Pressureless solid-state sintering of SiC ceramics with BN and C additives

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ABSTRACT
This study proposes BN as a new sintering aid for pressureless solid-state sintering of SiC ceramics. The full densification of SiC ceramics for 0.5–2.7 wt% BN addition showed the composition tolerance of the newly developed ceramics. The electrical resistivity decreased by an order of magnitude (10⁻¹⁻¹⁰⁻⁷ Ω-cm) as BN content increased from ~0.5 to ~0.9 wt% because of the increased BN-derived B doping in the SiC lattice. A further increase in BN content had no significant effect on the electrical resistivity, which is attributed to the limited solubility of B in the SiC lattice. The thermal conductivity decreased with increasing BN content owing to increased phonon scattering at B-doped sites and the thermally insulating BN phase located at the grain boundaries. The fracture toughness increased with increasing BN content owing to interfacial debonding at the weak SiC-BN interfaces. However, intrinsically weak BN grains with low hardness were responsible for reduced flexural strength and hardness with increasing BN content.

1. Introduction

Solid-state sintered SiC ceramics are important high-temperature structural materials because of their useful engineering properties such as strength retention at high temperatures, high oxidation resistance, high hardness, high thermal conductivity, and excellent thermal shock resistance \([1,2,3,4,5,6,7,8,9]\). Owing to the high covalency (~88%) of Si-C bonds, the self-diffusion coefficient of SiC is very low. Thus, a sintering temperature of ~2500 °C and a pressure of ~2000 MPa is required to fully densify a pure SiC powder [6]. Prochazka [5] first developed pressureless solid-state sintered SiC ceramics using B and C as sintering aids. B promotes densification by segregating at the grain boundaries, thus reducing the grain boundary energy, whereas the role of C is to increase the surface energy of SiC by removing the native SiO₂ film via a carbothermal reduction process.

Unlike liquid-phase sintered ceramics, where mechanical, thermal, and electrical properties can be tuned using a wide range of additive systems \([10,11,12]\), the scope of pressureless solid-state sintered SiC ceramics is constrained by the limited choice of sintering additives. Thus, it is interesting to develop new additive systems for the pressureless solid-state sintering of SiC ceramics.

To date, B and B₄C powders, BCl₃ gas, and LiBH₄ and H₃BO₃ solutions have been used as B sources for solid-state sintering of SiC ceramics \([5,6]\). It has been reported that B, B₄C, and BCl₃ are equally beneficial in achieving high relative density \([5,6]\). However, a high content of LiBH₄ is required, and H₃BO₃ is ineffective in densification owing to the high evaporation loss of B₂O₃ during sintering \([6]\). The present work investigates BN as a potential sintering aid for the pressureless solid-state sintering of SiC ceramics. To the best of our knowledge, the use of BN as a B source for pressureless solid-state sintering of SiC ceramics is rarely found. Li et al. \([3,4]\) fabricated SiC-0.25–8 wt% in situ BN composites via pressureless solid-state sintering using B₄C and C as sintering aids. Kim et al. \([13]\) fabricated SiC-1.4–27.3 wt% BN composites by hot pressing without sintering aids. Thus, the literature reveals that the solid-state consolidation of SiC–BN composites requires the use of additional sintering aids (B₄C/B) or the application of pressure.

The present work reports the successful pressureless sintering of SiC ceramics using BN (0.45–2.65 wt%) as a B source and phenolic resin as a C precursor. The electrical resistivity, thermal conductivity, flexural strength, hardness, and fracture toughness of the newly developed ceramics were characterized as a function of the BN content.

2. Experimental procedure

Commercially available α-SiC (0.5 μm, >98% pure, 0.05% Si, 0.20% C, 0.85% O, <830 ppm trace elements: Fe, Al, Ni, V, Na, Cr, Ca, Ti, Mg, K, S, SiNx 15 C, Sika Tech, Fiven, Norway), h-BN (~10 μm, 99.5% pure, 0.02% C, 0.60% O, A01, H.C. Starck, Germany), and phenolic resin (>99.9% pure, 0.02% S, TD-739, Kangnam Chemical Co., Ltd., Korea) were used as the starting materials. The phenolic resin had a carbon yield of 60%. The SiC to C weight ratio was fixed at 32:1 in all batches. As shown in Table 1, five batches of powders with varying BN content (0.45–2.65 wt%) were mixed by ball milling using SiC balls for 24 h in ethanol. After drying, the
powder mixture was granulated using a 60-mesh sieve. The sieved powders were uniaxially pressed at 25 MPa into cylindrical pellets (Ø 25 mm × 7 mm high), followed by cold isostatic pressing at 207 MPa. The green compacts were pyrolyzed at 900°C in flowing Ar gas for 1 h and subsequently pressureless sintered in a graphite resistance furnace at 2100°C for 2 h under flowing Ar gas. The rule of mixtures was used to calculate the theoretical density. The residual C left after complete reduction of native silica (0.75 wt%) was assumed in the theoretical density calculation. The sintered density was measured using the weight to volume ratio of the sintered specimens. The polished specimens were etched using a modified Murakami’s reagent [14]. For the phase identification, sintered samples were ground and analyzed using X-ray diffraction (XRD; D8 Discover, Bruker AXS GmbH, Karlsruhe, Germany). The Rietveld refinement method was used for the quantitative phase analysis of SiC polytypes. The etched microstructure and fracture surface were observed by scanning electron microscopy (SEM, S4300, Hitachi Ltd., Hitachi, Japan). The polished and chemically etched SBN5 sample was also analyzed by electron probe microanalyzer (EPMA, JXA-8900 R, Jeol, Tokyo, Japan) without conducting layer coating. Hall-effect measurements were performed on the specimens (10 × 10 × 0.7 mm³) at room temperature (RT) using the van der Pauw technique to obtain electrical resistivity. The thermal diffusivity (α) and heat capacity (C_p) were measured using a laser flash method (Model LFA 467, NETSCH GmbH, Selb, Germany) after applying a thin graphite coating over the samples (10 × 10 × 2 mm³). The measurements were conducted three times for each specimen in an argon atmosphere and the thermal conductivity (κ) was calculated using the following equation:

\[ κ = αρC_p \]  

where ρ denotes the density. The flexural strength was measured according to ASTM C 1161–13 [15]. Bar-shaped samples were cut and polished to a size of 2.0 × 1.5 × 25 mm³. The tensile surfaces of the bars were polished to a 1-μm diamond finish, and the tensile edges were chamfered to avoid stress concentration and large edge flaws caused by sectioning. Bending tests were performed at a cross-head speed of 0.2 mm/min using a four-point bending method with inner and outer spans of 10 and 20 mm, respectively. The fracture toughness was measured according to JIS-R1607 [16]. Bar-shaped samples were cut and polished to a size of 4.0 × 3.0 × 25 mm³. Vickers indentation (Model AVK-C2, Akashi Corp., Yokohama, Japan) was made on 4.0 × 25 mm² face polished to 1-μm using 30 kgf load with a dwell time of 10 s. The indent diagonals and radial cracks were measured, and the fracture toughness (K_c) was calculated using the following formula:

\[ K_c = 0.018 \left( \frac{E}{HV} \right)^{1/2} \left( \frac{P}{c^{3/2}} \right) \]  

where E, HV, P, and c denote elastic modulus, Vickers hardness, load, and crack length, respectively. The hardness of the polished specimens was measured by Vickers indentation (Model MVK-H1, Akashi Corp., Yokohama, Japan) under a load of 0.5 kgf with a dwell time of 10 s. Grain size measurement was carried out on SEM images using an image analysis software (Image-Pro plus 4.0, Media Cybernetics Inc., MS, USA). The average grain size was defined as the average length of diameters passing through the grain centroid measured at 2° intervals, and the average aspect ratio was defined as the ratio of the longest to shortest diameter.

### Table 1. Batch composition, theoretical density, and relative density of pressureless solid-state sintered SiC ceramics.

| Specimen | Batch composition (wt%) | Theoretical density (g/cm³) | Relative density (%) | SiC polytype content (wt%) |
|----------|-------------------------|-----------------------------|---------------------|--------------------------|
|          | α-SiC       | BN   | C    | | 6 H | 15 R | 4 H |
| SIC powder | 100 | -    | -    | - | 82.5 | 16.6 | 0.9 |
| SBN1     | 96.531 | 0.452 | 3.017 | 3.141 | 99.9 | -    | -    |
| SBN2     | 96.097 | 0.899 | 3.004 | 3.135 | 99.9 | 85.1 | 8.9  | 6.0 |
| SBN3     | 95.667 | 1.343 | 2.990 | 3.130 | 99.9 | 85.1 | 7.3  | 6.3 |
| SBN4     | 95.241 | 1.783 | 2.976 | 3.125 | 99.9 | 85.2 | 7.0  | 7.7 |
| SBN5     | 94.399 | 2.651 | 2.950 | 3.115 | 99.9 | 85.6 | 5.4  | 9.0 |
production. This study suggests that BN as a sintering aid, which exhibits compositional tolerant behavior, should be beneficial for the mass production of pressureless solid-state sintered SiC parts.

In this study, BN-derived B atoms diffused into the SiC lattice and lowered the grain boundary energy. Whereas the phenolic resin-derived C increased the surface energy ($\gamma_{SV}$) by reducing the native silica at $>1500^\circ$C via the following reaction:

$$\text{SiO}_2(s) + 3\text{C}(s) \rightarrow \text{SiC}(s) + 2\text{CO}(g) \quad (3)$$

The Gibbs free energies of formation ($\Delta_f G^\circ$ (T)) of Si, SiC, C, CO, CO$_2$, and SiO$_2$ at 1527°C, 0.1 MPa are 189.323, −55.895, 434.124, −269.242, −396.311, and −589.559 KJ/mol, respectively [17]. The calculated value of Gibbs free energy ($\Delta G$) for Reaction (3) is −1307.192 KJ/mol. The combined action of BN-derived B and C reduced $\gamma_{GB}/\gamma_{SV}$ to a subcritical value [5] leading to the successful densification of SiC ceramics via solid-state sintering.

Figure 1 shows the XRD patterns of the pressureless solid-state sintered SiC ceramics, which revealed the presence of SiC, BN, and C in the sintered specimens. Table 1 presents the SiC polytypes determined using the Rietveld refinement method. The raw α-SiC powder predominantly contained 6H (JCPDS No. 72–0018) and 15R (JCPDS No. 39–1196) polytypes. All specimens exhibited 6H as the major phase and 4H (JCPDS No. 29–1127) and 15R as the minor phases. The sintered specimens exhibited a 15R–4H phase transformation owing to B doping in the SiC lattice (refer to Table 1), and B is known to stabilize hexagonal (6H, 4H) phases [18,19]. The present results suggest that the BN addition accelerated the 15R to 4H polytypic phase transformation of SiC.

Figure 2 shows the SEM images of the etched SBN1 and SBN5 specimens. The microstructure exhibited elongated α-SiC grains. Flake-like BN grains were observed at the grain boundaries. The average grain size of SiC was constant at ~2.3 μm, whereas the average aspect ratio decreased slightly from 3.6 ± 0.6 in SBN1 to 3.3 ± 0.6 in SBN5 owing to increased steric
hindrance by BN grains located at the grain boundaries. Figure 3 shows the qualitative compositional map of SBN5 obtained using EPMA. It confirmed the presence of flake-like BN grains and residual C in the SiC matrix.

Figure 4 shows the electrical resistivity and thermal conductivity of the pressureless solid-state sintered SiC ceramics as a function of the BN content. The electrical resistivity was found to decrease from $1.6 \times 10^7 \, \Omega\text{-cm}$ in SBN1 to $1.4 \times 10^6 \, \Omega\text{-cm}$ in SBN4; it then increased to $1.7 \times 10^6 \, \Omega\text{-cm}$ in SBN5. Raw α-SiC powders contain N-impurities and exhibit n-type conduction \cite{20}. With a small amount of BN content (~0.5 wt%), the inherent n-type donors in the SiC lattice were compensated by BN-derived B-acceptors, causing high electrical resistivity ($1.6 \times 10^7 \, \Omega\text{-cm}$) in SBN1. However, excess

Figure 3. Scanning electron microscopy image and qualitative compositional map of SBN5 determined using EPMA: (a) SE image, (b) BSE image, (c) Silicon, (d) Carbon, (e) Boron, (f) Nitrogen, (g) Oxygen, and (h) etched microstructure of SBN5. The encircled region shows a typical spot used for elemental analysis of Si, C, B, N, and O in a SiC grain.

Figure 4. Electrical resistivity and thermal conductivity of pressureless solid-state sintered SiC ceramics as a function of BN content.
B-derived acceptors were responsible for the reduction in electrical resistivity with successive BN addition. The slight increase in the electrical resistivity of SBN5 is attributed to the trade-off between the beneficial effect of BN-derived boron doping in the SiC lattice and the deleterious effect of increased electrically insulating BN grains at the grain boundaries. These findings are consistent with those of B-doped SiC ceramics fabricated via spark plasma sintering, which exhibited a minimum electrical resistivity at 0.5 mol% B. The electrical resistivity then increased monotonically with increasing B content (1–5 mol%) [20]. It is worth noting that BN-derived N has ~3-fold higher solubility in SiC lattice than B [3,13]. However, B quickly reaches its solubility limit (0.2 mol%) owing to its high diffusivity compared to N, which requires prolonged annealing time to reach its solubility limit [5,6]. The quantitative analysis of SiC grains was performed using EPMA, which confirmed higher doping of B in SiC lattice than N. As shown in Figure 3(h), a circular region of ~1 μm diameter was probed on SiC grains for quantitative elemental analysis of Si, C, B, N and O. A total of seven grains were analyzed. The average atomic percentages of Si, C, B, N, and O in SiC grains were 41.7625 ± 1.9979, 57.5219 ± 1.9679, 0.5118 ± 0.4839, 0.1169 ± 0.1149, and 0.0869 ± 0.0539, respectively.

As shown in Figure 4, the thermal conductivity decreased from 115.7 Wm⁻¹K⁻¹ in SBN1 to 83.6 Wm⁻¹K⁻¹ in SBN2 owing to increased B doping with increasing BN content. B dopants act as phonon scattering sites and thus cause the thermal conductivity to deteriorate [19]. Whereas BN-derived N-dopants are not so detrimental to thermal conductivity [21]. It has been postulated that C₅(NC)₅ complexes are formed in N-doped SiC ceramics, which do not form any lattice vacancies and thus, have a negligible effect on thermal conductivity [22]. Similar to the electrical conductivity, the further addition of BN had no significant effect on the thermal conductivity owing to the limited solubility of B in the SiC lattice. A slight decrease (~9%) in thermal conductivity from 83.6 Wm⁻¹K⁻¹ in SBN2 to 75.9 Wm⁻¹K⁻¹ in SBN5 is primarily attributed to the increased content of thermally insulating BN grains located at the grain boundaries [18,19].

The mechanical properties of the solid-state sintered SiC ceramics are presented in Table 2. The flexural strength was found to decrease with increasing BN content, from 249.7 MPa in SBN1 to 223.9 MPa (four-point bending) in SBN5 owing to (1) the increased content of intrinsically weak BN grains with easy cleavage along the basal plane [3,18], (2) high interfacial residual strains owing to the large thermal expansion mismatch between SiC and BN [3,18], and (3) agglomeration of the BN grains with increasing BN content. As shown in Figure 5, all specimens exhibited predominantly transgranular fracture owing to strong interfacial bonding between the SiC grains. Figure 5(b) depicts a BN agglomerate in SBN5.

The newly developed ceramics exhibited flexural strengths of 224–250 MPa (four-point bending), which were relatively lower than those of solid-state sintered SiC ceramics with B₄C and C (307–556 MPa, four-point bending) [1,23,24]. The reported flexural strengths of SiC-0.5–3 wt% in situ BN composites sintered with B₄C and C were 371–402 MPa (three-point bending) [3] and those of solid-state sintered SiC ceramics with B and C were 205–400 MPa (three-point bending) [25]. According to Biswas’s work [26] on the flexural strength of hot-pressed SiC ceramics sintered

Table 2. Flexural strength, fracture toughness, and hardness of pressureless solid-state sintered SiC ceramics.

| Specimen | Flexural strength (MPa) | Fracture toughness (MPa m¹/₂) | Hardness (GPa) |
|----------|------------------------|------------------------------|----------------|
| SBN1     | 249.7 ± 10.5           | 2.1 ± 0.1                    | 25.6 ± 0.6     |
| SBN2     | 244.2 ± 13.6           | 2.2 ± 0.1                    | 25.2 ± 1.0     |
| SBN3     | 233.6 ± 9.4            | 2.6 ± 0.1                    | 25.0 ± 1.6     |
| SBN4     | 228.0 ± 12.0           | 2.8 ± 0.2                    | 24.7 ± 1.1     |
| SBN5     | 223.9 ± 22.4           | 3.2 ± 0.3                    | 23.4 ± 2.0     |

Figure 5. Typical fracture surfaces of pressureless solid-state sintered SiC ceramics: (a) SBN1 and (b) SBN5.
with Al–B–C additives, the flexural strength measured using the three-point bending method was ~150 MPa higher than that obtained using the four-point method. Thus, the flexural strength values (224–250 MPa) of the present specimens measured by four-point bending were comparable to those (371–402 MPa, three-point bending) of the SiC-0.5–3 wt% in situ BN composites sintered with B4C and C [3] and those (205–400 MPa, three-point bending) of solid-state sintered SiC ceramics with B and C [25].

As shown in Table 2, the fracture toughness of the pressureless solid-state sintered SiC ceramics varied from 2.1 to 3.2 MPa·m$^{1/2}$, and it increased with increasing BN content. Figure 6 shows the crack propagation behavior in SBN1 and SBN5. The elongated α-SiC grains fractured transgranularly and failed to cause crack deflection because of the high interfacial strength. As shown in Figure 6(b), interfacial debonding at the weak SiC-BN interfaces was responsible for the increased fracture toughness with increasing BN content. The high interfacial residual strains due to a large thermal expansion mismatch between SiC and BN phases resulted in weak SiC-BN interfaces [3,18]. Interfacial debonding at matrix-reinforcement interfaces dissipates the strain energy and thus, increases the fracture toughness [27]. The critical flaw sizes (c), calculated using $K_{\text{IC}} = 1.35 \sigma\sqrt{c}$, were approximately 39, 45, 68, 83, and 112 μm for SBN1, SBN2, SBN3, SBN4, and SBN5, respectively. Given that the critical flaw sizes (39–112 μm) were much larger than the average grain size (~2.3 μm), the fracture is thought to have originated from the processing defects [28]. The typical fracture origins of SBN1 and SBN5 were residual pores near the tensile surfaces in bending bars (Figure 7). As shown in Table 2, the hardness of the pressureless solid-state sintered SiC ceramics decreased from 25.6 ± 0.6 GPa in SBN1 to 23.4 ± 2.0 GPa in SBN5 owing to an increased volume fraction of the soft BN phase. The hardness of the newly developed ceramics was comparable with the hardness (24.2 GPa) reported for the SiC ceramics subjected to pressureless solid-state sintering at 2200°C using B and C [1].

4. Conclusions

SiC ceramics were fabricated by pressureless solid-state sintering using BN and phenolic resin-derived C as sintering aids. The major findings are summarized as follows:

1. The newly developed SiC ceramics exhibited semi-insulating characteristics. The electrical resistivity decreased by an order of magnitude ($10^7$→$10^6$ Ω·cm) with increasing BN content from ~0.5 to ~0.9 wt% owing to increased B doping in the SiC lattice. There was no significant change in electrical resistivity with a further increase in BN content owing to the limited solubility of B in the SiC lattice.
(2) The thermal conductivity decreased from 115.7 to 75.9 Wm⁻¹K⁻¹ with increasing BN content from 0.5 to ~2.7 wt% owing to increased phonon scattering at B-doped sites and the thermally insulating BN phase located at the grain boundaries.

(3) The fracture toughness was found to increase with increasing BN content owing to interfacial debonding at the weak SiC-BN interfaces. The flexural strength and hardness decreased monotonically with increasing BN content owing to intrinsically weak BN grains with low hardness located at the grain boundaries.

(4) The typical values of electrical resistivity, thermal conductivity, flexural strength, fracture toughness, and hardness for SiC ceramics sintered with 0.5 wt% BN were 1.6 × 10⁷ Ω·cm, ~116 Wm⁻¹K⁻¹, ~250 MPa, 2.1 MPa·m¹/², and 25.6 GPa, respectively.

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Disclosure statement

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