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# Slip casting using wet-jet milled slurry

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**Abstract**

Green bodies fabricated by slip casting using wet-jet milled slurries at different solid contents (10, 30, and 50 vol.%), which had low viscosity, had very high relative density and showed very small shrinkage during sintering, as comparing to the ball-milled slurries. The square of the thickness of green bodies was proportional to casting time indicating slip casting was implemented well. The relative density of the green body prepared by the wet-jet milled slurries was 67%, indicating much higher than that of the ball-milled slurries. After sintering, the linear shrinkage of the sintered bodies prepared by wet-jet slurries at different solid contents was constant at 11%. On the other hand, the linear shrinkage of the sintered bodies prepared by the ball-milled slurries was dependent on the solid contents in slurries. Thus, it was possible to produce not only green bodies with high density but also sintered bodies with low shrinkage by using the wet-jet milled slurries.


*Keywords:* Slip casting; Density; Milling; $Al_2O_3$

## 1. Introduction

Recently, the wet-jet mill process, in which suspensions collide mutually at high pressure and high speed, has been developed as a new method of mixing and dispersion[1] in food technology field. Because the high speed of this system causes the cavitation, turbulent flow, and shear flow, grain refinement and homogenization are achieved with no grinding media.

Ceramic shaping process, such as slip casting,[2–12] requires the dispersion of ceramic powder in an aqueous solution. In general, ceramic slurries must satisfy the following conditions to produce dense green bodies: (1) low viscosity, (2) high solid content, and (3) good dispersion. A good ceramic slurry is achieved by strong electrostatic, steric repulsion and weak attractive interparticle van der Waals forces.[13,14]

Large green bodies with complicated shapes can be produced by slip casting processing, which has been widely utilized in the forming of traditional ceramics. In general, ball-mill process is widely used for preparation of ceramic slurries.[15,16] However, it is known that ceramic particles after ball-milling have tendency to re-flocculate by an increase of the attractive force which is caused by an increase of the active sites such as lattice defects induced on the surface of particles after ball-milling.[17] As a result, the re-flocculation causes an increase of viscosity and lead inhomogeneous structures of green bodies.[18]

In the present work, we demonstrate that $Al_2O_3$ slurries prepared by wet-jet mill process have properties of low viscosity and stability. Furthermore, green bodies fabricated by the wet-jet milled slurries have high green density without depending on solid contents of slurry, as comparing to compacts preparing by the ball-milled slurries.

## 2. Experimental procedures

### 2.1. Materials and preparation of slurries

A commercially available α-$Al_2O_3$ powder (AKP-20, 0.5 μm, Sumitomo Chemical, Japan) was used in the present work. A commercially available $NH_4^+$ salt of poly(acrylic acid) (PAA, Aron A6114, Toagosei Co., Ltd., Japan) was utilized as deflocculants to obtained a dispersed slurry.

### 2.2. Preparation of slurries

Aqueous suspensions containing 10, 30, and 50 vol.% of $Al_2O_3$ powder were prepared with 0.12 wt.% dispersant in distilled water. The suspensions were stirred for 2 h before milling process. After then, the suspensions were wet-jet milled at pressure of 200 MPa. Wet-jet milling (PRE03-20-SP; Genus, Japan)

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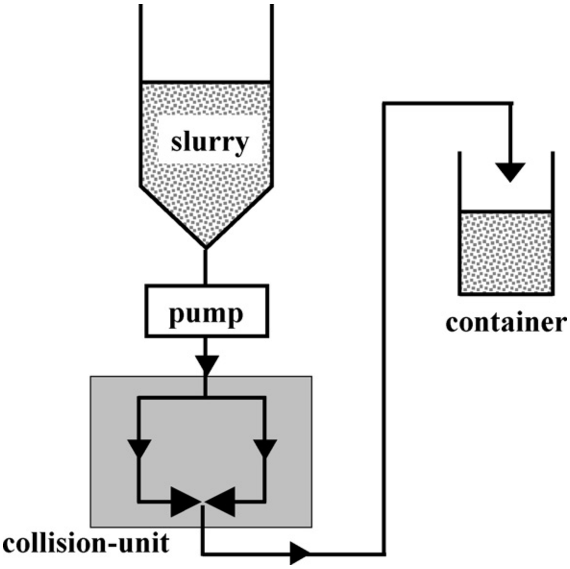


Fig. 1. Wet-jet milling system.

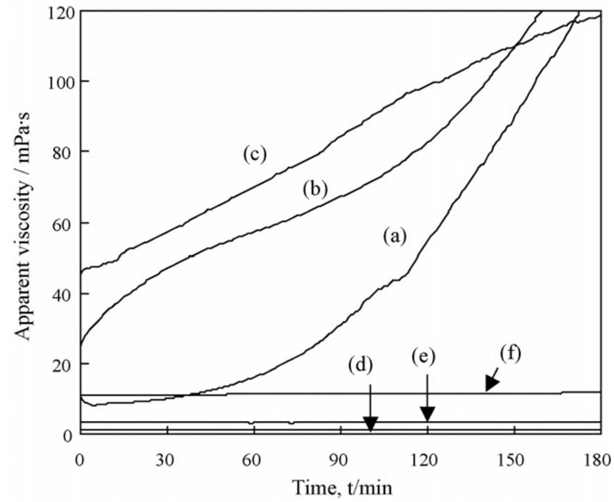


Fig. 2. Apparent viscosity of slurries with different solid contents prepared by (a)–(c) ball-milling and (d)–(f) wet-jet milling as a function of time. (a) and (d): 10 vol.%, (b) and (e): 30 vol.%, and (c) and (f): 50 vol.%.

system is shown in Fig. 1. The slurry was pumped into collision-unit at a pressure of 200 MPa, in which the particles in the slurry collide mutually. Then, the slurry was collected in a container. On the other hand, ball-milling process was performed for 24 h using high-grade $Al_2O_3$ balls with diameter of 10 mm.

The rheological characteristics of the slurries were measured using sin-wave vibro viscometer (SV-10, A and B, Japan) at 20 °C. The viscosity of slurries was calculated from the current value to move transducers put into the slurries by the frequency of 30 Hz and the amplitude of 0.2 mm.

### *2.3. Green bodies produced by slip casting and sintering*

The slurries were degassed before slip casting. The green bodies were formed in a gypsum mold (Wako Pure Chemicals Industries, Ltd., Japan). To prepare the gypsum mold, gypsum was added to water as 70 wt.% of the water content, and the gypsum slurry was stirred for 1 min, and then degassed under vacuum. The gypsum slurry was poured into a resin mold adjusted to a height of 2 cm and a diameter of 4 cm. The gypsum mold was dried in air at 40 °C before slip casting. The green bodies were dried at room temperature after slip casting and removed from the gypsum mold. Relative density of green bodies was estimated using Archimedes' method after calcination of 800 °C. The heating and cooling rates were both 100 °C/h. The sintered compacts were prepared at 1600 °C for 2 h in air.

## 3. Results and discussion

Fig. 2 shows apparent viscosity of slurries at different solid contents prepared by ball-milling (a)–(c) and wet-jet milling (d)–(f), as a function of time. The viscosity of the ball-milled slurries increases rapidly with time. In general, it is known that aggregated and flocculated slurry causes an increase of viscosity.[18] Hence, this rapid increase of viscosity indicates that the $Al_2O_3$ particles in the ball-milled slurries aggregate and flocculate. On the other hand, the viscosity of the wet-jet milled slurries is very low and constant without depending on the solid contents of slurry. Thus, it is found that the slurries produced by the wet-jet mill processing are stable as comparing to the ball milled slurries.

Slip casting was performed using the ball-milled slurries and the wet-jet milled slurries at different solid contents (10, 30, and 50 vol.%). Fig. 3 shows the relationship between square of the thickness of green body and casting time. The square of the thickness of the green body is proportional to casting time. The gradient of the straight line of wet-jet milled slurries is very lower than that of the ball-milled slurries. Furthermore, casting rate of the wet-jet milled slurries increases as increasing the slurry concentration, whereas that of the ball-milled slurries are not dependent on the solid contents. Generally, the casting

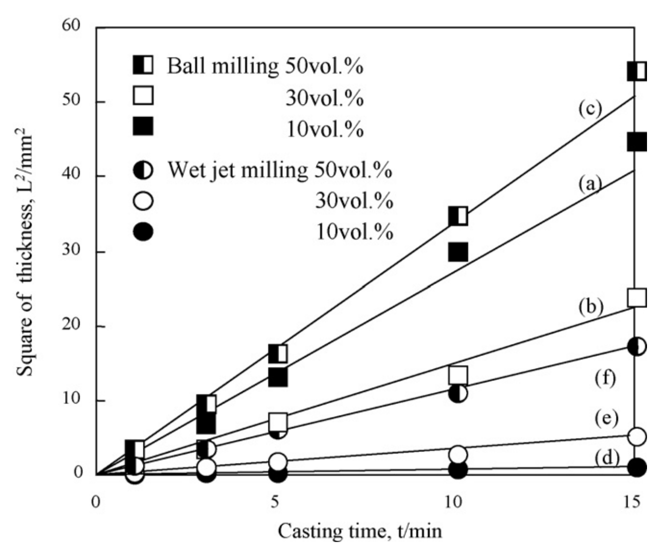


Fig. 3. Relationship between square of the thickness of green body and casting time using (a)–(c) ball-milled and (d)–(f) wet-jet milled slurries. (a) and (d): 10 vol.%, (b) and (e): 30 vol.%, and (c) and (f): 50 vol.%.

behavior can be quantified as a filtration process on the base of Darcy's law:[19]

$$Q = \frac{dV}{A\,dt} = \frac{k\Delta P}{L} \quad (1)$$

where $Q$ is the fluid flux (m/s), $V$ the filtrate volume ($m^3$), $A$ the filter area ($m^2$), $k$ the permeability of filter ($m^2$/Pa s), $\Delta P$ the pressure difference across filter (Pa), and $L$ is the thickness of the filter (m). From Eq. (1):

$$L^2 = (k\Delta P)t \quad (2)$$

where $t$ is the slip casting time. Eq. (2) indicates that the square of the thickness of green body formed by slip casting is proportional to the casting time. In all conditions, the square of the thickness of green bodies is proportional to casting time (Fig. 3). This result indicates that slip casting process is implemented well with the wet-jet milled and the ball-milled slurries. Moreover, the gradient of the straight line can be expressed by permeability of green body ($k$) and capillary pressure of gypsum mold ($\Delta P$):[20]

$$k = \frac{2E^3}{5S^2\rho\eta(y-1)(1-E)^2} \quad (3)$$

$$y = \frac{X}{V} \quad (4)$$

where $E$ is the porosity of green body, $S$ the specific surface area of raw material ($m^2$/g), $\rho$ the density of raw material (g/$m^3$), $\eta$ the apparent viscosity of solvent (Pa s) and $X$ is the volume of slurry containing volume $V$ of solids, i.e. $y$ is the reciprocal of slurry concentration. In this work, slip casting was carried out on the same conditions except the solid contents of slurry. This suggests that the casting rate of slip casting is influenced by only solid content and porosity of green body ($E$), i.e. the higher casting rate is caused by higher solid content and lower packing density of green body. Some parameters in the present work are given below:

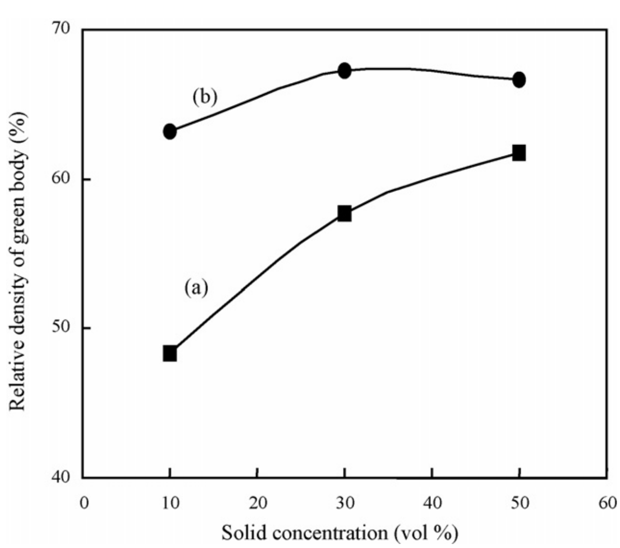


Fig. 4. Relative density of green bodies produced by (a) ball-milled slurries and (b) wet-jet milled slurries as a function of solid contents.

- specific surface area of $Al_2O_3$ particle ($S$): 4.0 $m^3$/g;
- density of $Al_2O_3$ ($\rho$): $3.98 \times 10^6$ g/$m^3$;
- viscosity of solvent ($\eta$): $1.1 \times 10^{-3}$ Pa s;
- capillary pressure of gypsum mold ($\Delta P$): $1.5 \times 10^5$ Pa;
- reciprocal of slurry concentration ($y$): 10.0 (10 vol.%); 3.33 (30 vol.%); 2.00 (50 vol.%).

The capillary pressure of gypsum mold ($\Delta P$) was calculated using Eq. (3) from the relative density of green body produced by stirred $Al_2O_3$ slurry. In this work, permeability of green bodies ($k$) produced by the wet-jet milled and the ball-milled slurries at different solid contents is $7.52 \times 10^{-15}$ and $3.31 \times 10^{-13}$ $m^2$/Pa s at 10 vol.%, $3.78 \times 10^{-14}$ and

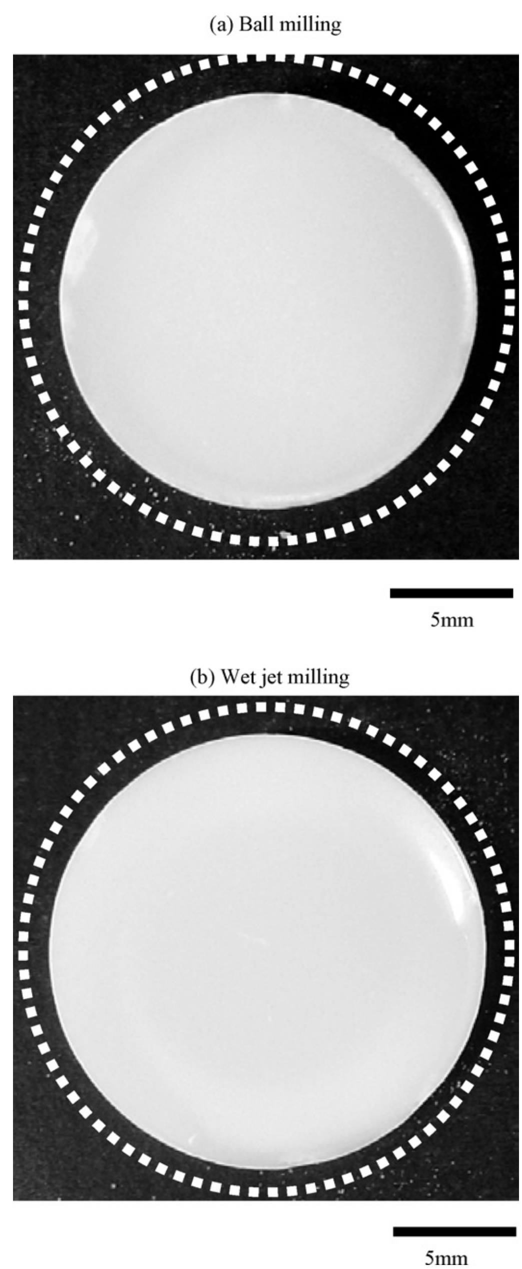


Fig. 5. Appearance photographs of the compacts, which are prepared from (a) ball-milled and (b) wet-jet milled slurries of 30 vol.%, sintered at 1600 °C for 2 h in air. The dotted lines express the size of green bodies before sintering.

$1.14 \times 10^{-13}$ m$^2$/Pa s at 30 vol.%, $1.29 \times 10^{-13}$ and $4.01 \times 10^{-13}$ m$^2$/Pa s, respectively. By substituting these parameters for Eq. (3), the porosity of green bodies fabricated by the wet-jet milled and the ball-milled slurries is estimated to be 0.288 and 0.643 at 10 vol.%, 0.313 and 0.445 at 30 vol.%, 0.340 and 0.443 at 50 vol.%, respectively. The green bodies fabricated by the wet-jet milled slurries have the same porosity approximately regardless of slurry concentration.

Fig. 4 shows relative density of green bodies as a function of solid contents. The relative density of green $Al_2O_3$ bodies prepared from the wet-jet milled slurries is very higher than that of prepared from the ball-milled slurries at all solid contents. Green bodies prepared from the wet-jet milled slurry with solid contents of 10, 30, and 50 vol.% have almost similar relative density of 63.2%, 67.2%, and 66.7%, respectively. Thus, the porosity of green bodies is 0.368, 0.328, and 0.333, respectively. These experimental values were agreement with the data calculated from Eq. (3). Hence, the casting rate is depended on the solid contents of slurry. On the other hand, since the relative density of green bodies fabricated by the ball-milled slurries increases with increase in the solid contents of slurries, the casting rate is influenced by not only the solid content of slurry but also the porosity of green body.

Fig. 5 shows appearance photographs of the compacts, which are prepared from 30 vol.% slurry, sintered at 1600 °C for 2 h in air. The circles drawn in dotted line express the size of compact before sintering. It is clearly seen that the shrinkage of sintered body produced using the wet-jet milled slurries (Fig. 5a) is smaller than that produced by the ball-milled slurries (Fig. 5b).

Fig. 6 shows linear shrinkage along the diametrical direction and relative density of sintered bodies as a function of solid contents. All the sintered bodies have similar high relative density of 95%. Linear shrinkages of sintered bodies produced by the ball milled slurries with 10, 30, and 50 vol.% solid contents are 20%, 17%, and 12%, respectively. The shrinkage is influenced by solid contents of slurry. On the other hand, shrinkage of sintered bodies fabricated by the wet-jet milled slurries is constant at 11% without depending on solid contents of slurries. Its shrinkage value is comparable to the case of the ball-milled slurry with 50 vol.% solid content. Viscosity of slurry produced by the wet-jet milling was constant as a function of time (Fig. 2) and the green bodies had high relative density (Fig. 4). Hence, particles in the wet-jet milled slurries have the characteristics for which it is difficult to re-flocculate and are well packed after slip casting without depending on solid contents, as comparing to the ball-milled slurry.

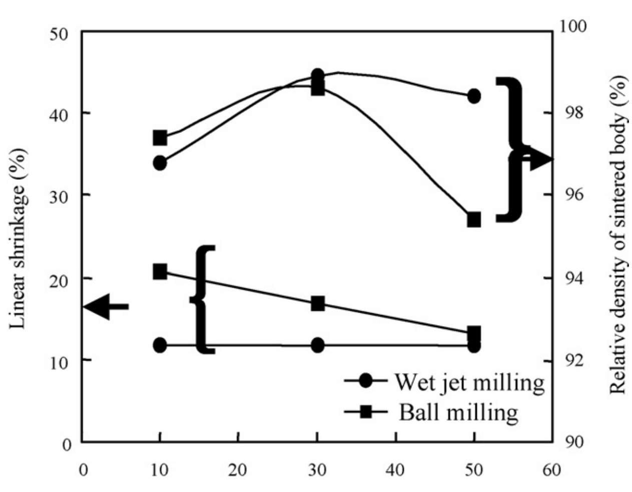


Fig. 6. Linear shrinkage along the diametrical direction and relative density of sintered bodies as a function of solid content.

## 4. Conclusion

Wet-jet milled slurries showed very low viscosity below about 10 mPa s and they were good stable for a long time, as comparing to the ball-milled slurries. The green bodies prepared from the wet-jet milled slurries had high relative density above 65% and they were independent with the solid contents of slurry. Moreover, the linear shrinkage during sintering at 1600 °C for 2 h was also same at 11% without depending on solid contents of slurry. These properties are very suitable for slip casting to produce a high dense green compact.

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