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ARTICLE

Rapid sintering of SiC ceramics assisted by high-frequency induction heating

Alin Yoshida [a] [*], Anna Gubarevich [b] and Katsumi Yoshida [b]

[a] *Department of Materials Science and Engineering, School of Materials and Chemical Technology, Institute of Science Tokyo, 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8550, Japan;* [b] *Laboratory for Zero-Carbon Energy, Institute of Integrated Research, Institute of Science Tokyo, 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8550, Japan*

This study explored the sintering of SiC using high-frequency induction heating and evaluated its impact on densification. A comparative analysis was performed against conventional pressureless sintering to elucidate differences in sintering behavior. Our experimental results demonstrated that high-frequency induction heating not only significantly accelerates the SiC sintering process, reducing processing time from hours to minutes, but also enhances the densification of SiC. Specifically, SiC sintered via induction heating exhibited a remarkable relative density increase of up to 28% compared to SiC sintered through conventional pressureless sintering. Scanning electron microscopy (SEM) revealed that induction heating promotes grain growth, particularly in the central region of the samples compared to conventional pressureless sintering. Additionally, X-ray diffraction analysis demonstrated that induction heating considerably promotes the transformation of SiC polytypes. In conclusion, the sintering behavior of SiC under induction heating exhibited distinct characteristics, including higher densification, increased grain growth, particularly in the central region, and a greater degree of polytype transformation in comparison with conventional sintering method.

Keywords: silicon carbide; sintering; induction heating; mircostructure; grain growth

1. Introduction

Materials play a crucial role in ensuring the safety of nuclear reactors. In order to achieve the desired reactor performance and reliable safety, it is necessary for the materials used in nuclear reactors to possess specific properties. Silicon carbide (SiC) stands out as a highly desirable material in various industries, including the nuclear sector, due to its exceptional thermal stability and mechanical properties at elevated temperatures [1]. Additionally, SiC exhibits remarkable resistance to oxidation, creep, chemicals, and radiation, making it a suitable material for severe environmental conditions in various applications [2-4]. Therefore, SiC is anticipated to play a significant role in enhancing nuclear safety. SiC has been utilized as a coating layer for TRISO (Tri Structural ISOtropic) fuels in high-temperature gas-cooled reactors, which serve as the primary pressure vessel and contain fission products [5]. Furthermore, SiC has been proposed as an alternative material for fuel cladding in light water reactors (LWRs) to enhance accident tolerance and mitigate severe accidents [6]. Additionally, SiC has gained attention as a potential material for the first wall in future fusion reactors [7].

Numerous efforts have been undertaken to enhance the densification of silicon carbide (SiC) and control grain growth, as ceramics with high density and fine grains generally exhibit superior mechanical properties [8]. However, achieving densification in SiC is challenging due to its strong covalent bonding and low self-diffusivity [9]. Initially, densification of SiC was attempted by subjecting it to extremely high temperatures exceeding 2000ºC [10]. Subsequently, sintering additives have been introduced to SiC to lower the sintering temperatures and facilitate densification. The inclusion of sintering additives, such as boron, is anticipated to enhance the sinterability of SiC by reducing grain boundary energy [9,11]. Additionally, additives like carbon play a role in eliminating SiO_2, which is typically formed on the surface of SiC due to oxidation in the presence of oxygen in the air. This elimination of SiO_2 leads to an increase in SiC diffusion rate and promotes the sintering process [9,12,13]. Furthermore, SiC exhibits various polymorphic crystal structures characterized by one-dimensional ordering sequences known as “polytypes,” and some additives have been found to influence the transformation of these polytypes [14,15].

The sintering technique that relies solely on external heating is referred to as pressureless sintering. However, in order to enhance sinterability, mechanical pressure has been employed as an external load to assist SiC sintering, giving rise to the technique known as hot-press sintering [16]. Pressureless sintering is typically more suitable for complex-shaped SiC components. In recent years, there

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*Corresponding author. E-mail: yoshida.a.124b@m.isct.ac.jp have been advancements in sintering techniques for SiC that involve the assistance of electric current and electric fields. Field-assisted sintering techniques include methods such as spark plasma sintering, microwave sintering, and induction heating sintering [17]. Several experimental studies have been conducted on SiC fabrication using spark plasma and microwave sintering. However, the literature on sintering by induction heating remains relatively limited.

Sintering by induction heating is introduced as a method to heat up a material via electromagnetic induction. An alternating current (AC) in the coil results in a magnetic field inducing eddy current in the material placed inside the coil. The eddy current produces heat by Joule heating [18]. Recently, some researchers reported fabricating ceramics assisted by induction heating due to its high heating rate, which improves productivity optimization [19,20]. However, the understanding of SiC sintering assisted by induction heating is still limited, and the effects of sintering additives on the sintering and polytype transformation behavior of SiC by induction heating are still unclear.

In this study, the focus was on investigating the pressureless sintering of SiC ceramics with the assistance of induction heating and evaluating its effectiveness in achieving densification compared to conventional pressureless sintering. In order to understand the process comprehensively, the polytype transformation among well-known polytypes like 3C, 4H, and 6H was evaluated.

2. Experimental procedure

Beta SiC powder (grade BF-17 graded, Höganäs, Sweden) with 0.4 ± 0.2 μm average grain size was used as the starting powder. Boron (Rare Metallic Co., Ltd., Japan) and carbon (Asahi Carbon Co., Ltd., Japan) were added as sintering additives. The composition of boron and carbon for SiC was 0.6-0.9 wt% and 0.5-1.0 wt%, respectively. The powder was mixed by wet ball-milling using 10 mmϕ SiC balls for 24 hours, followed by drying and sieving through a 300 μm mesh. The powder was then compacted by uniaxial pressing with pressure of 10 MPa for 1 minute. Subsequently, the isostatic pressure of 200 MPa was applied to the compacts for 20 minutes by cold isostatic pressing (CIP). The green pellets, referring to the pressed pellets before heating, were placed in carbon crucible and pressureless-sintered in the high-frequency induction heating furnace (SK Medical Electronic Co., Ltd., SK-TKY2-MU-αIV, Japan). For comparison, the green pellets were also pressureless-sintered using the conventional furnace (Hi-Multi 5000, Fuji Dempa Kogyo Co., Ltd., Japan).

The programs using both induction heating and conventional furnaces are shown in **Figure1**. Figure 1 shows the example of the sintering programs for a holding time of 10 min. At the beginning of sintering, the chamber was evacuated to 1 Pa in the induction heating furnace, and 1×10^{-4} Pa in the conventional furnace. Then, the green pellets were heated to 1500ºC as pre-sintering treatment. The heating rate was 800ºC/min and 30ºC/min in the induction heating system and the conventional system, respectively. This heat treatment was held at 1500ºC for 5 minutes under vacuum before changing to argon atmosphere. After holding at 1500ºC for 5 minutes, the temperature was raised to 2000ºC, and then held for 0-30 minutes. In this step, the heating rate for the induction heating was changed to 200ºC/min.

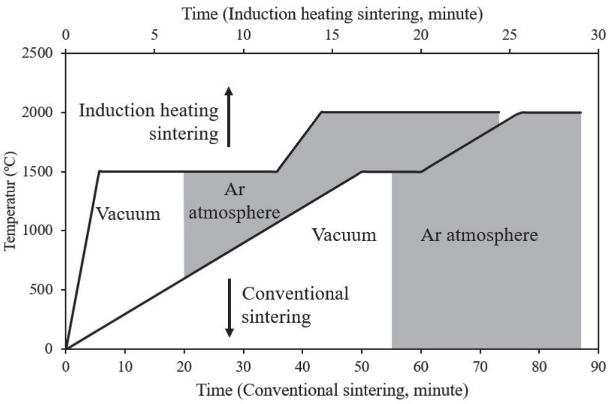

Figure 1. Sintering programs using induction heating and conventional furnaces (holding time for 10 minutes).

The density and porosity of the samples were measured using the Archimedes' method. The microstructure on the top surface of the sintered sample was observed by field emission scanning electron microscopy (FE-SEM, Hitachi Science Systems, Ltd., S-3500, Japan), and the grain size of 100 grains in each sample was measured using ImageJ software [21]. The length of 100 grains of each sample was measured. The average grain size and standard deviation were used to represent the grain size in this study. In addition, the samples were examined by X-ray diffractometry (XRD, Malvern Panalytical Ltd., Aeris Research edition, UK), and the XRD profile was used to determine the content of 3C, 4H, and 6H polytypes in the samples by Rietveld analysis using HighScore Pluse software [22].

3. Results and discussion

3.1. Effect of holding time at 2000ºC on densification and polytypes of SiC

The density and porosity of sintered SiC with 0.6 wt% boron (B) and 1 wt% carbon (C) additives are listed in **Table 1**. Without holding time at 2000ºC in induction heating, the relative density of sintered SiC already reached 85.5 % (the theoretical density of SiC was assumed to be 3.21 g/cm^3 [23]), and even increased to 94.1 % when the holding time was extended to 30 minutes. The improvement of its relative density appeared significantly for 10-minute holding time. Although the difference in density and porosity was small when the holding time at 2000ºC was extended from 10 to 30 minutes, the average grain size confirmed that grain growth still occurred.

For the holding time of 10 minutes, the conventional sintered sample had the relative density of only 66.9 %.

Table 1. Bulk density, relative density, open porosity, closed porosity, average grain size (mean ± standard deviation (SD)) and SiC polytype content of starting SiC powder and sintered SiC with 0.6wt% B and 1wt% C additives samples sintered at 2000ºC for 0-30 minutes.

Sample	Starting SiC powder	Sintered SiC samples			
Furnace		Induction heating			Conventional
Holding time at 2000 ºC (minute)		0	10	30	10
Bulk density (g/cm^3)		2.74	2.97	3.02	2.15
Relative density (%)		85.5	92.6	94.1	66.9
Open porosity (%)		7.8	0.4	0.1	29.6
Closed porosity (%)		6.7	7.0	5.8	3.4
Average grain size at the center	0.4±0.2	2.5±1.1	3.1±1.5	4.1±1.5	0.8±0.4
SiC polytype content					
6H (%)	31.8	81.0	68.6	67.7	25.2
3C (%)	67.2	2.3	0.1	0.0	57.1
4H (%)	1.0	16.7	31.3	32.3	17.7

The large open porosity of 29.6% suggested that the sintering process was not completed yet. Compared to the conventional pressureless-sintered sample, the pressureless-sintered sample by induction heating reached much higher density for the same holding time. The results emphasized that the sintering of SiC was greatly enhanced by induction heating sintering. The reason can be described by the difference in the mechanisms between conventional and induction heating sintering. In the case of conventional sintering, the external heating promotes the kinetics of sintering by grain boundary diffusion, lattice diffusion, and viscous flow [24]. In the induction heating system, the heat produced by the Joule effect affects the kinetic of sintering in the same way [17]. However, the high heating rate and the existence of an electric field and current which are characteristics of induction heating provide the following additional effects; high heating rate was reported to have an impact on the consolidation kinetic and the shrinkage kinetic through grain-boundary diffusion and powder-law creep [25]. The AC field has been reported to enhance the sintering rate and retard grain growth [26, 27]. Additionally, electric current has been suggested to affect mass transport supported by electromigration and increasing point defect concentration theory [28].

The polytype composition of the starting SiC powder originally consisted of 31.8% of 6H-SiC, 67.2% of 3C-SiC, and 1.0% of 4H-SiC. When SiC was sintered by the conventional method, the content of 3C-SiC and 6H-SiC decreased to 57.1% and 25.2% respectively. On the contrary, the 4H-SiC content increased to 17.7%. The change in SiC polytype content indicated that 3C-SiC and 6H-SiC were transformed to 4H-SiC.

In the case of the sample sintered by induction heating at 2000ºC without holding time, the content of 3C-SiC dramatically decreased to 2.3%. On the contrary, the 6H-SiC and 4H-SiC content increased to 81.0% and 16.7% respectively. The change in SiC polytype in the sample sintered without holding time points to the fact that most of cubic (3C) polytypes transform to hexagonal (4H, 6H) polytypes before the sintering step. When the holding time was extended, the content of 6H-SiC decreased, and it was 67.7% when the holding time was 30 minutes. On the other hand, the content of 4H-SiC continued to increase with increasing holding time. The highest content of 4H-SiC was 32.3% at the holding time of 30 minutes. The SiC polytype content of the samples sintered for 10 and 30 minutes suggested that 6H-SiC also changed to 4H-SiC.

The 3C to 4H and 6H to 4H polytype transformations have been reported to be influenced by the boron additive. The reason was suggested that, in the layer displacement mechanism, when boron is dissolved in SiC grains, it would promote the driving force of the transformation of cubic to hexagonal polytype [14,15]. Therefore, it can be explained why the transformation of 3C to 4H and 6H to 4H polytypes can be observed in both conventional and induction heating sintered samples. In addition, these results demonstrated that the phase transformation was more pronounced in induction heating sintering. It was suggested that, in the induction heating sintering, the diffusion of the boron additive into the SiC lattice was promoted and the 3C to 4H and 6H to 4H polytype transformations were accelerated at the same time. For the 3C to 6H polytype transformation, which was the most significantly promoted in induction heating sintering, it was possible that high heating rate or the presence of electrical current and field favor this transformation. The transformation mechanism is still unclear and requires further study.

3.2. Microstructure of SiC ceramics sintered by induction heating

Figure 2 presents the visual appearance of the sintered samples with 0.6 wt% B + 0.5 wt% C fabricated using induction heating (Figure 2 (a)) and conventional sintering (Figure 2 (b)) techniques. It is noteworthy that the samples

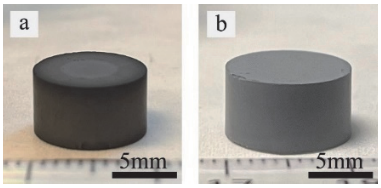

Figure 2. Appearance of SiC ceramics with 0.6wt% B+0.5wt% C sintered in (a) induction heating furnace, and (b) conventional furnace.

subjected to induction heating sintering exhibited a noticeable darker region along the edge and a light gray color ring at the center. In contrast, these distinct characteristics were absent in the samples sintered using conventional sintering method. The occurrence of both the dark region and the light gray-colored ring can be attributed to a specific current distribution resulting from induction heating [18]. Therefore, the microstructure developed differently depending on each region, and the difference was larger enough to show on the appearance of samples.

Theoretically, the current and temperature at the edge should be highest and promote the sinterability, but the SEM images shown in **Figure 3** and grain size listed in **Table 2** suggested that the grain growth was not promoted at the edge of the sample. The grains on the edge of the induction heating samples were almost the same size as those on the conventionally sintered samples.

The hypothesis is that the carbon additives would react with SiO_2 on the SiC surface during the pre-sintering treatment step [9], [12,13]. The reaction would be promoted more at the edge where the temperature is relatively high. The asymmetrical grains observed at the edges of the samples are considered to be evidence of gas-phase reaction. After the pre-sintering treatment, the remaining additive at the edge would be comparatively small. For this reason, the microstructure developed less when it reached the sintering temperature.

3.3. Effect of sintering additives on densification and polytype of SiC

SiC with three different compositions of sintering additives (0.6 wt% B + 1.0 wt% C, 0.9 wt% B + 1.0 wt% C, and 0.6 wt% B + 0.5 wt% C) were pressureless-sintered at 2000ºC for 10 minutes by high-frequency induction heating and conventional furnaces. The relative density of the sintered samples with various compositions of sintering additives is listed in Table 2. The relative density of the samples sintered by conventional sintering was 65.8-66.9%, and their open porosity was between 29.6-31.0%. The large open porosity showed that the sintering process was not finished with only 10 minutes of holding time. In contrast, the sample pressureless-sintered by induction heating reached higher relative density, and the value was in the range of 92.6-94.7%. The highest relative density belonged to SiC with 0.6% B and 0.5% C additives. The open porosity was obviously smaller than that of conventional pressureless-sintered samples. The open porosity of sintered SiC by induction heating was only 0.2-0.4%.

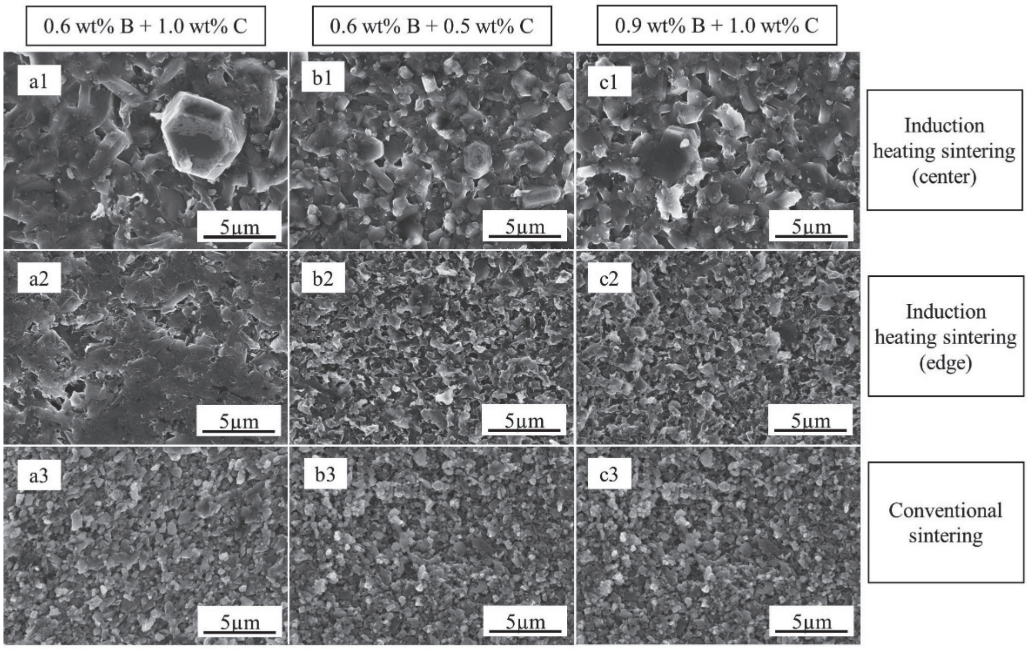

Figure 3. (a) Microstructures of SiC ceramics with 0.6 wt% B + 1.0 wt% C sintered by: (a1) induction heating sintering for 10 minutes at the center, (a2) induction heating sintering for 10 minutes at the edge, (a3) conventional sintering for 10 minutes. (b) Microstructures of SiC ceramics with 0.6 wt% B + 0.5 wt% C sintered by: (b1) induction heating sintering for 10 minutes at the center, (b2) induction heating sintering for 10 minutes at the edge, (b3) conventional sintering for 10 minutes. (c) Microstructures of SiC ceramics with 0.9 wt% B + 1.0 wt% C sintered by: (c1) induction heating sintering for 10 minutes at the center, (c2) induction heating sintering for 10 minutes at the edge, (c3) conventional sintering for 10 minutes.

Table 2. Bulk density, relative density, open porosity, closed porosity, average grain size (mean ± SD) and SiC polytype content of conventional and induction heating pressureless-sintered SiC with B and C sintering additives.

Additive (wt%)	Starting SiC powder	0.6 wt% B + 1.0 wt% C		0.6 wt% B + 0.5 wt% C		0.9 wt% B + 1.0 wt% C	
Furnace		Induction heating	Conventional	Induction heating	Conventional	Induction heating	Conventional
Bulk density (g/cm^3)		2.97	2.15	3.04	2.14	2.99	2.10
Relative density (%)		92.6	66.9	94.7	66.8	93.3	65.6
Open porosity (%)		0.4	29.6	0.2	29.9	0.3	31.0
Closed porosity (%)		7.0	3.4	5.1	3.3	6.4	3.4
Average grain size							
At center (µm)	0.4±0.2	3.1±1.5	0.8±0.4	0.8±0.5	0.5±0.3	1.0±0.4	0.4±0.3
At edge (µm)		0.5±0.3		0.5±0.3		0.4±0.2	
Polytype content							
6H (%)	31.8	68.6	25.2	79.4	23.1	81.1	20.7
3C (%)	67.2	0.1	57.1	1.4	59.1	2.0	54.6
4H (%)	1.0	31.3	17.7	19.2	17.8	16.9	24.7

The contrast shows that the induction heating system is able not only to heat up the samples with a high heating rate but also to result in SiC with high relative density. Samples with 0.6 wt% B + 0.5 wt% C sintered by induction heating provided the most effective improvement of the density, and its relative density became around 28% higher than the sample with the same sintering additive composition sintered by conventional method.

In conventional sintering, the small difference in grain size between samples with 0.6 wt% B + 1.0 wt% C and 0.6 wt% B + 0.5 wt% C reveals that the grain size became larger after carbon addition as shown in Table 2. Similarly, the average grain size in the center area of the induction heating sintered samples also increased as the carbon concentration increased, but the change was more drastic compared with the conventional sintered samples. In addition, in the induction heating system, the larger porosity of the sample with 0.6 wt% B + 1.0 wt% C resulted in its lower relative density.

When the carbon content was fixed at 1.0 wt% and the boron concentration exceeded 0.9 wt%, in conventional sintering, the relative density and the average grain size decreased from 66.9% to 65.6% and 0.8 µm to 0.4 µm, respectively. In the induction heating system, the trend of the change in grain size is similar to the conventional one. Although the average grain size in the center of the samples decreased from 3.1 µm to 1.0 µm, the boron concentration results in a small improvement in relative density of 92.6 to 93.3 %.

Regarding the experimental results, boron and carbon affected grain growth in the same trend in both conventional and induction heating sintering. Carbon addition promoted grain growth of SiC, but an increase in the boron concentration resulted in the declination of grain growth. However, the porosity related to the density was affected oppositely. The addition of carbon inhibited the densification of SiC in induction heating sintering. In contrast, boron addition promoted densification with inhibition of grain growth. Since highly dense microstructure consisting of fine grains is more preferred because it provides better machinal properties [8], SiC ceramics with small grain size and high density can be achieved successfully by induction heating with an appropriate amount of sintering additives.

The SiC polytype content of starting SiC powder and sintered samples is listed in Table 2. Compared with starting powder, the 3C-SiC content of conventional pressureless-sintered samples reduced from 67.2% to 54.6-59.1%. It can be suggested that some of 3C-SiC transformed to 6H-SiC during conventional pressureless sintering. On the contrary, in the case of sintered samples heated with induction heating, the content of the 3C-SiC drastically decreased. It showed that the transformation of the 3C polytype to 6H is more massive in the induction heating system.

When the content of 4H-SiC was focused, it was found that the amount of 4H polytype increased after sintered. This is applied to both conventional sintered and induction heating sintered samples. So, it was suggested that 3C to 4H and 6H to 4H transformations influenced by boron occurred in both systems. However, the percentage of 4H content is remarkable in the sample with 0.6 wt% B and 1.0 wt% C sintered by induction heating. On the other hand, in case of conventional sintering, SiC with 0.9 wt% C and 1.0 wt% B had the most significant 4H polytype content. These results suggested that the B/C ratio, which favors the polytype transformations, was different between conventional sintering and induction heating.

4. Conclusion

The experimental findings confirmed that induction heating sintering enhances the sinterability of SiC due to its unique characteristics, such as high heating rate, electric field, and current. Induction heating sintering enables the achievement of high-density SiC in a short holding time, thereby optimizing the sintering process productivity. In this study, a notable difference of around 28% was observed in the relative density between conventionally sintered samples and those sintered using induction heating. The sintering behavior was closely observed. When SiC was sintered using induction heating, the specimens attained a high density even without requiring a holding time. The results demonstrated that grain growth and densification occurred during the holding step. Following a holding time of 10–30 minutes, a noticeable reduction in porosity was observed. Notably, for SiC samples with additives of 0.6 wt% B and 1.0 wt% C, the most significant improvement in densification was achieved with a holding time of 10 minutes.

One characteristic of induction heating sintering is the presence of a non-uniform microstructure caused by uneven current distribution. Usually, the microstructure should be more developed at the edge of samples where the highest temperature takes place. However, the results obtained for SiC with B and C additives did not follow this pattern. The small grain size was observed at the edge. This phenomenon led to the hypothesis that a gas-phase reaction occurred, with the asymmetric grain at the edge indicating that the reaction between the additives and SiO_2 was promoted due to the relatively high temperature at the edges. Consequently, the amount of remaining additives was lower compared to the center, resulting in a less developed microstructure upon reaching the sintering temperature. Moreover, XRD results showed the unexpected effect of induction heating on polytype transformation. The transformation of the SiC polytype, especially the transformation of 3C to 6H, was enhanced by sintering with induction heating.

The boron and carbon additive affected grain growth in the same way as conventional sintering. The grain became larger as the amount of carbon increased. On the other hand, the grain growth was suppressed when boron is added more. Otherwise, in contrast to conventional sintering, carbon addition refrained densification in induction-heating sintering, and densification was promoted as the amount of boron increased. Furthermore, when SiC was sintered by induction heating, the transformations of the 3C to 4H and 6H to 4H polytypes influenced by the boron additive were promoted in a lower B/C ratio compared to conventional sintering.

In conclusion, this study demonstrated that induction heating enhances the productivity of SiC sintering by reducing processing time and promoting densification. The sintering behavior of SiC ceramics using induction heating was found to differ from the conventional pressureless sintering method.

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