Anisotropic sintering shrinkage and microstructural evolution of c-axis-oriented Si₃N₄ ceramics

Takuma TAKAHASHI¹,†, Junichi TATAMI¹,², Hiromi NAKANO³ and Satoshi TANAKA⁴

¹Kanagawa Institute of Industrial Science and Technology, 705–1 Shimoimaizumi, Ebina, Kanagawa 243–0435, Japan
²Yokohama National University, 79–1 Tokiwadai, Hodagaya-ku, Yokohama 240–8501, Japan
³Toyohashi University of Technology, 1–1 Hikarigaoka, Tempaku-cho, Toyohashi, Aichi 441–8580, Japan
⁴Nagaoka University of Technology, 1603–1 Kamitomioka-machi, Nagaoka, Niigata 940–2188, Japan

High-thermal-conductivity Si₃N₄ ceramics are desired for substrates in SiC power devices. We successfully fabricated highly c-axis-oriented Si₃N₄ ceramic by casting in a rotating-magnetic field and then sintering using a conventional gas pressure. As a result, the c-axis orientation of Si₃N₄ grains effectively improves the thermal conductivity. The sintering behavior of c-axis-oriented Si₃N₄ ceramics was investigated. The linear shrinkage ratio in the c-axis-oriented direction was smaller than that in the direction normal to the c-axis-oriented direction. Fine c-axis-oriented structures resulted from the orientation of β-Si₃N₄ seeds during the casting because they played as templates controlling the grain growth of β-Si₃N₄ nuclei and inducing the anisotropic sintering shrinkage.

Key-words : Silicon nitride, Magnetic field orientation, Sintering shrinkage, Thermal conductivity

‡ Corresponding author: T. Takahashi; E-mail: cp-t-takahashi@newkast.or.jp

1. Introduction

Silicon nitride (Si₃N₄) is a typical engineering ceramic because of its good mechanical properties such as high strength, hardness, toughness, and good chemical and thermal stabilities.¹⁻¹¹ Substrates with high thermal conductivities in the thickness direction are urgently needed for silicon carbide power devices.¹² Silicon nitride (Si₃N₄) ceramics are expected to be applied as substrates because they have better mechanical properties than conventional aluminum nitride ceramics. Si₃N₄ itself exhibits anisotropic thermal conductivity, i.e., the thermal conductivity of Si₃N₄ in the c-axis direction is higher than that in the a-axis direction,¹³,¹⁴ which means that having the c-axis of Si₃N₄ as the thickness direction should be effective in making Si₃N₄ a high-thermal-conductivity substrate.

In previous works, c-axis-oriented Si₃N₄ ceramics were fabricated via moulding processes in which external fields were applied. Hirao et al. reported Si₃N₄ ceramics with anisotropic microstructures fabricated by tape-casting and hot-pressing processes.¹⁵ Watari et al. also reported that crystal-oriented Si₃N₄ ceramics were fabricated using the tape-casting method, followed by hot isostatic pressing.¹⁶ Because the shape of the β-Si₃N₄ seed particles is rod-like and they are easily oriented in the in-plane direction by shear stress in the tape-casting process, the c-axes of β-Si₃N₄ grains cannot be oriented in the thickness direction of the substrate. c-axis-oriented Si₃N₄ ceramics were fabricated by applying stress fields and magnetic fields. In this process, the orientation of ceramic particles is not restricted by the moulding process and geometry of the component, which means that a substrate in which the c-axis of Si₃N₄ is oriented in the thickness direction is possibly obtained. Li et al. reported the first fabrication of c-axis-oriented Si₃N₄ ceramics using a magnetic field.¹⁷ Zhu et al. reported the fabrication of c-axis-oriented Si₃N₄ ceramics using a rotating magnetic field.¹⁸,¹⁹ In their work, 5 wt % β-Si₃N₄ whiskers were added to the powder mixture as seeds.¹⁸ Fine near-equiaxed 5 wt % β-Si₃N₄ particles were also used.¹⁹ These seeds were highly oriented using a rotating magnetic field of 12 T in green bodies. Because of sintering the green bodies, c-axis-oriented Si₃N₄ ceramics were successfully obtained. Particularly, the c-axis-oriented Si₃N₄ ceramics containing fine near-equiaxed particles exhibited very high thermal conductivities.¹⁹ Recently, Takahashi et al. reported the fabrication of c-axis-oriented Si₃N₄ ceramics via a low magnetic field orientation process using multilayered-graphene-coated β-Si₃N₄ seed particles prepared by mechanical treatment.²⁰

The anisotropic sintering shrinkage behaviour has often been reported in c-axis-oriented Si₃N₄ ceramics.¹⁸,²¹,²² Teshima et al. reported the anisotropic shrinkage of c-axis-oriented Si₃N₄ ceramics fabricated by extrusion moulding and gas pressure sintering.²¹ In their report, the sintering...
shrinkage ratios were 21% in the extrusion direction and 7% in the radial direction. Zhu et al. also reported anisotropic sintering shrinkages 22% in the parallel and 7% in the normal directions relative to an applied rotating magnetic field. 18) Park et al. reported a model based on the anisotropic sintering shrinkage of Si3N4 containing oriented β-Si3N4 whiskers. 21) In their report, the anisotropy of shrinkage became larger as the whiskers content increased. In these reports, only the property and microstructure of the final product were shown; the densification and microstructure evolution mechanism were not clarified. Densification of Si3N4 ceramics resulted from particle rearrangement, α-β phase transformation, and Ostwald ripening of β-Si3N4 grains. 23) The grain growth of β-Si3N4 grains in the c-axis direction is faster than in the a-axis direction. As a result, elongated β-Si3N4 grains are developed during densification of Si3N4 ceramics. Therefore, a green body having oriented Si3N4 seeds should exhibit anisotropic shrinkage and microstructure development.

It has been reported that densification and grain growth progress anisotropically in crystal oriented ceramics. 24)–27) The anisotropic sintering shrinkage completely depends on the preferential direction of grain growth and the packing structure of green compact. Patwardhan et al. examined the anisotropic sintering shrinkage of roughly oriented aluminium oxide (Al2O3) ceramics fabricated using ellipsoidal particles by the doctor blade technique. 28) Shui also examined the anisotropic sintering shrinkage of c-axis-oriented Al2O3 ceramics fabricated by applying a magnetic field. 29) Tanaka et al. reported c-axis-oriented zinc oxide (ZnO) powder compacts prepared using a high magnetic field orientation process. 30) Takahashi et al. examined the anisotropic sintering shrinkage and microstructural evolution of c-axis-oriented strontium barium niobate (SBN) powder compacts prepared by applying a high magnetic field. 31) In these reports, the mechanisms of anisotropic sintering shrinkage and microstructural evolution differ depending on the material. Although the anisotropic sintering shrinkage and microstructural evolution during solid phase sintering have been reported above, details of the anisotropic sintering shrinkage and microstructural evolution of c-axis-oriented Si3N4 via liquid phase sintering and phase transformation have not been investigated. In this work, the anisotropy of sintering shrinkage and microstructure evolution of c-axis-oriented Si3N4 ceramics were investigated.

Green compacts with c-axis-oriented β-Si3N4 seeds were prepared by casting in a magnetic field at 10 T. Specimens for measuring the sintering shrinkage normal and parallel to the applied magnetic field were prepared by cutting out from the green compact after dewaxing.

2. Experimental procedures

β-Si3N4 (NP-500, Denka Co., Ltd., Japan) and α-Si3N4 powders (SN-E10, Ube Industries Ltd., Japan) were used as raw materials. Figure 1 presents the morphology of β-Si3N4 seed particles observed using a scanning electron microscope (SEM, JSM-6390LV, JEOL, Japan). The average particle size (d50) measured by laser diffraction/scattering (SALD-7000, Shimadzu Co., Japan) was 0.6 μm. The average particle size was also measured from image analysis using Fig. 1, and was the same as the d50 measured by the laser diffraction/scattering method. The average aspect ratio calculated from image analysis was 1.5. Sintering aids are needed to fabricate dense Si3N4 ceramics. Li et al. have been reported that Y2O3–HfO2–SiO2 system can improve not only enhancement of the densification of Si3N4 but also formation of cubic HfO2 crystalline phases to reduce grain boundary glassy phase. 28) Therefore, the Si3N4–Y2O3–HfO2–SiO2 system should be expected to achieve high thermal conductivity. Y2O3 (RU-P, Shin-etsu Chemical Co., Ltd., Japan), HfO2 (HF001PB, Kojundo Chemical Laboratory Co., Ltd., Japan), and SiO2 (SO-C2, Admatechs Co., Ltd., Japan) were added as sintering aids. The starting composition was fixed at β-Si3N4:α-Si3N4:Y2O3:HfO2:SiO2 = 10:82:2.5:5:0.5 by weight. These powders were mixed and dispersed by ultrasonic radiation with polyethyleneimine (Mw = 10000) dispersant in distilled water, with solid loading of 20 vol %. The slurry was loaded in a plastic mould 25 mm in diameter and placed in a rotating magnetic field at 10 T. After drying, a green compact was obtained, which was calcined at 500°C for 3 h in air to burn the polymer dispersant. The dewaxed green body was fired at 1700–1900°C for 5 min or 6 h in 0.9 MPa N2.

Relative density was measured by the Archimedes method. Phases and crystal orientations of the Si3N4 ceramics were estimated by X-ray diffraction (XRD, Multitex 2kW, Rigaku Co., Japan). The Lotgering factor (L.F.), as the orientation degree, was calculated from the XRD profiles by the following equation: L.F. = (ρ − ρ0)/(1 − ρ0), where ρ0 = ΣIh(00l)/ΣI0(hkl) and ρ = ΣI(00l)/ΣI(hkl), where Ih and I are the diffraction peak intensities of non-oriented and oriented samples, respectively. The linear shrinkage ratio was evaluated from sample geometry. The plasma-etched surface was observed by SEM. A thin film sample for transmission electron microscopy observation was prepared by Ar ion milling and specimens were observed using a transmission electron microscope (TEM, JEOL-2100F, JEOL, Japan). The compositions of the specimens were measured by energy dispersive
X-ray spectrometry in scanning TEM (STEM-EDS). The $\beta$ ratio was calculated by the Rietveld method using RIETAN-FP.29

3. Results and discussion

3.1 Density and sintering shrinkage

Table 1 lists the relative density and sintering shrinkages of Si$_3$N$_4$ ceramics prepared with and without a rotating magnetic field. The relative densities of Si$_3$N$_4$ ceramics prepared with and without a rotating magnetic field were almost the same: 99 and 96%, respectively, which were estimated using the true density calculated from the raw powder composition. The thermal conductivity in the c-axis orientation direction was 100 W·m$^{-1}$·K$^{-1}$ (the non-oriented ceramics had 65 W·m$^{-1}$·K$^{-1}$). The anisotropic linear shrinkage ratio showed the same tendency as reported in previous studies; that is, the linear shrinkage ratio along the direction parallel to the rotational axis of the sample in the magnetic field was smaller than that in the direction normal to the rotational axis.

Figure 2 shows the densification and anisotropic sintering shrinkage behaviours of c-axis-oriented Si$_3$N$_4$ ceramics. The horizontal axis shows the sintering condition and the vertical axis shows the relative density and linear shrinkage ratio parallel and normal to the applied magnetic field. Increase in the firing temperature and soaking time increased the relative densities of the sintered bodies. Although the relative density obtained with a rotating magnetic field was the same as that without a magnetic field, the sintering shrinkage ratio strongly depended on the direction of the applied magnetic field, i.e., the linear shrinkage ratio normal to the rotational axis of the sample in the magnetic field was larger than that parallel to the rotational axis of the sample in the magnetic field. This anisotropic sintering shrinkage increased markedly with an increasing sintering temperature above 1800°C. The linear shrinkage ratios in the directions parallel and normal to the rotational axis of the sample in the magnetic field were 9 and 16%, respectively, at 1900°C. Finally, the linear shrinkage ratio in the direction parallel to the rotational axis of the sample in the magnetic field decreased to 3%, which means that the sample expanded in the direction normal to the applied magnetic field at 1900°C. Although the anisotropic shrinkage ratios of Si$_3$N$_4$ ceramic in final products have been reported previously,20,21,22 this strange anisotropic sintering shrinkage behaviour was discovered in the present study. The reason for the anisotropic sintering shrinkage should be related to the microstructural evolution of Si$_3$N$_4$ ceramic.

3.2 Phases present

Figures 3 and 4 show XRD patterns of the green compact and sintered bodies prepared with and without a rotating magnetic field (10 T). The plane parallel to the magnetic field was measured as shown in Fig. 4. A strong (002) peak of $\beta$-Si$_3$N$_4$ was observed in the pattern of the green compact prepared with a rotating magnetic field, but not in that of the green compact prepared without the magnetic field. This result indicates that the c-axes of the $\beta$-Si$_3$N$_4$ seed particles were oriented normal to the direction of the applied magnetic field. After firing at 1900°C for 6 h, the peaks of $\alpha$-Si$_3$N$_4$ disappeared and the (002) peak of $\beta$-Si$_3$N$_4$ increased. The orientation degree calculated using the Lotgering method was 0.70 after densification. It was confirmed that $\alpha$-$\beta$ phase transformation and orientation of $\beta$-Si$_3$N$_4$ occurred in association with densification.

Figure 5 shows the $\beta$ ratio changes of Si$_3$N$_4$ ceramics prepared with and without the magnetic field during sintering. The $\beta$ ratio change showed the almost same trend between the non-oriented and c-axis-oriented Si$_3$N$_4$ ceramics. As shown in Fig. 3, the relative density was also the same, independent of orientation. From these results, the driving force for sintering due to the $\alpha$-$\beta$ phase transformation was unchanged; that is, the $\alpha$-$\beta$ phase transition is not the principal factor causing the anisotropic sintering shrinkage behaviour.
3.3 Microstructural evolution

Figures 6(d) and 7(d) show the microstructures of sintered bodies prepared with and without the rotating magnetic field and which were fired at 1900°C for 6h. The observed plane was parallel to the rotational axis of the sample in the magnetic field. In the sintered body prepared with the magnetic field, the c-axes of the prismatic Si₃N₄ grains were dominantly along the direction normal to the applied magnetic field. In contrast, a non-oriented structure was observed in the sintered body prepared without magnetic field. These results are consistent with previous reports.¹⁵⁻²⁰

The densification of Si₃N₄ ceramics is related to the α-β phase transformation and the development of rod-like β-Si₃N₄ grains. Figures 4 and 7 present the XRD patterns and microstructures of c-axis-oriented Si₃N₄ ceramics fired at various conditions. As observed in Fig. 4, an α-β phase transformation occurred and the intensities of the (002) and (101) peaks increased with firing temperature and soaking time. During the initial sintering stage [Figs. 4(a) and 7(a)], the oriented β-Si₃N₄ seed particles grew with the α-β phase transformation via liquid phase sintering. Here, although the microstructures of the non-oriented and c-axis-oriented bodies were completely different [shown in Figs. 6(a) and 7(a)], their relative densities were the same at 66%. Figure 8 shows the grain size distributions of ceramics fired at 1700°C for 5 min, as shown in Figs. 6(a) and 7(a). The y-axis is the quantity of grain per unit area and the x-axis is the equivalent circle diameter. The grain size distribution in Fig. 8 is almost the same and it can be considered that the β-Si₃N₄ seeds grew individually. This result means that the effect of seeds on the formation of microstructure was the same during the initial stage of sintering in both the non-oriented and c-axis-oriented bodies. That is, in the c-axis-oriented Si₃N₄ ceramics, β-Si₃N₄ nuclei produced from the α-β phase transformation contributed to form the highly oriented microstructure via their orientation. In Figs. 4(b) and 7(b), the α-β phase transformation was completed and the β-Si₃N₄ nuclei produced from the α-β phase transformation grew between elongated β-Si₃N₄ grains. In particular, the intensity of the (101) plane increased. In Figs. 4(c) and 7(c), smaller β-Si₃N₄ grains with the same direction as the larger elongated β-Si₃N₄ grains grew preferentially, and the intensity of the (002) plane increased. Finally, the (002) plane peak remained [as shown in Fig. 4(d)], the oriented β-Si₃N₄...
grains grew markedly, and the oriented structure was completely formed, as shown in Fig. 7(d).

The microstructures were evaluated in more detail using TEM. **Figure 9** presents the high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) images of non-oriented and c-axis-oriented Si$_3$N$_4$ ceramics. The observed plane was normal to the rotational axis of the sample in the magnetic field. The bright area is an intergranular glassy phase consisting of SiO$_2$ and sintering aids. Comparing the HAADF-STEM images, the
thicknesses of the intergranular glassy phases were clearly different. The c-axis-oriented Si₃N₄ ceramics had very thin glassy phases between hexagonal grains. In contrast, the non-oriented Si₃N₄ ceramics had thick glassy phases around the grains. The difference in thickness of the glassy phase might result from the liquid phase being squeezed out from between the c-axis-oriented β-Si₃N₄ grains. On the other hand, if β-Si₃N₄ grains grow randomly, the glassy phase seems thicker because the space between β-Si₃N₄ grains was only filled by the liquid phase. 

Table 2 presents an analysis of the orientation direction of β-Si₃N₄ grains using Fig. 9(a). The degree of the tilt angle ϕ from the basis [000l] axis was calculated using the following equations:

\[
\varphi = \cos^{-1}(\cos x \cdot \cos y) \quad (1)
\]

\[
\theta = \tan^{-1}\left(\frac{\tan y}{\sin x}\right) \quad (2)
\]

ϕ of most of the oriented β-Si₃N₄ grains was small. High-integrity adjacent grains had similar and smaller ϕ (for example, the ϕ values between the β-Si₃N₄ grains Nos. 1 and 3 were 2.19 and 2.26, respectively). The grain boundary was round in such high-integrity β-Si₃N₄ grains.

In contrast, the grain boundary was straight for low-integrity β-Si₃N₄ grains (for example, the ϕ values between the β-Si₃N₄ grains Nos. 5 and 10 were 2.41 and 13.13, respectively).

3.4 Mechanism of anisotropic sintering shrinkage and microstructure evolution

In oriented Al₂O₃ ceramics, the linear shrinkage ratio along the direction with higher necking among particles per unit length in the powder compact was larger during the initial stage of sintering than that along the other direction. In addition, during the middle and final stages of sintering, the difference in shrinkage decreased gradually with densification and grain growth. This result indicates that the anisotropic sintering shrinkage of oriented Al₂O₃ ceramic depends on the packing structure of the powder compact. The anisotropic sintering shrinkage behaviour of Al₂O₃ ceramic is not the same as that of c-axis-oriented Si₃N₄ ceramics. Shrinkage occurred regardless of direction during the initial stage of sintering in the case of c-axis-oriented Si₃N₄ ceramics, which means that the packing structure of the powder compact is not dominant. Densification and grain growth occurred isotropically during the initial stage of sintering in c-axis-oriented ZnO ceramics. Anisotropic shrinkage and grain growth occurred simultaneously during the middle and final stages of sintering. The linear shrinkage ratio along
the direction of grain growth was larger than that normal to its direction.\textsuperscript{26)} That is, the grain growth of ZnO is strongly related to anisotropic sintering. For SBN, neck formation (densification) and grain growth along the c-axis direction dominated the anisotropic sintering shrinkage. The linear shrinkage ratios of the c-axis direction during initial and final sintering were larger than those of the a-axis direction.\textsuperscript{27)} It was the same as the behaviour of Si\textsubscript{3}N\textsubscript{4}: the anisotropy of sintering shrinkage in ZnO and SBN increased markedly in the final stage. However, in the case of Si\textsubscript{3}N\textsubscript{4}, the mechanism of anisotropic sintering shrinkage and microstructure development should be completely different from the above cases because densification and grain growth occur via liquid phase reaction.

This anisotropic sintering behaviour and formation process of the c-axis-oriented structure in Si\textsubscript{3}N\textsubscript{4} ceramics can be considered as in Fig. 10. Initially, the \(\beta\)-Si\textsubscript{3}N\textsubscript{4} seed particles grew markedly along the direction normal to the applied magnetic field. Therefore, sintering shrinkage was limited by the remarkably elongated grain growth of \(\beta\)-Si\textsubscript{3}N\textsubscript{4}; shrinkage along the direction parallel to the applied magnetic field (\(\Delta L_a\)) was larger than that along the normal direction (\(\Delta L_c\)). During hot pressing of Si\textsubscript{3}N\textsubscript{4} ceramics, it has been reported that elongated grain growth occurred in the direction normal to the applied stress.\textsuperscript{16)} In this work, although external stress was not applied during sintering, internal stress should result from anisotropic grain growth of the \(\beta\)-Si\textsubscript{3}N\textsubscript{4} seed particles, which means a higher sintering stress along the parallel direction than the normal direction. The anisotropic sintering stress from the seeds and nuclei formed by the \(\alpha\)-\(\beta\) phase transformation increased the grain growth of \(\beta\)-Si\textsubscript{3}N\textsubscript{4} nuclei and inducing the anisotropic sintering shrinkage.

4. Conclusion

Highly c-axis-oriented Si\textsubscript{3}N\textsubscript{4} ceramics were fabricated using magnetic field orientation and gas pressure sintering. Although the densification behaviours were almost identical, the sintering shrinkage behaviours differed depending on the c-axis orientation; the linear sintering shrinkage ratio in the c-axis direction was smaller than that in the direction normal to the c-axis. The \(\alpha\)-\(\beta\) phase transformation occurred similarly without relation to the c-axis orientation. Fine c-axis-oriented structures resulted from the orientation of \(\beta\)-Si\textsubscript{3}N\textsubscript{4} seeds during the casting because they played as templates controlling the grain growth of \(\beta\)-Si\textsubscript{3}N\textsubscript{4} nuclei and inducing the anisotropic sintering shrinkage.

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Takahashi et al.: Anisotropic sintering shrinkage and microstructural evolution of c-axis-oriented Si₃N₄ ceramics

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