Characterization of $\text{Al}_2\text{O}_3$ Slurries Prepared by Wet Jet Milling

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Abstract: Al$_2$O$_3$ slurries with low viscosity and thixotropic properties which is suitable for slip casting were prepared within a short period of time by adopting wet jet milling process. The viscosity of wet jet milled slurry was about 6 mPa·s and remained unchanged for long time, while the viscosity of ball milled slurry rapidly increased with time and reached a maximum value. The FWHM of (113) XRD peak was the same before and after milling indicating no change in crystal structure by the process of milling. Particle size distribution of wet jet milled slurry corresponded to that of the ball milled slurry. However, wet jet milled slurry had low re-flocculation properties and high packing density (more than 60%) than ball milled slurry. We also found that the Al$_2$O$_3$ particles after ball milling yielded many hydroxy groups on the surface as compared with the raw materials and wet jet milled particles.

Key-words: Wet jet milling, Viscosity, Thixotropy, Particle size distribution, Sedimentation

1. Introduction

In general, ceramics is manufactured via a lot of processes such as pulverization of raw materials, dispersion, mixing, drying, shape forming, sintering and machining. Hence, long time and large energy are consumed to fabricate ceramic products. For example, the ball milling which is known to be most general process for pulverization, dispersion and mixing, requires 24 h or more to produce a stable characteristic slurry. In order to reduce the manufacturing cost of ceramics, it is important to decrease the preparation time of stable ceramic slurry.

Slip casting, which has been used in the forming process of traditional ceramics, is suitable to form compacts with high density and complex shape easily. Furthermore, in order to improve productivity and thickness of the cast cake, new processes such as pressure casting and centrifugal compaction have been developed. In these processes, the packing density and casting rate are strongly influenced by the slurry properties, such as viscosity, thixotropy, concentration, and dispersion. The slurry must satisfy the following conditions to produce high dense green bodies: (1) low viscosity; (2) low thixotropy; (3) high solid content; (4) good dispersion and (5) optimum particle size distribution.

Recently, in chemical engineering and food technology fields, the wet jet milling process has been demonstrated as a new method of mixing and dispersion. In this process, the particles in the suspension or solution collide each other at high pressure and high speed. In addition, the cavitation, turbulent flow and shear flow are induced by the high speed. By using the above mentioned effects, the grain refinement, emulsifying and homogenization are achieved within a short period of time.

The aim of the present work is to produce good slurries having low viscosity, low thixotropy and high solid content by wet jet milling for a short time. The properties of slurry, such as viscosity, particle size distribution and sedimentation behavior were characterized. We will show that the good slurry can be produced within a short time by wet jet milling.

Fig. 1. Schematic illustration of wet jet mill system.
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The characterization of Al₂O₃ slurries prepared by wet jet milling and ball milling was performed. The slurry was prepared by adding Al₂O₃ powder to deionized water and stirring for 24 h. The particle size distribution and viscosity of the slurry were measured.

2. Characterization

2.1 Viscosity

The viscosity of the slurry was measured using a Sine-wave vibro viscometer (SV–10; A&D, Japan) at 20°C. The viscosity of slurry was calculated from the current value to move transducers put into the slurry by the frequency of 30 Hz and the amplitude of 0.2 mm. The viscosity of wet jet milled slurry is independent on the number of milling times.

2.2 Analysis of Slurries

The rheological characteristics of the slurries were measured using a Sine-wave vibro viscometer (SV–10; A&D, Japan) at 20°C. The viscosity of slurry was calculated from the current value to move transducers put into the slurry by the frequency of 30 Hz and the amplitude of 0.2 mm. The viscosity of wet jet milled slurry is independent on the number of milling times.

3. Results and discussion

Figure 2 shows apparent viscosity of slurries prepared by wet jet milling and ball milling. The viscosity of wet jet milled slurry is found to be almost constant and lower than that produced by ball milling. The viscosity of wet jet milled slurry is independent on the number of milling times. On the other hand, the viscosity of ball milled slurry increases rapidly with time. It is known that the increase in viscosity of dispersed slurry is caused by re-flocculation of ceramic particles and the flocculated slurry has thixotropic property.

Thus, this rapid increase in slurry viscosity indicates re-flocculation of Al₂O₃ particles in the ball milled slurry. On the other hand, the viscosity of slurries prepared by wet jet milling showed very low and constant value indicating non-flocculation and low thixotropic properties.

Figure 3 shows the particle size distribution of wet jet milled and ball milled Al₂O₃ slurries. The particle size distribution of slurries prepared by wet jet milling is identical with that of ball milled slurry. Regardless of the number of milling times, average particle size is 570 nm as well as the primary particle size. This indicates that it is possible to pulverize raw materials till primary particle size by wet jet milling for only 1 time.

Figure 4 shows the appearance of slurries after sedimentation test. For the comparison of sedimentation test, the slurry stirred for 24 h was also carried out. It is seen that the ball milled slurry settles slightly. On the other hand, the wet jet milled slurries and stirred slurry are separated into two regions with the supernatant on the top and the sedimentation cake. The height of sedimentation cake of wet jet milled slurries is lower than that of stirred slurry. Table 1 represents the normalized height percent for wet jet milled, stirred, ball milled and wet jet milled slurries. The cake height of slurries prepared by wet jet milling is about 50% of initial slurry height. From this result, the packing densities of these cakes were estimated to be more than 60%. On the other hand, the packing density of cake produced by stirred slurry was estimated to be about 50%. It is known that the packing density of sedimentation cake increases with decrease in the amount MPa, in which the particles in the slurry collide each other. The slurry was passed into the collision-unit for 1, 3 and 5 times.

For the purpose of comparison, Al₂O₃ slurry was prepared by ball milling also. The ball milling was performed for 12 h using high grade Al₂O₃ balls with a diameter of 3 mm.

Table 1. Percentage of Cake Height to Initial Height of Slurry

<table>
<thead>
<tr>
<th>Method</th>
<th>Stirred</th>
<th>Ball mill</th>
<th>Wet jet mill</th>
</tr>
</thead>
<tbody>
<tr>
<td>Condition</td>
<td>24 h</td>
<td>12 h</td>
<td>1 time</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3 times</td>
<td>5 times</td>
</tr>
<tr>
<td>Normalized height (%)</td>
<td>58.8±0.1</td>
<td>97.7±0.1</td>
<td>47.7±1.8</td>
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<td></td>
<td></td>
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<td>48.6±2.1</td>
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of flocculated particles. This indicates that amount of flocculated particles in the wet jet milled slurry is lower than that in the stirred slurry. On the other hand, the cake height of ball milled slurry is about 98%. This is due to flocculated network structure. It is also reported by Tsetsekou et al. that the flocculated network structure impeded particle sedimentation.

Figure 5 shows X-ray diffraction patterns of Al₂O₃ particles before and after milling. All the specimens show similar diffraction pattern and show pure α-Al₂O₃ phase. Furthermore, the full width at half maximum of the (113) peak is same before and after milling (Fig. 5(b)). Generally, the peak of X-ray diffraction pattern is known to become broad by damage of crystal structures. This indicates that Al₂O₃ particles prepared by wet jet milling and ball milling are not damaged its crystal structure by milling.

Figure 6 presents FT–IR diffuse reflection spectra of Al₂O₃ particles before and after milling. Intense peaks are observed at around 3650 and 3550 cm⁻¹ in the spectrum of ball milled Al₂O₃, while less intense peaks are observed in raw Al₂O₃. These peaks are assigned to OH stretching vibrations (νOH). On the other hand, a broad band in the wave number range 3000–3500 cm⁻¹ is observed for wet jet milled Al₂O₃, and the peak intensities are found to be smaller than that of ball milled Al₂O₃. This result suggests that more OH groups are induced on the Al₂O₃ surface by ball milling, since the particle surface is damaged by high impact energy of the ball milling. We believe that the increase of OH groups on the surface of Al₂O₃ after ball milling affected the slurry properties such as viscosity, thixotropy and sedimentation. On the other hand, the surface of Al₂O₃ particles after wet jet milling is identical with raw Al₂O₃. This suggests that surface of particles is not damaged by wet jet milling and the interaction force between particles is weak. The weak interaction contributes to the low viscosity, low thixotropy and high packing density.

4. Conclusion

Wet jet milled slurry had very low viscosity and low re-flocculate properties compared to the ball milled slurry. Particle size distribution was identical for wet jet milled and ball milled slurries. Packing density of sedimentation cake produced by wet jet milled slurries was estimated to be more than 60% indicating less flocculation.

The crystal and surface structures of particles were not influenced before and after wet jet milling. On the other hand, the particles after ball milling had more OH groups on the surface which accelerates re-flocculation.

References


