Development of Advanced Ceramics by Powder Composite Process

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Abstract

A powder composite process was applied to develop several kinds of advanced ceramics. TiO2 nanoparticles and Si3N4 particles were mixed using a powder composite process to disperse TiN nanoparticles in Si3N4 ceramics, which are expected to be used as novel materials for next-generation hybrid ceramic bearings. TEM observations showed that the TiO2 nanoparticles were directly bonded to submicron Si3N4 particles. Si3N4 ceramics with uniformly-dispersed TiN nanoparticles were fabricated using the composite particles. The amount of damage caused by the Si3N4 ceramics with TiN nanoparticles to the mating metals in a ball-on-disk test was comparable to the damage caused by Si3N4 ceramics without TiN particles. Nanocomposite particles of Al2O3-doped ZnO prepared by a powder composite process were also used for fabricating ZnO ceramics. TEM observations revealed the uniform presence of Al2O3 nanoparticles on ZnO particles. A sintering body fabricated using the composite powder prepared by this powder composite process had more uniform and finer microstructures than that fabricated using a powder mixture prepared by conventional wet mixing. The ZnO ceramics prepared by the powder composite process exhibited higher electrical conductivity than those prepared by the conventional wet ball milling process. CNT-dispersed Al2O3 ceramics were fabricated using a powder mixture of CNT and fine Al2O3 powder prepared by the powder composite process. It was shown that CNTs were uniformly dispersed in the developed CNT-dispersed Al2O3 ceramics, and that they had high electrical conductivity and strength.

Keywords: powder composite process, silicon nitride, titanium nitride, zinc oxide, alumina, carbon nanotube

1. Introduction

Advanced ceramics are very significant materials with the potential to solve present and future environmental and energy problems. The properties of these ceramics depend, not only on the atomic bonding and crystal structure, but also on the microstructural properties such as the grain size, grain boundary, impurity, secondary phase, pores, and defects in the sintered body. In particular, the powder processing has a large influence on the latter because advanced ceramics are fabricated by shaping and sintering fine raw powders. Therefore, the advancement of ceramic powder processing techniques, including the homogeneous dispersion of raw materials, formation of a uniform green body, and analytical control of the sintering shrinkage behavior, is very important to make better ceramics. The homogeneous mixing of additives can be better achieved by using nanoparticles rather than large particles as the raw material. However, the use of the wet milling process with liquid dispersion media such as water and ethanol might lead to the reagglomeration of nanoparticles during drying. We have focused on the fabrication of advanced ceramics by using a powder composite
process in which nanoparticles are mechanically mixed using only a dry mixing process, which allows us to obtain nanocomposite particles. In this process, nanoparticles are bonded to submicron particles by applying an external mechanical force, specifically a shear force.2

Many types of powder composite processes have been developed commercially.1,2 These nanocomposite particle processes are divided into two types: the bonding of fine particles to a core particle and the homogeneous mixing of various fine particles. The former is regarded as surface modification on the microscale and nanoscale. The powder composite process is characterized by (a) a simple dry process, (b) unlimited raw material, (c) a large amount of treatment, and (d) the easy control of the coating conditions. The particle composite behavior depends on several factors. Naito et al. reported the effects of the rotor shape, mechanical treatment conditions (rotation speed and time), and raw material particle sizes on the particle composite process.3-6 Nogi et al. also estimated the relationship between the temperature of the container and the interface of the particles.

Some researchers have studied the synthesis of ceramic particles and the preparation of porous ceramics using a powder composite process.7-10 We also developed several kinds of dense advanced ceramics and controlled their microstructures to improve their properties.11-12 In this paper, we will introduce the microstructure and properties of TiN-particle-dispersed Si₃N₄ ceramics, Al₂O₃-doped ZnO ceramics, and CNT-dispersed Al₂O₃ ceramics fabricated by the particle composite process.

2. TiN-Nanoparticle-Dispersed Si₃N₄ Ceramics

Over 40 years have passed since Si₃N₄ ceramics were first developed. In the meantime, Si₃N₄ ceramics with high strength and fracture toughness have been developed as a result of using newly discovered SiAlONs; the invention of sintering aids such as Y₂O₃; the development of fine, pure, and highly sinterable Si₃N₄ powder; the invention of a gas pressure sintering technique; the advancement of the science and technology for microstructure control, etc. Si₃N₄ ceramics have been applied to automobile components such as glow plugs, hot chambers, and turbocharger rotors. Around the same time, cutting tools and bearing components were also developed. Si₃N₄ ceramic bearings were put to practical use in 1983. Although the cost of Si₃N₄ ceramics was high, they were used as bearing materials in machine tools because of properties such as high strength, high toughness, high elastic modulus, high hardness, light weight, and good corrosion resistance. Furthermore, Si₃N₄ ceramics fabricated from Si₁₇₃N₄-Y₂O₃-Al₂O₃-TiO₂-Al₂O₃ have been widely used for bearing applications. The authors have studied the sintering behavior and microstructure control of Si₃N₄-Y₂O₃-Al₂O₃-TiO₂-Al₂O₃. We found that the densification is enhanced at lower temperatures by simultaneous additions of TiO₂ and AlN to Si₃N₄-Y₂O₃-Al₂O₃. It was also shown that TiN is formed by a reaction between TiO₂ and AlN or Si₃N₄, and that its size is almost the same as that of the added TiO₂. Furthermore, it was clarified that the contact fatigue of Si₃N₄ ceramics is suppressed by the dispersed TiN particles.

Hybrid bearings composed of Si₃N₄ ceramic balls and metal rings are more popular than all-ceramic bearings because of their cost. In hybrid bearings, it is possible that hard and large TiN particles damage the mating metals in a manner analogous to the wear map concept. The dispersion of TiN nanoparticles is expected to be effective in solving this problem. TiN nanoparticles can be formed from TiO₂ nanoparticles because the size of TiN should be almost the same as that of TiO₂. However, it is difficult to realize TiN nanoparticle-dispersed Si₃N₄ ceramics even if TiO₂ nanoparticles are completely dispersed in the slurry. This difficulty might be because of the reagglomeration of TiO₂ nanoparticles during the drying process. We applied the powder composite process to develop TiN-nanoparticle-dispersed Si₃N₄ ceramics and lower the aggressiveness to the mating metals in a wear test.

To fabricate TiN-nanoparticle-dispersed Si₃N₄ ceramics, high-purity, fine Si₃N₄ powder (SN-E-10, Ube Co., Ltd., Japan), Y₂O₃ (RU, Shinetsu Chemical Co., Japan), Al₂O₃ (AKP-30, Sumitomo Chemical Co., Japan), AlN (F grade, Tokuyama Co., Japan), and TiO₂ (Aeroxide P 25, Nippon Aerosil Co., Ltd, Japan) were used as the raw materials. First, TiO₂ nanoparticles were dispersed in ethanol according to our previous study, using polyethyleneimine (EPOMIN, MW1200, Nippon Shokubai Co., Ltd., Japan) by ball milling for 48 h. The Si₃N₄ powder was then mixed into the TiO₂ slurry by ball milling for 48 h, followed by the elimination of the ethanol. The pre-mixed powder was mechanically treated using a powder composer (Nobilta NOB-130, Hosokawa Micron Co., Japan) to prepare composite particles. After the powder composite process, the other sintering aids were added by ball mill-
ing in ethanol for 48 h using $\beta$-sialon balls in a silicon nitride container. After removing the ethanol, 4 wt% paraffin (melting point: 46-48°C, Junsei Chemical Co., Japan) and 2 wt% dioctyl phthalate (DOP, Wako Junyaku Co., Japan) were added as a binder and lubricant, respectively. For reference, five types of powder mixtures were also prepared using only mechanical treatment without premixing, using conventional wet ball milling with 20-, 200-, or 540-nm TiO$_2$ particles, and not using TiO$_2$ and AlN (i.e., no TiN formation in Si$_3$N$_4$ ceramics). The notations for these powder mixtures are listed in Table 1.

Fig. 1 shows SEM images of the powder mixtures before and after the mechanical treatment. The TiO$_2$ nanoparticles formed aggregates before the mechanical treatment [Fig. 1(a)]. On the other hand, as shown in [Fig. 1(b)], no TiO$_2$ nanoparticle aggregates were found in the powder mixture after the mechanical treatment, i.e., the TiO$_2$ nanoparticles were well dispersed. Fig. 2 presents TEM images of the powder mixtures before and after the mechanical treatment. Although the TiO$_2$ nanoparticles were dispersed in ethanol by wet mixing, as reported in our previous paper 32), they reagglomerated as a result of mixing with Si$_3$N$_4$ powder and/or drying. As shown in Fig. 2(b), the mechanical treatment resulted in the uniform dispersion of the TiO$_2$ nanoparticles, thus suggesting that they might be strongly attached to the Si$_3$N$_4$ particles. A high-resolution TEM (HRTEM) image of a composite particle is shown in Fig. 3. It can be seen that a TiO$_2$ nanoparticle is directly bonded onto a submicron Si$_3$N$_4$ particle. At the atomic scale, the interface between the TiO$_2$ and Si$_3$N$_4$ was flat. Such a direct-bonded interface should be stronger than the interface of physically adsorbed particles. Si was detected in the TiO$_2$ particles by

Table 1 Composition and mixing process for TiN-dispersed Si$_3$N$_4$ ceramics

| Notation | Composition (weight ratio) | Mixing process | Remarks |
|----------|---------------------------|----------------|---------|
| PM-N     | 92 5 3 5 5               | Powder composite process | TiO$_2$:540nm |
| W-N      | 92 5 3 5 5               | Powder composite process | TiO$_2$:540nm |
| W-SM     | 92 5 3 5 5               | Conventional wet ball milling | TiO$_2$:540nm |
| S-M      | 92 5 3 5 5               |                        | TiO$_2$:540nm |
| ST       | 92 5 3 0 0               |                        | No TiN in Si$_3$N$_4$ ceramics |

Fig. 1 SEM images of powder mixture (a) before and (b) after a powder composite process.

Fig. 2 TEM images of powder mixture (a) before and (b) after a powder composite process.
an EDS analysis. In addition, neck growth occurred between the TiO$_2$ and Si$_3$N$_4$ particles, similar to the initial stage of sintering, in spite of the mechanical treatment at ambient temperature. This phenomenon should result from the reaction between the TiO$_2$ and TiO$_2$ and/or Si$_3$N$_4$. Nogi et al. reported the relationship between the temperature of the container and the particle interface\(^7\). In this case, because the container temperature is 50—60°C, the interfacial temperature is expected to be around 700—800°C. On the other hand, the crystalline phase of the TiO$_2$ was anatase, both before and after the mechanical treatment, indicating that the temperature of the particle itself was not very high. Therefore, a mechanochemical reaction occurring just at the interface resulted in diffusion and sintering, forming the flat interface and the neck between the TiO$_2$ and SiO$_2$ layer and/or Si$_3$N$_4$ particles.

The mixed powders were sieved using a #60 nylon sieve and then molded into \(\phi 15 \times 7\) mm pellets by uniaxial pressing at 50 MPa followed by cold isostatic pressing at 200 MPa. After binder burnout in air at 500°C for 3 h, the green bodies were fired at 1800°C in 0.9 MPa N$_2$ for 2 h using a gas pressure sintering furnace (Himulti 5000, Fujidenpa Kogyo Co., Japan). The sintered bodies were hot isostatically pressed at 1700°C for 1 h under 100 MPa N$_2$. The density of the fired samples was measured using the Archimedes method. The phase present in the samples was identified by X-ray diffraction (RINT2000, Rigaku Co., Japan). The microstructure was observed by a transmission electron microscope (TEM, JEM-3000F, JEOL, Japan) equipped with an energy-dispersive spectroscopy (EDS, Voyager III, NORAN instruments). The Si$_3$N$_4$ ceramics were machined using a grinding machine (SG-45FIHH, Wasino Engineering Co., Japan) and then polished using diamond slurry. The wear property was estimated by a ball-on-disk test, which consisted of a polished Si$_3$N$_4$ disk and a steel ball bearing (SUJ-2). The testing conditions were a relative humidity of 50 ± 2% and a temperature of 22-24°C. The radius of the SUJ-2 ball was 3 mm. The rotation speed and radius were 10 cm s$^{-1}$ and 3 mm, respectively. The weight was 5 N, and the running distance was 250 m. The polished and worn surfaces of the specimens were observed by scanning probe microscopy (SPM, SPA-400, Seiko Nanotechnologies, Japan).

The relative density of all of the specimens was over 98%, which was sufficiently high to measure the mechanical properties. \(\beta\)-Si$_3$N$_4$ and TiN were also identified as the main phases of the products in the sample, in addition to TiO$_2$ and AlN. Fig. 4 shows TEM images of the TiN-dispersed Si$_3$N$_4$ ceramics. As shown in Fig. 4 (a), the size of the TiN particles in the Si$_3$N$_4$ ceramics fabricated using just wet mixing was 300-500 nm. On the other hand, in the case of using composite particles prepared by premixing and

![Fig. 3](image1)  
**Fig. 3** High-resolution TEM image of composite particle prepared by a powder composite process.

![Fig. 4](image2)  
**Fig. 4** TEM images of Si$_3$N$_4$ ceramics. (a) Sample W-N and (b) sample PM-N.
mechanical treatment, 20-100-nm TiN nanoparticles were found in Si₃N₄ grains and in the grain boundary (Fig. 4 (b)). Thus, it was shown that TiN nanoparticle-dispersed Si₃N₄ ceramics were fabricated using composite powder prepared by premixing followed by mechanical treatment.

Fig. 5 and 6 show optical micrographs of the worn surfaces of the SUJ2 balls and Si₃N₄ disks after the ball-on-disk tests. It can be observed that the areas of the SUJ2 balls worn by sample PM-N were smaller than all of the other areas, even though the wear track width of the Si₃N₄ ceramics is almost the same. The wear volume was calculated from the wear area of the ball and the worn surface profile of the Si₃N₄ disk. As listed in Table 2, the wear volume of the Si₃N₄ disk was independent of the Si₃N₄ ceramics, except for sample W-M. On the other hand, the wear volume of the SUJ2 ball depended on the mating Si₃N₄ ceramics, i.e., the wear volume of the ball worn by sample PM-N was not only smaller than those worn by samples M-N and W-N but was also the same as that worn by sample NT. Furthermore, the wear volumes of the steel balls worn by samples M-SM and M-M were higher than those of PM-N, thus suggesting that TiN nanoparticle dispersion should cause less damage to the mating metals.

The wear behavior was evaluated by SPM observations of the worn surfaces. Fig. 7 shows SPM images of the worn surfaces of the steel balls after the ball-on-disk tests. It was found that there were many grooves on the worn surface and that the distance between the grooves was comparable to the size of the TiN. Fig. 8 shows the polished surface of sample W-M. Even though the surface was polished, it can be observed that a large TiN particle projects from the surface because of its high hardness. This indicates that aggressive wear was caused by hard and large TiN particles during the ball-on-disk test, similar to abrasive wear. In other words, because sample PM-N had much smaller TiN particles, the abrasiveness toward the mating metallic ball was considered to be comparable to Si₃N₄ ceramics without TiN particles.

Fig. 9 shows SPM images of the worn surfaces of the TiN-dispersed Si₃N₄ ceramics. The agglomerates

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**Table 2** Wear volume of Si₃N₄ disk and SUJ2 ball after ball-on-disk test

| Sample | Wear volume / 10⁻¹²m³ |
|--------|-----------------------|
|        | Si₃N₄ disk | SUJ2 ball |
| PM-N   | 9.5          | 1.5       |
| M-N    | 9.4          | 2.7       |
| W-N    | 9.8          | 3.9       |
| W-SM   | 10.9         | 20.7      |
| W-M    | 4.4          | 3.5       |
| NT     | 10.0         | 1.6       |
observed on the worn surface of sample W-M might consist of metallic wear particles. Because of the adhesion of metallic particles on the worn surface of Si₃N₄ ceramics, the wear volume of sample W-M appears to be smaller than the other samples. Because the grain boundary phase is easily removed, the Si₃N₄ grain shape is emphasized. The Si₃N₄ ceramics in this study were composed of Si₃N₄ grains, TiN grains, and a grain boundary phase. It is well known that softer materials are more easily worn. Therefore, a softer grain boundary phase is considered to be preferentially worn during a ball-on-disk test. Furthermore, because the Si₃N₄ ceramics used in this study were fabricated from the same composition of sintering aids, it seems that the wear volumes of the Si₃N₄ ceramics were comparable. Thus, it was shown that TiN nanoparticle-dispersed Si₃N₄ ceramics were successfully developed using TiO₂ and Si₃N₄ composite particles prepared by pre-mixing and mechanical treatment, and that the dispersion of TiN nanoparticles lowered the aggressiveness to the mating metals in the wear test.

The developed Si₃N₄ ceramics need high mechanical properties for bearing application. ISO 26602:2009 provides a classification defining the physical and mechanical properties of silicon nitride preprocessed ball bearing materials. These materials are classified in three categories by the specification of their characteristics and microstructures. Fig. 10 shows

![Fig. 8 SPM images of polished surface of sample W-M.](image)

![Fig. 7 SPM images of worn surface of steel ball after ball-on-disk test.](image)

(a) sample PM-N, (b) M-N, (c) W-N, (d) W-SM, (e) W-M and (f) NT

![Fig. 9 SPM images of worn surface of Si3N4 disk after ball-on-disk test.](image)

(a) sample PM-N, (b) M-N, (c) W-N, (d) W-SM, (e) W-M and (f) NT
the Weibull plot of the bending strength of sample PM-N. The shape factor in the Weibull plot of the bending strength was 13, and the average bending strength was 1109 MPa. The fracture toughness and Vickers hardness were 6.7 MPam^{1/2} and 15.4 GPa, respectively. These are higher than the Class I values in ISO 26602:2009. We prepared the developed material to measure the crushing strength. The crushing strength of this material was about 31.7 N, which is 1.5 times that of the conventional Si₃N₄ ceramics used for bearings. A rolling fatigue test was carried out using 3 10-mm diameter balls under a pressure of 5.9 GPa. The rolling fatigue lifetime of the developed TiN-nanoparticle-dispersed Si₃N₄ ceramics was longer than 107 cycles. Consequently, it was confirmed that the developed material has sufficient mechanical properties for use in the next generation of ceramic bearings.

3. Al-Doped ZnO Ceramics

ZnO is a typical n-type semiconductor. Numerous researchers have studied the electrical properties of ZnO ceramics for many years. Because of the advantages offered by the particular properties of ZnO ceramics, they have been used to manufacture varistors^{30}, sensors^{30}, thermoelectric transducers^{30}, transparent conductive films^{30}, light emitting diodes^{30}, etc. The electrical properties of ZnO ceramics are controlled by adding small amounts of additives. For example, it has been reported that Cr₂O₃, CoO, MnO, Bi₂O₃, and Pr₂O₃; additives exhibit nonlinear electrical conductivity^{30,40}. In addition, because Al₂O₃ dissolves in ZnO, the carrier concentration can be controlled (controlled valency). Therefore, Al₂O₃-doped ZnO improves the electrical conductivity^{41,42}.

Many researchers have fabricated ZnO ceramics using a solid reaction; here, a ball milling technique is normally used to mix the powder for the fabrication. The homogeneous dispersion of additives is one of the most important processes in powder processing; this process results in the production of better ZnO ceramics. This homogeneous mixing of additives can be better achieved by using nanoparticles rather than large particles as the raw material. However, the use of the wet milling process with liquid dispersion media such as water and ethanol might lead to the reagglomeration of nanoparticles during drying. Consequently, the amount of mixed material obtained might not be sufficient to take advantage of the characteristics of nanoparticles. We developed Al₂O₃-doped ZnO ceramics using nanocomposite particles composed of γ-Al₂O₃ nanoparticles and ZnO submicron particles prepared by mechanical treatment and investigated the effect of the mixing process on the electrical conductivity of ZnO ceramics.

ZnO powder (grain size: 0.2−0.6 μm, JIS 1, Hakusui Tech Co., Ltd.) and γ-Al₂O₃ powder (grain size: 7 nm, TM-300, Taimei Chemicals Co., Ltd.) were used in this study. The amounts of γ-Al₂O₃ used were 0.1, 1.0, and 3.7 mol%. These powders were premixed for 30 min by dry ball milling. This mixing was carried out using Al₂O₃ balls of φ 10 mm. The premixed powder was mechanically treated using a powder composer (Nobilta NOB-130, Hosokawa Micron Co.) working at 2000 rpm for 10 min to prepare composite powders. For reference, raw material powders of the same compositions were prepared by carrying out wet ball milling in ethanol using Al₂O₃ balls of φ 10 mm for 24 h.

The mechanically treated powders were observed using a transmission electron microscope (TEM, JEM-3000F, JEOL Ltd.). Fig. 11 shows the TEM images of the ZnO-Al₂O₃ powder mixture after the mechanical treatment (Figs. 11 (a) and (b)) and wet ball milling (Figs. 11 (c) and (d)). Figs. 11 (b) and (d) are enlarged TEM photographs. The angular and dark pieces of grains shown in Figs. 11 (a) and (c) represent the ZnO grains. Smaller particles can be seen on the surface of a ZnO particle in Fig. 11 (a). Fig. 11 (b) shows that the size of the smaller particles on the ZnO particles was less than 10 nm, which was almost the same as that of the γ-Al₂O₃ used in this study. Therefore, it appears that the γ-Al₂O₃ nanoparticles were directly bonded to the ZnO particles. On the other hand, a large agglomeration of γ-Al₂O₃ was observed after wet ball milling (Fig. 11 (c)), and it was concluded that the γ-Al₂O₃ nanoparticles were not directly bonded to the ZnO particle.

Fig. 10 Weibull plot of bending strength of sample PM-N.
Fig. 11 shows the XRD profiles of the ZnO-Al₂O₃ powder before and after the mechanical treatment, as well as the raw materials used. The characterization of the composite powder was carried out by X-ray diffraction using Cu Kα radiation. It was observed that the phases present in the composite powder consisted exclusively of ZnO and γ-Al₂O₃, which were the same as the raw materials. Therefore, we inferred that γ-Al₂O₃ bonded to the ZnO grain surface because of the mechanical treatment. In this experiment, we were also able to establish the phenomenon reported above. The estimated temperature of the particle interface must be maintained below 700 °C. It is known that the sintering shrinkage of ZnO starts at a temperature of approximately 600 °C. It should be noted that γ-Al₂O₃ is phase transformed to α-Al₂O₃ with an increase in temperature and grain growth. Therefore, the temperature of the γ-Al₂O₃ and ZnO particles themselves should be very low, but the temperature of the interface between the ZnO and γ-Al₂O₃ should be slightly higher because of the shear force.

The specific surface area was measured by the BET method using a surface area analyzer (Quan tasorb, Quantachrome Instruments, USA). Table 2 lists the specific surface area of the ZnO-Al₂O₃ powder, which was measured using the BET method after the mechanical treatment. For reference, the specific surface areas of the raw materials (ZnO and Al₂O₃) are also listed in the table. The specific surface areas of ZnO and γ-Al₂O₃ were 4.2 m²/g and 196 m²/g, respectively. When 3.7 mol% Al₂O₃-doped ZnO was used, the specific surface area was 12.1 m²/g before the mechanical treatment and 10.3 m²/g after the mechanical treatment. When 1.0 mol% and 0.1 mol% of Al₂O₃ were added, the specific surface area changed from 6.7 m²/g to 5.5 m²/g and from 4.6 m²/g to 4.2 m²/g, respectively. The above decreases in the specific surface areas were assumed to be because of the formation of a bonding area between the ZnO particles and γ-Al₂O₃ during the mechanical treatment. The specific surface area, S₀, before the mechanical treatment was estimated as follows:

\[ S_0 = S_{\text{submicron}} (1 - f) + S_{\text{nano}} f \]  

(1)

where \( f \) is the weight fraction of the nanoparticle, \( S_{\text{submicron}} \) and \( S_{\text{nano}} \) are the specific surface areas of the submicron-sized particle and nanosized particle, respectively. If a cubic-shaped nanoparticle of a phase bonds to the spherical submicron particle, the total surface area should be reduced by \( 2L^2 \). When all of the nanoparticles bonded to the submicron particles, the decrease in surface area, \( \Delta S \), per unit of weight was as follows:

\[ \Delta S = \frac{2L^2}{6L} S_{\text{nano}} f \]  

(2)
Hence, the specific surface area after bonding, \( S \), was estimated as follows:

\[
S = S_{\text{submicron}} (1 - f) + \frac{2}{3} S_{\text{nano}} f
\]

\[
= S_{\text{submicron}} + \left( \frac{2}{3} S_{\text{nano}} - S_{\text{submicron}} \right) f
\]

(3)

The values of the specific surface areas after the mechanical treatment, as calculated in eq. (3), are also listed in Table 3. The calculated values of the specific surface areas were in good agreement with the experimental values. Therefore, it was concluded that the specific surface area decreased because of the bonding between the ZnO and \( \gamma \)-Al\(_2\)O\(_3\) particles during the mechanical treatment and that almost all of the added \( \gamma \)-Al\(_2\)O\(_3\) bonded to the ZnO particles. These results agreed well with those obtained by the TEM analysis.

In order to fabricate granules, paraffin (4 mass\%, melting point: 46–48 °C, Junsei Chemical Co., Ltd.), dioctyl phthalate (DOP, 2 mass\%, Wako Pure Chemical Industries, Ltd.), and cyclohexane (S grade, Wako Pure Chemical Industries, Ltd.) were added to these mixed powders, after which the powder mixture was sieved using a 250-μm-mesh nylon sieve. The powder was then molded into cylindrical pellets by uniaxial pressing at 50 MPa followed by cold isostatic pressing at 200 MPa. After the binder was burned out in air at 500 °C for 3 h, the green bodies were fired at 1400 °C in air for 2 h.

We fabricated sintering bodies using the prepared composite powder and the powder obtained by the wet ball milling of 1.0 mol\% Al\(_2\)O\(_3\)-doped ZnO. The relative densities of the sintering bodies prepared from the composite powder and the powder obtained by wet ball milling were 98.9% and 99.2%, respectively. A phase analysis using X-ray diffraction showed that the main phase was ZnO, and the secondary phase was ZnAl\(_2\)O\(_4\) (Fig. 13).

The microstructures of the ZnO ceramics were observed using the SEM and TEM. The SEM images in Fig. 14 show the thermally etched surfaces of 1.0 mol\% Al\(_2\)O\(_3\)-doped ZnO ceramics. The dark particles represent ZnO, and the small bright particles represent ZnAl\(_2\)O\(_4\). It appears that the particle size of the

| Sample          | Specific surface area (m\(^2\)/g) | Experimental value | Estimated value |
|-----------------|-----------------------------------|--------------------|-----------------|
|                 |                                   |                    |                 |
| \( \gamma \)-Al\(_2\)O\(_3\) | 196                               | 196                | 196             |
| ZnO             | 4.2                               | 4.2                | 4.2             |
| Powder composite process |                       |                    |                 |
| Before | After | After | After |
| 3.7mol\% Al\(_2\)O\(_3\) | 12.1                               | 10.3               | 9.8             |
| 3.7mol\% Al\(_2\)O\(_3\) | 6.7                                | 5.5                | 5.8             |
| 3.7mol\% Al\(_2\)O\(_3\) | 4.6                                | 4.2                | 4.4             |

Fig. 13 XRD profiles of 1.0 mol\% Al\(_2\)O\(_3\)-doped ZnO ceramics. prepared by (a, c) a powder composite process and (b, d) wet ball milling.

Fig. 14 SEM images of 1.0 mol\% Al\(_2\)O\(_3\)-doped ZnO ceramics prepared by (a) a powder composite process and (b) wet ball milling.
ZnO prepared from the wet ball milled powder was slightly larger than that of the ZnO prepared from the mechanically treated powder. The grain size of the powder obtained after the mechanical treatment was 5.3 μm, whereas that obtained by wet ball milling was 5.6 μm. The sintered body fabricated from the mechanically treated powder had a slightly smaller and more uniform grain size than that fabricated from the powder obtained by wet ball milling. Furthermore, the particle size of the secondary ZnAl₂O₄ phase in the ZnO ceramics fabricated by the mechanical treatment process appeared to be considerably smaller and more uniform than that of the secondary ZnAl₂O₄ phase in the ZnO ceramics fabricated by the wet ball milling process. It has been reported that adding Al₂O₃ to ZnO restricted the densification and grain growth of the ZnO. Therefore, the homogeneous and fine microstructure of the ZnO ceramics obtained by the mechanical treatment process can be attributed to a uniform dispersion of the nanosized γ-Al₂O₃ particles.

The electrical conductivity of the 1.0 wt% Al₂O₃-doped ZnO ceramics was measured using the four-terminal method. The electrical conductivities of the sintered bodies obtained by wet ball milling and the powder composite process were 333 S/cm and 588 S/cm, respectively. In other words, the electrical conductivity of the composite powder obtained by the powder composite process was almost twice that of the powder obtained by wet ball milling. The high electrical conductivity of the Al₂O₃-doped ZnO was attributed to the dissolution of the Al₂O₃ in the ZnO to form electrons as carriers. The grain boundary structure and impurity segregation also influenced the electrical conductivities of the ZnO ceramics. TEM images revealed that there was no difference between the sintering bodies obtained by the powder composite process and wet ball milling (Fig. 15). The solubility limit of Al in ZnO is 0.3 mol%, which is smaller than the amount of Al₂O₃ added. The excess amount of Al might be segregated in the grain boundary and could be present as a secondary phase (ZnAl₂O₄). However, there were no considerable differences in the grain boundaries and secondary phases in a comparison of the mechanical treatment and ball milling processes, except for the size of the secondary phase. This implied that the microstructure had a small effect on the difference in the electrical conductivity.

4. CNT-Dispersed Al₂O₃ Ceramics

Carbon nanotubes have high strength, high elastic modulus, and excellent thermal and electrical conductivities, resulting in their use as fillers for composites to improve their mechanical, electrical, and thermal properties. In our previous research, electrically-conductive CNT-dispersed Si₃N₄ ceramics were developed. In the case of the CNT composite process, one of the most important problems to be solved is the uniform dispersion of CNTs. Here, we tried to prepare a powder mixture of CNTs and Al₂O₃ using the powder composite process and then fabricate CNT-dispersed Al₂O₃ ceramics using the nanocomposite particles.

The raw materials were multiwall carbon nanotubes and an alumina powder (Taiimei Chemical, TM-DAR, 0.2 μm). 2 wt% (4 vol%) of the carbon nanotubes were mixed with alumina using a powder composer (Nobiruta, Hosokawa Micron Co., Japan). Fig. 16 (a) shows the CNT-Al₂O₃ powder mixture prepared by the powder composite process. It was found that granules composed of CNTs and Al₂O₃ particles were formed, and there were no agglomer-

![Fig. 15](image1) TEM images of 1.0 mol% Al₂O₃-doped ZnO ceramics prepared by (a) a powder composite process and (b) wet ball milling.

![Fig. 16](image2) TSEM images of powder mixture of CNTs and Al₂O₃ using a powder mixture prepared by a powder composite process. (a) As-received powder. (b) After ultrasonification in ethanol. (c) and (d) Enlarged view of (b).
ates of CNTs. Figs. 16 (b) and (c) are SEM photographs of the powder mixture shown in Fig. 16 (a) after ultrasonification. The granules were easily pulverized by ultrasonic irradiation, and the CNTs were homogeneously dispersed in the Al$_2$O$_3$ powder. This indicated that the CNTs and Al$_2$O$_3$ were uniformly mixed by the particle composite process. Fig. 16 (d) is an enlarged view of the CNT-Al$_2$O$_3$ powder mixture. Fine Al$_2$O$_3$ particles were fixed on the CNTs. This phenomenon is similar to that seen with the SiO$_2$ nanoparticle-SiO$_2$ fiber prepared by a powder composite process, as reported by Naito et al.

The powder mixture was fired at 1750°C for 1 h in an Ar atmosphere using a hot pressing technique. The applied pressure was 30 MPa. A 20 × 20 × 5 mm ceramic plate was obtained. After cutting the sample into 3 × 4 × 20 mm pieces, the relative density, bending strength, and electrical conductivity were evaluated. The microstructure was observed by SEM. Fig. 17 shows the microstructure of the CNT-Al$_2$O$_3$ composite fabricated by hot pressing. There were no large pores in the sintered body in spite of the inhibition of the densification by the CNTs. The bulk density of this sample was 3.82 g/cm$^3$, and the relative density was 92%. The grain size of the Al$_2$O$_3$ in this composite was about 2 μm, which is much smaller than that of pure Al$_2$O$_3$ ceramics hot-pressed under the same conditions (Fig. 17 (b)). The inhibition of grain growth resulted in the lowering of grain boundary migration because of the dispersed CNTs. In the enlarged photograph (Fig. 17 (c)), many projecting CNTs and small holes can be observed in the sintered body. These is evidence of the bridging or pullout of CNTs, which is similar to fiber reinforced ceramics.

The bending strength and electrical conductivity of the CNT-Al$_2$O$_3$ composites using nanocomposite particles are listed in Table 3. The bending strength of the CNT-Al$_2$O$_3$ composites was 423 MPa, which is higher than that of pure Al$_2$O$_3$ ceramics sintered by hot-pressing at the same temperature. This strengthening should be caused by the limitation of the grain growth and the reinforcement by the bridging and pullout of CNTs. The electrical conductivity of the developed CNT-Al$_2$O$_3$ composites was 448 S/m. This value was almost the same as the 12 wt% CNT-dispersed Si$_3$N$_4$ ceramics. The achievement of such a high electrical conductivity by the addition of only 2 wt% CNTs is attributed to the homogeneous dispersion of the CNTs by the powder composite process, which formed numerous electrically conductive paths in the ceramics. Consequently, CNT-dispersed Al$_2$O$_3$ ceramics with high strength and high electrical conductivity were developed using nanocomposite particles prepared by the powder composite process.

5. Conclusions

A particle composite process was applied to develop several kinds of advanced ceramics. TiO$_2$ nanoparticles and Si$_3$N$_4$ particles were mixed using this powder composite process to disperse TiN nanoparticles in Si$_3$N$_4$ ceramics, which are expected to be used as novel materials for next-generation hybrid ceramic bearings. TEM observations showed that the TiO$_2$ nanoparticles were directly bonded to submicron Si$_3$N$_4$ particles. Si$_3$N$_4$ ceramics in which TiN nanoparticles were added by the particle composite process were investigated. It was confirmed that the TiN nanoparticles were uniformly dispersed in the ceramic matrix. The bending strength of the developed TiN-dispersed Si$_3$N$_4$ ceramics was 800 MPa, which is higher than that of pure Si$_3$N$_4$ ceramics sintered by hot-pressing at the same temperature. The electrical conductivity of the developed TiN-dispersed Si$_3$N$_4$ ceramics was 448 S/m. This value was almost the same as the 12 wt% CNT-dispersed Si$_3$N$_4$ ceramics. The achievement of such a high electrical conductivity by the addition of only 2 wt% CNTs is attributed to the homogeneous dispersion of the CNTs by the powder composite process, which formed numerous electrically conductive paths in the ceramics. Consequently, CNT-dispersed Al$_2$O$_3$ ceramics with high strength and high electrical conductivity were developed using nanocomposite particles prepared by the powder composite process.

Table 4  Density, bending strength and electrical conductivity of CNT-dispersed Al$_2$O$_3$ ceramics

| CNT content | 2wt% |
|-------------|------|
| Bulk density| 3.82 g/cm$^3$ |
| Relative density | 97.8 % |
| Bending strength (3-point bending test) | 423 MPa |
| Electrical conductivity (DC 4 terminal method) | 448 S/m (=0.22 Ω cm) |
particles were uniformly dispersed were fabricated using the composite particles. The amount of damage caused by the Si₃N₄ ceramics with TiN nanoparticles to the mating metals in a ball-on-disk test was comparable to the damage caused by the Si₃N₄ ceramics without TiN nanoparticles. Nanocomposite particles of Al₂O₃-doped ZnO prepared by the powder composite process were also used to fabricate ZnO ceramics. TEM observations revealed that Al₂O₃ nanoparticles were uniformly present on ZnO particles. The sintering body fabricated using the composite powder prepared by this powder composite process had more uniform and finer microstructures than that fabricated using the powder mixture prepared by conventional wet mixing. The ZnO ceramics prepared by the powder composite process exhibited higher electrical conductivity than those prepared by the conventional wet ball milling process. CNT-dispersed Al₂O₃ ceramics were fabricated using a powder mixture of CNTs and a fine Al₂O₃ powder prepared by the powder composite process. It was shown that the CNTs were uniformly dispersed in the developed CNT-dispersed Al₂O₃ ceramics, and they had a high electrical conductivity and strength.

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