

# Pulverization of $Y_2O_3$ nanoparticles by using nanocomposite particles prepared by mechanical treatment



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## ABSTRACT

Nanoparticles are being extensively investigated in industries and academia due to their widespread applications in various areas of science and manufacturing. Unfortunately, nanoparticles easily aggregate. This problem of nanoparticle aggregation creates challenges for various applications. In this study, we focus on using nanocomposite particles prepared via mechanical treatment to pulverize the aggregated  $Y_2O_3$  nanoparticles.  $CaCl_2$  and aggregated  $Y_2O_3$  nanoparticles were mechanically treated using a process in which high shear and compressive stress are applied to them in order to generate nanocomposite particles. In order to rinse out  $CaCl_2$ , the nanocomposite particles were then added to distilled water to dissolve the  $CaCl_2$  cores, and they were centrifuged before supernatant solution was decanted. The recovered  $Y_2O_3$  nanoparticles had a significantly smaller particle size and larger specific surface area than the aggregates. Thus, we confirmed that the aggregated  $Y_2O_3$  nanoparticles were effectively pulverized in a short time by using nanocomposites prepared by mechanical treatment. Further, the relative density of the sintered body prepared from the pulverized  $Y_2O_3$  nanoparticles was higher than that of the as-received and ball-milled  $Y_2O_3$  nanoparticles.

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## 1. Introduction

The shape, dispersion, and manufacturing of nanoparticles have been extensively studied for realizing a wide range of applications in various areas of science and manufacturing. In particular, their optical, chemical, physical and electronic properties have led to many developments, e.g., in developing nanocarriers for controlled drug delivery, catalysis, biological imaging, selective treatment of severe diseases, formation of extremely hard and scratch-proof coatings, and production of low-sintering temperature ceramics [1–4]. However, nanoparticles form aggregates or agglomerates easily due to interparticle interactions. These interactions are either dominated by weak van der Waals forces, stronger polar and electrostatic interactions, or covalent interactions during the synthesis process [5–7]. Aggregate formation degrades the properties of the final products. For example, when the particles are aggregated,

the resulting powder compact forms an inhomogeneous structure. Furthermore, if the size of the aggregated particles increases, the internal pore size of the powder compact increases. As a result, the sinterability of the material decreases and the properties of the final product are degraded [8–10]. Therefore, it is crucial to control nanoparticle dispersion and aggregate formation. Unfortunately, it is very difficult to pulverize and disperse aggregated nanoparticles perfectly. Thus, the pulverization and dispersion of nanoparticles have become important techniques for handling nanoparticles in industries [11]. Various methods have been developed for pulverization of nanoparticle aggregates in liquids, including use of agitator discs, colloid mills, high-pressure homogenizers, triple roller mills, ball mills, attrition mills, and bead mills [12,13]. These pulverization methods operate under mild operating conditions, and may be used for large-scale production. In the past few years, many researchers have studied ball milling and bead milling to pulverize nanoparticles to almost primary particle size [13,14]. However, the contamination of the nanoparticles with ground ball media after ball milling and bead milling was ten times higher than that caused by a dry process [14]. In our previous study, we used mechanical treatment, i.e., dry process in which strong shear and compressive stresses are applied to powders in order to obtain a uniform dispersion of nanoparticles [15–23]. With this result, we

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confirmed that application of a strong shear force can pulverize and disperse aggregated nanoparticles onto the surface of core particles. We considered the possibility that we might obtain only the pulverized nanoparticles if the core particles were removed from the nanocomposite particles after mechanical treatment. In order to disperse nanoparticles, Gotoh et al. have proposed a method for dispersing dry nanoparticles in a liquid using large carrier particles ( $>90\ \mu\text{m}$ ), which involves coating the large carrier particles with the nanoparticles, followed by sonication of the composite particles and sedimentation of the larger particles [24]. However, yield of the nanoparticles should be very small because separation by the sedimentation process needs the large difference in the particle size. We selected micron-sized  $\text{CaCl}_2$  particles as host particles because they have a relatively high melting point, and they are inexpensive and are easy to dissolve in water.

In this study, we focus on mechanical treatment as a method of pulverizing aggregated nanoparticles.  $\text{CaCl}_2$  and aggregated  $\text{Y}_2\text{O}_3$  nanoparticles were mechanically treated by application of a strong shear and compressive stress to prepare nanocomposite particles. Then, pulverized  $\text{Y}_2\text{O}_3$  nanoparticles were obtained after removal of the  $\text{CaCl}_2$  particles. We also fabricated powder compacts and sintered bodies using the  $\text{Y}_2\text{O}_3$  nanoparticles.

## 2. Experimental procedure

$\text{CaCl}_2$  powder (Kanto Chemical, Japan, purity: 95.0%) and  $\text{Y}_2\text{O}_3$  powder (ShinEtsu, BB-type, Japan, purity:  $>99.9\%$ ,  $\text{CaO} <10\ \text{ppm}$ ) were used as raw materials in this study. 95 and 5 vol% of  $\text{CaCl}_2$  and  $\text{Y}_2\text{O}_3$  powders were used. The crystallite size of the as-received  $\text{Y}_2\text{O}_3$  powder estimated with the Scherrer equation and FWHM values in the X-ray diffraction pattern was 22 nm. The powders were mechanically treated using a powder composer (NOB-130, Hosokawa Micron Co.). This process allowed us to produce nanocomposite particles under dry conditions, without any binder, by applying strong shear and compressive stress due to the rotating blade and the small gap between the blade and chamber [15–23]. The experimental procedures used are briefly shown in Fig. 1. First, the  $\text{CaCl}_2$  powder was put into the chamber and pre-treated at approximately 1000 rpm for 1 min. After that, the  $\text{Y}_2\text{O}_3$  powder was added to the chamber and pre-mixed using the same conditions. Finally, the powders in the chamber were mechanically treated. The rotation speed of the blade was about 4000 rpm, the power applied was 5 kW, and the soaking time was 1 min.

In order to rinse out  $\text{CaCl}_2$ , the resulting powder mixtures were added to distilled water, and then centrifuged at 3000 rpm for 10 min before the supernatant solution was decanted. This centrifuging and decantation procedure was carried out ten times.

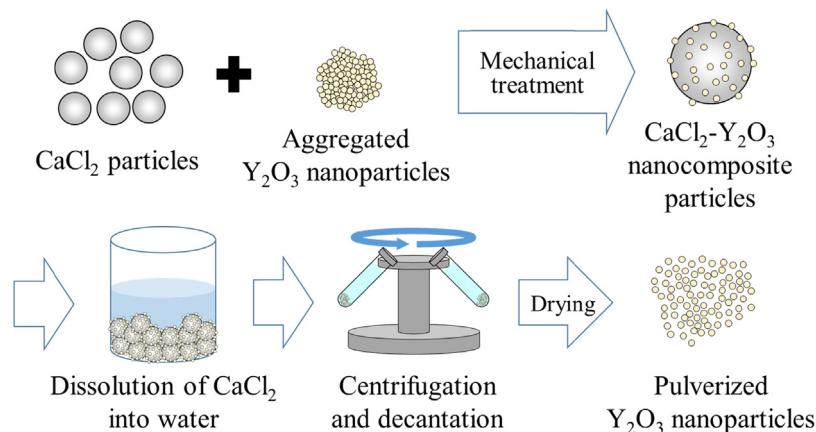


Fig. 1. Schematic diagram of our mechanical treatment procedure.

The resulting powders were dried at  $100^\circ\text{C}$  in oven. The specific surface areas of the  $\text{Y}_2\text{O}_3$  powder were measured using the Brunauer–Emmett–Teller (BET) method (BELSORP-mini II, BEL Japan, Inc., Japan), and the  $\text{Y}_2\text{O}_3$  powder was analyzed using an X-ray fluorescence spectrometer (XRF; JSX-3202EV, JEOL Ltd., Japan) and X-ray diffraction (XRD; RINT 2000, Rigaku Co., Japan) in order to confirm the extent of contamination. Particle size and shape were observed using a field emission scanning electron microscope (FE-SEM, JSM-7001F, JEOL Ltd., Japan).

In order to make granules of these powders, 8 wt% paraffin (melting point:  $46\text{--}48^\circ\text{C}$ , Junsei Chemical Co., Japan) and 4 wt% dioctyl phthalate (DOP, Wako Pure Chemical Industries, Ltd., Japan) were added to the powder as a binder and a lubricant, respectively. The resulting material was sieved using a nylon sieve of  $250\ \mu\text{m}$  mesh. The granules were molded to  $\phi 11 \times 3\ \text{mm}$  by uniaxial pressing under 20 MPa, followed by cold isostatic pressing under 200 MPa. The cold isostatic pressing procedure was repeated 10 times. SEM was used to observe the fracture surface of the powder compacts. In addition, a specimen with a thickness of  $400\ \mu\text{m}$  was subjected to a liquid immersion technique, which uses the transmitted light from an optical microscope (OM, Olympus BX51, Japan) to observe the internal structure of the green body. The liquid used for immersion was diiodomethane.

Organic compounds in the powder compacts were eliminated by firing at  $500^\circ\text{C}$  for 3 h in an air flow of 4 L/min. After dewaxing, the powder compacts were sintered in an electric furnace (M-1700, Watanabe Manufacturing Co., Japan) at  $1500^\circ\text{C}$  for 2 h. The heating rate was  $5^\circ\text{C}/\text{min}$ . The relative density of the sintered  $\text{Y}_2\text{O}_3$  body was measured by the Archimedes method. Microstructure observations were carried out via SEM of a thermally etched surface that was first prepared by firing at  $1300^\circ\text{C}$  for 30 min, and a fracture surface. As references, the as-received  $\text{Y}_2\text{O}_3$  powder and the  $\text{Y}_2\text{O}_3$  powder were ball milled with  $\text{Si}_3\text{N}_4$  balls. Ball milling was performed in ethanol for 24 h at a milling speed of 110 rpm in a polyethylene container. Ball milling was followed by elimination of ethanol.

## 3. Results and discussion

Fig. 2 shows the FE-SEM images of the three kinds of  $\text{Y}_2\text{O}_3$  nanoparticles and  $\text{CaCl}_2\text{-Y}_2\text{O}_3$  nanocomposite particles. The as-received  $\text{Y}_2\text{O}_3$  particles were found to be significantly aggregated (Fig. 2(a)). After ball milling, many of the  $\text{Y}_2\text{O}_3$  nanoparticles remained aggregated (Fig. 2(b)). On the other hand, after our mechanical treatment, aggregates of the  $\text{Y}_2\text{O}_3$  nanoparticles disappeared and nanosized  $\text{Y}_2\text{O}_3$  was detected on the surface of  $\text{CaCl}_2$  particles. This indicates that  $\text{CaCl}_2\text{-Y}_2\text{O}_3$  nanocomposite particles

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