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Effect of sintering additive on crystallographic orientation in AlN prepared by slip casting in a strong magnetic field

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Abstract

The preparation of oriented AlN bulk ceramics with and without additives was achieved by slip casting in a high magnetic field. The *a* and *b* axes of the AlN were aligned parallel to the direction of the magnetic field. The degree of crystallographic orientation was controlled by the viscosity of the slurry and the grain growth during sintering attributed to the sintering additives. The mechanical properties of the textured AlN depended on the direction of the crystallographic orientation.

Keywords: Slip casting; Sintering; Grain growth; Suspensions; Mechanical properties

1. Introduction

The development of the crystallographic orientation is an interesting topic in ceramics and one of the effective ways to improve their properties. Both the microstructural and crystallographic orientations of monolithic ceramics and composites can be controlled by hot-working[1–3] and templated grain growth.[4–6]

The crystallographic orientation even in diamagnetic ceramics has been successfully achieved by utilizing a strong magnetic field, and this processing can be applied to a wide variety of feeble magnetic ceramics such as Al_2O_3, TiO_2, ZnO and so on.[7–14] An asymmetric crystal structure must give anisotropic magnetic susceptibilities. When the particles with an anisotropic magnetic susceptibility are placed in a magnetic field, a magnetic torque is generated from the interaction between this anisotropy and a magnetic field. The reduction of magnetic energy upon rotation is defined by $\Delta E = -\Delta\chi V \boldsymbol{B}^2/2\mu_0$, where $\Delta\chi$ is the difference between the susceptibility along each axis, V is the volume of each particle, and μ_0 is the permeability in a vacuum. Particles will rotate to an angle that minimizes the system energy when placed in a magnetic field, $\boldsymbol{B}$. A crystal of AlN with a hexagonal structure exhibits an anisotropic susceptibility, and the texture of the AlN microstructure could be controlled by a strong magnetic field.

However, it is generally difficult to effectively apply a magnetic field in order to rotate fine diamagnetic particles, since fine particles tend to spontaneously agglomerate due to their strong attractive interactions (van der Waals' forces). Particle dispersion is important in this magnet alignment process. Colloidal processing is an effective technique for controlling the stability of particles in a slurry. It makes use of repulsive surface forces in order to avoid agglomeration.[15]

Aluminum nitride (AlN) is an attractive material as the substrate for electronic packaging because of its excellent properties, such as its high thermal conductivity and a coefficient of thermal expansion that is well matched to that of silicon. Tailoring its microstructure is very important in order to improve these properties, because a strong relationship exists between the properties of a material and its microstructure. Although, there have only been a few studies that have examined the development of texture in bulk AlN ceramics,[16] we have already reported that the textured AlN can be produced by colloidal processing in a strong magnetic field.[17] However, AlN is difficult to densify by sintering because of a covalent bonded material and the additives are necessary to densify AlN during sintering. Therefore,

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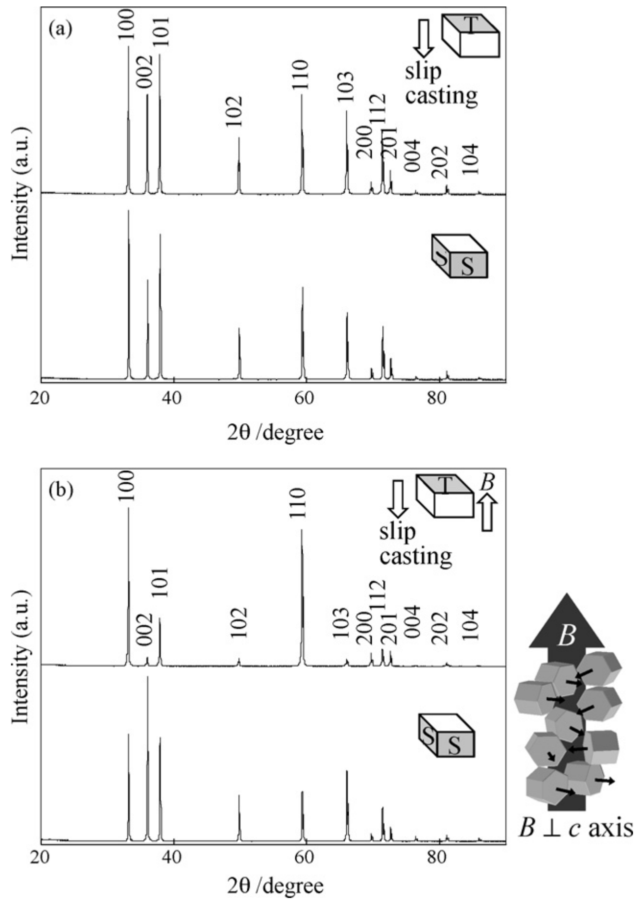

Fig. 1. X-ray diffraction patterns of T-plane perpendicular to the magnetic field and S-plane parallel to the magnetic field for the additive-free AlN sintered at 1900 °C for 2 h in N_2: (a) prepared by slip casting without a magnetic field and (b) are prepared by slip casting in a 10 T magnetic field.

the investigation of the role of the sintering additives for the texture is necessity.

In this report, we demonstrate the effect of Y_2O_3 as the sintering additives on the crystallographic orientation in AlN prepared by slip casting in a strong magnetic field and their properties.

2. Experimental procedure

A commercially available AlN powder (Grade H, Tokuyama Co., Ltd., Japan) and Y_2O_3 powder (Nanotech, C. I. Kasei Co., Ltd., Japan) with average particle sizes of 0.7 μm and 30 nm, respectively, were used as the starting materials.

Ethyl alcohol slurries were prepared that contained 40 vol% solids; the solids consisted of the additive-free AlN and the AlN containing 0.5 mass% and 5 mass% Y_2O_3 as the sintering additives. Re-dispersion is an indispensable technique for the proper dispersal of fine particles in slurries that might otherwise undergo spontaneous agglomeration.[18,19] The slurries were ultrasonicated for 10 min and stirred for more than 8 h. The slurries were then consolidated by slip casting after outgassing in a vacuum. A strong magnetic field of 10 T was applied to the slurry during slip casting at room temperature. The direction of the magnetic field was parallel to the gravitational sedimentation of the casting direction. For comparison, other samples were prepared by slip casting without the application of a magnetic field. The green compacts were further densified without disturbing the particle orientation by cold isostatic pressing (CIP) at 392 MPa, and were then isothermally sintered at the desired temperatures for 2 h in a nitrogen atmosphere.

The degree of crystallographic orientation was estimated by X-ray diffraction and electron backscattering diffraction (EBSD). The degree of crystalline orientation was estimated using Eq. (1) in conjunction with the intensities of the X-ray diffraction measurements from the surface perpendicular to the magnetic field (T in Fig. 1(b)). In general, the Lotgering factor evaluates the degree of orientation of the $(0\,0\,l)$ planes.[20] In contrast, Eq. (1) indicates to what extent the $(0\,0\,l)$ planes are perpendicularly aligned.[5]

$$F = \frac{(1-p)-(1-p_0)}{1-(1-p_0)} = \frac{p_0 - p}{p_0} \qquad (1)$$

where p or $p_0 = \sum I_{(0\,0\,l)} / \sum I_{(h\,k\,l)}$. The values of p were calculated from the intensities of the surface perpendicular to the direction of the magnetic field or of the casting direction with or without a magnetic field, respectively (T-surface in Fig. 1(a) and (b)). The value of p_0 was calculated from the ICDD card as randomly oriented specimens. Distributions of the tilting angle, θ, made by the c axis and the direction of the magnetic field were calculated from the EBSD analysis and the distribution profiles were fitted by the March–Dollase function[21] modified for Eq.

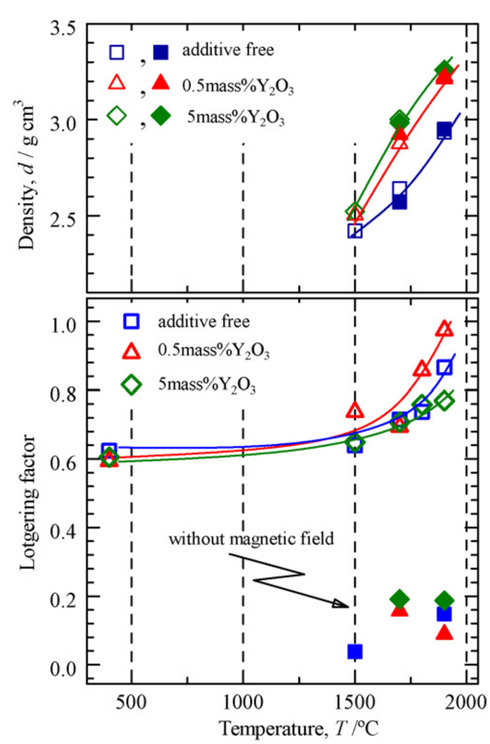

Fig. 2. Effect of the sintering temperature on the degree of crystalline texture and density in the AlN prepared by slip casting in a 10 T magnetic field. The open plots were prepared using a strong magnetic field and the closed plots were prepared without a magnetic field.

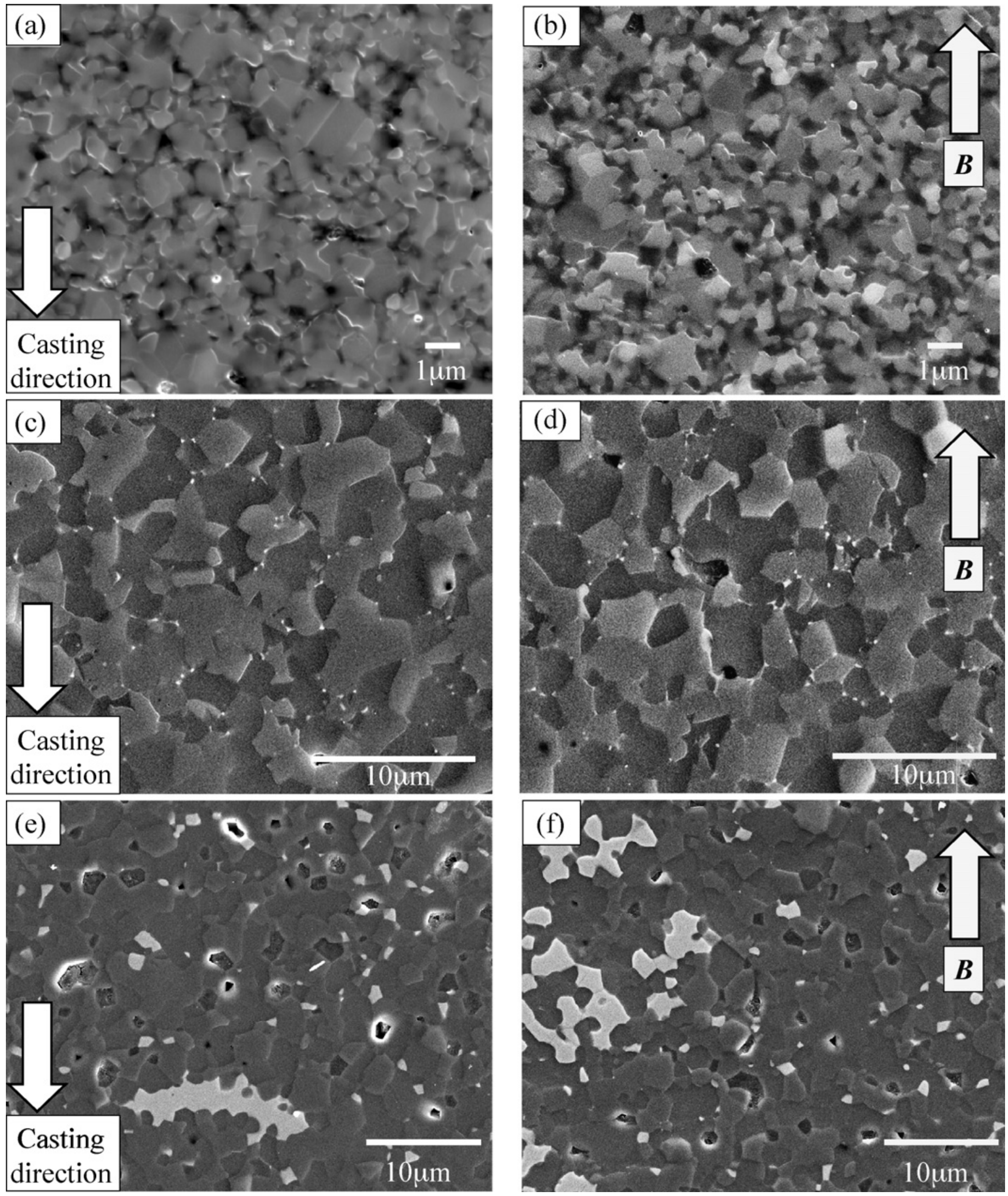

Fig. 3. Microstructure of the polished surface of AlN sintered at 1900 °C for 2 h in N_2: (a) the additive-free AlN without a magnetic field, (b) the additive-free AlN with a magnetic field, (c) the 0.5 mass% Y_2O_3-AlN without a magnetic field, (d) the 0.5 mass% Y_2O_3-AlN with a magnetic field, (e) the 5 mass% Y_2O_3-AlN without a magnetic field, and (f) the 5 mass% Y_2O_3-AlN with a magnetic field.

(2),

$$f_{\mathrm{MD}}(r,\theta) = \left(r^2\ \cos^2(90-\theta) + \frac{\sin^2(90-\theta)}{r}\right)^{-(3/2)} \qquad (2)$$

where r is the orientation parameter.

The flexural strength of the samples was determined using the 4-point bending test with upper and lower span lengths of 10 and 30 mm, respectively, and a crosshead speed of 0.5 mm/min. Their fracture toughness was evaluated using the single edge precracked beam (SEPB) method. The precracks were introduced into the SEPB samples using the bridge-indentation method.[22] The thermal conductivity of the AlN ceramics was determined by the flash diffusivity method.

3. Results and discussion

Fig. 1(a) shows XRD profiles of the additive-free specimen that was compacted by slip casting external to a magnetic field, followed by sintering at 1900 °C for 2 h in a N_2 atmosphere. Both profiles were taken from the top and side surfaces, perpendicular and parallel to the casting direction, respectively, and showed good agreement to each other. This result confirms that an aluminum nitride sample without additives external to a magnetic field has a randomly oriented polycrystalline structure.

Fig. 1(b) illustrates the XRD profiles of the additive-free specimen that was compacted by slip casting in a 10 T magnetic field, followed by sintering at 1900 °C for 2 h in a N_2 atmosphere. On the surface perpendicular to the magnetic field (T in

Fig. 1(b)), the intensities of the 1 0 0 and 1 1 0 reflections were very high, whereas on the surface parallel to the magnetic field (S in Fig. 1(b)), the intensity of the 0 0 2 reflection was high. This result shows that the development of the AlN orientation was controlled by the magnetic field and that the *a*, *b* axes were aligned parallel to the magnetic field, that is to say, the *c* axis was perpendicular to the magnetic field.

Fig. 2 illustrates the degree of crystalline orientation calculated from Eq. (1), together with the densities as a function of the sintering temperature for specimens prepared by slip casting in a 10 T magnetic field and specimens prepared without applying a magnetic field. For those specimens not exposed to the magnetic field, the degree of crystalline texture is approximately 0.1, irrespective of the sintering temperature. In the specimens exposed to a strong magnetic field, the orientation factor F was much larger than that of the randomly oriented material. At 400 °C the degree of the orientation of the additive-free AlN was larger than that of the other samples, but the difference was very small. This is due to the reason why we have some problems in the estimation of the orientation in the green bodies. First, the particles may rotate in polishing the surface in order to make it smooth and flat for XRD measurements. When the intensities of the 0 0 1 reflections decrease, it is easy to overestimate the orientation using the modified Lotgering equation and the differences in the orientations are reduced. However, the tendency of the orientation was obtained. The values for the textured specimens slightly increase with the increasing temperature, likewise, the change in the density. The degree of crystalline orientation of the specimen containing 0.5 mass% Y_2O_3 was highest at the high temperature. When comparing the additive-free AlN and the 5 mass% Y_2O_3-AlN, the degree of orientation of the additive-free AlN was higher than that of the 5 mass% Y_2O_3-AlN with the increasing temperature.

Fig. 3 shows the microstructures of the samples sintered at 2173 K for 2 h in a N_2 atmosphere. The fine microstructures were observed in the additive-free AlN with and without an applied magnetic field, while grain growth occurred in the AlN with Y_2O_3 addition. White particles (Al–Y–O phases as will be shown later) with size of a few hundred nm were finely and homogeneously dispersed in the 0.5 mass% Y_2O_3-AlN. However, the large white particles were inhomogeneously agglomerated in the 5 mass% Y_2O_3-AlN. The average grain sizes of the additive-free, 0.5 mass% Y_2O_3 and 5 mass% Y_2O_3-AlN estimated from the cross-sectional microstructure were 0.71, 2.5 and 3.0 μm, respectively, in the random samples, and those of the additive-free, 0.5 mass% Y_2O_3 and 5 mass% Y_2O_3-AlN were 0.79, 2.7, 2.9 μm, respectively, in the textured samples. Regardless of the application of a magnetic field, the average grain sizes are almost the same in the samples containing each Y_2O_3 content.

EDS analyses of the surface coated with carbon in the 5 mass% Y_2O_3-AlN sintered at 1900 °C are reported in Fig. 4. In the gray matrices (points 1 and 2), only Al and N were detected, whereas Al, N, Y and O could be detected in the white particles (points 3 and 4). This provided evidence that the white particles were Al–Y–O phases in the microstructures of AlN with Y_2O_3 addition. XRD analysis revealed that the Al–Y–O phases were $Al_5Y_3O_{12}$ and $Al_2Y_4O_9$ in the 0.5 mass% Y_2O_3-AlN and the 5 mass% Y_2O_3-AlN, respectively, regardless of applying a magnetic field. The intensities of the X-ray diffraction from these Al–Y–O phases were too weak to investigate the crystallographic orientation of the second phase.

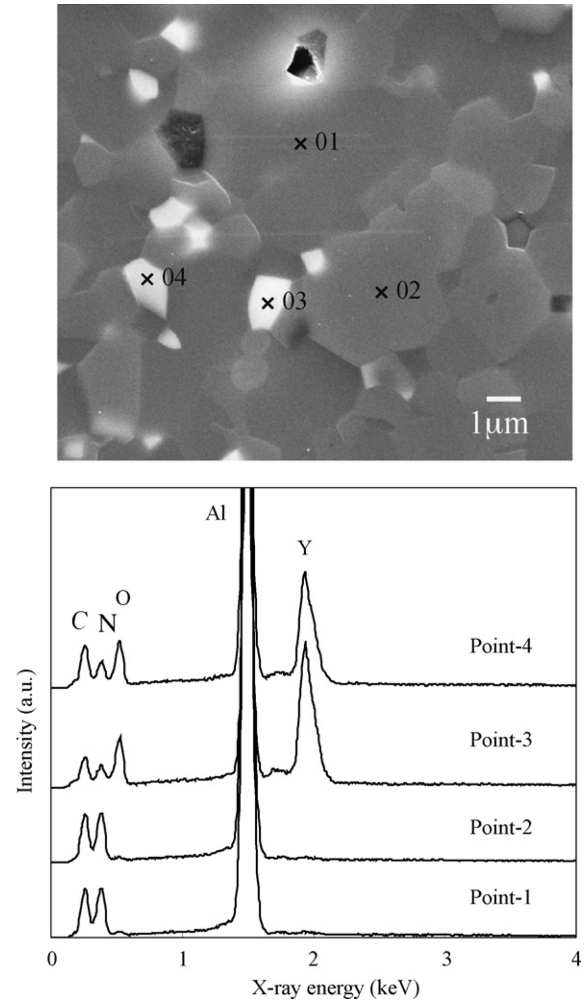

Fig. 4. EDS results for the polished surface of the 5 mass% Y_2O_3-AlN sintered at 2173 K for 2 h in N_2.

In order to estimate the degree of orientation, distributions of the tilting angle, θ, are shown in Fig. 5. These samples were sintered at 1900 °C for 2 h in a N_2 atmosphere. The orientation parameters, r, calculated from the March–Dollase function were 0.525, 0.465 and 0.610 for the additive-free, 0.5 mass% Y_2O_3 and 5 mass% Y_2O_3-AlN, respectively. The orientation parameter of $r = 0.525$ in the additive-free AlN indicates that 61.8% of grains are aligned with θ of more than 70°. The $r = 0.465$ and 0.610 in the Y_2O_3-doped AlN indicate that 69.4% and 51.8% of grains are aligned with θ of more than 70°, respectively. The trend in the degree of the crystallographic orientation agreed with that of the Lotgering factor is shown in Fig. 2.

We have reported that the required conditions were the low viscosity of the slurry for easy rotation of the particles and the grain growth during sintering.[23,24] Hence, we will discuss the cause of this trend in the texture from the point of view of the slurry conditions and the grain growth. The rheological behav-

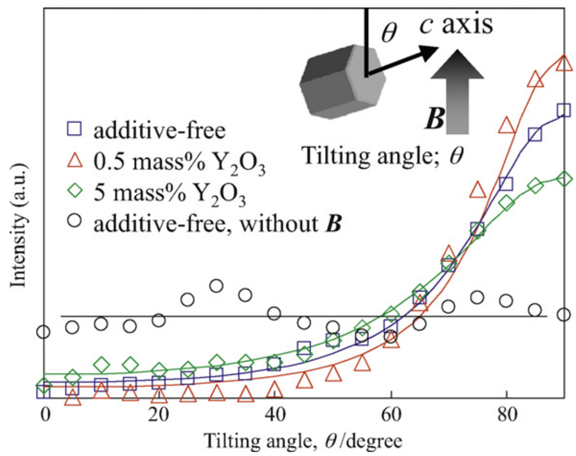

Fig. 5. Distribution of the tilting angle between the *c* axis of AlN and direction of the magnetic field. The ratio of grains with each tilting angle between the *c* axis and the sample surface perpendicular to the magnetic field was calculated from the EBSD results.

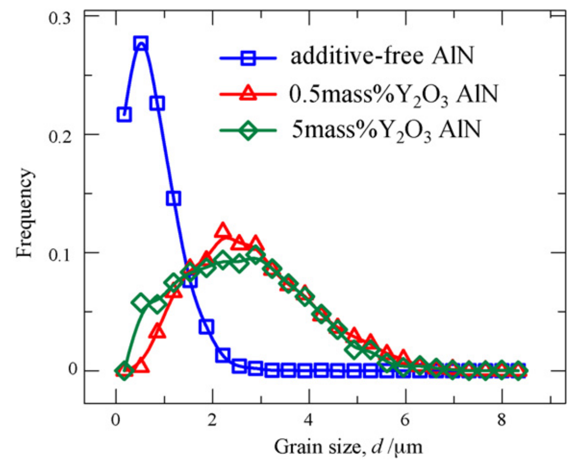

Fig. 7. Effect of the amount of Y_2O_3 on the grain size distribution of the specimens sintered at 2173 K for 2 h in N_2.

iors of the slurries are shown in Fig. 6. The shear stress linearly increased as the shear rate increased in all slurries. The viscosity of these slurries increased with the increasing Y_2O_3 contents. Consequently, it was more difficult to rotate the AlN particles in the slurries with the increasing Y_2O_3 contents. Fig. 7 shows the distribution of the grain size of the textured AlN after sintering at 1900 °C for 2 h in a N_2 atmosphere. The distributions of the grain size in both AlNs containing Y_2O_3 as an additive were similar to each other. However, the size distribution of the additive-free AlN was narrow and the number of fine grains of less than 1 μm increased when compared with the AlN containing Y_2O_3. No obvious grain growth in the additive-free AlN was observed.

In 0.5 mass% Y_2O_3-AlN, the particles easily rotated in the slurry because of the low viscosity. According to previous reports regarding other systems,[7–9] the development of texture has a close relationship to the microstructure; i.e., grain growth enhances the degree of orientation. It is also believed that the development of a crystallographic texture is promoted by grain growth during heating in the AlN ceramics. Consequently, the degree of orientation was the highest after sintering in the 0.5 mass% Y_2O_3-AlN. It was also easy to rotate the particles in the additive-free AlN; however, the change in the degree of orientation was limited during sintering, because of grain growth suppression. In the 5 mass% Y_2O_3-AlN, it was difficult to rotate the particles even in a strong magnetic field because of the higher viscosity than the others, hence, in this case, the development of a texture hardly increases even if grain growth occurred. When we use the nano size powders, their strong attractive interactions due to van der Waals' forces suppress the dispersion. Therefore, we try to improve the dispersion in a slurry using Y_2O_3 powder with an average particle size of 1.0 μm. The Lotgering factor in the green bodies is almost same as that in the additive-free AlN and the factor at 1800 °C was 0.95. This is because good dispersion in a slurry was obtained and the grain growth enhanced the orientation.

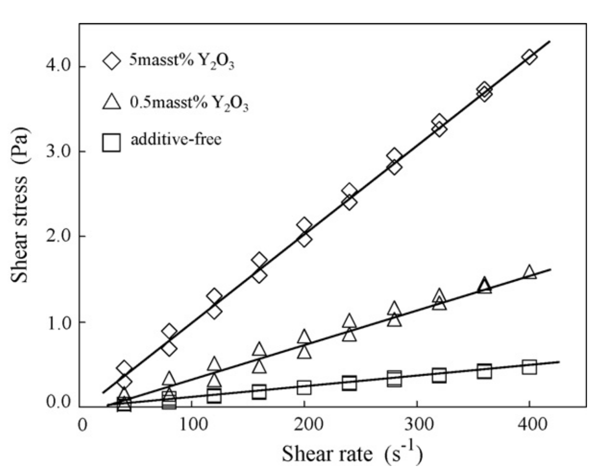

Fig. 6. Effect of the amount of Y_2O_3 on the rheological behaviors of the slurries.

The flexural strength and fracture toughness for the textured AlN containing Y_2O_3 sintered at 1900 °C for 2 h were estimated for the crack-growth directions parallel and perpendicular to the applied magnetic field, as shown in Figs. 8 and 9. The strength of 0.5 mass% Y_2O_3-AlN was higher than that of the 5 mass% Y_2O_3-AlN, because the dispersion of the oxide grains as the second phase was fine and homogeneous in the 0.5 mass% Y_2O_3-AlN. In contrast, the oxide phase was inhomogeneously agglomerated in the 5 mass% Y_2O_3-AlN as shown in Fig. 3. Furthermore, the flexural strength depends on the direction of the crystallographic orientation. The strength for the crack-growth direction parallel to the magnetic field was higher than that perpendicular to the magnetic field, regardless of the Y_2O_3 contents. The fracture toughness also depends on the crack-growth direction. The fracture toughness for the crack-growth direction parallel to the magnetic field was higher than that perpendicular to the magnetic field.

Fig. 10 illustrates the fracture surfaces of the textured AlN containing Y_2O_3 for the crack propagation perpendicular to the magnetic field. The fracture mode of the 0.5 mass% Y_2O_3-AlN, whose degree of orientation was higher than that of the others, was a mixture of intergranular and intragranular fractures. On the other hand, the fracture mode of the 5 mass% Y_2O_3-AlN was almost an intergranular fracture. It seemed that the crack easily

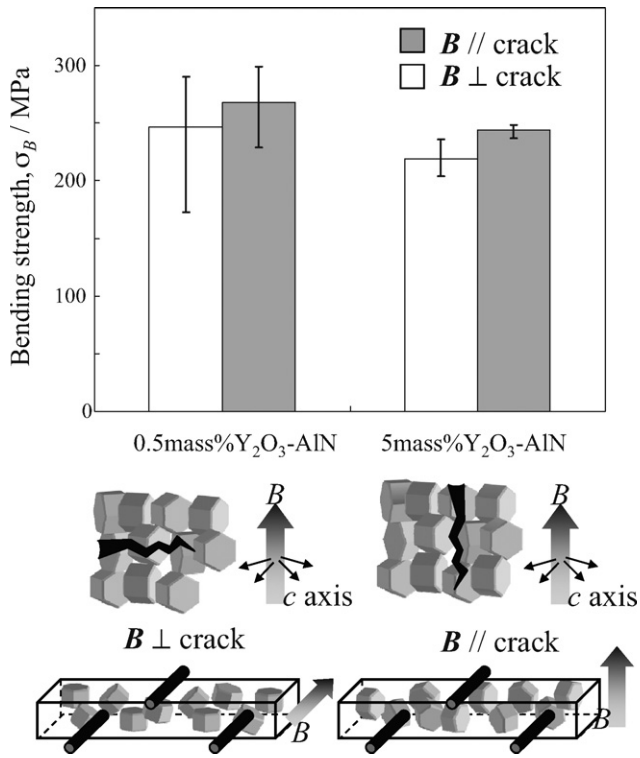

Fig. 8. Effect of the crystallographic orientation on the flexural strength of the textured AlN containing Y_2O_3 sintered at 1900 °C for 2 h in N_2.

propagated on the {1 1 0} cleavage plane aligned by a strong magnetic field in the 0.5 mass% Y_2O_3-AlN. In contrast, it was difficult for the crack to propagate on the cleavage plane in the 5 mass% Y_2O_3-AlN because of the lower orientation.

The thermal conductivities of the randomly oriented and the textured AlN were measured in the directions parallel and perpendicular to the magnetic field. In the 0.5 mass% Y_2O_3-AlN, the thermal conductivity of 86.3 and 83.7 W/(K m) in the directions parallel and perpendicular to the applying magnetic field, respectively, were similar to 86.3 W/(K m) for the randomly oriented sample.

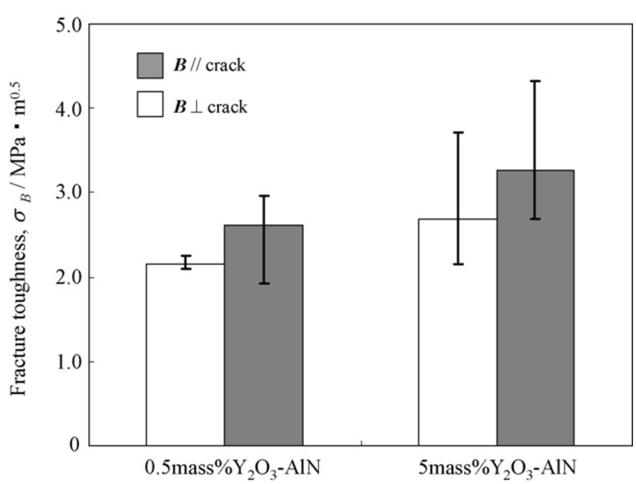

Fig. 9. Effect of the crystallographic orientation on fracture toughness of the textured AlN containing Y_2O_3 sintered at 1900 °C for 2 h in N_2.

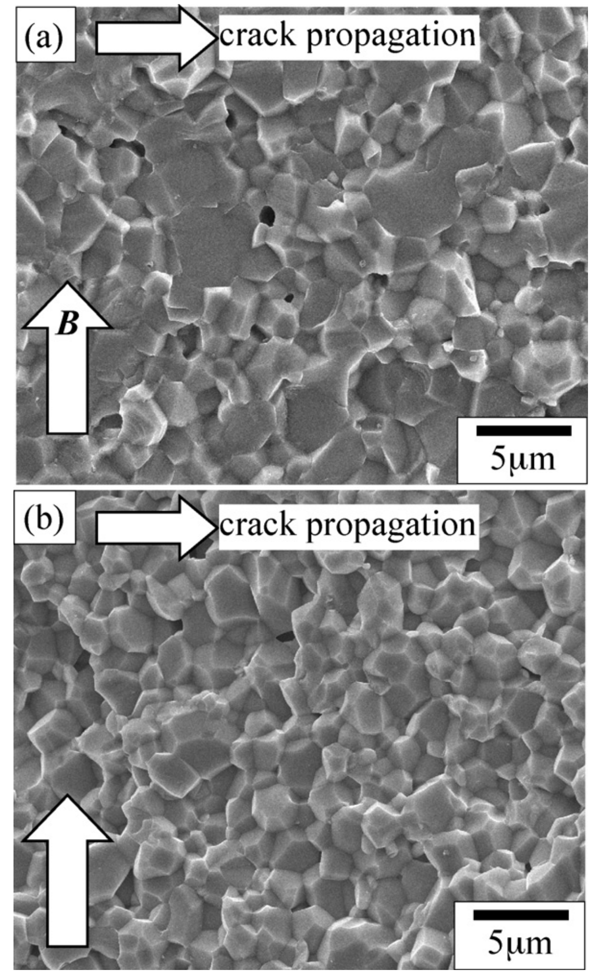

Fig. 10. Fracture surface of the textured AlN containing Y_2O_3 sintered at 1900 °C for 2 h in N_2: (a) 0.5 mass% Y_2O_3-AlN and (b) 5 mass% Y_2O_3-AlN.

4. Summary

Control of the crystallographic orientation in AlN bulk ceramics was achieved using a magnetic field and a colloidal filtration process. The *a* and *b* axes and the *c* axis were parallel and perpendicular to the magnetic field, respectively, i.e., the *c* axis was randomly aligned on the plane perpendicular to the magnetic field. We demonstrated the effect of the sintering additives on the crystallographic orientation of AlN with a view toward both the stage of slurry and sintering. High viscosity due to the sintering additives prevents particles from rotating in slurries, while the grain growth due to the sintering additives enhances the degree of the crystallographic orientation. The flexural strength for the direction parallel to the magnetic field was higher than that for the direction perpendicular to the magnetic field, following the same trend as observed for the fracture toughness described above. This indicates that the mechanical properties of the material can be controlled by tailoring the crystallographic orientation, and that the use of a magnetic field is very effective for developing a textured microstructure. The anisotropic mechanical properties of the textured AlN prepared using a magnetic field suggest that there is a good possibility that these properties can be simultaneously improved.

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