porous SiC ceramics sintered for 2 h in argon as a function of porosity.

specimens at porosities ranging from 35% to 40%. Moreover, the 1750 °C-sintered specimens showed higher strength than the other specimens at porosities between 45% and 50%. In particular, the S10 specimen sintered at 1750 °C showed an average strength of 63 MPa at 45% porosity. In contrast, the S6 specimen sintered at 1900 °C showed an average strength of 37 MPa at 44% porosity. The S9 specimen sintered at 1750 °C showed an average strength of 57 MPa at 50% porosity, whereas the S4 specimen sintered at 1900 °C showed an average strength of 34 MPa at 49% porosity. Therefore, some specific combinations of the SiC:PDSiC ratio and sintering temperature resulted in higher strength than the other specimens with equivalent porosity but fabricated under different conditions. In general, the flexural strength of the other porous ceramics and porous SiC ceramics decreased with increasing porosity.^{15,26–29} However, the present results suggest that judicious selection of the processing conditions and starting SiC:PDSiC ratio can improve the strength at an equivalent porosity.

Flexural strengths of 28 MPa at 44% porosity and 24 MPa at 43% porosity were reported in reaction-bonded porous SiC ceramics³⁰ and mullite-bonded porous SiC ceramics,³¹ respectively. A flexural strength of 45 MPa at 50% porosity was reported in polysiloxane-derived porous SiC ceramics¹⁵ and 40 MPa at 50% porosity in powder-processed porous SiC ceramics.³² The specimens reported in references 15 and 31 were sintered with Al₂O₃–Y₂O₃ or their compound (Y₃Al₅O₁₂). The flexural strength obtained in the 1750 °C-sintered S9 specimen was 57 MPa at 50% porosity. The superior strength of this porous ceramic was attributed to the homogeneous microstructure of the ceramic, which was obtained by adding the optimum amount of polysiloxane and carbon to the SiC powder (SiC:PDSiC ratio = 9:1), as well as by the beneficial effects of the Al₂O₃–Y₂O₃–MgO additive system compared with the Al₂O₃–Y₂O₃ additive system.

4. Conclusions

It is possible to tune the porosity of porous SiC ceramics within a range of 32–64% by adjusting the sintering temperature and SiC:PDSiC ratio in the starting composition, while keeping the sacrificial template content to 50%. The controllable range of porosity can be increased by adjusting the sacrificial template content.

Some specific combinations of the SiC:PDSiC ratio and sintering temperature resulted in excellent strength, e.g. 57 MPa at 50% porosity for the 1750 °C-sintered S9 specimen and 63 MPa at 45% porosity for the 1750 °C-sintered S10 specimen. These results suggest that judicious selection of the processing conditions and starting SiC:PDSiC ratio can improve the strength of porous SiC ceramics.

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