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Effect of ball size and powder loading on the milling efficiency of a laboratory-scale wet ball mill

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Abstract

Alumina powder was wet-milled by zirconia balls with varying diameter at varying rotation speed, and the resultant particle size of the milled powder was analyzed. At a given rotation speed, there exists an optimum ball size to yield minimum particle size of alumina. The optimum ball diameter decreases as the rotation speed increases. This result has been interpreted in light of the competition between the reduced kinetic energy of the smaller balls (a negative source for milling efficiency) and the increased number of contact points of the smaller balls (a positive source), which yields the optimum ball diameter at an intermediate size. As the rotation speed increases, kinetic energy of the balls increases, which, in turn, shifts the optimum ball size toward a smaller value. As the powder loading increases from 1 to 35 g at a given rotation speed and ball size, the milling efficiency decreases monotonically.

Keywords: Ball size; Particle size; Powder loading; Rotation speed; Wet ball mill

1. Introduction

Wet ball mill is one of the most predominantly used method for the purpose of mixing and grinding of raw materials in laboratories and industry [1–3]. The ball mill process is very complicated process governed by many parameters, such as ball size, ball shape, ball filling, slurry loading (with respect to ball amount), powder loading with respect to the amount of total slurry (slurry viscosity), and rotation speed. There has been a high industrial interest in optimizing such ball mill parameters from the viewpoint of the comminution of ores [1]. In ceramic laboratories, ball mill is often carried out to mainly achieve a thoroughly mixed state of starting powders with initial average particle size (d_{50}) of 1–20 μm. In laboratories, a polyethylene-based small bottle container (nominal volume of approximately 250 ml) and zirconia balls with nominal diameter of approximately 1–10 mm are frequently employed. However, reports on the efficient combinations of ball size—rotation speed have been sparse in the literature. Here we report that there exists an optimal ball size for efficient milling at a given rotation speed, based on a laboratory-scale wet ball mill. Also, the effect of powder loading on the particle size reduction has been investigated at given conditions of ball size and rotation speed.

There have been investigations on the influences of parameters associated with grinding balls such as ball size distribution [4,5] and ball shape [6] on the particle comminution. Salili et al. [7] reported the importance of the small ball size and small mass ratio of powder to ball for the efficient ball mill, while only two sets of experimental data for these variables were provided. Thus, the optimization of ball size could not be pursued. In the experimental work of Shinozaki and Sennai [8], the stored energy per gram of milled powder

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(gamma Fe_2O_3), ΔH_s, was given by $24.2N(d/D)^{2.57}$, where N is the number of steel balls, d is the diameter of ball, D is the diameter of the milling pot. In the modeling work of Kurlov and Gusev [9], it was shown that a fraction of the energy deposited in the processed material is consumed for the creation of microstresses, which slows down the comminution process.

As for the influence of the characteristics of wet medium on the milling efficiency, several groups [10] investigated the influence of liquid physical properties such as density, surface tension and viscosity on the rate of grinding, while some other groups [11] investigated the effect of bulk slurry rheology on the efficiency of grinding. There also have been efforts to investigate the kinetics of particle breakage [12] and modeling of the grinding process [13]. The particle breakage rate increases with milling time [12].

2. Experimental procedure

10 g of alumina powder (99.6%, CA-5M, KC Corp., Youngam, Jeonnam, Korea) with average diameter of 6.0 μm (d_{50}) was loaded to a polyethylene-based bottle (approximately 60 mm in inner diameter and 250 ml in nominal volume) with 500 g of zirconia balls and 70 ml of distilled water. The 500 g of balls reached approximately 50% of the bottle height regardless of the ball size due to the same packing factor of the randomly packed different-sized spherical balls [14]. The 50% ball filling of mill container was efficient for particle breakage in an existing work [15]. The total volume of the balls, Al_2O_3 powder, and water reached approximately 60% of the bottle height. Five types of ball diameter were used: nominal diameters of 1, 2, 3, 5, and 10 mm. By using the balls with each size, ball mill was carried out for 12 h at three different rotation speeds: 50, 100, and 153 rpm. These speeds are 29.0%, 57.9%, and 86.9% of the critical speed given by the relation, $N_c = 42.29D^{-1/2}$ [16], where N_c is the critical rotation speed (in rpm) and D is the inner diameter of the ball container (in meter). The rotation of the bottle was carried out along the central axis of the bottle in height direction. Average particle size of the milled powder was measured by a particle size analyzer (Model S3500, Microtrac, Largo, FL, USA). For the Al_2O_3 powder after milling at optimal conditions (rotation speed and ball size), the degree of contamination of ZrO_2 by zirconia balls was measured by using wavelength-dispersive X-ray fluorescence (XRF) spectrometer (Model Tiger S8, Bruker, Kalkar, Germany).

For the investigation of the effect of powder loading on the milling efficiency, varying quantity (5–35 g) of the same alumina powder was loaded to the same mill containers with 500 g of zirconia balls. The same quantity of distilled water (70 ml) was added as before. Ball milling was performed for 12 h at 153 rpm and 100 rpm for the ball diameters of 2 and 3 mm, respectively. The powder loading to the total volume of slurry was 1.8%, 3.5%, 5.1%, 6.7%, 8.3%, 9.8%, and 11.2%, respectively, for the powder loadings of 5, 10, 15, 20, 25, 30, and 35 g, respectively. The powder filling factors to the ball interstitial space were approximately 2.5%, 5.1%, 7.6%, 10.1%, 12.7%, 15.2%, and 17.7% for the powder loadings of 5, 10, 15, 20, 25, 30, and 35 g, respectively (here we used the porosity value of 0.4 for balls according to Ref. [12] to determine the powder filling factor to the ball interstitial space).

3. Results and discussion

3.1. Effect of ball size

The effect of ball size on the particle size reduction has been investigated first for varying rotation speed of the container. Percent passing and size distributions of the milled Al_2O_3 powder are shown in Figs. 1 and 2, respectively, as a function of particle size for varying ball size. The average particle sizes (d_{50}) of the milled Al_2O_3 powder are shown in Fig. 3 as a function of ball size for varying rotation speeds.

In Fig. 3, as anticipated, at a given ball size, a higher rotation speed (rpm) yields a finer average particle size due to the higher number of rotations within a given period of milling time (12 h). Interestingly, at each rotation speed, there exists an optimal ball size at which the most reduced average particle size is achieved among other investigated ball sizes. The ball diameter of 5 mm is optimal at 50 rpm of rotation speed (the resultant average particle size of the milled powder is 2.3 μm), ball diameter of 3 mm is optimal at 100 rpm (1.4 μm), and

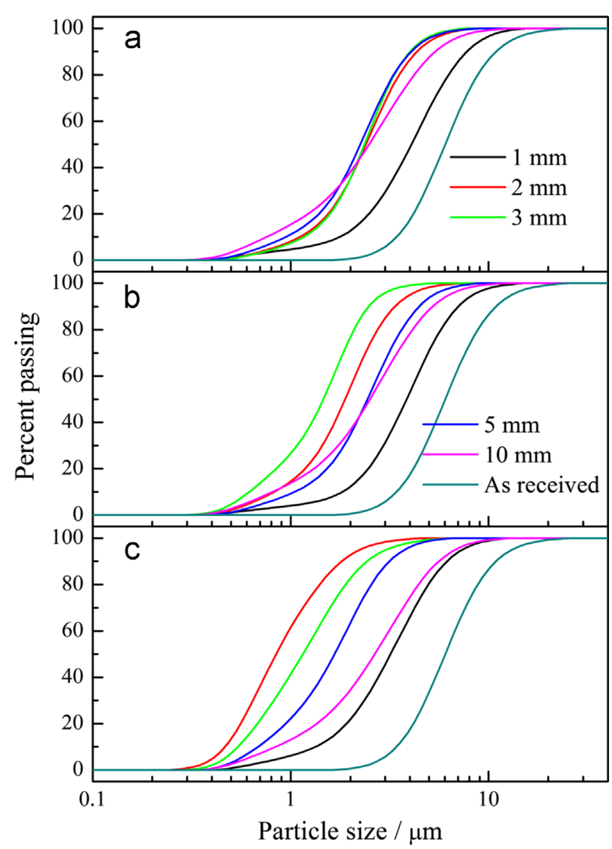

Fig. 1. Percent passing of milled Al_2O_3 powder as a function of particle size for varying ball diameter. Rotation speeds are (a) 50 rpm, 100 rpm, and (c) 153 rpm.

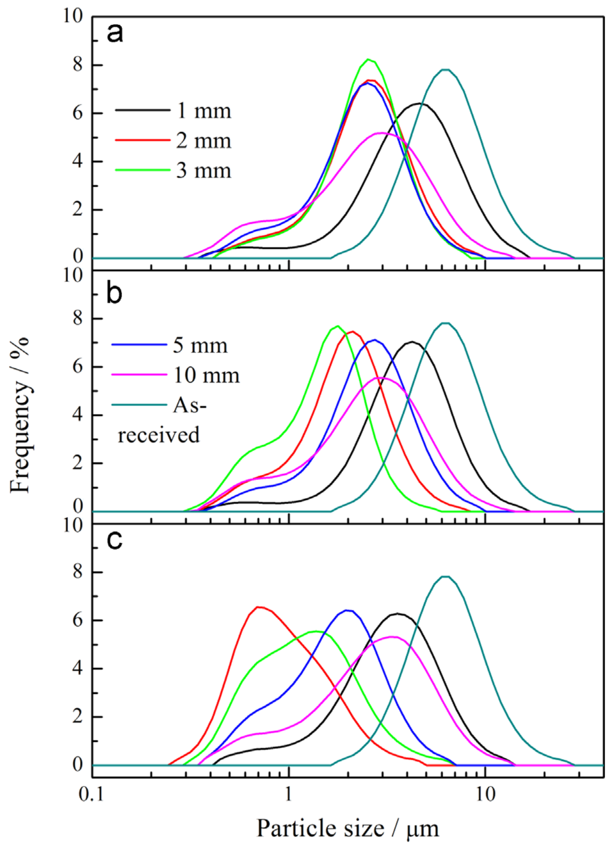

Fig. 2. Particle size distributions of milled Al_2O_3 powder for varying ball diameter. Rotation speeds are (a) 50 rpm, (b) 100 rpm, and (c) 153 rpm.

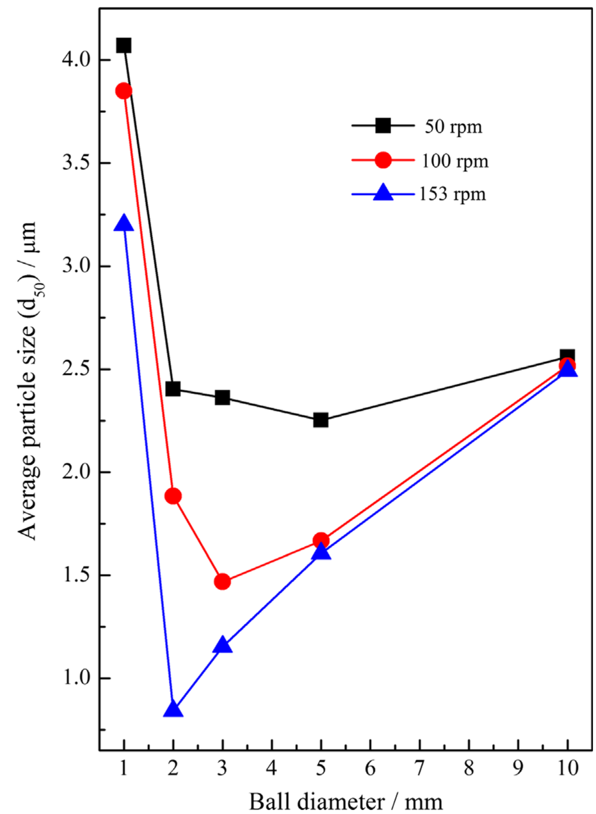

Fig. 3. Changes in average particle size (d_{50}) of milled Al_2O_3 powder for varying rotation speed (rpm) as a function of ball diameter.

2 mm is optimal at 153 rpm (0.84 μm). From this result, it can be said that the optimal ball size for efficient milling decreases with the rotation speed of the mill.

The particle size reduction results from the complicated dynamic interaction of the balls with turbulent slurry during the ball mill process, imposing a difficulty in quantitative interpretation of the observed phenomena of: (1) the existence of the optimized ball size at a given rotation speed; and (2) the decreased optimum ball size with increased rotation speed (rpm). As seen in Fig. 4, particle breakage may take place by the collision of balls in many places. It is believed that the collision between newly falling/entering balls to the slurry and the balls existing already in the slurry medium (marked as A in Fig. 4) would be most efficient for particle size reduction, while the ball collision between the balls existing in the turbulent slurry medium (marked as B in Fig. 4), that between the balls moving up along the container wall (marked as C in Fig. 4), and that between the balls falling down after reaching maximum height before entering the turbulent slurry (marked as D in Fig. 4) would also contribute to the particle size reduction. If the container wall were refractory material unlike the case herein (a soft polyethylene-based material), the collision between the balls and the refractory container wall would also be one of the efficient places of particle breakage.

In order to explain the observed phenomena in the present study, here we consider the case of the collision between newly falling/entering balls to the slurry and the balls existing

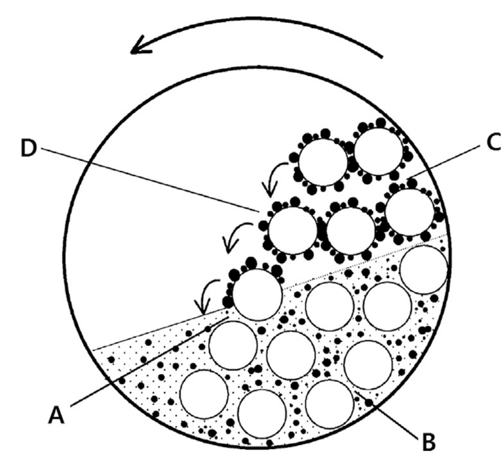

Fig. 4. Schematic cross sectional view of the ball mill process. Adapted from Ref. [12], and modified in the present study.

already in the slurry medium (marked as A in Fig. 4), which would be the most efficient source of the particle size reduction in the present study. A qualitative explanation for the observed phenomena is pursued hereinafter for the case of the collision at A. Among many conceivable factors influenced by the change of ball size, here we consider the changes in: (1) the total number of contact points between balls; and (2) kinetic energy of the colliding balls, both by the change in ball size.

The number of balls (n) in a given total mass (500 g in the present study) increases with reduced ball size (d) by the relation, $n \propto d^{-3}$. The number of contact points for a given ball (coordination number, CN) depends on the mode of packing: CN=6 for simple cubic packing with packing factor of 0.52, CN=8 for body centered cubic packing with packing factor of 0.68, and CN=12 for face centered cubic packing and hexagonal close packing with packing factor of 0.74, and $6 < CN < 8$ for random packing of sphere based on the packing factor of 0.6 [14] or the theoretical limit of 0.634 [17]. The coordination number is independent of ball size. Thus, the total number of contact points (N) in a given total mass (500 g) of balls can be regarded as being proportional to the number of balls (n),[1] which leads to the reasoning that the total number of contact points (N) increases with reduced ball size: $N \propto n \propto d^{-3}$. As the particle breakage certainly takes place mostly at the contact sites of the balls, smaller balls possess a positive influence to improve the milling efficiency because of the simply increased number of contact points between balls. This qualitative relation is illustrated conceptually in Fig. 5. In this figure, the positive influence of the smaller ball size on the milling efficiency is drawn by an arbitrary negative slope with ball size rather than the dependency of d^{-3}, because the dependency of d^{-3} is valid only in a stationary state of ball packing while the actual ball milling is a dynamic event taking place under the presence of the powder slurry existing between the balls. Actually the increased number of contact points by the reduced ball size is not limited to the case of collision at A. It increases the milling efficiency at B, C, and D as well.

For the analysis of the change of kinetic energy of the colliding balls (at A) by the change of ball size, one needs to consider the changes of ball mass and colliding velocity. The mass (m) of individual balls is proportional to the ball size by the relation, $m \propto d^3$. Thus, the smaller diameter certainly reduces the mass of individual balls.

As for the change of collision velocity by the change of ball size, note that, after the balls reach a maximum height which is fixed at a given rotation speed (Fig. 4), they fall to enter the turbulent slurry medium and experience sedimentation thereafter. The sedimentation speed is greatly reduced from the falling speed due to the drag force of the viscous slurry medium. Without the hindrance of other balls existing already in the slurry, the sedimentation velocity of the newly entered balls will certainly decrease until the sedimentation velocity reaches a terminal velocity [19]. It is true that the balls entered to the slurry medium collide with other balls existing already in the slurry medium before the newly entered balls reach the terminal velocity. However, the relation between the ball diameter and the terminal velocity will be informative to understand the collision velocity of balls in terms of their size, because a higher terminal velocity will yield a higher collision velocity between the balls. In a turbulent flow where Reynolds number is high, e.g. >2100, the terminal velocity (v_t) of falling balls in a viscous medium is proportional to the square root of ball diameter ($v_t \propto d^{1/2}$) [20,21]. Thus, the smaller diameter certainly reduces the collision velocity.

[1]The analytical expression for the total number of contacts varies depending on the number of balls [18].

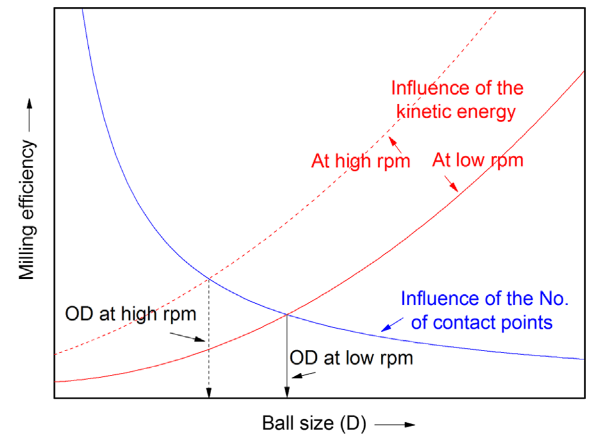

Fig. 5. Conceptual diagram illustrating the positive influence of the smaller balls (number of contact points) and the negative influence of smaller balls (collision velocity) on the milling efficiency. OD denotes optimum diameter of the balls.

By combining the mass dependency on ball size ($m \propto d^3$) with that of collision velocity ($v_t \propto d^{1/2}$), the kinetic energy ($KE = 1/2\, mv^2$, v is the collision velocity which is proportional to the terminal velocity v_t) of the colliding balls is proportional to the ball size by the relation, $KE \propto d^4$. Thus, the smaller balls will yield a reduced kinetic energy of collision to the balls existing already in the slurry medium, which will certainly decrease the milling efficiency. The reduced kinetic energy of the smaller balls is a negative influence of the smaller balls on the milling efficiency. This qualitative relation is drawn in Fig. 4 with the source of positive influence (increased number of contact points) of the smaller balls as aforementioned. In this figure, the negative influence of the smaller ball size on the milling efficiency is drawn by an arbitrary positive slope with ball size rather than the dependency of d^4, because in an actual dynamic ball mill situation, the balls already existing in the turbulent medium is moving rather than in stationary state, and thus the effect of the kinetic energy of the sedimenting balls on the milling efficiency would not be simply given by the dependency of d^4.

From above considerations, the experimentally observed phenomenon that there exists an optimal ball size for efficient milling at a given rotation speed (rpm) is interpreted to be resulted from the competition between the positive influence of a smaller ball size (increased number of contact points) and the negative influence of the smaller balls (decreased kinetic energy of the sedimenting balls). The best (optimal) milling efficiency will then appear at an intermediate ball size (Fig. 5). In Fig. 5, the optimum diameter was assumed to be the ball diameter at the intersection of the positive and negative influences.

As for the reason why the optimal ball size for efficient milling decreases with the rotation speed of the mill, it is first conceived that the higher rotation speed will increase the average height of the balls from which the balls start to fall down, unless the balls rotate simultaneously with the container (centrifugation) due to an overly high rotation speed. Then, the entering (falling) speed of the balls into the surface of the turbulent slurry medium will increase. This increased initial velocity of the balls in the slurry medium will lead to a higher collision velocity to the balls already located in the slurry medium, unless the terminal velocity of the balls is reached before the collision. The increased collision velocity of the balls at higher rotation speed will certainly increase kinetic energy of the sedimenting balls at a given size of the ball (Fig. 5), which attenuates the negative influence of the smaller balls, thereby yielding the optimal ball size at a lowered value.

Although the current work seeks optimal ball size for efficient milling at varying rotation speed, the degree of ZrO_2 contamination by the zirconia balls at the efficient milling conditions (optimal ball size at a given rotation speed) is of interest from the viewpoint of reserving the original composition of powder after the milling. Based on XRF analysis, the amounts of ZrO_2 in the milled Al_2O_3 powder at the condition of 2 mm-153 rpm was 3.69 wt%, while no ZrO_2 was identified in the as-received Al_2O_3. 'From this result, it is acknowledged that the zirconia balls used in the current study was a low-quality balls, but the conclusion of the current work on the efficient milling condition will not be changed. As the efficient milling is always associated with the increased contamination by grinding balls, care has to be taken to check the contamination especially when the conditions of the efficient milling are employed. Investigation on the optimal milling conditions for other material types of balls and the resultant degree of ball contamination was not carried out in the current work, while such an investigation will be necessary as a future work.

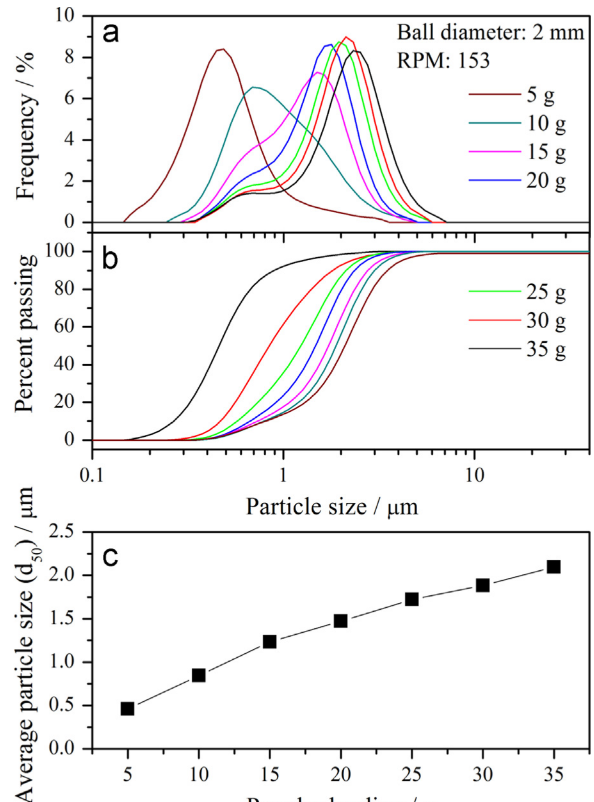

Fig. 6. Particle size distribution (a), percent passing (b), and average particle size (c) of milled powder for varying powder loading. Ball size and rotation speed are 2 mm and 153 rpm, respectively.

3.2. Effect of powder loading

Fig. 6 shows the change in average particle size (d_{50}) of the milled powder as a function of powder loading when the milling parameters are 2 mm in ball diameter (the optimum ball size at 153 rpm) and 153 rpm in rotation speed. The case when the milling parameters are 3 mm in ball diameter (the optimum ball size at 100 rpm) and 100 rpm in rotation speed is shown in Fig. 7. In both cases, the average particle size of the milled powder increases monotonically up to 35 g of powder loading. Such decreasing milling efficiency with increased powder loading observed in two sets of experiments herein (Figs. 6 and 7) is consistent with the observation of Tangsathitkulchai [12] who observed that particle breakage rate was diminished at a higher solid loading (up to 56%) to the total volume of slurry.

For the interpretation of the decreasing trend of the milling efficiency with powder loading observed in the current work, at least two physical origins can be conceived of. First, the increased powder loading will increase the viscosity of the slurry. It is known that the viscosity is linearly proportional to the powder loading up to a critical powder loading at which the viscosity increases abruptly. The critical powder loading is approximately 40 vol% of the slurry for the case of limestone–water slurry [22]. The increased viscosity herein due to the increased powder loading will then reduce the collision velocity of the balls in the slurry due to the increased drag force. The increased viscosity decreases the Reynolds number, which increases the tendency of laminar-type flow (the degree of turbulence is decreased) where sedimentation velocity is inversely proportional to the viscosity of medium [20,21]. Thus, the collision velocity will be reduced by the increased viscosity (increased powder loading), resulting in a decreased milling efficiency with powder loading as seen in Figs. 6 and 7. Second, an increased powder loading will increase the probability that multiple particles are loaded between the colliding balls. The multiple particles with water in inner spaces will show a kind of cushion effect, reducing the milling efficiency at a higher powder loading.

4. Conclusions

10 g of alumina powder (average diameter of 6.0 μm) was ball milled in aqueous medium (70 ml of distilled water) by 500 g of zirconia balls with varying diameter (1, 2, 3, 5, and 10 mm) in polyethylene bottles (500 ml) for 12 h at varying rpm, and the resultant particle size of the milled powder was analyzed. At a given rpm, there exists an optimum ball size to yield minimum particle size. The optimum ball diameter decreases from 5 to 2 mm as the rpm increases from 50 to 153. This result has been interpreted in light of the competition between the reduced kinetic energy of the colliding balls in the slurry (a negative source for milling efficiency) and

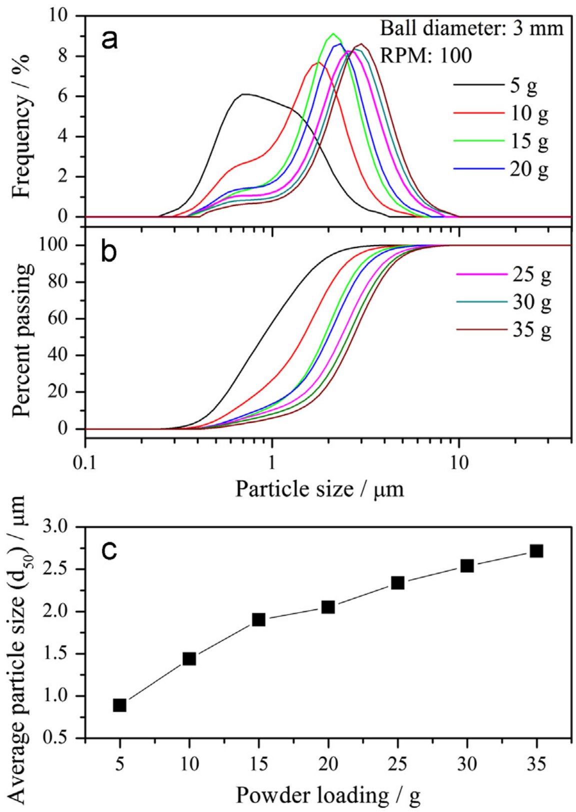

Fig. 7. Particle size distribution (a), percent passing (b), and average particle size (c) of milled powder for varying powder loading. Ball size and rotation speed are 3 mm and 100 rpm, respectively.

the increased number of contact points of the smaller balls (a positive source for milling efficiency), which yields the optimum ball diameter at an intermediate ball size. As the efficient milling is always associated with the increased contamination by grinding balls, care has to be taken to check the contamination by the balls especially when the conditions of the efficient milling are employed. As the rotation speed (rpm) increases, kinetic energy of the balls increases, which, in turn, shifts the optimum ball size toward a smaller value because the negative influence is alleviated. As the powder loading increases from 1 to 35 g at a given rotation speed (rpm) and ball size, the milling efficiency decreases monotonically. This phenomenon is interpreted to be resulted from (1) the increased viscosity, and (2) the increased cushion effect of the multiple particles existing between the colliding balls, with the increase of powder loading.

Acknowledgments

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