Advanced control of crystallographic orientation in ceramics by strong magnetic field

Tohru S. SUZUKI

1Ceramics Processing Group, Research Center for Functional Materials, National Institute for Materials Science, 1–2–1 Sengen, Tsukuba, Ibaraki 305–0047, Japan

1. Introduction

The tailoring of microstructures is essential to enhance the mechanical and functional properties, as well as reliability, in ceramics. Tailoring microstructure is performed with regard to various parameters, such as fine grain, particle dispersion, and grain boundary control. Solid materials are unlikely to have isotropic properties, and anisotropy often can be used effectively to enhance their mechanical and functional properties. Anisotropy can enhance properties such as texture control in steel and other metals, single-directional strengthening by dispersion of fiber or whiskers, and anisotropic polarization in piezoelectric materials. Therefore, crystallographic orientation is a significantly effective method for improving their properties and reliability. Techniques that control metal texture through work or recrystallization have been developed, but it is difficult to apply these to brittle materials such as ceramics.

Anisotropy can be controlled in bulk ceramics by external field with driving forces such as gravity, magnetic field, stress, and shear stress. The templated grain growth (TGG) method, in which platelet particles or rod-like particles as anisometric particles are aligned by tape casting, and aligned grains grow as seeds for controlling orientation in the whole bulk sample, can be used as one of the methods of crystallographic orientation by shear stress.1)

The TGG and reactive templated grain growth (RTGG) method2) can be applied to control the texture in anisotropic ceramics in which it is possible to prepare anisometric particles, such as bismuth layer structured compound ceramics, piezoelectric ceramics with perovskite-type structures, alumina, and silicon carbide.3)–7) It has been reported that thermal conductivity can be improved by controlling the anisotropy in textured silicon nitride prepared by unidirectional extrusion.8) Hot forging, which deforms bulk samples at high temperatures by uniaxial compression, is also one of the methods for imparting anisotropy by an external field.9) It was shown that applying hot forging to silicon carbide and alumina resulted in the improvement of properties such as compatibility of both strength and toughness.10)–12)

2. Orientation control under a magnetic field

It is well known that ferromagnetic materials such as ferrite are affectable by external magnetic fields to the extent that their orientation can be controlled. In contrast, diamagnetic and paramagnetic materials such as alumina, aluminum nitride, and titania have notably low magnetic susceptibility. Therefore, it is difficult to control the orientation of these materials with a magnetic field. However, due to the remarkable development of superconducting technology and cooling systems, it has recently become easier to use a strong magnetic field in a wide variety of contexts, such as physical and chemical analysis in a strong magnetic field, protein synthesis by magnetic levitation, and magnetic separation. Many researchers have

Corresponding author: T. S. Suzuki; E-mail: SUZUKI.Tohru@nims.go.jp
Furthermore, the range of application of magnetic fields has been expanding in material processing fields other than solid-state physics.15),16) Material with asymmetric crystal structure, such as hexagonal, tetragonal, and layered structures, have anisotropic magnetic susceptibilities depending on the direction of the crystal axis. The magnetic field rotates a single crystal particle with anisotropic susceptibility to minimize the energy of the system when placed in a magnetic field, $B$, according to Eq. (1).17)

$$ T = -\frac{\Delta \chi V B^2}{2\mu_0} \sin 2\theta \quad (1) $$

where $\Delta \chi = \chi_a - \chi_c$ is the anisotropy of the magnetic susceptibility, $V$ is the volume of each particle, $\theta$ is the angle between the magnetic field and the easy magnetization axis, and $\mu_0$ is the permeability in a vacuum. When the magnetic susceptibility of the $c$-axis ($\chi_c$) is larger than that of the other axes ($\chi_a, b$), the $c$-axis is aligned parallel to the magnetic field, and in the opposite case, the $c$-axis is aligned perpendicular to the magnetic field. However, the direction is randomly oriented on the plane perpendicular to the magnetic field. This is the driving force for the magnetic alignment, as shown in Fig. 1. However, the magnetic susceptibility is quite small in paramagnetic and diamagnetic materials, and the magnetic torque becomes notably small even if a strong magnetic field is used. Therefore, it is difficult to effectively apply a magnetic field to rotate fine particles because fine particles tend to spontaneously agglomerate, and rotation is prevented due to strong attractive interactions (van der Waals’ forces). Therefore, it is important to control the dispersion of particles in a slurry and rotate them by using colloidal processing to control the interaction between particles. The general procedure to control the crystallographic orientation by a magnetic field is as follows.

Synthesized or commercial powder can be used as the starting material. Ceramic powder is dispersed in solvent by mutual electrostatic repulsion due to adjusting the pH or dispersant. The slurries are mixed with a magnetic stirrer and ultra-sonicated to break the agglomerates.18),19) The slurries are then consolidated by slip casting, electro-phoretic deposition (EPD), tape casting, etc.20) A strong magnetic field is applied to these consolidation processes at room temperature for rotating particles and controlling the crystallographic orientation. The static and rotating magnetic fields can be applied, and the various directions can be selected, as shown in Fig. 2. The orientation direction of the easy magnetization axis to the shape of parts can be controlled by the direction of the magnetic field. Furthermore, the hard magnetization axis can be aligned by the rotating magnetic field, which means that consolidation is performed on a turntable placed at the center of a horizontal magnetic field, as described in detail in Section 3.2. The green compacts are sintered for densification outside of the magnet by various sintering methods such as pressureless sintering, spark plasma sintering, hot pressing, and milliwave sintering.

3. Orientation control in various ceramics using a strong magnetic field

3.1 Easy magnetization axis orientation based on static magnetic field

Silicon carbide is a ceramic widely used as a structural and functional material in varying applications due to its notable qualities. Many studies have reported the control of its microstructure.21-23) Control of the crystallographic orientation in polycrystalline SiC was achieved by slip casting in a static magnetic field of 12 T.24) Figure 3 shows the pole figures on the VT plane perpendicular to the magnetic field from the electron back scattering diffraction (EBSD) analysis. The intensity reached maximum at the center, and the $c$-axis of SiC was aligned parallel to the magnetic field. Figure 4 illustrates that the distribution of the angle made at the meeting of the $c$-axis and the vertical direction parallel to the magnetic field was obtained from the pole figure. Approximately 70 and 88% of
the grains were aligned with a tilt angle of less than 10 and 20°, respectively. With regard to the dense SiC prepared using the sintering additives and hot-pressing at 1900 °C for 2 h, the 3-point bending strengths were 799 and 907 MPa for the loading directions perpendicular and parallel to the c-axis in the oriented SiC, respectively. For dense SiC with random orientation, the strength was 724 MPa. The bending strength depends on the orientation direction, and the bending strength for the crack-growth directions parallel to the c-axis was higher than that perpendicular to the c-axis.

The c-axis of alumina can also be aligned parallel to the static magnetic field.\(^25\).\(^26\) Figure 5 illustrates the degree of c-axis orientation as a function of heating temperature together with the relative densities and grain sizes for alumina formed by slip casting in a static magnetic field of 12 T (B // casting direction) and the samples formed outside of a magnetic field. The degree of orientation was calculated using Eq. (2) from the intensities of each peak in the XRD measurements.

\[
P = \frac{I_{006}}{I_{006} + I_{110}}
\]  

where \(I_{006}\) and \(I_{110}\) are the intensities from the 006 and 110 reflections, respectively, on the surface perpendicular to the magnetic field. This value, \(P\) approaches unity as the degree of crystalline orientation in alumina increases with the c-axis orientation. For the samples without a magnetic field, the degree of orientation was approximately 0.025, consistent with that calculated from the value of ICDD. The samples not exposed to a magnetic field were confirmed to be polycrystalline alumina with random orientation. By comparison, the degree of crystalline orientation in the samples prepared in a magnetic field gradually increased with grain growth occurring from 1200 °C or less and increased more rapidly at temperatures above 1300 °C. This phenomenon indicates that the magnetic torque can rotate large particles more easily than small particles during slip casting because the torque is proportional to the particle volume according to Eq. (1), and according to Ostwald ripening, the large grains with high orientation grow by consuming fine grains with low orientation during sintering.\(^27\).\(^28\) Finally, large grains with a high orientation remained in the sintered materials. Therefore, grain growth enhanced the degree of orientation during sintering.

Transparent polycrystalline alumina has been produced and investigated since its initial development by Coble.\(^29\) Transparent Al\(_2\)O\(_3\) can be fabricated by hot pressing and SPS for removing residual pores. Dr. Kim found that a slow heating rate is sufficient to produce transparent alumina with a fine microstructure.\(^30\) This technique can be applied to green compacts with orientation. Alumina has a trigonal crystal structure, and birefringence prevents the penetration of incident light due to its asymmetric structure in polycrystal alumina ceramics. Consequently, orientation can be expected to improve the in-line transparency of
polycrystal alumina.\textsuperscript{31,32} We applied magnetic field alignment to transparent alumina with a fine microstructure, as shown in Fig. 6, and indicated that the $c$-axis alignment reduced the different of the refractive index and suppressed birefringence at the grain boundary.\textsuperscript{33}

In the case of titania, the $c$-axes of rutile and anatase were aligned parallel to a static magnetic field.\textsuperscript{34,35} When using rutile as the starting powder, the $c$-axis in rutile was aligned parallel to the magnetic field. Figure 7 shows the (0001) pole figure on the VT plane perpendicular to the magnetic field. Maximum intensity was observed at the center, it indicates that the $c$-axis was aligned parallel to the magnetic field, and the grain elongation was aligned parallel to the magnetic field during sintering. When using anatase powder as the starting material, the alignment of the $c$-axis in anatase was parallel to the magnetic field before the transformation to rutile. However, after this transformation, the $c$-axis of some rutile grains had a tilt of approximately 30° because rutile grew within its [100] planes parallel to the anatase [112] planes,\textsuperscript{36} and the anatase [112] planes are tilted 29.4° from the $c$-axis aligned by the magnetic field.

Since the orientation is maintained past the transformed phase, it is possible to control the orientation even in materials possessing crystal structures close to cubic, which possess notably small anisotropic susceptibility, such as barium titanite (BT).\textsuperscript{37,38} BT has a hexagonal phase at high temperature, and the phase can be stable at room temperature by reduction. These hexagonal BT particles can be rotated easily by the magnetic field of 12 T, and the $c$-axis is aligned parallel to the magnetic field. The BT powder transforms into the tetragonal phase during sintering, from which the (111) oriented tetragonal BT can be obtained. In the case of hematite with a corundum structure, orientation can be controlled by a magnetic field, but it is difficult to densify the particle packing in the hematite green compact because of the particle connections to each other and the formation of a chain structure due to weak ferromagnetic properties in a magnetic field. Therefore, goethite was used for orientation in the green body by a magnetic field, and then dense hematite was produced by transformation during sintering.\textsuperscript{39} Furthermore, insufficient anisotropic susceptibility can also be overcome by reaction sintering of green bodies prepared in a magnetic field. It is difficult to induce the orientation in yttria-doped tetragonal zirconia owing to the very small tetragonality; accordingly, monolithic zirconia particles mixed with yttria particles were used as starting materials for magnetic rotation, as textured tetragonal zirconia can be fabricated by reaction sintering after consolidation.\textsuperscript{40,41} Tanaka et al. reported that textured BaBi$_4$Ti$_4$O$_{15}$ can be fabricated by magnetic field alignment and reactive sintering.\textsuperscript{42} Textured $\beta$-alumina can be fabricated by reaction sintering among Na$_2$O, MgO, and porous textured $\alpha$-alumina prepared by slip casting in a magnetic field.\textsuperscript{43}

3.2 Hard magnetization axis orientation by rotating the magnetic field

Owing to the easy magnetization property of AlN, the $a$-axis is aligned by applying a static magnetic field. It was reported that a hard magnetization axis can be aligned by a rotating magnetic field, and this technique can be applied to ceramic consolidation.\textsuperscript{44,45} Figure 8 shows the [0001] pole figure on the T plane perpendicular and parallel to the static magnetic field and the rotating magnetic field, respectively. This indicates that the $a$-axis was aligned parallel to the static magnetic field and the $c$-axis was aligned perpendicular to the rotating plane. The $c$-axis of Si$_3$N$_4$ can also be aligned by a rotating magnetic field, and the thermal conductivity could be improved along the aligned $c$-axis in Si$_3$N$_4$.\textsuperscript{46} The rotating magnetic field can be applied to various ceramics, such as ZnO, hydroxypatite, and strontium barium niobite.\textsuperscript{47-50} Furthermore, the modulated-rotation magnetic field (time-dependent elliptic magnetic field) enables the tri-axis orientation of materials with three different magnetic susceptibilities depending on each axis.\textsuperscript{51,52}

Lithium-ion batteries are one of the most popular rechargeable battery options; therefore, scientists and
engineers worldwide are attempting to improve their power density and durability. A rotating magnetic field was used to fabricate the cathode consisting of an ideal textured microstructure with individual LiCoO$_2$ grains: a perpendicular alignment of the $c$-plane (vertical side) and random orientation of the $a$-axis. Unlike the cathode with both the random $c$-plane and the random $c$-axis orientation, lithium ions in specialized grains were easily accessible while relaxing the stress associated with intercalation.\(^{53}\) It is easy to combine magnetic orientation with other processes; consequently, multi-axis orientation can be controlled by a combination of the geometric effect of anisotropic particles and the magnetic field. When the $c$-axis is the easy magnetization axis, the $c$-axis is aligned parallel to the magnetic field, and the other axis is random in the plane perpendicular to the magnetic field even if the platelet particles are used as shown in Fig. 9(a). These two effects are expected to govern the multi-dimensional orientation if the easy magnetization axis is different from the axis aligned by the geometric effect, as shown in Fig. 9(b). In the case of Bi$_4$Ti$_3$O$_{12}$, biaxial orientation could be achieved by slip casting of anisometric particles in a strong magnetic field where the plane of the anisometric particles was aligned by gravity, and the alignment of the $a$- and $b$-axes was controlled by the magnetic field.

3.3 Multiaxis orientation by combination of magnetic field and other orientation effects

It is easy to combine magnetic orientation with other processes; consequently, multi-axis orientation can be controlled by a combination of the geometric effect of anisometric particles and the magnetic field. When the $c$-axis is the easy magnetization axis, the $c$-axis is aligned parallel to the magnetic field, and the other axis is random in the plane perpendicular to the magnetic field even if the platelet particles are used as shown in Fig. 9(a). These two effects are expected to govern the multi-dimensional orientation if the easy magnetization axis is different from the axis aligned by the geometric effect, as shown in Fig. 9(b). In the case of Bi$_4$Ti$_3$O$_{12}$, biaxial orientation could be achieved by slip casting of anisometric particles in a strong magnetic field where the plane of the anisometric particles was aligned by gravity, and the alignment of the $a$- and $b$-axes was controlled by the magnetic field.

**Figure 10** shows the {001} and {100} pole figure on the T plane perpendicular to the gravity and parallel to the magnetic field.\(^{54}\) In the {001} pole figure, one strong peak is at the center, indicating that the $c$-axis is aligned perpendicular to the T plane. In the {100} pole figure, four strong peaks were on the S1 and S2 planes parallel and perpendicular to the magnetic field, indicating that the (100) axis was aligned parallel to the magnetic field and showed a two-dimensional orientation. Although Bi$_4$Ti$_3$O$_{12}$ has an orthorhombic crystal structure, it was difficult to distinguish between the $a$- and $b$-axes because their lattice parameters are similar to each other, which makes it difficult to control the tri-axial orientation.

Next, we attempted to control the tri-axial orientation in MgTi$_2$O$_4$ with an orthorhombic crystal structure.\(^{55}\) Rod-like MgTi$_2$O$_4$ particles were used as the anisotropic particles, but it was difficult to align the rod-like particles because the particle size and aspect ratio were insufficient to control the alignment of the long sides of rectangular cuboid particles by gravity. Accordingly, tape casting was used for the alignment of the long side of rod-like particles, and one of the short sides was oriented by the magnetic field. The spherical powder was added with an equal weight of the rod-like particles into a slurry. The slurry was cast onto a glass substrate using a film applicator. The sheet dried in a magnetic field was cut and stacked in the casting direction and laminated by CIP followed by sintering. **Figure 11** illustrates the EBSD map of orientation-controlled MgTi$_2$O$_4$, clearly indicating the tri-axial orientation. The colors of most grains were green, red, and blue on the tape casting plane (T), S1 (side of the casting plane and parallel to the magnetic field), and S2 (normal to the magnetic field), respectively. Therefore, the $c$-axis was aligned on the casting plane by geometric effect and the shear stress during tape casting, and then the $b$-axis was aligned by the magnetic field while maintaining the orientation of the $c$-axis, and as a result the $a$-axis was aligned by the alignment of the other two axes.
3.4 Electrophoretic deposition in a magnetic field

Combining texture and layered structures is an effective approach to developing their properties in natural context, such as the exocuticle of the lobster.[56] Laminar ceramics with single components and different crystalline-oriented layers were fabricated by the EPD method in a magnetic field. The layered structure and texture were controlled simultaneously by EPD and a magnetic field, respectively.[57],[58] The direction of the electric field relative to the magnetic field (the angle between the vectors E and B, $\varphi_{B-E}$) was altered at constant intervals to control the dominant crystal directions in each layer. Figure 12 shows the EBSD map of the laminar structure of alumina with different crystalline-oriented layers prepared by EPD in a magnetic field of 12 T, followed by sintering at 1600 °C. This composite was produced by alternately changing $\varphi_{B-E} = 0$ and 90° layer by layer during EPD in a magnetic field. The bending strength depended on the crack growth direction. The strength of the crack propagation normal to the deposition direction was higher than that in the direction parallel to the deposition.

4. Summary

Microstructure control is important in the improvement of ceramic properties. A wide variety of parameters exist to control microstructure, and among them, crystallographic orientation is especially promising. The control of the crystalline orientation by a strong magnetic field was introduced in this review. Although the device was restricted owing to the use of a superconducting magnet for generating a strong magnetic field, this technique is applicable to
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Tohru S. Suzuki is a Group Leader of Ceramics Processing Group, Research Center for Functional Materials, National Institute for Materials Science (NIMS), Tsukuba, Japan. He earned his Bachelor (1990), Master (1992) and Doctor of Engineering (1995) degrees all from Waseda University, Japan. After working for Waseda University, he moved to National Research Institute for Metals that was reorganized to NIMS in 2001. He earned CerSJ Fellow Awards (2020), Global Star Award from the Engineering Ceramics Division of the American Ceramic Society (2017), Award for Outstanding Papers Published in the Journal of the Ceramic Society of Japan (2012) and JSPM Award for Innovatory Research from Japan Society of Powder and Powder Metallurgy (2005). His current research interests include the processing for a tailored microstructure in bulk ceramics, especially crystallographic orientation by a magnetic field, for improving their properties, such as transparency, ion conductivity, mechanical properties, etc.