Microstructure and mechanical properties of porous SiC ceramics by carbothermal reduction and subsequent recrystallization sintering

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ABSTRACT

Porous SiC ceramics were prepared by a new approach combination of carbothermal reduction and subsequent recrystallization sintering. Firstly, micro-sized SiC particles were used as the skeleton, and SiC spherical nanocrystals were in-situ synthesized by the vapor-solid reaction between carbon nanoparticles and silicon monoxide vapor. The shape and diameter of SiC nanocrystals are related to the pristine carbon nanoparticles, showing a shape memory effect. After recrystallization sintering, high purity a-SiC porous ceramics with tailored necking area were obtained by the evaporation-condensation of SiC nanocrystals. A linear relation was revealed between the flexural strength and the value of \(d/d_0\) of porous recrystallized SiC (RSiC) ceramics (the neck diameter to coarse micron-sized grains diameter ratio). The necking area and \(d/d_0\) value increased with the SiC nanocrystals content, due to the higher saturated vapor pressure resulted in high mass mobility to neck area of SiC nanoparticles. As a result, a remarkable value of \(d/d_0 \sim 99\%\) and outstanding flexural strength of 75.7 MPa could be achieved for the porous RSiC ceramics with \(~42\%\) porosity by adding 20 wt.% nano-sized SiC sintered at 1950°C for 2 h.

1. Introduction

Porous silicon carbide (SiC) ceramics are hence an ideal candidate for filters, catalytic supports, separation membranes, acoustic and thermal insulators, high-temperature structural materials, kiln furniture, thermoelectric energy conversion, and reinforcement of composites [1–5]. This is due to their unique combination of properties such as excellent mechanical properties, good chemical resistance, high thermal conductivity, low thermal expansion coefficient, and high thermal shock resistance.

A wide range of processing routes have been proposed for the production of porous SiC ceramics, such as partial sintering [6–9], replica method [10,11], sacrificial template [12,13], direct foaming [14,15], and bonding technique [16–18]. Among them, porous SiC ceramics fabricated by high-temperature recrystallization (one of the partial sintering methods) possesses high creep resistance and strength maintained at elevated temperatures, which was attributed to its high purity (99.9% pure) [8,9]. More importantly, pore size and porosity could be controlled by the size of starting powders and the degree of necking area between SiC particles during the recrystallization process [5,8,9]. Consequently, a very homogeneous pore structure with narrow pore size distribution could be prepared through recrystallization. Besides it, the non-densifying mechanism of evaporation-condensation during the recrystallization process enables the production of large parts with high dimensional accuracy, which is a key factor for the industry. Porous recrystallized SiC (RSiC) ceramics have been considered as one of the most favorable candidates for diesel particulate filters (DPF) [1,8,9]. For these filter applications, high porosity and precise pore size control are required. In addition, mechanical properties are important for filtration reliability, because high strength can allow high-pressure operation.

Generally, increasing the porosity will considerably sacrifice mechanical properties. The strength of porous ceramic is strongly dependent on the pore volume fraction and the microstructure of porous ceramics. Among them, the neck area between SiC grains is a key factor for porous RSiC ceramics [18,19]. Well-developed neck regions between SiC grains will lead to the high strength of porous RSiC ceramics. During the recrystallization process, the vaporized gas-phase materials such as Si and SiC\(_2\) transport in the components led to structural bonding, and the growth of SiC “like-necks” between the SiC grains occurred [20–22].

Growth and consolidation of SiC components generally occur at high temperature up to 2000 – 2400°C [23]. It is well know that the utilized RSiC material consisted of bimodal grain size distributions of SiC powder could reduce the sintering temperature. During consolidation,
the fine-grained fraction begins to decompose to gas species at relatively low temperature. The finer grains lead to higher saturated vapor pressures of these gaseous species. It is related to the difference between mean curvature and the average mean curvature. The addition of nano-sized SiC particles suggests that the promoted mass transport from the nanoparticles to neck regions could provide a well-developed neck area [24]. As a result, high strength could be achieved. However, silica is always present as a native layer on the surface of nano-size SiC grains, and this layer prevents necks connections between SiC grains. It has been demonstrated that SiC nanoparticles can be synthesized by the carbothermal reduction between SiO gas and nano-carbon [25,26]. Unlike the traditional methods, the vapor-solid (VS) reaction method for in-situ synthesis of SiC is direct, easy and can be used for mass production. By using coarse micro-size SiC particles as the skeleton, uniformly dispersed SiC nanoparticles could be in-situ synthesized by carbothermal reduction between SiO gas and nano-carbon in C-SiC system. On the other hand, the porosity of RSIC ceramics could be tailored by adjusting the weight content of in-situ synthesized SiC nanoparticles addition.

In the present study, we intend to fabricate porous RSIC ceramics with well-developed neck areas via evaporation-condensation of SiC nanoparticles, which was in-situ synthesized by carbothermal reduction between SiO and carbon nanoparticles. By adjusting the content of SiC nanoparticles, the necking area between SiC grains could be tailored. The effect of the necking area parameter on the microstructure and flexural strength of porous RSIC ceramics was investigated. The porous SiC ceramics with enlarged neck areas were expected to possess excellent mechanical properties and good reliability.

2. Experimental procedure

Commercial silicon carbide powders (median particle size: 3.5 μm, Ningxia Orient Tantalum Industry Co. Ltd.), SiO powders (median particle size: 16.5 μm, Taiyuan Heng Xin Technology Industry Co. Ltd.) and carbon powders (40 nm, Brand N330) were used as starting powders. Vapor-solid reaction between SiO and nano-sized carbon was developed to synthesize nano-sized SiC. In this study, to ensure the completion of the VS reaction between SiO and carbon, the molar ratio of SiO/C was selected as 1/1. The starting compositions are given in Table 1. SiC powder with a particle size of 3.5 μm was designated as Micro-SiC, and the in-situ synthesized nano-sized SiC by carbothermal reduction between nano-carbon and SiO particles was designated as Nano-SiC. The mixtures were wet-milled with agate balls in ethanol for 12 h in a plastic bottle. After mixing, the slurry was dried with a rotary evaporator and a vacuum oven at 110°C for 4 h and screened through a 75 mesh sieve. The green compacts with a dimension of 50 mm × 5 mm × 5 mm were formed by uniaxial pressing of the powder mixture under pressure of 120 MPa for 1 min. As shown in Figure 1(a), the green bodies were put on a graphite plate with holes located in the gas outlet direction in the furnace (High multi-5000, Fujidempa Co. Ltd., Osaka, Japan), and sintered at 1700°C for 2 h under an argon pressure of 0.225 MPa. SiO vapor was first generated via sublimation of SiO powders at high temperature, and then the SiO vapor reacted with the carbon black to form nano-sized SiC according to the following reaction: 2C(s) + SiO(v)→SiC(s) + CO(v). For comparison, pure nano-sized carbon black was sintering in the same condition. The heating rate from room temperature to 1200°C was 20°C/min, and 5°C/min from 1200°C to 1700°C. After that, the compacts were introduced into a medium-frequency vacuum induction furnace (3.5 kHz, Model ZGRS-160/2.55 Jinzhou Electric Furnace Co., Ltd., Jinzhou, China), and sintered at 1950 ~ 2100°C for 1 ~ 2 h under an argon pressure of 0.1 MPa. The heating rate from room temperature to the final sintering temperature was 20°C/min. As shown in Figure 1(b), the mass transport from SiC nanoparticles to the necking area results in bonding of coarse SiC grains because of the difference between mean curvature and the average of mean curvature during evaporation-condensation processing.

Phase identification was performed by X-ray diffraction (XRD, XPert PRO, Holland). The pore size and pore size distribution were determined by mercury porosimetry (AutoPore IV 9500, Micromeritics, GA, USA). The microstructure was observed by using scanning electron microscopy (SEM, S-4800, Japan). The necking area between SiC grains was measured by the line-intercept method using the SEM images. The calculated diameter of the necking area was multiplied by a ratio of 1.225 [27] to represent the real grain size. The value of d/d0 (the neck diameter to coarse micron-sized grains diameter ratio, as shown in Figure 1(b)) was used to evaluate the degree of necking area after evaporation-condensation processing. Each value was averaged over 100 ~ 200 diameters of the necking area. The density and porosity were measured by the Archimedes method. The flexural strength was measured by 3-point bending method with a 20 mm span at a cross-head

| Sample ID | Molar ratio of nano-SiC/Micro-SiC | α-SiC(3.5 um) wt.% | Carbon wt.% | SiO wt.% |
|-----------|---------------------------------|--------------------|-------------|----------|
| S10       | 10/90                           | 93.75              | 6.25        | 22.92    |
| S15       | 15/85                           | 90.43              | 9.57        | 35.09    |
| S20       | 20/80                           | 86.96              | 13.04       | 47.81    |
| S30       | 30/70                           | 79.55              | 20.45       | 74.98    |
speed of 0.5 mm/min at room temperature. Each final value was averaged over five measurements.

3. Results and discussion

Figure 2 shows the XRD patterns of the green body and porous RSiC ceramics. For the pure nano-sized carbon black, as shown in Figure 2(a), no other peaks except β-SiC (3C-SiC) were observed after VS reaction, indicating that SiC nanoparticles were successfully synthesized as a result of completed carbothermal reduction between silicon monoxide gas and carbon nanoparticles at 1700°C for 2 h. Figure 2(b) shows that the starting powder including α-SiC (6H-SiC and 4H-SiC) and graphite. After carbothermal reduction at 1700°C for 2 h, as shown in Figure 2(c), β-SiC phase was confirmed. After recrystallization at 1950°C, only α-SiC phase was observed for the sintered samples (Figure 2(d)). It should be mentioned that there was not β-SiC phase because the β to α phase transformation at high temperature. As the recrystallization temperature increased to 2100°C (Figure 2(e)), beside α-SiC phase, graphite was observed, indicating the silicon carbide decomposition [27] has occurred according to the reaction $2\text{SiC}(s) \rightarrow \text{C}(s) + \text{Si}_2\text{C}_2(g)$; $2\text{SiC}(s) \rightarrow \text{Si}(g) + \text{SiC}_2(g)$.

Figure 3 shows the SEM micrographs of the products after carbothermal reduction at 1700°C for 2 h. As shown in Figure 3(a), for the mixture of SiC and carbon nanoparticles, the micron-sized SiC keeps the primary irregular shape and it has a uniform size of ~3.5 μm. After the siliconization reaction at 1700°C, SiC nanospheres with an average grain size of ~70 nm formed between the micron-size SiC grains (Figure 3(a,b)). The carbothermal reduction reaction between SiO and carbon nanospheres...
was according to the following reaction:

$$2C(s) + SiO(v) \rightarrow SiC_P(s) + CO(v).$$

Here, the subscript "p" referred to particle form. The shape and diameter of SiC nanocrystals are related to the pristine carbon nanoparticles, showing a shape memory effect from carbon to SiC [28]. It is interesting that SiC whiskers with a diameter of

Figure 2. XRD patterns of the (a) powder mixture of SiO and carbon nanoparticles after VS reaction at 1700°C, (b) starting powder, (c) sintered compacts after VS reaction, and (d-e) porous SiC ceramics after recrystallization at different temperatures.

Figure 3. SEM morphologies of fractured surfaces of (a-c) S-20 samples and (d) powder mixture of SiO and carbon nanoparticles after VS reaction at 1700°C for 2 h. SiC nanoparticles and nano-whiskers are obtained via carbothermal reduction.
~70 nm and a length of ~3 μm are formed between coarse micro-size SiC grains (Figure 3(c)). Generally, the whiskers were synthesized by using an iron catalyst through the vapor-liquid-solid (VLS) mechanism [29]. However, it was found that alloy or metal droplets were never formed at the tips of SiC whiskers, suggesting the VLS mechanism never took place in the present study. The formation process of SiC whiskers could be explained as follows. During sintering, two vapors, SiO and CO, would generate as the reactants in an inert atmosphere, the gap between the micron-sized SiC particles provided the flow passage of SiO vapors and CO vapors, as the CO vapors diffused and met with the SiO vapors, the SiO vapors reacted with CO vapors for SiC growth in whisker form by the following reaction: SiO(v) + 3CO(v) → SiC\text{w}(s) + 2CO₂(v) [30]. Here, the subscript “w” referred to whisker form. The SiC form was related with the partial pressure of CO vapor (P_{CO}). In the present study, the SiC whisker-forming reaction was quite sluggish in an argon atmosphere with low P_{CO}. Figure 3(d) shows the micrograph of the products sintered by using pure spherical carbon nanoparticles as starting powders. As shown in it, spherical SiC nanoparticles with a uniform size of ~70 nm in diameter are observed, reflecting a genetic effect.

Figure 4 shows the SEM images of the fractured surfaces of the porous RSiC ceramics sintered after carbothermal reduction at 1700°C for 2 h and recrystallization sintered at 1950°C for 2 h. As shown in it, as compared with the microstructure of as starting powders (as shown in Figure 3), finer SiC nanoparticles disappeared. The primary irregular coarse micro-sized SiC particles became ellipsoidal, and the sintering neck was formed between the SiC particles. It was indicated that the evaporation-condensation of in-situ synthesized SiC nanoparticles occur at 1950°C.

This mass transport was attributed to the difference between mean curvature and the average of mean curvature. When the total volume of SiC particles is conserved, the surface motion in the sintering by evaporation-condensation is expressed by \( v = M(k - k_{\text{av}}) \), where \( M \) is the mobility, the average of mean curvature \( k_{\text{av}} \) is given by \( k_{\text{av}} = \int k dS \), where \( S \) is the surface area. This is a motion by the difference between mean curvature and the average of mean curvature. The equation is appropriate when surface diffusion of adatoms is infinitely fast compared to attachment kinetics in the sintering by surface diffusion [31–33]. During recrystallization, the normal velocity of the surface depended only on the position and local shape of the surface. There is a tendency for material mass transfer because of the differences in surface curvature of SiC particles and consequently the differences in vapor pressure at various parts of the system. And the material is transported from the high vapor pressure to the
low vapor pressure position. The silicon carbide, which lies on the convexity of granules, was vaporized and the vapor of silicon carbide was condensed on concave and flat surfaces in packed SiC granules at high temperatures. The reason is that on the surface of a particle there is a positive radius of curvature so that the vapor pressure is somewhat higher than that in a concave or flat surface. Consequently, the sharp corners of primary irregular coarse micro-size SiC disappeared and the particles became ellipsoidal. On the other hand, the difference between mean curvature of SiC nanoparticles and the average of mean curvature is larger so that the vapor pressure is higher than that of coarse particles, results in high mobility of SiC nanoparticles. This mass transfer of substance results in a symbiotic phenomenon, which takes place on the place of contact of granules and SiC was recrystallized. Finally, coarse micro-sized SiC particles (3.5 μm) were well bonded by an individual or multiple sintering neck. Simultaneously, porous structure with an average pore size of ~3.7 μm and uniform pore size distribution could be obtained. With increasing the content of SiC nanoparticles addition, the necking area increased.

Figure 5 shows the high magnification SEM images of the fractured surfaces of the porous RSiC ceramics after carbothermal reduction at 1700°C and recrystallization sintered at 1950°C for 2 h. As shown in Figure 5, the necking area of S20-1950°C × 2 h sample was obviously larger than that of S10-1950°C × 2 h sample. It was due to the higher vapor pressure and high mobility to the neck area of SiC nanoparticles.

Figure 6 shows the necking area and the value of d/d₀ of the porous RSiC ceramics after carbothermal reduction at 1700°C and recrystallization sintered at 1950°C for 2 h. The value of the average necking area and d/d₀ (the diameter of necking/the diameter of micron-sized SiC grains) was used to evaluate the degree of necking. As shown in it, with increasing the content of SiC nanoparticles addition from 10% to 20%, the necking area increased and the value of from 8.57 to 15.85 μm², and the value of d/d₀ increased from 73% to 99%. And then, as the SiC nanoparticles addition increased to 30%, it slightly decreased to 15.02 μm² and 97%, respectively. The feature of recrystallization sintering was the active participation of the gaseous phase (SiC₂ and Si₃C et al). In the present study, the difference between mean curvature of SiC nanoparticles and the average of mean curvature is larger so that the vapor pressure is higher than that of coarse particles, results in high mass mobility to the neck area of SiC nanoparticles. Consequently, completely contact of coarse micron-size SiC granules (d/d₀, 99%) could be obtained for the porous RSiC ceramics with 20% SiC nanoparticles. As the content of SiC nanoparticles addition further increased to 30%, the gaseous phase participated on the surface of the micron-size SiC grains (as shown in Figure 4(d)) led to slight grain growth of micron-size SiC grains and decrease of the value of d/d₀.

Figure 7 show the relation between the value of d/d₀ and flexural strength of porous RSiC ceramics after carbothermal reduction at 1700°C and recrystallization sintered at 1950°C. As shown in Figure 6 (a), the value of d/d₀ and the flexural strength increased linearly with the content of SiC nanoparticles addition. Besides it, the participation of the gaseous phase to the “neck” area between the coarse SiC particles increased with the holding time. It was indicated that full equilibrium was reached for a long time as 2 h. As shown in Figure 6 (b), a linear relation was revealed between the flexural strength and the value of d/d₀ of porous RSiC ceramics. The flexural strength increased linearly from 36.1 MPa to 75.7 MPa, as the value of d/d₀ increased from 67% to 99%. It was indicated that the degree of necking area is the most critical factor for the mechanical properties of porous recrystallized SiC ceramics. The bending strength could be improved by tailored the necking area between SiC grains.

Figure 8 shows the total porosity and flexural strength of porous RSiC ceramics after carbothermal reduction at 1700°C and recrystallization sintered at 1950°C for 2 h. Table 2 shows the properties of porous SiC ceramics after
carbothermal reduction at 1700°C and recrystallization sintered at different temperatures. As shown in Table 2, the porosity slightly decreased with the nano-size SiC addition content, due to the relative density of green body slightly increased. Besides it, although the evaporation-condensation is a "non-densifying mechanism",

Figure 6. The necking area and the value of d/d0 of the porous RSiC ceramics after carbothermal reduction at 1700°C and recrystallization at 1950°C for 2 h. The neck radius and d/d0 increase with the content of in-situ synthesized SiC nanocrystals.

Figure 7. The value of d/d0 and flexural strength of porous RSiC ceramics after carbothermal reduction at 1700°C and recrystallization at 1950°C. A linear relation was revealed between the flexural strength and the value of d/d0 of porous RSiC ceramics.
slight shrinkage occurred due to the motion of mass centers of particles in a long time during evaporation-condensation processing [31]. The flexural strength decreased from 75.7 MPa of S20-1950°C × 2h sample to 27.9 MPa of S10-2100°C × 1h sample with increasing the porosity slightly from 42% to 50%. According to the Spriggs equation based on the minimum solid area approach [34,35], the strength of a porous material was related to its porosity through the expression \( \sigma = \sigma_0 \exp(-b \cdot P) \), where \( \sigma_0 \) and \( \sigma \) were the strengths of fully dense and porous materials, respectively; \( P \) was the porosity of porous material, and \( b \) was a constant that is dependent on the pore characteristics. The values of \( b \) were reported to be 6 for cubic stacking and 9 for rhombic stacking [36]. Linear-regression fitted for the plots of flexural strength versus pore volume fraction in Figure 8(b) yield \( \sigma_0 = 60,215 \), and \( b = 15.21 \) for the porous RSiC ceramics, which was greater than those reported in the literature for porous ceramics [37–39]. The most likely explanation for this deviation from the model was that the production of a different solid area for each specimen irrespective of the material porosity, which was similar to the reported results in Kim’s research \( b = 16.03 \) for porous SiC ceramics from preceramic polymers [40], and \( b = 10.52 \) for porous sodium borate-bonded SiC ceramics [41]). Moreover, the strengths of the specimens sintered at 1950°C show higher than that of the specimens sintered at 2100°C with the same starting composition. It was explained by the partial volatilization and decomposition of the SiC grains, which was confirmed by the presence of graphite in the specimen sintered at 2100°C (as shown in Figure 2).

The S20-1950°C × 2 h sample with a porosity of 42% showed a maximal strength of 75.7 MPa.

4. Conclusions

Porous recrystallized SiC ceramics with tailored neck area were successfully fabricated by carbothermal reduction and subsequent recrystallization sintering. Silicon carbides spherical nanocrystals and whiskers were in-situ synthesized by vapor-solid reaction and vapor-vapor reaction between carbon nanoparticles and silicon monoxide vapor during carbothermal reduction processing at 1700°C for 2 h, respectively. Well-developed necking area \( (d/d_0, 99\%) \) between coarse micro-size SiC grains skeleton was obtained by evaporation-condensation of 20% in-situ synthesized SiC nanocrystals. It was due to the difference between mean curvature of SiC nanoparticles and the average of mean curvature is larger so that the vapor pressure is higher than that of coarse particles, results in high mobility to neck area. A linear relation was revealed between the flexural strength and the value of \( d/d_0 \) of porous RSiC ceramics. The flexural strength increased with the content of SiC nanoparticles and attained a relatively high value of 75.7 MPa for the S20-1950°C × 2 h sample with ~42% porosity due to the formation of well-developed neck regions \((d/d_0, 99\%)\).

Table 2. The properties of porous RSiC ceramics after carbothermal reduction at 1700°C and recrystallization at different temperatures.

| Sample ID | Sintering procedure | Porosity (%) | Flexural strength (MPa) | Necking area \( d/d_0 \) (%) |
|-----------|---------------------|--------------|------------------------|-----------------------------|
| S20       | 1950°C×1 h          | 42.6         | 72.5 ± 4.5             | 14.78 97                    |
|           | 1950°C×2 h          | 42.4         | 74.2 ± 2.2             | 16.85 99                    |
|           | 2000°C×1 h          | 42.1         | 75.7 ± 3.0             | 16.91 99                    |
| S15       | 1950°C×1 h          | 44.2         | 51.5 ± 3.9             | 11.94 84                    |
|           | 1950°C×2 h          | 43.8         | 54.8 ± 3.3             | 12.79 90                    |
|           | 2000°C×1 h          | 43.5         | 56.5 ± 3.4             | 13.06 92                    |
|           | 2100°C×1 h          | 46.52        | 36.9 ± 0.81            | / 90                       |
| S10       | 1950°C×1 h          | 49.0         | 30.8 ± 2.6             | 7.79 67                     |
|           | 2100°C×1 h          | 50.5         | 27.9 ± 3.5             | / 90                       |

Disclosure statement

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Figure 8. The total porosity and flexural strength at room temperature of porous RSiC ceramics after carbothermal reduction at 1700°C and recrystallization at 1950°C. The relation of the strength to porosity can be approximated by Spriggs equation.
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Data availability

The raw data required to reproduce these findings cannot be shared at this time due to technical or time limitations.

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