Real-Time Slurry Characteristic Analysis During Ball Milling Using Vibration Data

Hyeondeok Jeong  
KICET: Korea Institute of Ceramic Engineering and Technology

Jungwon Yu  
ETRI: Electronics and Telecommunications Research Institute

Youngjae Lee  
ETRI: Electronics and Telecommunications Research Institute

Sung-min Lee  
KICET: Korea Institute of Ceramic Engineering and Technology

Sung-Soo Ryu  
KICET: Korea Institute of Ceramic Engineering and Technology

Seiki Kim  
kimseiki@kicet.re.kr  
KICET: Korea Institute of Ceramic Engineering and Technology

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Research Article

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Abstract

The characteristics of an internal slurry were analyzed during ball milling, which is commonly utilized in ceramic processing. We used a device with a capacity of 50 L because this is the size employed in industries, and built a circulation system to collect the slurry during the milling process. The properties of the slurry were characterized in terms of their particle size and viscosity, while vibration data were collected from the side of the ball mill drum in real time. A fast Fourier transform was performed on the vibration data, allowing the energy to be calculated and compared with the slurry characteristics. The vibration data in the 3–4 kHz range showed a strong negative correlation with the slurry viscosity. Our results confirm that the characteristics of the internal slurry can be monitored in real time using vibration data collected during ball milling.

1. Introduction

Concomitant with the advancements in data processing and machine learning technologies, big data are being increasingly used in the manufacturing field for process optimization and equipment maintenance [1–3]. The introduction of big data-based technologies requires high-quality data collected under various conditions. Consequently, such technologies are mainly applied in mass-producing industries, such as the semiconductor and automobile industries, which are led by large companies. Big data-based technologies are difficult to apply in industries led by small and medium enterprises, such as ceramics. Therefore, a technology capable of a unified method of data collection is desirable for big data-based production in the ceramic field.

The ball milling process is widely used in both industry and academia, for example, to pulverize or mix raw materials. Industries involved in mass production require large-scale ball milling equipment is required for mass production. Thus, shortening the ball milling time while maintaining the quality of the produced powder are important for reducing production costs, increasing productivity, and ensuring quality control. To this end, it is necessary to determine the optimum ball milling conditions to maximize its effectiveness and minimize the milling time by analyzing the change in the properties of the material during the milling process. However, field workers currently empirically determine the process conditions, and the process consumes more energy and time than necessary. Therefore, a method that can analyze changes in the properties of the slurry in real time during the milling process is required to optimize or manage the process.

Because the characteristics of the internal material cannot currently be determined during the ball milling process, process optimization studies have been conducted via ex situ characterization of the material properties, including the particle size and viscosity [4–7]. However, an ex situ analysis requires several experiments to optimize the milling process, and experimental errors are unavoidable because of the time difference between the acquisition of the slurry and the measurement. Conversely, simulations using the discrete element method have been performed to calculate the motion of the ball and raw material depending on the milling conditions [8–11] and to estimate the changes in the properties of the internal
material by collecting sound or vibration signals [12–18]. In particular, in the field of mineral production, process control through acoustic data monitoring has achieved the highest efficiency ball milling conditions using the acoustic signal strength [12–14]. These studies are meaningful in that they optimized and controlled the ball milling process using real-time data; however, these studies did not correlate the data with the actual material properties.

In this study, the changes in the particle size of the raw material and the viscosity of the slurry with the ball milling time were measured, and the properties were evaluated by preparing spray-dried granules from slurries milled for different lengths of time. In addition, vibration data generated during the ball milling process were collected in real time, and the correlation between the viscosity was analyzed. This method allows for the real-time analysis of the characteristics of the internal slurry, which vary during the ball milling process.

2. Materials And Methods

A ball milling process was performed to prepare a slurry by mixing Al₂O₃ (AES-11, Sumitomo Chem, Japan) and deionized water (DIW) with a dispersant (5468CF, San Nopco, Japan). In the milling process, alumina balls (60 kg) with a diameter of 10 mm, Al₂O₃ (13.6 kg), DIW (9 kg), and a dispersant (81.6 g, 0.6 wt.% of powder) were mixed in a 50 L ball mill for 24 h with a rotation speed of 40 rpm. Figure 1 shows an image and schematic of the milling equipment used in this experiment. A circulation path was constructed in the milling machine using rotary joints at both ends of the rotating shaft, and the slurry was obtained during the milling process. After 1, 4, 12, and 24 h of milling, approximately 100 ml of the slurry was obtained, to which 3 wt.% of a binder (HS-BD 20A, San Nopco, Japan) was added before being stirred for 1 h. Granules were then obtained by drying the slurries using a mini spray dryer (B-290, Büchi, Switzerland).

The analysis of the slurry's particle size and viscosity in terms of the ball milling time was conducted using a particle size analyzer (LA-350, Horiba, Japan) and a viscometer (DV2T, Brookfield, USA), respectively, and the properties of the granule powder were analyzed using a particle size analyzer and scanning electron microscopy (SEM, JSM-6390, JEOL, Japan).

During the ball milling process (Fig. 1b), a vibration sensor (352C33, PCB Piezotronics, USA), a DAQ (data acquisition, Ni-9234, National Instrument, USA) with chassis (cDAQ-9191, National Instruments, USA), and a battery pack were attached to the side of the ball mill to collect vibration data in real time. Vibration data were recorded using LabVIEW software (National Instrument, USA) for 10 s every 30 min at a sampling rate of 51.2 kS/s, giving a total of 48 data points collected over 24 h.

3. Results And Discussions

Figure 2 shows the variations in the particle size and viscosity of the alumina slurry with the milling time. The particle size variation results without ultrasonic treatment (Fig. 2a) clearly show dispersion of soft
agglomerations of the alumina powder during the milling process, indicating a rapid size reduction up to 8 h. Conversely, with sonication treatment, the particle size (Fig. 2b) did not significantly change (0.48 to 0.44 µm) based on the D50 value. This is because the milling process in this experiment was not designed to grind particles, but to disperse them in the DIW as a slurry for the spray dryer. The viscosity results based on the shear rate after 1, 4, 12, and 24 h of milling (Fig. 2c) show a shear thinning behavior, and the viscosity increases proportionally with the milling time when the shear rate is fixed at 24 s$^{-1}$ (Fig. 2d). The change in the viscosity with time increased rapidly up to approximately 4 h, after which it increased at a lower rate until the end of the milling process. Considering the observed particle sizes and viscosities (Fig. 2), the reason for the initial increase in the viscosity is likely the breaking of the soft agglomerations of the particles and their dispersion through the DIW. Conversely, after the initial rapid increase in the viscosity, the subsequent gradual increase seen can be attributed to the grinding of alumina particles and wear of the used balls.

Figure 3 shows the particle size analysis of the alumina granules subjected to spray drying with the slurry after 1, 4, 12, and 24 h of ball milling, hereafter referred to as G01, G04, G12, and G24, respectively. In the case of G01 and G04, particles similar in size to the raw powder were produced in addition to the granulated particles, indicating a bimodal particle size distribution. This occurs because the particles in the slurry are not sufficiently dispersed [19–21], as indicated by the variations in the particle size and viscosity (Fig. 2). Conversely, G12 showed a granule size of approximately 18 µm, larger than those of G01 and G04, and no fine particles of less than 1 µm were observed. Although G24 did not produce fine particles, the particle size was lower than that of G12. The increased viscosity as milling continues is thought to reduce the fluidity of the slurry, which reduces the feeding rate of the slurry and droplet size under the same spray dry conditions.

Figure 4 shows the SEM images of the surface morphology and polished cross section of the granules after impregnation in a polymeric resin. The G01 powder includes a number of irregularly shaped particles, such as dimpled and hollow particles, instead of spherical granules (Fig. 4a). These atypical granules readily break down to produce fine particles (Fig. 3). Conversely, the number of irregularly shaped particles decreases with increasing milling time of the slurry (Fig. 4b–4d), because the Al$_2$O$_3$ raw powder is better dispersed through the DIW with longer milling times, causing homogeneous shrinkage during drying.

During the ball milling process described above, the vibration signal was collected from the side of the ball mill drum. Before collecting the vibration data during the actual process, the data were collected from operations with only balls, balls + DIW, and balls + slurry (DIW + powder), as shown in Fig. 5, to determine the cause of the observed signals. The intensity of the raw vibration data decreased in the order of balls + DIW (Fig. 5b), balls + slurry (Fig. 5c), and only balls (Fig. 5a) because the propagation of vibrational energy to the drum wall is greater through a medium such as DIW than through air, and the vibration is attenuated with the balls + slurry mixture owing to the powder. Moreover, strong signals were periodically observed in the vibration data of the three different cases, indicated by the red arrows in Fig. 5a–5c. These periodic signals are observed at approximately 0.6 Hz in the fast Fourier transform (FFT) data.
(Fig. 5d–5f), which corresponds to a rotational speed of 40 rpm, indicating that the periodic vibrations originate from the equipment, i.e., due to friction, whenever the ball mill rotates. Excluding the signal caused by the rotation of the ball mill, the case where milling is done with only the ball (Fig. 5d) shows high vibrational intensity in the 0–7 kHz range. Conversely, when fluids such as DIW or slurry are included in the milling process (Fig. 5e and 5f), the vibrational intensity in the 7–10 kHz range is high. It can be inferred that the signal due to the collision of the balls appears in the range of 0–7 kHz, while the signal due to the fluid appears in the range of 7–10 kHz. In addition, when there a fluid is present, the vibration signal in the 0–7 kHz range caused by the balls is attenuated. Because the overall intensity is lower when the fluid is a slurry rather than water, the attenuation is greater in the case of slurry than in the case of water.

Figure 6 shows the vibration data collected at 1 and 24 h after ball milling. The raw vibration data (Fig. 6a) shows no significant difference over time, which is similar to the result shown in Fig. 5c, in which no milling was performed. After FFT (Fig. 6b), the shape was again similar to that shown in Fig. 5f, which was obtained before milling, and a signal due to drum rotation was detected at 0.6 Hz. On the other hand, there is a difference with respect to the milling time in the data after FFT, which is the reduction in the signal intensity in the 2–7 kHz region.

To analyze the energy change with respect to the vibration frequency, the frequency band was divided into intervals of 1 kHz. The energy changes in each band are shown in Fig. 7. The energy was calculated by summing the power values taken from the power spectrum obtained from the vibration data after FFT. The calculated energies showed three different characteristics in the 0–2, 2–7, and 7–10 kHz frequency ranges. In the 0–2 kHz region, the energy tends to increase; in the 2–7 kHz region, the energy gradually decreases after an initial rapid reduction in energy, and in the 7–10 kHz region, irregular energy changes with high deviation are observed, before the energy decreases after approximately 12 h of milling. The change in energy evidenced by the signal in the 2–7 kHz range was inversely proportional to the change in viscosity shown in Fig. 2d.

The vibrational signals in the low-frequency region of 0–7 kHz are due to the collision of the balls; thus, the reduction in the intensity is assumed to be caused by the buffering action of the increased slurry viscosity on the collisions of the balls. Conversely, the signals in the 7–10 kHz region, which are thought to be caused by the propagation of the ball collision signal through the slurry, are complex because the energy change is affected by the collision energy of the ball and the propagation characteristics of the vibration signal are changed according to the viscosity of the slurry [22, 23].

Figure 8a shows a scatter plot of the viscosity change (in Fig. 2d) and the vibration energy in the 3–4 kHz range (in Fig. 7) as a function of the milling time. The vibration energy in the 3–4 kHz range shows the highest Pearson correlation coefficient of −0.951 with respect to the viscosity. However, the other vibration energies in the 2–3, 4–5, and 5–6 kHz ranges, which showed similar trends, also showed high correlation coefficients of −0.898, −949, and −898, respectively. The correlation coefficients in the frequency ranges of 0–1 and 9–10 kHz were low at only 0.666 and −0.126, respectively. This suggests
that the viscosity of the internal slurry can be determined from the vibration energy in a specific frequency range. Energy data corresponding to the 3–4 kHz range were also obtained from additional milling experiments with only balls or balls and DIW at the same rotation speed of 40 rpm for 12 h (Fig. 8b), to determine whether the energy change is due to the slurry. Because the absolute energy values are different in terms of the scale, the data were normalized by dividing them by the initial value to express the change from the initial energy. In the case of operation with only the ball or ball and DIW, the energy value slightly decreased, which was assumed to be due to the presence of alumina particles generated by the friction between the balls. However, there was no sudden change, as in the case involving the slurry, which indicates that the change in the vibration energy generated during the ball mill process is due to the slurry. Additionally, although the normalized vibration energies for the three repeated experiments under the same conditions were not completely consistent (Fig. 8c), the results showed similar trends in which the energy decreased rapidly and then decreased at a lower rate. These results indicate that the data are reproducible and can be used to determine the slurry viscosity during the milling process.

4. Conclusions

This study analyzed the correlation between vibration data collected in real time and the characteristic changes in the slurry with the ball milling time. The results can be summarized as follows.

1. During ball milling, the particle size decreased, and the slurry viscosity increased. The properties of the slurry significantly influenced granule production in the spray drying process.
2. By collecting vibration data in real time and calculating the energy for each frequency band, a specific frequency range was identified that is strongly correlated with the viscosity data.
3. Three additional experiments were conducted under the same conditions, and the data in the aforementioned frequency range, which had a strong correlation with the viscosity, showed a similar trend.
4. The characteristics of the slurry can be monitored during ball milling using vibration data, which allows the optimum milling conditions and time to be determined.

The results of this study show that the viscosity of the slurry can be inferred during the ball milling process without directly measuring it. It is expected that the same analysis will be possible for other properties such as particle size and slurry load, which will form the basis of our future work in this area.

Declarations

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Figures

![Image and Schematic](image.png)

Figure 1

(a) Image and (b) Schematic of the milling equipment used in this study
Figure 2

Particle size of the powder in terms of the milling time (a) without sonication and (b) with sonication; slurry viscosity under different (c) shear rates and (d) milling times
Figure 3

Particle size distributions of G01, G04, G12, and G24 powders
Figure 4

Surface morphology and polished cross-sectional SEM images of (a) G01, (b) G04, (c) G12, and (d) G24 granules

Figure 5

Various vibration data (a–c) as raw state and (d–f) after FFT milling with ball only, ball + DIW, and ball + slurry, respectively
Figure 6

Vibration data collected during ball milling at milling times of 1 and 24 h: (a) raw data and (b) data after FFT
Figure 7

Vibration energy change in each frequency band with respect to the milling time

Figure 8

(a) Scatter plot of the viscosity change and vibration energy in the 3–4 kHz range, (b) Vibration energy change in the 3–4 kHz range with the milling time, and (c) Results of three more replica runs for the
vibration energy change in the 3–4 kHz range with the milling time.