Effects of ZrB$_2$ addition on texture development and properties of porous Si$_3$N$_4$–ZrB$_2$ composites by magnetic field alignment

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ABSTRACT

Textured porous Si$_3$N$_4$–ZrB$_2$ composites were prepared by the gel-casting method assisted by magnetic field alignment technology and subsequent pressureless sintering. These results showed that when a 6 T magnetic field was applied, the ZrB$_2$ grains also formed a c-axis orientation in the textured green bodies, except for β-Si$_3$N$_4$ grains, which formed an a- or b-axis orientation. The increasing ZrB$_2$ content inhibited the degree of texture of the Si$_3$N$_4$ and ZrB$_2$ grains in the green bodies, respectively. After sintering at 1800 °C, new ZrN and BN phases without grain orientation characteristics formed by the reaction between Si$_3$N$_4$, ZrB$_2$ and N$_2$ and the ZrB$_2$ phase disappeared completely. The sintering process promoted the degree of texture of Si$_3$N$_4$ as the matrix increases. Meanwhile, the variations in the degree of texture of Si$_3$N$_4$ with changes in the ZrB$_2$ content were consistent with that in the green body. Additionally, when 10 wt% ZrB$_2$ content was introduced, the apparent porosity reached its highest value and the bulk-density reached its lowest value, leading to the appearance of least strength. In textured porous composites, the strength was mainly affected by the densification process instead of the grain orientation. Also, no evident difference in strength existed between the two directions owing to the function of high porosity.

KEYWORDS

Grain orientation; porous composite; magnetic field alignment; Si$_3$N$_4$; ZrB$_2$

1. Introduction

Porous Si$_3$N$_4$ ceramics have been widely researched owing to their excellent properties, such as high strength, low density, high thermal shock resistance and low coefficient of thermal expansion. These properties enable them to be used successfully in many fields, including separation membranes, catalyst supports, gas filters and heat exchangers [1–4]. Recently, in order to meet requirements for further applications under special conditions, porous Si$_3$N$_4$ ceramics with tailored microstructures are being developed and made integrant.

Today, many technologies have been developed to prepare porous ceramics by tailoring the microstructure. These include emerging technologies for controlling the microstructures of porous ceramics by magnetic field alignment, which can give them special microstructures that include grain orientation and special pore channels [5,6]. These unique microstructures are expected to give porous ceramics excellent structural and functional properties [7]. Magnetic field technology has been successfully applied in the preparation of textured ceramics for decades. Grains with anisotropic magnetic susceptibility will form an orientation along one direction with minimal system energy when acted upon by a magnetic force [6,8,9]. At present, these technologies are mainly used to prepare textured dense ceramics. However, their application in the preparation of porous ceramics is rare. In an earlier report, we prepared textured porous single-phase Si$_3$N$_4$ ceramics using a new technology, in which Si$_3$N$_4$ grains show a high degree of orientation [7,10]. So far, in response to the limitation of the properties of materials prepared by single-phase composition, porous ceramic composites have been rapidly developed to meet demand for special applications by improving the properties. ZrB$_2$ is an attractive candidate for hypersonic aerospace applications owing to its excellent properties of a high melting point (>2500 °C), high thermal properties and good chemical stability. Based on previous studies, ZrB$_2$ grains showed anisotropic magnetic susceptibility and their c-axis could be oriented to align along the magnetic field direction [11,12]. Current research into textured ceramics by magnetic orientation technology focuses mainly on single-phase ceramic systems. The effect of the secondary phase on texture development of composites is rarely reported. The orientation of ZrB$_2$ gains as a secondary phase could affect the orientation of Si$_3$N$_4$ grains via the rotation competition process. Additionally, ZrB$_2$ as the secondary...
phase could react with Si$_3$N$_4$ to form a new phase. The effect of the reaction process between two textured phase compositions on the orientation and microstructure is important for the preparation of textured composites. Therefore, in this paper, we focus mainly on the research on the effects of ZrB$_2$ addition as the secondary phase on the grain orientation of porous Si$_3$N$_4$ composites.

Additionally, the gel-casting method is a suitable processing route for preparing porous ceramics with special shapes and high quality. The gel-time (formation time) can be controlled by the initiator and catalyuster content. Gelled green bodies have a uniform microstructure and high green strength [13,14]. By combining the gel-casting method and magnetic field alignment technology, highly textured porous ceramics or composites with excellent properties can be prepared in a reasonable time. Earlier, we also fabricated many kinds of textured ceramics, including Si$_3$N$_4$, SiC and TiB$_2$-(Ti, Zr)B$_2$ ceramics with a highly defined grain orientation by gel-casting assisted by magnetic field alignment technology [7,15,16]. Thus, the combined method is an effective approach for preparing textured porous Si$_3$N$_4$-ZrB$_2$ composites.

In this study, textured porous Si$_3$N$_4$-ZrB$_2$ composites were prepared by gel-casting assisted by magnetic field alignment technology. The effects of the ZrB$_2$ content on the grain orientation and properties of textured porous composites were examined. The effect of sintering processing on the phase composition of porous composites was also investigated.

2. Experimental procedure

Commercially available β-Si$_3$N$_4$ grains with an average diameter of 0.8 μm were used as the matrix and ZrB$_2$ grains with an average diameter of 1 μm were employed as the secondary phase. Y$_2$O$_3$ and amorphous SiO$_2$ at a weight ratio of 6.5:3.5 (10 wt%, relative to the total powders) were used as sintering aids. The ZrB$_2$ contents were 0, 5, 10, 15, 20 wt% (relative to the total powders). Tetramethylammonium hydroxide (TMAH) solution was used as the dispersant and distilled water as the solution medium. 5 wt% NaOH and 5 wt% HCl were used to tailor the pH of the solution or slurry.

To begin with acrylamide (AM) as a monomer and methylenebisacrylamide (MBAM) as a crosslinking agent were introduced to the distilled water to form a premix. After the powders with 30 vol%, solid content were added to the premix, the slurries were ball-milled in a plastic jar for 6 h using ZrO$_2$ as the milling medium. Next, the pH of the slurries was adjusted to about 11.5 with NaOH and HCl solution. After the slurries were degassed for 10 min to remove the pores, the initiator and catalyst were introduced into the slurries to adjust the polymerization time to about 15 min. The prepared slurries were placed in the central position of the magnetic field, in which its direction was parallel to the direction of gravity and the magnetic field intensity was adjusted to 6 T. After the slurries were completely solidified, they were removed from the magnetic field. The prepared textured green bodies were dried at 70 °C and 100 °C, respectively, to inhibit nonuniform shrinkage. The samples were first sintered at 600 °C for 2 h at a 2 °C/min heating ratio to remove the polymer and then at 1800 °C for 2 h at a 5 °C/min heating ratio in an N$_2$ atmosphere of 0.1 Mpa.

The textured green bodies and sintered bodies were cut along the directions parallel and perpendicular to the magnetic field direction. The orientation and phase compositions of the prepared samples were analyzed by X-ray diffraction (XRD). The logging orientation factor, f, was used to evaluate the degree of texture of Si$_3$N$_4$ and ZrB$_2$ based on XRD. The bulk density and apparent porosity of the sintered samples were measured according to Archimedes theory in distilled water. The theoretical density was calculated based on the raw powder compositions, including Si$_3$N$_4$, ZrB$_2$, Y$_2$O$_3$ and SiO$_2$. Next, the relative density was obtained from the ratio between the bulk density and theoretical density. The bending strength of the samples was measured by the three-point bending test. The span was 30 mm and the cross-head speed was 0.5 mm/min. The above value was the average of five samples. The fracture morphologies of the sintered samples were scanned by SEM.

3. Results and discussion

XRD images of Si$_3$N$_4$-ZrB$_2$ green bodies prepared by gel-casting without and with a magnetic field of 6 T are shown in Figure 1. The top surface is perpendicular to the magnetic field direction and the side surface is parallel to the magnetic field direction. In Figure 1(a), the phase compositions consist of β-Si$_3$N$_4$ and ZrB$_2$. In Figure 1(b)-(e), however, the ZrB$_2$ phase also appears, except for the existence of β-Si$_3$N$_4$ and Y$_2$O$_3$. The amorphous SiO$_2$ phase cannot be detected in the XRD pattern containing the crystallized phase with high intensity. In Figure 1(a), (hk0) peak intensities such as the (200) and (210) peaks are higher on the top surface than on the side surface. A lower (101) peak on the top surface becomes highest on the side surface. The a- or b-axis of β-Si$_3$N$_4$ was oriented parallel to the magnetic field direction. These results were consistent with those of a previous study [6,7]. In Figure 1(b-e), except for the orientation of β-Si$_3$N$_4$, ZrB$_2$ grains also show an orientation behavior. For ZrB$_2$ grains, the peak intensities of the (001) crystal face perpendicular to the c-axis of ZrB$_2$ grains are highest on the top surface, while the intensities are lowest on the side surfaces such as the (001) and (002) crystal faces. On the contrary, the peak intensities of the (hk0) crystal face parallel to the c-axis of ZrB$_2$ grains are lowest on the top surface, while the
intensities are highest on the side surface. In conclusion, applying a magnetic field of 6 T makes the ZrB$_2$ grains form a higher orientation during gel-casting. The c-axis of ZrB$_2$ grains is oriented to align along the magnetic field direction. ZrB$_2$ grains have a hexagonal crystal structure and show anisotropic magnetic susceptibility. Because the magnetic susceptibility along the c-axis is higher than that along the a- or b-axis, the magnetic energy along the c-axis is smaller than that along the a- or b-axis when ZrB$_2$ grains are functioned by the magnetic field. That is, the c-axis of ZrB$_2$ grains is an easily magnetized axis, which is consistent with analyses by other researches for magnetization in 12 T [11,12].

Additionally, it can be seen from Figure 1(b-e) that with increasing the ZrB$_2$ content, all the peak intensities of β-Si$_3$N$_4$ on the top and side surfaces decrease gradually while the peak intensities of ZrB$_2$ increase gradually. These changes show only the difference in the contents of each phase, but do not illustrate the orientation changes in each phase in the samples with ZrB$_2$ content. Therefore, we need to calculate the Lotgering orientation factor of each phase, including β-Si$_3$N$_4$ and ZrB$_2$, to evaluate the respective degrees of texture. The degree of texture, $f_{(00l)}$, of β-Si$_3$N$_4$ is calculated according to equations from the literature [7]. For ZrB$_2$, owing to the formation of a c-axis orientation, we must calculate the degree of texture, $f_{(00l)}$, using the following equations [11,17].

$$f_{(00l)} = \frac{P - P_0}{1 - P_0}$$

(1)

$$P_{\beta_{Si_3N_4}} = \frac{\sum I_{(00l)}^{(00l)}}{\sum I_{(u,v)}}$$

(2)

where $\sum I_{(00l)}$ is the sum of all the (00l) peak intensities on the surface perpendicular to the magnetic field.
field direction, and \( \sum I_{(hkl)} \) is the sum of all the (hkl) peak intensities on the surface perpendicular to the magnetic field direction. \( P \) is calculated from the prepared samples and \( P_0 \) is calculated from the standard PDF cards. If \( I_{(001)} \) is equal to 1, ZrB\(_2\) grains form the orientation completely. If \( I_{(001)} \) is equal to 0, no ZrB\(_2\) grains form the orientation.

The Lotgering orientation factors of \( \beta\)-Si\(_3\)N\(_4\) and ZrB\(_2\) in the samples prepared in 6 T are shown in Figure 2(a and b), respectively. It is seen that with increasing the ZrB\(_2\) content in the samples, the Lotgering orientation factors both show a decreasing trend. These results show that during gel-casting in a magnetic field, the addition of ZrB\(_2\) grains exerts a negative effect on the degree of texture of each phase, and increasing the ZrB\(_2\) content make the degree of texture decrease more evidently.

In the textured \( \beta\)-Si\(_3\)N\(_4\)-ZrB\(_2\) green bodies, \( \beta\)-Si\(_3\)N\(_4\) grains form the a- or b-axis orientation and ZrB\(_2\) grains as the secondary phase form the c-axis orientation. Si\(_3\)N\(_4\) and ZrB\(_2\) grains show different anisotropic magnetic susceptibility. \( \beta\)-Si\(_3\)N\(_4\) crystal cells consist of non-metal Si and N, and ZrB\(_2\) crystal cell consists of transition metal Zr and B. It is reported that the anisotropic magnetic susceptibility is close to \( 1 \times 10^{-8} \) for Si\(_3\)N\(_4\) \([9]\). The measured \( \chi_c \) and \( \chi_{ab} \) are \( 0.094 \times 10^{-4} \) and \( -0.671 \times 10^{-4} \) for ZrB\(_2\), respectively \([12, 18]\). It is determined that the anisotropic magnetic susceptibility of ZrB\(_2\) is higher than that of \( \beta\)-Si\(_3\)N\(_4\). Under the same magnetic field conditions, ZrB\(_2\) grains can form an orientation more easily. Owing to the low dispersibility of ZrB\(_2\) these grains easily form an agglomeration in the medium, leading to difficulty in forming an orientation, as shown in Figure 3(a). Meanwhile, the presence of grain agglomeration easily causes an increase in the viscosity of the slurry. As shown in Figure 3(b), increase in the ZrB\(_2\) contents cause the viscosity of the slurry to increase gradually. In a system of grain orientation, greater viscosity of the slurry can hinder the rotation of the grains, leading to a decrease in the degree of texture. Additionally, in slurry consisting of \( \beta\)-Si\(_3\)N\(_4\) and ZrB\(_2\), owing to the co-existence of different magnetic susceptibilities and grain sizes or shapes, their magnetic torques are different when they are placed in the magnetic field. That is, during the rotation of grains in a magnetic field, orientation competition between grains occurs. The grains with higher orientation energy will rotate the equilibrium position first, while the adjacent grains with weaker orientation energy cannot reach an equilibrium position because they suffer from the hindrance and collision effects of previous grains. As a result, the degree of texture of the samples decreases.

**Figure 2.** Lotgering orientation factor of green bodies prepared with a strong magnetic field of 6 T: (a) \( \beta\)-Si\(_3\)N\(_4\) and (b) ZrB\(_2\).

**Figure 3.** (a) SEM images of ZrB\(_2\) powders and (b) viscosity of the 30 vol% slurry with different ZrB\(_2\) contents.
Figure 4 shows XRD patterns of the textured composites prepared in 6 T with subsequent sintering at 1800 °C for 2 h. In Figure 4(a), evident differences are seen between the peak intensities of the top and side surfaces. On the top surface, the (210) and (200) peak intensities are highest and the (002) peak almost disappear. But on the side surface, (101) peak intensity is highest and the (002) peak intensity is enlarged. It is observed that high a- or b-axis textured single-phase β-Si$_3$N$_4$ ceramics are prepared under the function of the magnetic field. It is seen from Figure 4(b-e) that in the samples with different ZrB$_2$ contents, the ZrB$_2$ phase disappears completely and new ZrN, ZrSi$_2$, and BN phases appear. The (101) and (200) peaks of ZrN overlap with the (101) and (111) peaks of β-Si$_3$N$_4$, respectively. Further, the main product is ZrN and the minor products are ZrSi$_2$ and BN. With increases in the ZrB$_2$ content, the peak intensities of the ZrN phase increase gradually in the sintered samples, illustrating an increasing formation of ZrN. It is found that no evident change exists by analyzing the peak intensities of ZrN in the top and side surfaces in the samples. That is, the formed ZrN grains have no orientation.

Owing to the peaks overlapping, the (101) and (111) peak intensities of β-Si$_3$N$_4$ are calculated based on the following equations:

$$I_{(101)} = \frac{I_{\text{total}}}{\theta - 33.5° - I_{(220)}} \times 2.20$$

$$I_{(111)} = \frac{I_{\text{total}}}{\theta - 39.5° - I_{(220)}} \times 1.87$$

The Lotgering orientation factors calculated for β-Si$_3$N$_4$ in the samples are shown in Figure 5. It is seen that when the ZrB$_2$ content is lower than 10 wt%, the degree of texture maintains a constant value. When the content is more than 10 wt%, the degree of texture decreases slightly. Compared with that in green bodies, the sintering process still promotes increases in the degree of texture of β-Si$_3$N$_4$, although new phases form during sintering.

Figure 6 shows SEM BSE images of textured porous composites. In Figure 6(a), it is seen that only gray Si$_3$
N₂ grains exist. In Figure 6(b–e), bright white grains appear in addition to gray Si₃N₄ grains. Based on previous analyses, the white grains are the ZrN phase. Owing to the formation of a smaller amount of ZrSi₂ and the BN phase observed by XRD data, these grains are not observed in the SEM BSE images. With increases in the ZrB₂ content, the number of white phase (ZrB₂) grains gradually increases. This result is consistent with those for the XRD pattern. Because no evidently special shaped β-Si₃N₄ and ZrB₂ grains appear, no difference is observed in the grain orientation of the microstructure.

Based on previous research [19], it is determined that a reaction between Si₃N₄ and ZrB₂ occurs in the N₂ atmosphere. When the temperature is more than 1500 °C, ZrB₂ will react with N₂ to form the ZrN and BN phases in accordance with the following equation:

\[
2\text{ZrB}_2(s) + 3\text{N}_2(g) \rightarrow 2\text{ZrN}(s) + 4\text{BN}(s)
\]  

When not enough N₂ exists, only part of the ZrB₂ participates in the reaction, leading to the co-existence of ZrB₂, ZrN and BN. When enough N₂ exists, ZrB₂ will be completely transformed into ZrN and BN. In our research, flowing N₂ is used as a protection gas, making the reaction complete. Thus, the ZrB₂ phase is not detected in

![Figure 5. Lotgering orientation factor of the β-Si₃N₄ composition in sintered samples.](image)

![Figure 6. SEM BSE images of sintered sample: (a) SN; (b) SNZ5; (c) SNZ10; (d) SNZ15 and (e) SNZ20.](image)
XRD and SEM. As is known, at high temperatures, decomposition of Si$_3$N$_4$ will occur to form Si and N$_2$ in accordance with the following equation:

\[
\text{Si}_3\text{N}_4(s) \rightarrow 3\text{Si}(s) + 2\text{N}_2(g)
\]  

(6)

Guo et al. [18] calculated the Gibbs free energy of Equation (6). They found that when the N$_2$ pressure is 1 atm, Si$_3$N$_4$ will decompose at temperatures above 1828 °C. And when the N$_2$ pressure is 100 Pa, Si$_3$N$_4$ will decompose at temperatures above 1395 °C. That is, decreasing the N$_2$ pressure decreases the decomposition temperature of Si$_3$N$_4$. During sintering, the reaction of ZrB$_2$ and N$_2$ decreases the partial pressure of N$_2$ around the Si$_3$N$_4$-ZrB$_2$. To reach system equilibrium, N$_2$ will be offered by decomposition of Si$_3$N$_4$. Therefore, the existence of ZrB$_2$ promotes the decomposition of Si$_3$N$_4$. The N$_2$ in Equation (5) is derived from N$_2$ formed by the decomposition of Si$_3$N$_4$ and added flowing N$_2$.

It is observed that in textured green bodies, Si$_3$N$_4$ and ZrB$_2$ grains both form a high orientation. During sintering, however, the ZrN products obtained by the reaction of equation of (5) form no orientation. In these reactions, reestablishment of the crystal structure occurs. The Zr-B chemical bonds in the ZrB$_2$ crystal cells break, for example, new Zr-N, Zr-Si and B-N chemical bonds form. As this case, the grain orientation of the products has no heritability. Therefore, the ZrN grains show no orientation. These results show that this reestablishment reaction process is not beneficial for improvement of the grain orientation in textured composites. In our previous research, however, we found that the formed (Ti,Zr)B$_2$ phase shows high grain orientation characteristics with the solid solution reaction of TiB$_2$ and ZrB$_2$. And the orientation of the (Ti,Zr)B$_2$ grains is determined by highly oriented TiB$_2$ grains as template grains [16]. Therefore, it is concluded that different reaction mechanisms play different roles in the orientation development of the formed phase.

Our previous research confirmed that the grain orientation has no effect on the densification process [7,10]. Thus, only textured samples are used here to analyze their densification. Figure 7 shows the relative density and apparent porosity of textured samples. It can be seen that with increases in the ZrB$_2$ content, the relative density of sintered samples gradually decreases. When the ZrB$_2$ content is 10 wt%, the relative density reaches the minimum. On a contrary, with further increases in the ZrB$_2$ content, the relative density increases. However, the apparent porosity of sintered samples shows the opposite change trend with respect to ZrB$_2$ content. When the 10 wt% ZrB$_2$ is added, the apparent porosity of sintered samples has a maximum value of 47.15%. The pore size distribution of sintered samples with 10 wt% ZrB$_2$ is shown in Figure 8. The mean pore diameter is about 1.48 μm. The pore diameter is in the range from 0.2 to 3.8 μm. These changes are mainly caused jointly by reactions described by Equations (5 and 6).

Figure 9 shows the bending strength of sintered samples along different directions at room temperature. It is found that in the SN (without ZrB$_2$) sample, the bending strength in the direction where the load is parallel to the magnetic field direction is slightly greater than that in the direction where the load is perpendicular to the magnetic field. When the ZrB$_2$ (ZrN) content is increased in the samples, however, there is no evident difference between the bending strengths in the two directions. When the ZrB$_2$ content is 10 wt%, the bending strength of the sample is lowest. On the contrary, when the ZrB$_2$ content is less or more than 10 wt%, the bending strength increases. As the prepared textured composites have high porosity and the grain shape shows no anisotropy in the

![Figure 7. Relative density and apparent porosity of sintered samples.](image-url)
microstructure of the textured samples, the bending strength of the samples depends mainly on densification rather than the degree of texture. Lower density causes lower bending strength in samples with 10 wt % ZrB₂ content.

4. Conclusions

Textured porous Si₃N₄-ZrB₂ composites were prepared by gel-casting assisted by magnetic field alignment technology and sintering without a magnetic field. In textured green bodies, except for β-Si₃N₄ grains which formed the a- or b-axis orientation, ZrB₂ grains formed the c-axis orientation. The increasing ZrB₂ content caused the degree of texture of the Si₃N₄ and ZrB₂ phases to decrease gradually, mainly due to the increase in rotation resistance. After sintering at 1800 °C, the ZrB₂ phase reacted with N₂ to form the ZrN and BN phases. Owing to reestablishment of the crystal structure during the reaction process, the grain orientation showed no heritability, and ZrN products did not form an orientation. The ZrB₂ content played a key role in the densification process of sintered samples by the corresponding chemical reaction, accompanied by changes in bending strength at room temperature. When the ZrB₂ content was 10 wt%, the densification and bending strength reached extreme values, respectively. Further research on the heritability of grain orientation will be conducted in our future works.

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