NASA TECHNICAL MEMORANDUM   NASA TM-77397

EFFECTS OF APPLIED PRESSURE ON HOT-PRESSING OF β-SiC

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Translation of Yokyo Kyokai-shi (Journal of the Ceramic Society of Japan), Vol. 89, No. 6, 1981, pp. 302-309.

(NASA-TM-77397) EFFECTS OF APPLIED PRESSURE ON HOT-PRESSING OF BETA-SiC (National Aeronautics and Space Administration) 20 p HC A02/MF A01 CSCL 11G
N84-33593
Unclas
G3/27 22665

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON, D.C. 20546   FEBRUARY 1984

ORIGINAL PAGE IS
OF POOR QUALITY

STANDARD TITLE PAGE

1. Report No. NASA TM-77397

2. Government Accession No.

3. Recipient's Catalog No.

4. Title and Subtitle
EFFECTS OF APPLIED PRESSURE ON HOT-PRESSING OF β-SiC

5. Report Date February 1984

6. Performing Organization Code

7. Author(s)
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8. Performing Organization Report No.

10. Work Unit No.

9. Performing Organization Name and Address
Leo Kanner Associates
Redwood City, California 94063

11. Contract or Grant No. NASW-3541

13. Type of Report and Period Covered
Translation

12. Sponsoring Agency Name and Address
National Aeronautics and Space Administration, Washington, D.C. 20546

14. Sponsoring Agency Code

15. Supplementary Notes
Translation of Yokyo Kyokai-shi (Journal of the Ceramic Society of Japan), Vol. 89, No. 6, 1981, pp. 302-309.

16. Abstract
The effects of applied pressure on the densification during hot pressing of β-SiC compacts were investigated. β-SiC powder used in this study is Starck made and has the average particle size of about 0.7 μm. Hot-pressing experiments were carried out in graphite dies at the temperatures of 1700° to 2300°C and at the pressures up to 1000 kg/cm^2. Experiments were also done with the compacts containing 1 weight percent B_4C. Sintered compacts were then subjected to the observation of microstructure and to the measurement of Rockwell A-scale hardness.

(1) The densification of the compacts was accelerated by increasing pressure. This pressure effect was clear in hot-pressing at around 2000°C for β-Sic. However, to obtain the compacts of sufficiently high density, over 3.0 g/cm^3, severe hot-pressing conditions, above 2200°C at 200 kg/cm^2 or 2100°C at 600 kg/cm^2, are needed. B_4C addition was very effective to mitigate the hot-pressing conditions. (cont'd)

17. Key Words (Selected by Author(s))
Silicon carbinde
Hot-pressing
Densification
Rockwell hardness

18. Distribution Statement
Unclassified - Unlimited

19. Security Classif. (of this report) Unclassified

20. Security Classif. (of this page) Unclassified

21. No. of Pages

22.

Abstract Continued

(2) Empirical relations between hot-pressing temperature and pressure to realize 95 percent density were deduced as follows: $T = 2610 - 185 \log P$ for β-SiC and $T = 2780 - 280 \log P$ for β-SiC containing B_4C, respectively.

(3) The compacts of high densities near the theoretical value showed Rockwell A-scale hardness of about 96. Compared with microVickers or Knoop hardness, Rockwell hardness is thought to be macroscopic and to reflect the bonding strength between the particles of the compacts. The hardness of the compacts was simply dependent on its density, not on the hot-pressing condition. In other words, the compacts prepared under different conditions but having the same density have the same hardness. This means that the densification takes place accompanying the strengthening of the bonding and is not likely to occur by the deformation of the particles due to concentrated stress.

EFFECTS OF APPLIED PRESSURE ON HOT-PRESSING OF β-SiC

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Introduction

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Silicon carbide (SiC) and silicon nitride (Si_3N_4) have been recognized as superior high temperature materials, and diligent researches have been conducted on them in many areas. Because these materials are extremely difficult to sinter, pressure sintering hot-pressing has been performed generally when a compact with sufficient density must be obtained. However, this technique has some shortcomings: it lacks good productivity, and it is difficult to produce a compact in a complete form. In the case of SiC, for example, research has been conducted on the method for obtaining a high density compact by the conventional sintering technique using sintering assistants and SiC powder that is characterized by easy sinterability and that was produced as a result of research on production and processing methods of the raw material powder. Thus, the conventional sintering technique is in the process of being developed. However, pressure sintering is still performed in the current stage whenever high density compacts of high strength and reliability need to be manufactured.

We have studied the effect of pressure in pressure sintering before, specifically using high temperature materials such as zirconium boride (ZrB_2) [6,7]. In this paper, the result of studies on the effect of pressure in sintering SiC and its compact macro hardness is reported.

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*Numbers in the margin indicate pagination in the foreign text.

2. Experimental Method

2.1 Raw Material Powder

The SiC powder of H. Starck, W. Germany, was used as obtained. Although it was understood that this SiC was β-type with average granule diameter of 0.7 μm, its granule size distribution seemed to be spread out, since many small size granules of approximately 0.1 μm and large size granules that were larger than 2 μm were also observed by SEM. According to the analysis table made by the manufacturer, it contained 0.65% boron (B) as well as a slight amount of Fe.

As is clear from the experimental result, sintering conditions are required to be restrictive when only SiC is used, even if the pressure sintering technique is applied, although it is advantageous for high densification. We also conducted experiments on the powder which contained boron carbide (B_4C) as a sintering assistant. Boron carbide is said to be as effective as boron or boron+carbon in pressure sintering. The B_4C that we used was powder #1500, manufactured by Elektroschmelz of W. Germany. This was much more coarse than the SiC powder that we used. We put both powders in a polyethylene bottle, and mixed them using a bowl of super-hard alloy.

2.2 Pressure Sintering

Among pressure sintering techniques, there is what is called the super-high pressure sintering method using 5 (or 6) to 50 (or 60) kbar pressure, which has also been applied to SiC sintering. However, this is not a good technique for industrial use. In this experiment, pressure sintering was performed using graphite as a die and the standard single axis pressure was applied. The applicable pressure is restricted by the resistance of the graphite when this technique is applied. The maximum pressure of the conventional quality graphite die was 300 kg/cm^2: however, the pressure can be increased safely up to 600 kg/cm^2 if a die of super-strong graphite material, which has become available recently, is used. In this experiment, a die and punch made of this super-hard graphite material* was used when over 400 kg/cm^2 pressure was applied. /304

The graphite die was heated from room temperature by high frequency induction as a fixed pressure was applied in the range of 100-1000 kg/cm^2. After the die reached the expected temperature, it was left 30 minutes in the same condition. Then, the applied pressure was released and the die was left to cool. The temperature of the bottom of the hole made on the die was measured by a photo pyrometer, and the temperature was controlled by adjusting the pyrometer high frequency output. The maximum temperature observed in this experiment was 2300°C. The obtained compacts were 20x20x10 squares, or they were cylinders with diameter 15 mm and height 6-7 mm when sintering was performed with pressure above 800 kg/cm^2, since the pressure area was decreased because of the capacity of the oil pressurizer that was used.

2.3 Observation of the Sintered Compact

The carbon attached to compacts was taken off by grinding the surface lightly with a diamond wheel, and the compacts were boiled in water. The bulk density of compacts was obtained from the weights measured in water and air. The broken-out sections of compacts were observed by SEM, and X-ray diffraction was also performed.

2.4 Measuring of Rockwell Hardness

In general, hard materials such as ceramics are measured either by micro Vickers or Knoop hardness. However, these measurements represent microhardness. Because the impression area is small, these measurements represent the material or granule hardness in some cases. In order to reflect the degree of sintering development, which specifically means density, bonding among granules, and

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*Toyo carbon IVD-3 or Owada carbon OC-7660.

hardness of granules, it is desirable to measure macro hardness using a heavy weight and large impression area comprising a sufficient number of holes. Thus, it was decided to use the Rockwell A-scale hardness (HrA) for measuring using a diamond cone, with a standard load of 10 kg and testing load of 60 kg. Hardness was measured at approximately 10 places of the testing compact surface, which was ground in parallel motion by the diamond wheel, and their mean hardness was obtained.

3. Experimental Result and Its Examination

3.1 Density When SiC Only (no additive) Was Used

The densification process of the testing material filling the graphite die during pressure sintering may be found by observing the ram table movement on which the die was loaded. Table 1 shows the movement of the ram table when pressure sintering was performed as the constant pressure 400 kg/cm^2 and various temperatures were applied. Although the ram table moved upward as the testing material shrank and densified, swelling of the whole instrument including the die and punch caused by the temperature rise intensified this movement in such a way that the downward movement was intensified and made stronger than the upward movement until the density of the testing compacts grew prominent, while the temperature was rising or it was maintained at a fixed height to stabilize heat. Density of the testing material at an arbitrary time can be obtained from the following three measurements: the ram table movement when the testing material was not yet filled and was heated while other conditions were maintained the same; the ram table movement during the sintering experiment; and the final density and size of the testing material. However, the constant density change of the testing material during sintering of this method could not be obtained because the refractory material supporting the die deformed during pressure sintering upon applying over 2000°C temperature as was done in this experiment, and because the quantity of the refractory material usage was varied in each experiment.

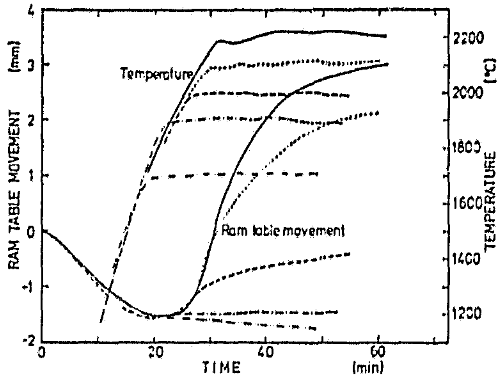


Fig. 1. Ram table movements during hot-pressing experiments on Starck β-SiC at 400 kg/cm^2.

From Fig. 1, it may be observed that densification begins at an approximate temperature of 1500°C, and at 1900°C density increases to the point where the ram table reverses its rotation direction. These temperatures did not change, it seemed, even when different pressures were applied. Densification was rapidly accelerated when the temperature rose past 1900°C and at the beginning of the period when the temperature was maintained at a certain level, but the testing material densification was increased very little after the temperature was stabilized for 30 minutes. The ram table movement during the temperature stabilization period, as shown in Table 1, indicates that densification steadily progressed; however, this movement was caused, as mentioned before, by the deformation of the refractory materials. Densities of the testing compacts that were sintered for 30 minutes at various temperatures and pressures are illustrated in Fig. 2, treating temperature as a parameter. Naturally, a higher pressure gives higher density compacts. When SiC only (no additives) is pressure sintered, sintering conditions are required to be more restrictive compared to other regular ceramic raw materials. For example, when a compact of density over 3 g/cm^3 /305
(approximately 93% relative density) is to be made, the temperature needs to be above 2200°C at 200 kg/cm^2 or 2100°C at 600 kg/cm^2.

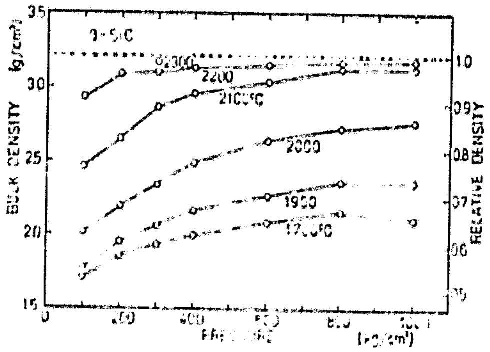


Fig. 2. Relations between applied pressure and density (β-SiC, hot-pressing time: 30 min).

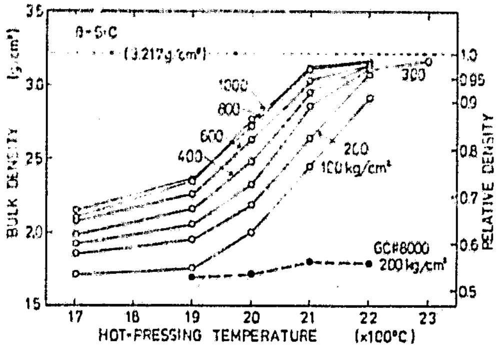


Fig. 3. Relations between hot-pressing temperature and density (β-SiC, hot-pressing time: 30 min).

Fig. 3 shows the relation between sintering temperature and density, treating pressure as a parameter. In all cases of different applied pressures, density varies most in the temperature range of 1900-2100°C, and the effect of pressure shows clearly. The figure also shows the result of testing on a commercial SiC grinder (Hujimi Grinder, GC #8000, α-type, average granule diameter approximately 0.9 μm). This grinder was sintered with application of the same pressure, for comparison purposes. This SiC showed very poor densification, while that of Starck densified well. It is considered that this difference was caused by different impurities that were contained, distribution of granule sizes, oxidation conditions on the granule surface, rather than being caused by the crystal phase of the powder. Especially the SiC of Starck contained a non-negligible amount of B, although its condition is not known. B is well known to be effective for promoting sintering. It is said that 0.3% B is sufficient to be effectively used as a sintering assistant [1] and the SiC of Starck contained B at a higher rate. As for granule size distribution, GC #8000 contained granules of more homogeneous diameters, while the SiC of Starck showed wider distribution because it contained many small particles, making the powder more active. No judgement can be made on the oxidation condition of the granule surface because the fluoric acid process was not performed. It is considered that SiC_2 on the surface of granules interferes with sintering.

3.2 SiC Densification Containing B_4C

B_4C was added as a sintering assistant before sintering was performed. Sintering was performed at 2000°C and 200 kg/cm^2; this is the condition that is considered to show most clearly the effect of the additive in order to determine the optimal amount of the additive. The density of the compacts containing different amounts of additive was examined. The result is shown in Fig. 4.

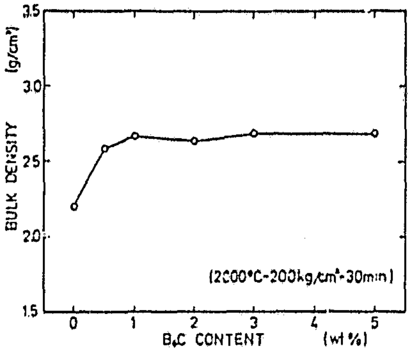


Fig. 4. Densities of SiC compacts containing various amounts of B_4C, hot-pressed at 2000°C and 200 kg/cm^2 for 30 min.

Although density grows as more B_4C is added, density becomes constant when more than 1 wt.% assistant is added. Thus, the B_4C amount was determined to be 1 wt.% for the following sintering experiments.

Figure 5 shows the ram table movement when SiC with B_4C added was sintered at 400 kg/cm^2 and at various temperatures. Densification was accelerated at a lower temperature than SiC with no B_4C; densification accelerated as temperature exceeded 1800°C. It was observed that the ram table moved irregularly at around 1600°C while the temperature rose. It is considered that this was some kind of reaction caused by B_4C, which occurs around this temperature.

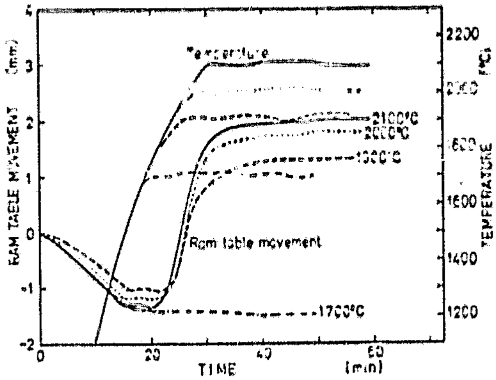


Fig. 5. Ram table movements during hot-pressing experiments on Starck β-SiC+1 wt.% B_4C at 400 kg/cm2.

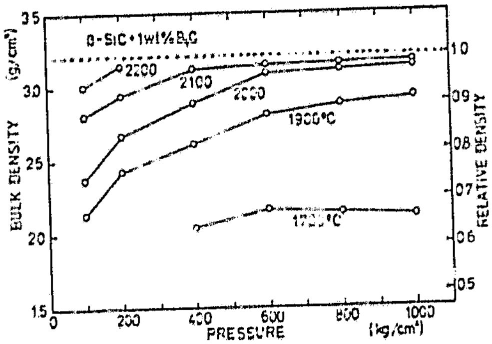


Fig. 6. Relations between applied pressure and density (β-SiC+1 wt.% B_4C, hot-pressing time: 30 min).

Density of the sintered compacts with added B_4C is shown in Figs. 6 and 7. Adding B_4C makes the sintering condition less restrictive. And when the pressure is the same, the same density can be obtained at a temperature 150-200°C lower than that required /306 by SiC that contains no additive. The pressure also has a greater effect. Although SiC powder contains B, it can be said that adding more B_4C gives a good effect for densification.

3.3 Effect of Pressure

The greater the pressure applied during sintering, the more advantageous it is for sintering and the lower the sintering temperature can be. When we studied ZrB_2 and NbB_2, it was found that pressure applied for sintering when a certain compact is to be made can be defined by the linear relation between the logarithm of pressure and the temperature. The following expression was found to be effective.

$$T(°C)=A-B\log P\,(kg/cm^2) \qquad (1)$$

Here, T represents the sintering temperature; P, pressure; and A and B, constants.

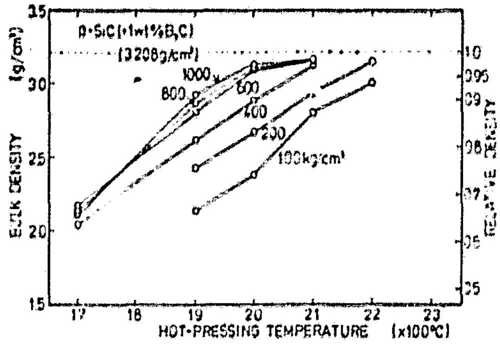


Fig. 7. Relations between hot-pressing temperature density (β-SiC+1 wt.% B_4C hot-pressing time : 30 min).

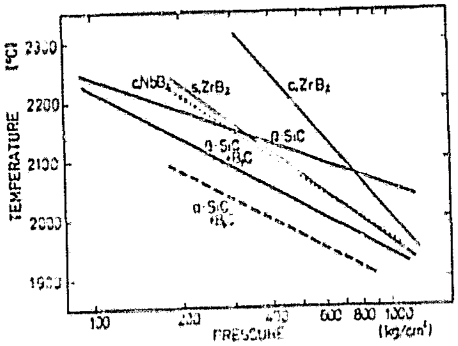
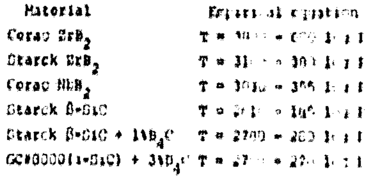


Fig. 8. Pressure-temperature conditions for realizing 95% density.

High temperature sintering materials such as SiC are defined to have 95% relative density. The expected sintering conditions for producing this density were calculated using Figs. 2, 3, 6, and 7 (within the range of conditions of this experiment). The relation between the logarithm of pressure and temperature in this sintering condition was found to be linear also with this SiC. This relation of SiC and other compacts that have been obtained is shown in Fig. 8, and their expressions are listed in Table 1.

Some of the constants in expressions differ according to different raw materials, even if the raw materials consist of the same elements. Particularly, the fact that constant 3, representing the effect of pressure, varies with each expression is considered to reflect the characteristics of the raw material powder. When constant B has a great value, it means, in these expressions, that increasing pressure has a great effect on densification. The effect of pressure on this SiC, compared to other materials, was not great, and the sintering temperature was only able to be decreased by no more than 185°C when the pressure was increased ten times higher within the range of experimental conditions applied. The effect of pressure was increased when B_4C was added, and temperature was decreased by 280°C. As is

Table 1. Empirical relations between hot-pressing temperature, T (°C) and pressure, P (kg/cm²) to realize 95% density.



Material	Empirical equation
Cerac ZrB_2	[illegible]
Starck ZrB_2	[illegible]
Cerac NbB_2	[illegible]
Starck β-SiC	[illegible]
Starck β-SiC + 1%B_4C	T = 2700 − [illegible]
GC#8000(α-SiC) + 3%B_4C	[illegible]

clear, also, adding B_4C makes sintering conditions less restrictive. Although we studied the relation between the constants in these expressions, melting points and elasticity rate of each material, we did not achieve a clear answer. It is considered that the constants relate to physical properties, particularly hardness and creep, which are activities induced under stress. However, the possible measurement of hardness is in microVickers hardness only up to 1400°C (Hv = 1000, 100 grf at 1400°C); therefore, it is desirable to measure creep within the range of sintering temperatures.

The figure shows the case of SiC containing 3 wt.% of B_4C, the optimal amount of additive determined for GC #8000 SiC. The sintering condition is less restrictive in this case than in the SiC of Starck which contains 1 wt.% B_4C. It is thought that sintering promotion by adding B_4C is generated by the reactions between B_4C and SiC_2 on the surface of SiC granules, transforming them to SiC and increasing surface energy and decreasing intergranular energy by melting into SiC. It is considered that the optimal quantity of B_4C is high when it is added in GC #8000 SiC because this SiC is oxidized to a higher degree, requiring more B_4C to generate the reaction with SiC. Also, it is possible that B-O type compounds promote SiC sintering in some way even though they may evaporate during sintering.*

3.4. Structure

Figure 9 shows SEM photographs of broken-out sections of

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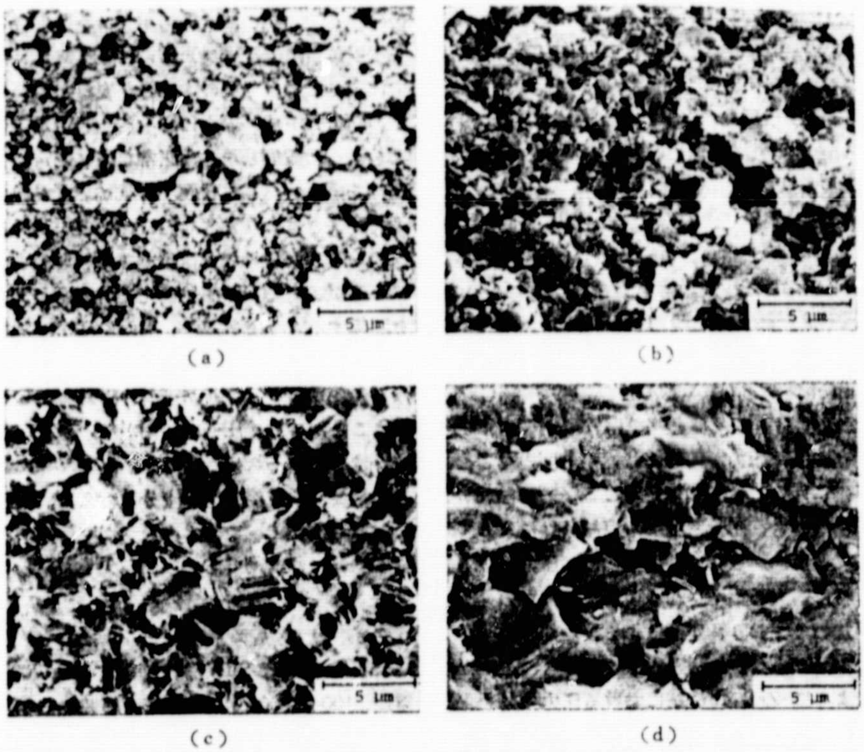
*According to pressure sintering transportation in which a large amount of C and B_2O_3 are added to SiC, a reduction in weight of the test material beyond the weight of the additives is observed, indicating an insignificant particle growth. It appears that B_2O_3 acts chemically upon SiC at high temperature.

sintered compacts. At low sintering temperatures, the conditions in compacts are more similar to the condition where raw material powder is simply filled. Compacts with density higher than 2.5 g/cm^3 show clearly the bonding junction among granules. This means a temperature above 2000°C in the case of SiC with no additive and above 1900°C in the case of SiC containing B_4C. The /307 intergranular field cannot be observed on broken-out sections, which means that breaking took effect intergranularly. If the granule size is inferred from the distribution of air bubbles, it is thought to be about 1-1.5 μm in the case of SiC containing no additive that was sintered at 2000°C, and 4 μm at 2200°C. It seems that sintering temperature or density determines granule size.



(a) (b)

(c) (d)

Fig. 9. Scanning electron micrographs of fractured surfaces of the compacts. (a) β-SiC powder.
(b) 1900°C-200 kg/cm^2 - 30 min (+1 wt.% B_4C).
(c) 2100°C-800 kg/cm^2 - 30 min. (d) 2200°C-200 kg/cm^2 - 30 min.

EPMA analysis was conducted on the B_4C additive. Although B_4C granules were recognized in compacts sintered at low temperatures, there was no clear information obtained regarding distribution of B_4C or B-element originated in B_4C in case of compacts sintered at high temperatures above 2000°C.

According to X-ray diffraction, it was observed that there was only the original β-type crystal phase in compacts sintered below 2100°C, and part of the phase was transformed into α-type among compacts sintered at temperatures above this. α-type crystals increase as the temperature rises and time passes, but this transformation was a slow process. Although it is considered that adding B_4C promotes this transformation [9], a significant difference was not observed.

3.5 Compact Hardness

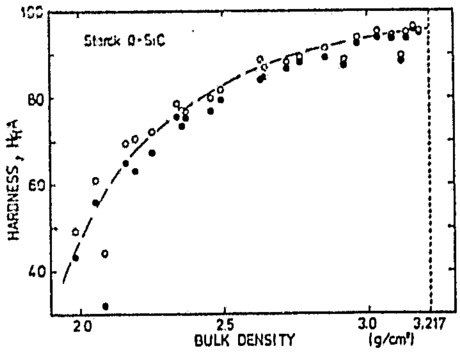


Fig. 10. Relations between density and Rockwell hardness of the compacts.

Figures 10 and 11 show the relation between density and Rockwell hardness of compacts. White circles in the figures represent the average hardness on the upper surface of compacts, and black circles the lower surface of compacts. The hardness of the lower surface is smaller. Probably it is because pressure was decreased and density was lowered at the bottom area of the compacts that were sintered to be in a shape of height and length ratio approximately 1 to 2, but still they were not the floating type when one axle pressure was applied.

The relation between hardness and density can be shown by one curved line. Here it is indicated that SiC compact hardness is not related to sintering conditions, and it directly relates to density. The general principle is that sintering is to be performed

at low temperatures when the pressure is great, or at high temperatures when the pressure is low when the same density compacts are to be obtained. However, this result specifically regarding SiC indicates that the same density gives the same sintered result no matter what sintering conditions are applied. As separating the sintering process into processes of density and strength increases, they are found to be directly related to each other in the case of SiC. This means that a density increase occurs as physical materials move, generating bonding among granules no matter what temperature /308
and pressure are applied. On the other hand, in the case of ZrB_2 that was reported in our previous paper, hardness differed even though density remained the same when different sintering conditions were applied [6]. It is considered that this was because granule deformation caused by pressure, particularly the deformation in the joint areas of granules where stress was concentrated, increased density, and the deformed joint areas were not sufficiently bonded. This probably increased the effect of pressure in sintering ZrB_2, as shown in Fig. 1. It is thought that granule deformation by pressure rarely occurred in SiC pressure sintering within the range of the experimental conditions.

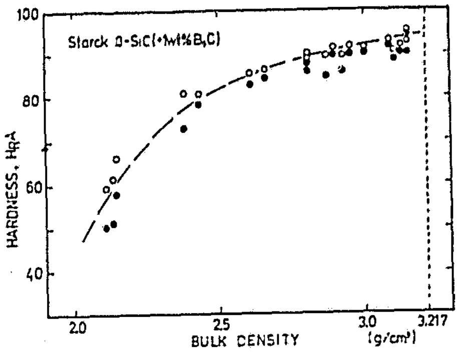
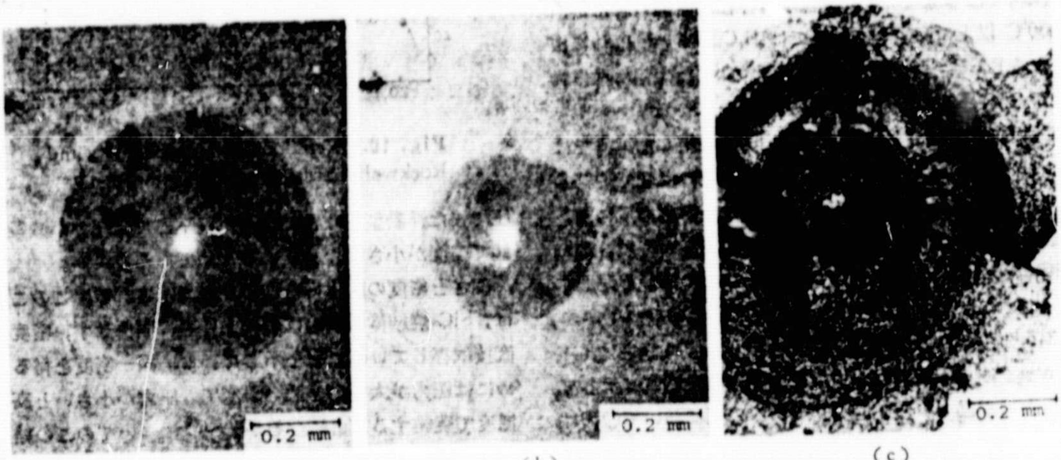


Fig. 11. Relations between density and Rockwell hardness of the compacts containing 1 wt.% B_4C.

The same density-hardness relation is indicated by compacts containing B_4C, but its hardness was slightly lower compared to the hardness of SiC with no additive, which was HrA = 96.

The implications of this measured hardness are not clearly known. When the hardness is small, it can be converted from HrA to Vick by using the hardness table of metals as a reference [11], but over 86 HrA is outside the application range of this table. Also, the condition of impressions differs according to different hardness. As shown in Fig. 12, compacts of low hardness and low density had cylinder-shaped impressions where the cone was directly pushed in, and the impression size varied according to hardness, while compacts of over 90 HrA had cracks and were chipped in the impressed area, making the measured values unreliable. If there were no cracks or chipping, the measured hardness would have had greater values.



(a) (b) (c)

Fig. 12. Impressions by Rockwell hardness test.
(a) = 2.08 g/cm^3, HrA=56, (b) = 2.23 g/cm^3, HrA=72,
(c) = 3.04 g/cm^3, HrA=94.

When measuring was performed with compacts of high hardness using a heavy weight load, the tip of the diamond core was broken. It seemed that this breakage was related to chipping. Micro Vickers hardness was measured on several high density compacts.

Rockwell hardness measurement was easily conducted by simply reading the dial gauge, and discrepancies of measured values were less common than in Vickers hardness measuring, as long as the tip was not broken. This kind of macro hardness measuring method would be more effective for low density compacts since grinding cannot be conducted to a sufficient degree because of a high rate of existence of air bubbles, making it impossible to measure impressions.

4. Summary

Single-axis pressure sintering was performed in the temperature range 1700-2300°C and pressure 100-1000 kg/cm^2 using β-SiC powder of Starck, and the effect of pressure on densification was studied. Rockwell hardness of the produced SiC compacts was measured.

1. Densification was promoted by increasing pressure, and it was most effective when pressure sintering was performed at 1900-2100°C. However, fairly strict sintering conditions were required to produce compacts with sufficiently high density. The conditions for producing 95% relative density β-SiC could be expressed by a linear relation of the logarithm of pressure and temperature, and the following formula was obtained:

$$T(°C)=2610 - 185 \log P \ (kg/cm^2) \qquad (2)$$

2. When B_4C was contained at 1 wt.%, required sintering conditions became less strict and the effect of pressure was largely increased. The sintering condition can be expressed as follows. /309

$$T=2780-280 \log P$$

This means that the necessary sintering temperature can be decreased by 185°C when 10 times higher pressure is applied in the case of SiC with no additives, and by 280°C in the case of SiC with the additive.

3. When compacts had the same density, they showed the same Rockwell hardness no matter what pressure and temperature were applied during sintering. In other words, it was understood that density increase promoted strength, and that the same density generated the same condition in sintered compacts.

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sintered compacts. At low sintering temperatures, the conditions in compacts are more similar to the condition where raw material powder is simply filled. Compacts with density higher than 2.5 g/cm^3 show clearly the bonding junction among granules. This means a temperature above 2000°C in the case of SiC with no additive and above 1900°C in the case of SiC containing B_4C. The /307 intergranular field cannot be observed on broken-out sections, which means that breaking took effect intergranularly. If the granule size is inferred from the distribution of air bubbles, it is thought to be about 1-1.5 μm in the case of SiC containing no additive that was sintered at 2000°C, and 4 μm at 2200°C. It seems that sintering temperature or density determines granule size.

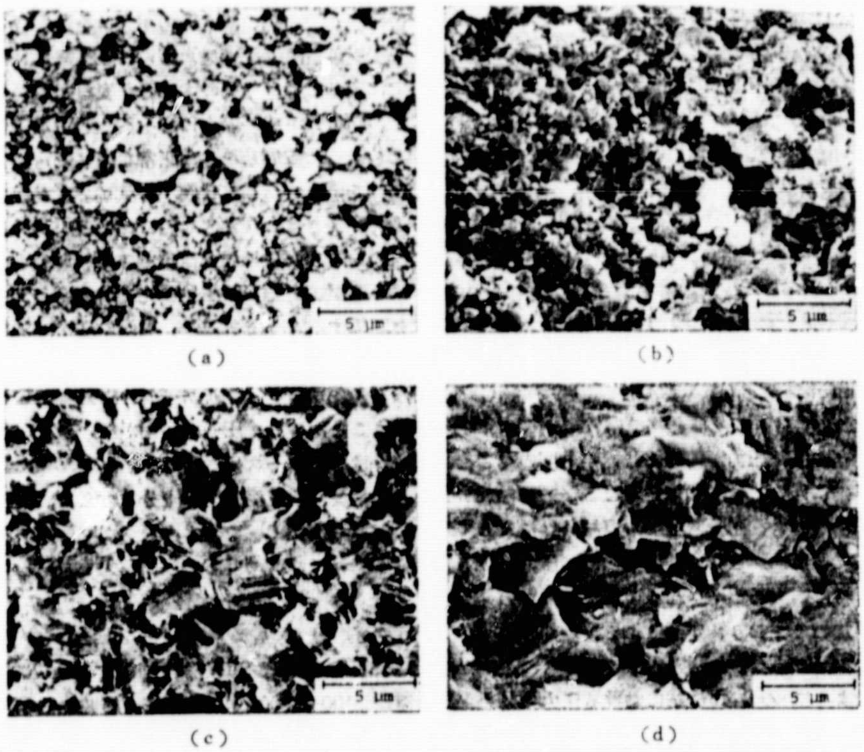
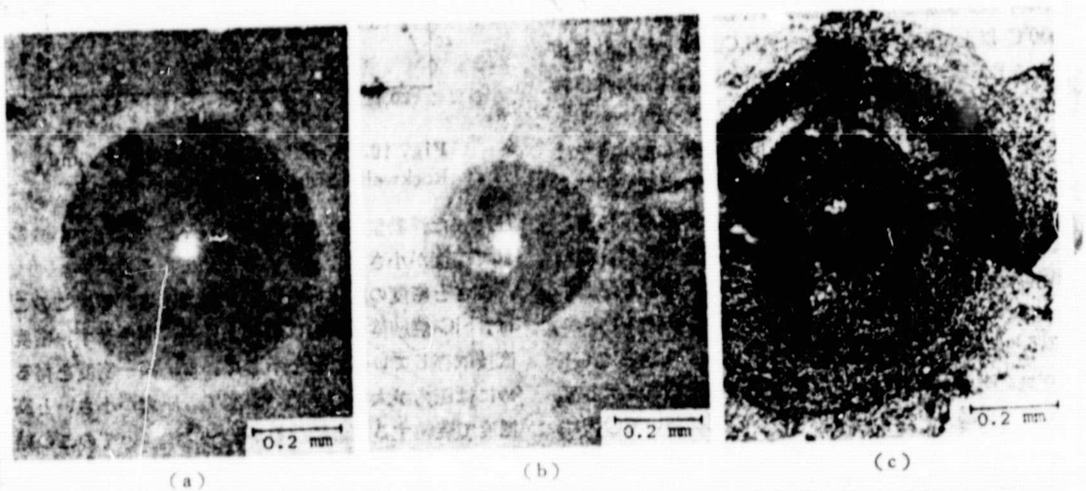


Fig. 9. Scanning electron micrographs of fractured surfaces of the compacts. (a) β-SiC powder.
(b) 1900°C-200 kg/cm^2 - 30 min (+1 wt.% B_4C).
(c) 2100°C-800 kg/cm^2 - 30 min. (d) 2200°C-200 kg/cm^2 - 30 min.

hardness table of metals as a reference [11], but over 86 HrA is outside the application range of this table. Also, the condition of impressions differs according to different hardness. As shown in Fig. 12, compacts of low hardness and low density had cylinder-shaped impressions where the cone was directly pushed in, and the impression size varied according to hardness, while compacts of over 90 HrA had cracks and were chipped in the impressed area, making the measured values unreliable. If there were no cracks or chipping, the measured hardness would have had greater values.



(a) (b) (c)

Fig. 12. Impressions by Rockwell hardness test.
(a) = 2.08 g/cm^3, HrA=56, (b) = 2.23 g/cm^3, HrA=72,
(c) = 3.04 g/cm^3, HrA=94.

When measuring was performed with compacts of high hardness using a heavy weight load, the tip of the diamond core was broken. It seemed that this breakage was related to chipping. Micro Vickers hardness was measured on several high density compacts.