

Effects of sintering atmosphere on grain morphology of liquid-phase-sintered SiC with Al₂O₃ additions

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Abstract

The effects of sintering atmospheres of Ar and N₂ on grain morphology were investigated for pressureless liquid-phase-sintered (LPS) SiC with Al₂O₃ additions. When increasing the sintering temperature, the SiC grain size and its aspect ratio increased in both sintering atmospheres. With a 2 mass% Al₂O₃ addition, no distinct difference was observed between the grain morphology of SiC sintered in the Ar atmosphere and that sintered in the N₂ atmosphere. With a 15 mass% Al₂O₃ addition, sintering in a N₂ atmosphere led to retarded grain growth and this resulted in a fine homogeneous microstructure, whereas sintering in an Ar atmosphere enhanced the grain growth compared with that in 2 mass% Al₂O₃. The effects of atmosphere on the grain morphology depend on the amount of Al₂O₃ addition, and this also affects the grain growth process of solution-reprecipitation. The mechanical properties of the SiC are also considered.
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1. Introduction

Silicon carbide (SiC) has excellent properties as a structural ceramic for high temperature applications, including its high strength, high thermal shock resistance and high chemical stability up to elevated temperatures, and for use in wear resistant components due to its high hardness. However, SiC is difficult to densify because of its strong covalent bond and low self-diffusion coefficient. Therefore, many kinds of sintering additives have been developed for hot-pressing and pressureless sintering. Pressureless sintering is a more effective process than hot-pressing, for producing a large or complicated shape at a relatively low cost.

Boron (B)–carbon (C) is now the most typical additive for pressureless solid-state sintering of SiC, and was first found by Prochazka.¹ Subsequently, various other additives for pressureless liquid-phase-sintered (LPS) SiC have been

demonstrated, such as Al₂O₃,^{2–4} Al₂O₃–Y₂O₃,^{5–7} yttrium-aluminum garnet (YAG)⁸ and AlN–Y₂O₃.^{9–11} The advantages of LPS-SiC are its higher fracture toughness due to crack deflection or crack bridging caused by the intergranular fracture mode and its lower sintering temperature, compared with solid-state-sintered SiC which has a substantially transgranular fracture mode and requires a high temperature for densification.

The authors have showed that β-SiC powder compacts with additions of Al₂O₃ are pressurelessly densified to higher than 97% of the theoretical density in an Ar atmosphere.^{2,3} The microstructure development of the SiC sintered with Al₂O₃ was also reported and it was concluded that the elongated plate-like SiC grains interlocking with relatively high aspect ratios result in a higher fracture toughness, 5–6 MPa m^{1/2}, than those of SiC sintered with B and C additions. Microscopic studies of the Al₂O₃-added SiC elucidated that a well-developed flat basal plane exists along the grain boundary with an extremely thin Al-rich layer of 0.5–1.0 nm thickness, which may contribute to the intergranular fracture.¹²

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As a sintering atmosphere, argon gas (Ar) has mainly been used for SiC. A nitrogen gas (N₂) atmosphere has been investigated for the purpose of comparison with an Ar atmosphere, in addition to applying a N₂ atmosphere in the case of the sintering of SiC with AlN containing additives. Prochazka et al. showed that N₂ retards the densification of B-doped β-SiC, depending on the N₂ pressure.¹³ They concluded that nitrogen is incorporated into the SiC, resulting in the retardation, and it also modifies the electronic properties by the formation of n-type defects. Seo et al. observed the differences in the phase transformation and grain growth behaviors of β-SiC heat-treated under Ar and N₂ atmospheres.¹⁴ They proposed that the retarded β to α phase transformation and grain growth in the N₂ atmosphere are due to the suppression of the mass transport rate under an enhanced lattice strain caused by nitrogen incorporation into β-SiC.

In addition, with respect to LPS-SiC, N₂ atmosphere effects have been reported to be substantially similar to those for solid-state-sintered SiC. Omori et al. sintered SiC with Al₂O₃–Y₂O₃ additions pressurelessly in N₂ at 2100 °C.⁵ Dijen et al. compared the pressureless sintering atmosphere of N₂ with that of Ar for SiC samples containing Al₂O₃–Y₂O₃(–C) additives.¹⁵ In this case, for densification, sintering in a N₂ atmosphere needed the higher temperature of 2100 °C than that of 1900 °C which is sufficient in an Ar atmosphere. They found that when sintering in N₂, AlN is formed, and they assumed that the AlN would dissolve in the SiC grains during sintering and precipitate at the grain boundaries. Jun et al. reported that a N₂ atmosphere significantly retarded the β to α transformation, and nearly inhibited the grain growth in pressureless sintering of β-SiC with Al₂O₃–Y₂O₃ additions.¹⁶ They demonstrated that fine-grained SiC with high strength is produced by AlN addition to a SiC–Al₂O₃–Y₂O₃ composition, in combination with atmosphere switching from N₂ to Ar. In recent years, several attempts have been made to achieve hot-pressing of SiC with liquid phase, and subsequent heat treatment or tension testing at high temperatures, in Ar and N₂ atmospheres.^{17–19} Nagano et al. reported that tension testing in a N₂ atmosphere of hot-pressed SiC with Al₂O₃–Y₂O₃–CaO brings about crystallization of the grain boundary phase to form AlN and prevents severe vaporization of the sample.¹⁸

Many studies have been carried out on sintering conditions including the atmosphere of LPS-SiC, mainly with Al₂O₃–Y₂O₃ containing additives such as the examples mentioned above. However, the effect of the interaction between the additive amount and the sintering atmosphere on the resulting microstructure has not been reported in detail for LPS-SiC. Clearly, it is important to study microstructure control with various sintering factors, in order to obtain a dense SiC with higher mechanical properties. In the present work, the effects of N₂ atmosphere versus those of the Ar one are examined with respect to the pressureless sintering of β-SiC with Al₂O₃ additions. The grain morphology changes occurring in both Ar and N₂ atmospheres with small and relatively large

liquid amounts are investigated. In addition, the mechanical properties of the SiC are reported.

2. Experimental

As the starting materials, SiC powder (β-type, Ultrafine, Betarundum, Ividen Corp., Gifu, Japan) and Al₂O₃ powder (α-type, RA-30, Iwatani Chemicals Industry Co., Osaka, Japan) were used. The average particle sizes of SiC and Al₂O₃ were 0.3 and 0.8 μm, respectively. The SiC and Al₂O₃ powders were mixed using a rotating plastic jar with steel-ball-implanted plastic balls and ethanol. The amounts of Al₂O₃ addition were 2 and 15 mass% of the mixture of the SiC and Al₂O₃ powders. Two mass% of Al₂O₃ is the minimum addition amount for densifying β-SiC to above 97% of the theoretical density, and 15 mass% is the amount with which the

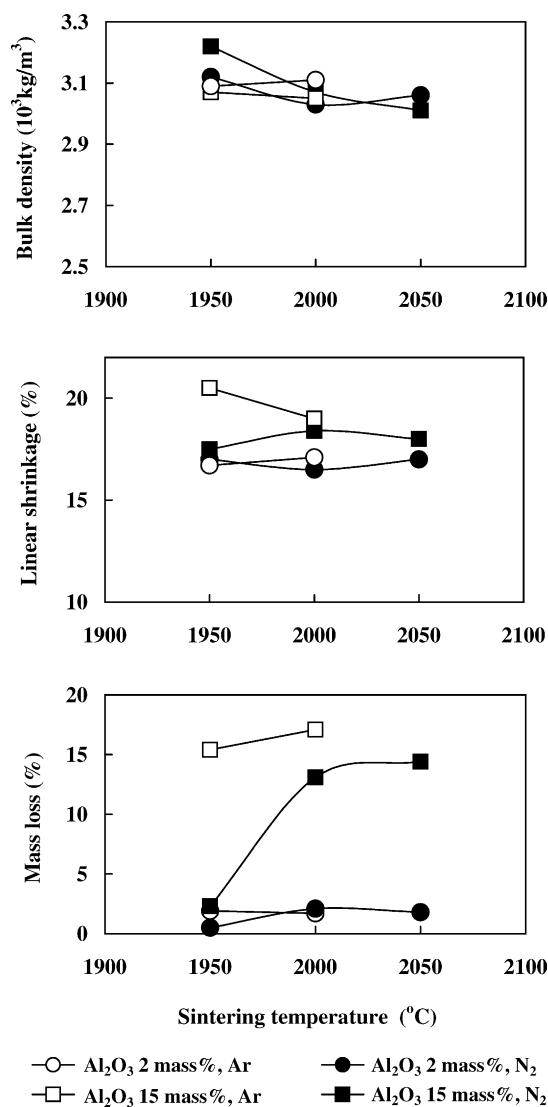


Fig. 1. Bulk density, linear shrinkage and mass loss of SiC with Al₂O₃ additions as a function of sintering temperature.

optimum mechanical properties are obtained.² The amount of dried powder of 70 g was uniaxially pressed under 20 MPa in a steel die of 40 mm × 70 mm. Subsequently, the powder compacts were cold-isostatically rubber pressed under 200 MPa.

The sintering was performed using a graphite resistance furnace. The green compacts were embedded in a powder bed in a graphite container with a lid. The powder bed used was a mixture of 80 mass% graphite grit (particle size: –3 mm, purity: 98–99%, Toyo Carbon Co., Japan) and 20 mass% Al₂O₃ powder (A-21, Sumitomo Aluminum Co., Japan).²⁰ The sintering conditions were 1950–2050 °C and 5 h in atmospheric Ar and N₂.

Bulk densities of the sintered bodies were determined by the Archimedes method. Shrinkage and mass loss during sintering were measured from the changes in the dimensions and masses of the samples. The sintered samples were cut to 3 mm × 3 mm × 30 mm to eliminate the as-sintered surface part, and the cut surfaces were mirror polished.

Microstructures were observed using a scanning electron microscope (SEM) on the polished and etched surface. The etching was carried out with a boiling mixed solution of NaOH and K₃[Fe(CN)₆]. The SEM images were analyzed using image processing software, NIH Image, which is avail-

able in the public domain. The average grain size was defined as the average diameter of the circles, which have the same areas as the sectional areas of grains in the micrographs, and the average aspect ratio was defined as the average value of the ratios of long axis to short axis of the ellipses, which are equivalent to the sectional grain shapes. They were calculated from 150 to 200 grains in each case. Transmission electron microscopy (TEM) was conducted to examine the microstructure in more detail using an analytical electron microscope (JEM-100CX) for mechanically and ion-thinned specimens of selected sintered samples. Microscopic phase identification was accomplished by performing a selected area diffraction (SAD) pattern analysis using the unit installed on the electron microscope. Oxygen contents of selected sintered samples were determined using a selective hot-gas extraction method.

Three-point flexural strength measurement was carried out using the above mentioned test bars (3 mm × 3 mm × 30 mm) at a crosshead speed of 0.5 mm/min, with a span of 20 mm. Four bars of each sintered sample were tested at room temperature (RT) and 1400 °C. Fracture toughness measurement was performed at RT by the chevron notch method²¹ using the identical test bars. Hardness was measured by using a Vicker's indenter under 500 g loading and 30 s holding time on the polished surface of the specimens.

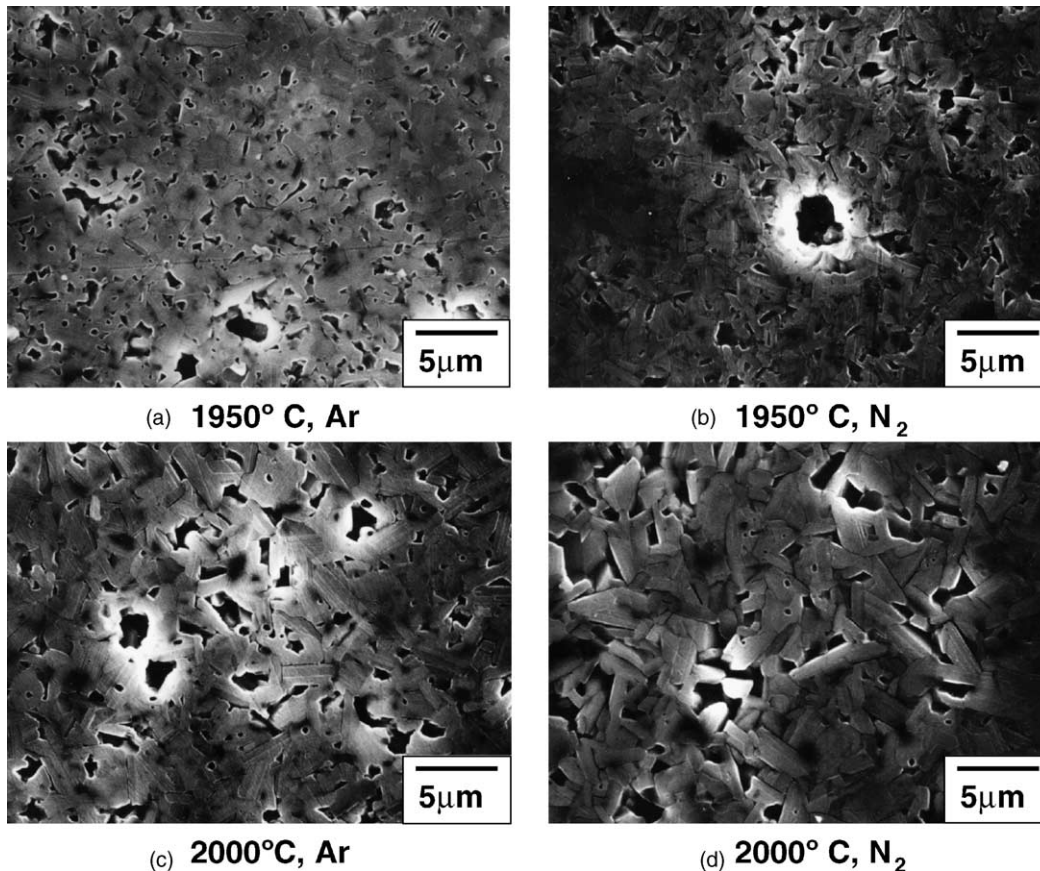


Fig. 2. SEM micrographs of polished and etched surfaces of SiC with 2 mass% Al₂O₃ addition.

3. Results and discussion

Fig. 1 shows the bulk densities, linear shrinkages and mass losses of the SiC samples with 2 and 15 mass% Al_2O_3 , sintered at 1950–2050 °C in Ar and N_2 atmospheres. All of the bulk densities obtained were above $3.0 \times 10^3 \text{ kg/m}^3$. The bulk density of the SiC samples with 15 mass% Al_2O_3 sintered in N_2 decreased with increasing sintering temperature. The mass losses of SiC with 2 mass% Al_2O_3 added were low, not above 2 mass%, whereas those of SiC with 15 mass% Al_2O_3 added changed depending on the sintering temperature and atmosphere. In the case of a 15 mass% Al_2O_3 addition, the

SiC sintered in N_2 had lower mass losses than that sintered in Ar. When increasing the sintering time of SiC with Al_2O_3 in Ar, the Al_2O_3 content in the sintered body decreased and the mass loss increased, which resulted in a porous surface layer with increased thickness.²⁰ This tendency was also observed when increasing the sintering temperature in both Ar and N_2 in this study. Alumina contents in the sintered SiC are shown in Table 1. With increasing the sintering temperature, the Al_2O_3 content decreased, which led to a thickening of the porous surface layer. The decrease in the Al_2O_3 content of the SiC with 15 mass% Al_2O_3 during sintering was smaller in the case of N_2 than in that of Ar. The bulk density of SiC with

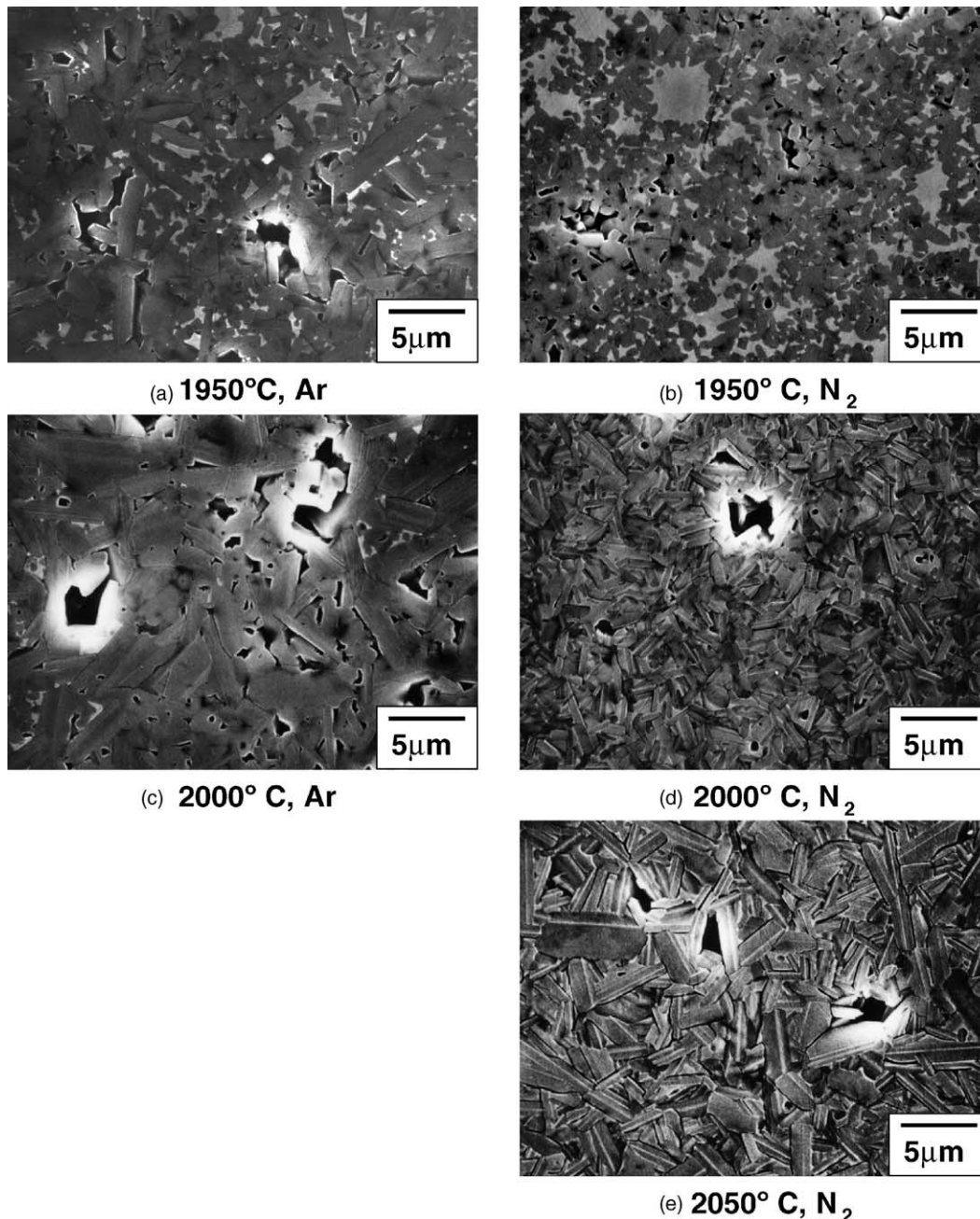


Fig. 3. SEM micrographs of polished and etched surfaces of SiC with 15 mass% Al_2O_3 addition.

Table 1
Al₂O₃ content in the sintered SiC with Al₂O₃ additions

Al ₂ O ₃ addition (mass%)	Sintering atmosphere	Sintering temperature (°C)		
		1950	2000	2050
2	Ar		1.6	
2	N ₂		0.8	
15	Ar	6.9	3.9	
15	N ₂	13.3	4.3	2.7

Al₂O₃ content was calculated from O content, which was determined by analysis.

15 mass% Al₂O₃ sintered at 1950 °C in N₂, which is 3.22×10^3 kg/m³, is estimated as being 97.6% of the theoretical density as calculated from the Al₂O₃ content. Although the SiC with 15 mass% Al₂O₃ sintered at 2050 °C in N₂ had the low bulk density of 3.01×10^3 kg/m³, the relative density of the SiC without the porous surface layer would be above 99%, taking into account the pore distribution described later.

SEM micrographs of the polished and etched surfaces of SiC with 2 and 15 mass% Al₂O₃ additions are shown in Figs. 2 and 3, respectively. Fig. 4 shows the average grain size and the average aspect ratio of the SiC grains versus sintering temperature. The grain morphology significantly depended on the amount of Al₂O₃ addition, sintering temperature and atmosphere. The grain growth of the SiC proceeded to change from equiaxed to elongated plate-like grains with increasing the sintering temperature. A similar tendency was reported with increasing the sintering time.³ In the case of a 2 mass% Al₂O₃ addition, the average grain size of SiC sintered in Ar

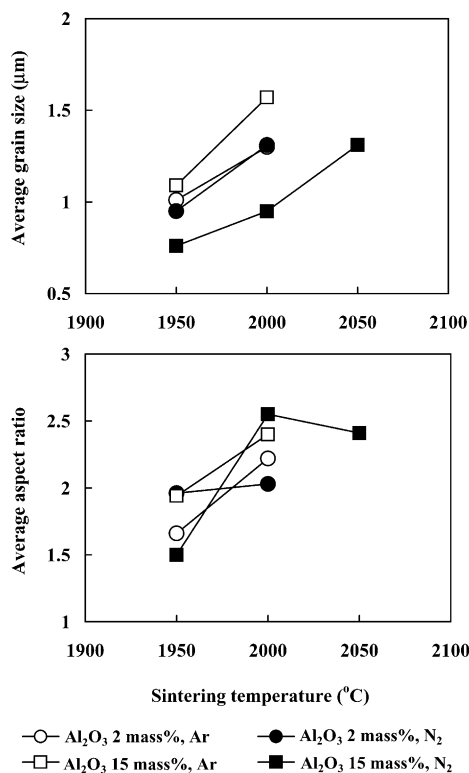
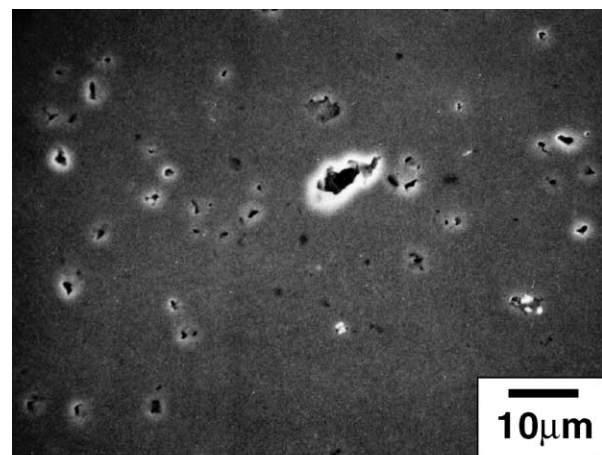
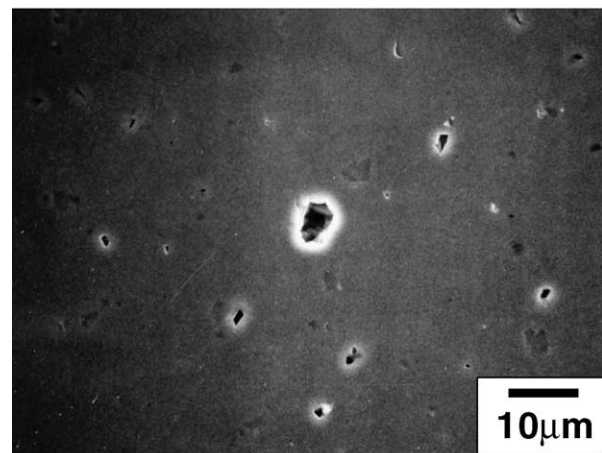


Fig. 4. Average grain size and average aspect ratio of the SiC grains for SiC with 2 and 15 mass% Al₂O₃ additions.

and that in N₂ were almost the same under the sintering temperatures of 1950 and 2000 °C, respectively. In the case of a 15 mass% Al₂O₃ addition, the average grain sizes in Ar and N₂ were distinctly different. The average grain size of SiC in N₂ was smaller than that in Ar when comparing data obtained at the same sintering temperature. This finding is consistent with the results of many studies, which have indicated that a N₂ atmosphere retards SiC grain growth compared with an Ar atmosphere. However, it is noteworthy that at the sintering temperatures of 1950 and 2000 °C, the average grain size of SiC with 15 mass% Al₂O₃ in Ar was larger and that in N₂ was smaller than the average grain sizes of SiC with 2 mass% Al₂O₃ in Ar and N₂. Fig. 5 shows the pores on the polished surfaces of SiC with 15 mass% Al₂O₃ sintered in N₂. When the sintering temperature was increased from 2000 to 2050 °C in N₂, the pores became fewer. This finding reveals that densification proceeds along with the grain morphology development for SiC with 15 mass% Al₂O₃ sintered in N₂. The porosities estimated from the pore sizes of the specimens shown in Fig. 5 were low, being below 0.2%.



(a) 2000 °C



(b) 2050 °C

Fig. 5. SEM micrographs of polished surfaces of SiC with 15 mass% Al₂O₃ addition sintered at 2000 and 2050 °C in N₂.

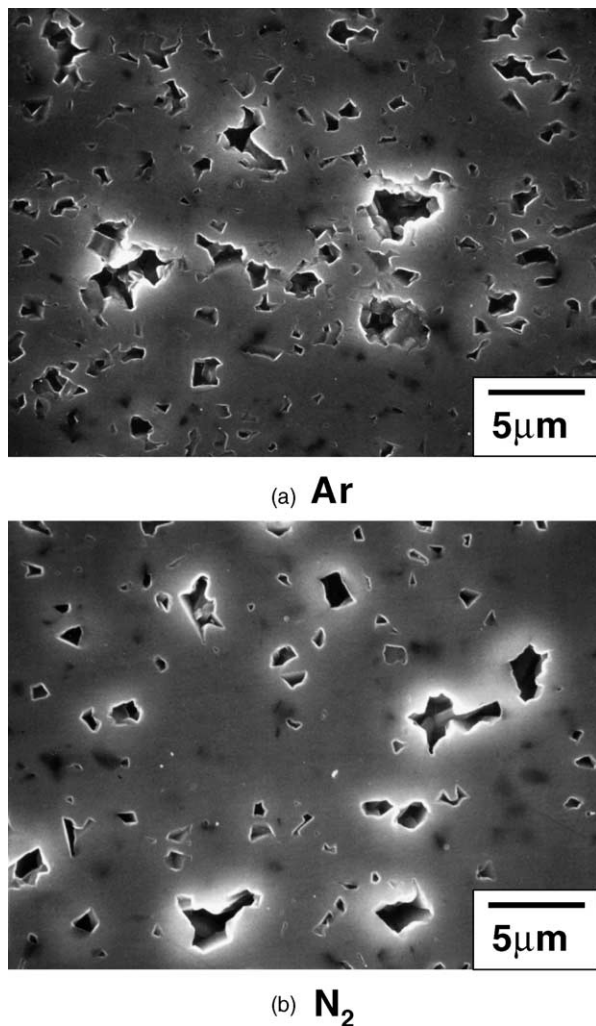


Fig. 6. SEM micrographs of polished surfaces of SiC with 2 mass% Al₂O₃ addition sintered at 2000 °C in Ar and N₂.

Generally, in liquid phase sintering when the liquid amount is too small, the densification is insufficient and exaggerated grain growth often occurs, similarly to growth in solid-state sintering, whereas when the liquid amount becomes large, the densification proceeds rapidly by grain rearrangement followed by grain growth through solution-precipitation, which results in a homogeneous microstructure. Two mass% of Al₂O₃ addition is the minimum amount with which the liquid phase sintering of SiC can progress to a high density above 97%.² Fig. 6 shows the pore structures of SiC with a 2 mass% Al₂O₃ addition sintered at 2000 °C in Ar and N₂. The SiC sintered in N₂ had fewer pores, with a simpler shape than that sintered in Ar. In the case of a 2 mass% Al₂O₃ addition, no retardation effect by N₂ was observed on grain growth, but we can recognize that densification with fewer pores would occur more slowly in the case of N₂ than that of Ar. These findings suggest that a relatively small amount of liquid, compared with the amount of liquid in the case of 15 mass% Al₂O₃, would significantly decrease the effect of N₂ on solution-precipitation; this will be further

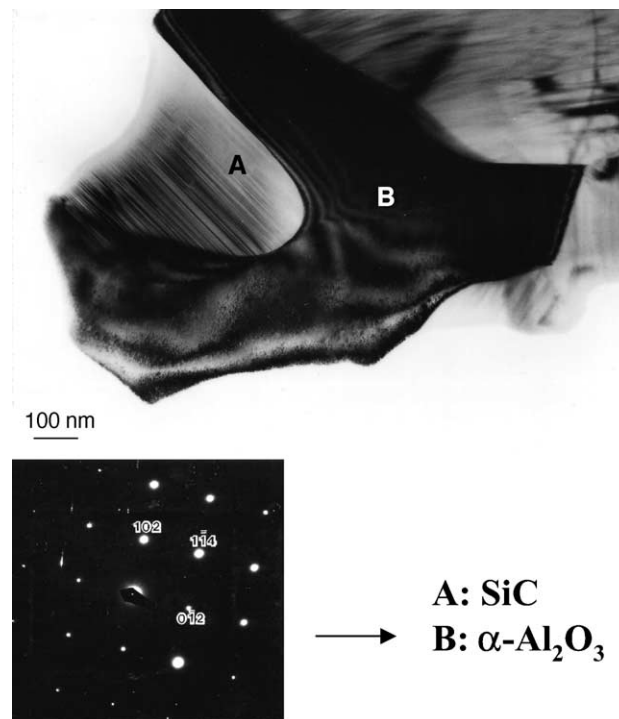


Fig. 7. Bright-field TEM image and SAD pattern of the second phase for SiC with 15 mass% Al₂O₃ addition sintered at 1950 °C in N₂.

described later. In addition, owing to the presence of liquid, nitrogen incorporation to SiC, which would suppress grain growth in solid state contacts,¹⁴ seems unlikely in this study.

For SiC with 15 mass% Al₂O₃, there should be some interaction between the liquid mainly containing Al₂O₃ and atmospheric gas. It is known that the introduction of nitrogen to oxide glass results in an increase of the softening temperature and viscosity of the glass, causing an increase in the densification temperature of LPS-SiC and a decrease in mass loss during sintering.^{19,22} Moreover, it has been reported that the solubility of SiC to an additive liquid in a N₂ atmosphere would decrease under the assumption of a preferred nitrogen solubility in the liquid, compared with an Ar atmosphere.²³ These phenomena are thought to be the N₂ effects which retard grain growth on solution-precipitation and are considered to coincide with the case of a 15 mass% Al₂O₃ addition to SiC in this study.

A TEM image and SAD pattern of the second phase are shown in Fig. 7 for SiC with 15 mass% Al₂O₃ sintered at 1950 °C in N₂. The second phase apparent at the junctions of the SiC grains was α-Al₂O₃, not AlN or AlON. Dijen et al. reported that the second phases of SiC with Al₂O₃ and Y₂O₃ sintered in N₂ were Y₂O₃ and Y–Al–O, and at the grain boundary some small inclusions of Y₂O₃ and AlN were observed.¹⁵ In this investigation, further analysis has not been conducted, but a similar inclusion including some nitrogen containing phase can be assumed in the SiC.

The flexural strengths of SiC with 2 mass% Al₂O₃ sintered at 1950–2050 °C in Ar and N₂ were almost the same,

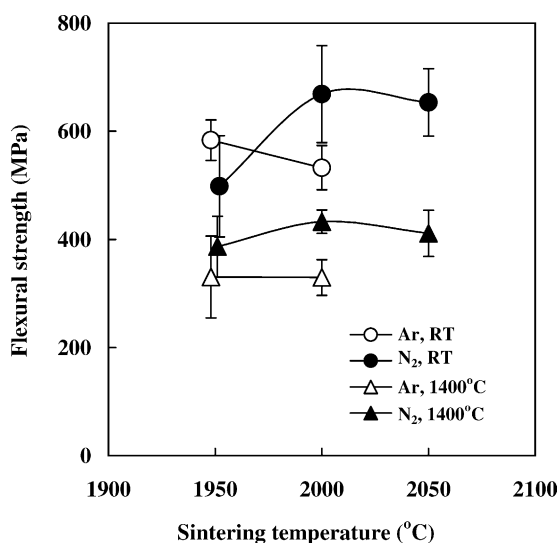


Fig. 8. Flexural strength at RT and 1400 °C of SiC with 15 mass% Al₂O₃ addition as a function of sintering temperature.

Table 2

Vicker's hardness and fracture toughness of the sintered SiC with 15 mass% Al₂O₃ addition

Sintering atmosphere	Sintering temperature (°C)	Vicker's hardness (GPa)	Fracture toughness, K _{IC} (MPa m ^{1/2})
Ar	1950	21.9	4.3
N ₂	1950	25.8	5.5
N ₂	2000	23.1	5.9
N ₂	2050	-	6.0

being 490–540 MPa at RT and 290–370 MPa at 1400 °C. The flexural strength of SiC with a 15 mass% Al₂O₃ addition is shown in Fig. 8 as a function of sintering temperature. The flexural strengths of SiC with 15 mass% Al₂O₃ sintered in N₂ were higher than those of SiC sintered in Ar under most conditions. The higher strengths can be explained by the smaller flaw size due to the homogeneous smaller elongated grain structures of SiC sintered in N₂.

The Vicker's hardnesses and fracture toughnesses of some samples of SiC with 15 mass% Al₂O₃ are listed in Table 2. The difference in hardness between SiC sintered at 1950 °C in Ar and N₂ is considered to be due to the difference in porosity. The SiC sintered at 1950 °C in N₂ showed a higher fracture toughness than that sintered in Ar, although the former had a microstructure with a lower average aspect ratio of the grains than the latter. This might be explained by that the SiC sintered at 1950 °C in N₂ is a composite of SiC and Al₂O₃ with different thermal expansion coefficients, in which micro-cracks can be easily produced.

4. Conclusions

Pressureless sintering of β-SiC with Al₂O₃ additions was examined. Differences between the grain morphologies ob-

tained through sintering in atmospheres of Ar and N₂ were investigated for the LPS-SiC.

- (1) Weight loss and decomposition of Al₂O₃ during sintering for SiC with 15 mass% Al₂O₃ are suppressed, resulting in a higher density when the SiC is sintered in an N₂ atmosphere compared with that obtained with sintering in an Ar atmosphere.
- (2) The SiC grains grow and the aspect ratio of the grains increases with an increase in sintering temperature from 1950 to 2000 °C in both Ar and N₂. With a small liquid amount of 2 mass% Al₂O₃ addition, no distinct difference in grain morphology is observed between Ar and N₂. With a relatively large liquid amount of 15 mass% Al₂O₃ addition, N₂ retards grain growth, whereas Ar enhances it, compared with that observed at 2 mass% Al₂O₃ addition.
- (3) The flexural strengths of SiC with 15 mass% Al₂O₃ sintered in N₂ are higher than those of SiC sintered in Ar, in most cases. The fracture toughness of SiC with 15 mass% Al₂O₃ sintered in N₂ is over 5.5 MPa m^{1/2}, regardless of the grain morphology.

We can conclude that applying a N₂ atmosphere and an adequate amount of Al₂O₃ addition would enable more effective control of the microstructural development of SiC, compared with that which is possible under an Ar atmosphere, and would allow us to obtain a finer homogeneous grain morphology which has the potential for higher mechanical properties. These results may serve as a starting point for further research on pressureless LPS-SiC with inexpensive additives such as Al₂O₃.

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