



## Noisy-power dissipation and efficiency in NiO/Fe<sub>2</sub>O<sub>3</sub> wet grinding by planetary ball mill

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**Abstract:** The relationship between the efficiency of NiO/Fe<sub>2</sub>O<sub>3</sub> wet grinding and noisy-power dissipation was studied. The optimal grinding parameters were found as: a slurry water content of 64.10%–85.47%, ball number ratio of 360/20, revolution speed of 300.9 r/min, powder-filling ratio of 10.88%, ball-filling ratio of 20.53%–23.88%, and grinding time of approximately 6 h. The discrete element method (DEM) was employed to analyze relationship between the noisy-power dissipation and the grinding efficiency, and equations describing the relationship were derived. The mean particle size of the ground powder decreased with a decrease in the degree of noisy-power dissipation, while the grinding efficiency and the amount of specific impact power used decreased with an increase in the degree of noisy-power dissipation.

**Key words:** noisy-power dissipation; NiO/Fe<sub>2</sub>O<sub>3</sub> powder; planetary ball mill; mean particle size

### 1 Introduction

Interest in alloy and cermet materials for fabricating inert anodes for use in industrial aluminum electrolysis has increased in recent years because of their significant advantages in terms of energy, cost, productivity, and environmental benefits [1–8]. NiFe<sub>2</sub>O<sub>4</sub>-based cermet is one of the most promising materials studied in recent years, due to its high electrical conductivity and corrosion resistance [6,9]. However, the performance of the inert anode fabricated from NiFe<sub>2</sub>O<sub>4</sub>-based cermet could not satisfy the requirements for aluminum electrolysis because of unsuitable mechanical properties and insufficient corrosion resistance and electrical conductivity.

Generally, ball milling and sintering techniques have been used to produce NiFe<sub>2</sub>O<sub>4</sub>-based cermet. However, well-blended, fine granular NiO/Fe<sub>2</sub>O<sub>3</sub>, which is the precursor of NiFe<sub>2</sub>O<sub>4</sub>, must be prepared first. The planetary ball mill is one of the most popular equipments for grinding NiO/Fe<sub>2</sub>O<sub>3</sub> because of its moderate cost and simple setup and handling [7,10,11]. Researchers identified that the grinding efficiency of planetary ball

mill depends on several factors, such as pot diameter, pot depth, ball diameter, ball-filling ratio, revolution radius, material-filling ratio, and rotational speed [12,13]. Further, the impact energy between balls and powders, frequency of impacts, and abrasion are major factors that influence the working of planetary ball mills, and these factors are related to the ball–ball, ball–container, ball–powder, and powder–powder impact energy and collision frequency [14,15]. The specific impact energy is a useful parameter for analyzing the grinding mechanism and granular media performance [12–14,16]. However, few systematic experimental investigations conducted to date have focused on technical parameters, noisy-power dissipation, and efficiency in NiO/Fe<sub>2</sub>O<sub>3</sub> grinding because observing the motion of the balls within the mills and evaluating the performance of the mills are difficult. In fact, studies on NiO/Fe<sub>2</sub>O<sub>3</sub> grinding and their noisy-power dissipation are seldom reported nowadays.

In recent years, the discrete element method (DEM) [10,14,15,17–19], particle element method (PEM) [20], and modified version of the distinct element method (MDEM) [21] have been utilized to investigate specific impact energy dissipation in the case of materials such as

gibbsite [13,14,22], limestone [22], and talc [16]. In particular, the DEM is one of the most powerful techniques for investigating the granular-media grinding mechanism and performance.

Therefore, in this study, the DEM is employed to analyze the water-filling ratio, number ratio of balls with diameters of 10 mm and 20 mm, revolution speed, powder-filling ratio, ball-filling ratio, grinding time, and the relationship between grinding efficiency and noisy-power dissipation in the grinding of NiO/Fe<sub>2</sub>O<sub>3</sub>. Three major mechanical energy dissipation modes were found during planetary ball milling operation: efficient energy absorption by the ground powder, noisy-power dissipation, and thermal energy generation as a result of heating of the granular media, pots, and gas during grinding. The efficient energy absorbed by ground powder and noisy-power dissipation should not be omitted in the balls motion, but thermal energy of heating granular media grinding, pots and gas is very small because the temperature is homogeneous during the grinding.

## 2 Experimental

### 2.1 Equipment and chemical reagents

The grinding mill used in this study was a planetary ball mill (XQM-4L, China). The mill consisted of four pots made of stainless steel (density,  $\rho=8.2\times10^3$  kg/m<sup>3</sup>) placed on a revolving disk. The volume, inner diameter, and height of the pots were 1l, 118, and 92 mm, respectively. The grinding media were dual-sized balls (diameters of 10 mm, 20 mm) made of stainless steel. The rotational speed,  $r$ , which was determined by the rotation-to-revolution speed ratio of the planetary ball mill, was set to be 1.00 for the study ( $r=N_p/N_r$ , where  $N_p$  is the rotation speed of the pots and  $N_r$  is the revolution speed of the disk). The revolution speed of the disk could be varied from 50 to 400 r/min, and the rotation speed of the disk could be varied from 100 to 800 r/min. The planetary ball mill was set to alternately rotate and revolve in the clockwise and anticlockwise directions at intervals of 5 min. The pot was set to rotate counter to the direction of revolution of the disk.

The acoustic test instrument employed in the study was an AR824 digital sound level meter (Smart Sensor, China) and was used to test the level of noisy-power dissipation in the planetary ball mill. During the working of the ball mill, the output of the digital meter increased with an increase in the amount of power dissipated. Thus, the digital sound level meter could evaluate the efficiency of grinding indirectly.

The sizes of the particles of the samples to be

ground were measured using the sedimentation method. Ethanol (CH<sub>3</sub>CH<sub>2</sub>OH; minimum concentration of 99.8%) was employed as the liquid for the measurements. The sedimentation method for determining the particle size is based on the fact that particles were smaller in size when displace in larger volume of suspension ( $V_s$ ) of ethanol absolute at any point of time. The specific surface areas of the ground powers were characterized by a Nova Win surface area analyzer (Quantachrome Instruments, USA).

The test samples to be ground were a ferric oxide (Fe<sub>2</sub>O<sub>3</sub>) powder (minimum concentration of 95.76%; Liuzhou Yuejin Chemical Industry Limited Company, China) and a nickel protoxide (NiO) powder (minimum concentration of 99%, Chengdu Nickel Powder Materials LLC, China). The initial diameters of the Fe<sub>2</sub>O<sub>3</sub> and NiO powder particles were 47 and 75  $\mu$ m, respectively. The two powders were mixed in a Fe<sub>2</sub>O<sub>3</sub>/NiO mass ratio of 57.9/42.1. The Fe<sub>2</sub>O<sub>3</sub>/NiO mixture was then dispersed in water in different concentrations, and the resulting slurries were used as the starting samples. These samples were placed in each pot of the planetary ball mill and ground. The blended slurries were dried in an oven at 100 °C for 24 h. The blended powders obtained after drying the slurries were dispersed using an agate mortar. As mentioned previously, the particle sizes of the blended powders were measured using the sedimentation method, and their specific surface areas were measured using a surface area analyzer.

### 2.2 Single-factor experiments

#### 2.2.1 Effect of water content of slurry

200 small balls (diameter of 10 mm) and 40 big balls (diameter of 20 mm) were placed in each stainless steel pot along with 234 g of the powder mixture to be ground. The grinding time and rotation speed were set to be 4 h and 299.5 r/min, respectively. The amount of water added to each pot ranged from 50 g to 450 g. That is to say, the water content of the NiO/Fe<sub>2</sub>O<sub>3</sub> slurries was 21.4%–192.3%.

#### 2.2.2 Effect of number ratio of big to small balls

The small (diameter of 10 mm) and big (diameter of 20 mm) steel balls were placed at different number ratios in the stainless steel pots. However, the total volume of the balls was kept constant at  $\sim 272.3$  cm<sup>3</sup>. The numbers of small and big balls used are listed in Table 1. At every stage of the experiment, each pot contained the same number of balls. 234 g of the dry powder was placed in each pot. The grinding time and rotation speed were set to be 4 h and 299.5 r/min, respectively. The amount of water added to each pot was 150 g. That is to say, the water content of the NiO/Fe<sub>2</sub>O<sub>3</sub> slurry in each pot was 64.1%.

**Table 1** Number ratio of balls with diameters of 10 mm and 20 mm

No.	Number		Number ratio of balls of different diameters
	Small balls (diameter=10 mm)	Big balls (diameter=20 mm)	
1	40	60	40/60
2	80	55	80/55
3	120	50	120/50
4	160	45	160/45
5	200	40	200/40
6	240	35	240/35
7	280	30	280/30
8	320	25	320/25
9	360	20	360/20
10	400	15	400/15

### 2.2.3 Effect of rotation speed

360 small balls and 20 big ones were placed in each pot. 234 g of the dry powder mixture was also placed in each pot. The grinding time was set to be 4 h. The amount of water added to each pot was 150 g. That is to say, the water content of the NiO/Fe<sub>2</sub>O<sub>3</sub> slurry in each pot was 64.1%. The rotation speed was varied from 260.6 to 460.8 r/min.

### 2.2.4 Effect of powder-filling ratio

360 small balls and 20 big balls were placed in each pot. The total mass of all the balls was 2151.33 g. The amounts of dry powder added to each pot were 116.79, 234.00, 351.00, 468.00, and 585.30 g for ball/powder mass ratios of 5.44%, 10.88%, 16.32%, 21.75%, and 27.21%, respectively. The grinding time and rotation speed were set to be 4 h and 300.9 r/min, respectively. The amount of water added to each pot was 150 g. That is to say, the water content of the NiO/Fe<sub>2</sub>O<sub>3</sub> slurry in each pot was 64.1%.

### 2.2.5 Effect of ball-filling ratio

Balls were placed in the pots in different ball-filling ratios,  $J$ . The ball-filling ratios used are listed in Table 2. The amount of dry powder added to each pot was 234.2 g. The grinding time and rotation speed were set to be 4 h and 300.9 r/min, respectively. The amount of water added to each pot was 150 g. That is to say, the water content of the NiO/Fe<sub>2</sub>O<sub>3</sub> slurry in each pot was 64.1%.

### 2.2.6 Effect of grinding time

360 small balls and 20 big ones (the ball-filling ratio was 27.23%) were placed in each pot. The amount of dry powder added to each pot was 234 g. The grinding time,  $t$ , was varied from 2 h to 12 h with the mean grinding time being 2 h. The rotation speed was set to be 300.9 r/min. The amount of water added to each pot was 150 g. That is to say, the water content of the NiO/Fe<sub>2</sub>O<sub>3</sub> slurry in each pot was 64.1%.

**Table 2** Conditions corresponding to different ball-filling ratios

Sample No.	Number of small ball	Number of big ball	Volume of ball/mL	Volume of mill pot/mL	Ball-filling ratio/%
1	200	0	104.72	1000	10.47
2	232	4	138.30	1000	13.83
3	264	8	171.81	1000	17.18
4	296	12	205.31	1000	20.53
5	328	16	238.82	1000	23.88
6	360	20	272.32	1000	27.23
7	392	24	305.82	1000	30.58

## 3 Results and discussion

During planetary ball milling operations, mechanical energy is dissipated through three major paths. These are the absorption of energy by the powder to be ground, noisy-power dissipation, and the thermal energy released by the heating of the granular media being ground and the pots and gas used in the grinding process. On the basis of the results of this study, the mechanical energy dissipated could be represented by Eq. (1). In this equation, the last term on the right-hand side is usually very small, as the temperature during the grinding process remains constant.

$$P_W = P_{W,m} + P_{W,n} + P_{W,h} \quad (1)$$

where  $P_W$  is the specific impact energy of the planetary ball mill;  $P_{W,m}$  is the energy absorbed by the granular media to be ground;  $P_{W,n}$  is the energy dissipated owing to noisy-power dissipation;  $P_{W,h}$  is the thermal energy released by the heating of the granular media to be ground and the pots and gas used.

### 3.1 Analysis of $V_s$ and $P_{W,n}$ for different slurry water contents

Figure 1 shows the effects of the slurry water content on  $V_s$  and  $P_{W,n}$  for the parameters listed in Table 3. It can be seen that  $V_s$  and  $P_{W,n}$  increase initially with an increase in the slurry water content. The value of  $V_s$  peaks at 27 mL when the content is 85.5%; the corresponding value of  $P_{W,n}$  is 0.0026 J/s. The value of  $P_{W,n}$  reaches the maximum (0.0044 J/s) when the content is 42.7%; the corresponding  $V_s$  is 18 mL. Further, both  $V_s$  and  $P_{W,n}$  decrease with further increase in the water content.

The above-described phenomena were analyzed using the DEM. Using the DEM, MORI et al [16] had found that the motion of the balls during wet milling is affected by the drag force,  $F_D$ , and the buoyancy of the mill balls.

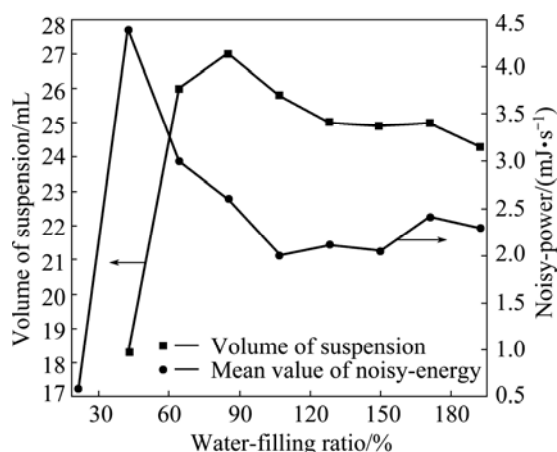


Fig. 1 Relationship among slurry water content,  $V_s$ , and  $P_{W,n}$

Table 3 Parameters for single-factor experiments

Slurry water content/%	Number ratio of balls	Rotational speed/(r·min <sup>-1</sup> )	Powder amount/g	Ball-filling ratio/%	Grinding time/h
Variable	200/40	299.5	234.0	27.23	4
64.1	Variable	299.5	234.0	27.23	4
64.1	360/20	Variable	234.0	27.23	4
64.1	360/20	300.9	Variable	27.23	4
64.1	360/20	300.9	234.2	Variable	4
64.1	360/20	300.9	234.0	27.23	Variable

The value of  $F_D$  can be calculated using Eq. (2). It should be noted that  $C_d$  in Eq. (2) is calculated using Eq. (3) and it is a function of the Reynolds number,  $Re$ , which is given by Eq. (4).

$$F_D = C_d A \rho_s \frac{u_R^2}{2} \quad (2)$$

$$C_d = \left(0.63 + \frac{4.8}{\sqrt{Re}}\right)^2 \quad (3)$$

$$Re = \frac{d_B |u_R| \rho_s}{\alpha} \quad (4)$$

where  $C_d$  is the drag coefficient;  $A$  is the projection area of a ball;  $\rho_s$  is the density of the slurry;  $u_R$  is the relative velocity of an individual ball and the slurry;  $d_B$  is the diameter of an individual ball;  $\alpha$  is the viscosity of the slurry.

According to Eqs. (2)–(4), the phenomenon of grinding shown in Fig. 1 is related to  $\rho_s$ ,  $A$ ,  $u_R$ ,  $\alpha$ , and  $d_B$ .  $d_B$  is assumed to be constant for this experiment. When the water content of the slurry is very small (i.e., the powder mixture in the mill pots is dry),  $u_R$  remains constant and  $Re$  increases with an increase in water content. It should be noted that  $Re$  is very small since the viscosity of the slurry is high when it is dry, that is, its

water content is very low. Therefore, the last term on the right-hand side of Eq. (3) cannot be neglected. Hence,  $C_d$  decreases with an increase in  $Re$ . In addition,  $F_D$  decreases with a decrease in  $C_d$ , as suggested by Eq. (2). The buoyancy of the balls is also very low when the slurry water content is low. The energy dissipated by the powder is very large because  $F_D$  and buoyancy are very small. The first term on the right-hand side of Eq. (1), which results from the force acting on the powder, is very large. Therefore, the value of  $P_W$  is determined primarily by the  $P_{W,m}$  term, which increases with an increase in  $V_s$ .

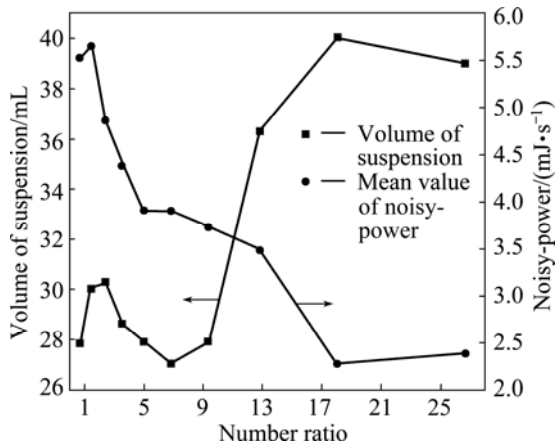
Secondly,  $C_d$ ,  $\rho_s$ ,  $A$ , and  $\alpha$  decrease and  $u_R$  increases with an increase in the slurry water content, that is, as the dry powder becomes a slurry (or as the ground material starts to cover the grinding balls). The grinding process corresponds to a transition state that is affected by both  $F_D$  and the buoyancy together. The energy dissipated by the powder is the maximum in this transition state. Therefore,  $P_W$  is determined by  $P_{W,m}$ . Further,  $V_s$  is the maximum in this transition state.

In the end, the NiO/Fe<sub>2</sub>O<sub>3</sub> powder does not cover the balls completely as the buoyancy of the balls is at the highest value. The value of  $Re$  is very large, and thus the last term on the right-hand side in Eq. (3) can be neglected. Further,  $\rho_s$ ,  $A$ ,  $u_R$ ,  $\alpha$ ,  $d_B$ , and  $C_d$  are constant. Hence,  $F_D$  does not vary with an increase in the slurry water content when the water content is very large. Therefore,  $V_s$  remains almost constant, and  $P_W$  is mostly determined by the  $P_{W,n}$  term.

According to Eqs. (2)–(4),  $V_s$  decreases (i.e., the particle size is large) when  $F_D$  increases. Similarly,  $V_s$  increases (i.e., the particle size is small) when  $F_D$  decreases. From Fig. 1, it can be seen that  $V_s$  reaches the maximum value of 27 mL when  $P_{W,n}$  and the water content are 0.003–0.0026 J/s and 64.10%–85.47%, respectively. In this case, the grinding process is the most efficient, and  $P_{W,n}$  has the smallest value. Hence, the slurry water content should be controlled to be 64.10%–85.47%. Further, an interesting phenomenon can be seen in Fig. 1, in which the grinding efficiency of the planetary ball mill decreases as the energy dissipated in the form of noisy power increases.

### 3.2 Analysis of $V_s$ and $P_{W,n}$ for different ball number ratios

Figure 2 shows that  $V_s$  and  $P_{W,n}$  vary with the number ratio of the balls with diameters of 10 mm and 20 mm as the other parameters listed in Table 3, remain constant. It can be seen from the figure that  $V_s$  increases to 40 mL (i.e., the particle size decreases as  $V_s$  increases) with an increase in the number ratio; the corresponding  $P_{W,n}$  is 0.0023 J/s. Further,  $V_s$  decreases with the number



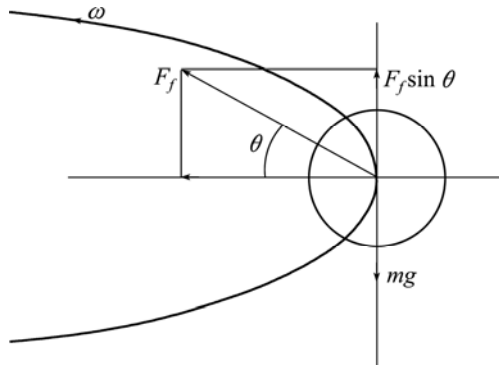
**Fig. 2** Relationship among number ratio of balls with diameters of 10 mm and 20 mm,  $V_s$ , and  $P_{W,n}$

ratio of the balls. However,  $P_{W,n}$  increases with an increase in the number ratio.

On the basis of the DEM, ROSENKRANZ et al [10] and MIO et al [12] found that the motion of the mill balls is effective in grinding powders when the motion shifts from cascading-type to cataracting-type during grinding; further, they also noted that the rolling motion of the mill balls was ineffective for grinding. This phenomenon can be interpreted using the Newton's Second Law. For simplicity, considering that a single-mill ball spirals upwards, as shown in Fig. 3. According to the Newton's Second Law, this type of cascading motion can be described by Eq. (5).

$$\mu R \omega^2 \sin \theta \geq g \quad (5)$$

where  $\mu$  is the friction coefficient;  $R$  is the rotation-to-revolution speed ratio;  $\omega$  is the angular velocity;  $\theta$  is the angle between the horizontal and vertical directions;  $g$  is standard gravity.



**Fig. 3** Force analysis of spiral motion of a mill ball

In Fig. 2 and Eq. (5),  $R$  and the rotation and revolution speeds are constants.  $\mu$  and  $\theta$  increase with an increase in the number ratio of the balls with diameters of 10 mm and 20 mm. Cascading-type motion takes

place when the term on the left-hand side of Eq. (5) is greater than the term on the right-hand side. At this point, the motion shifts from cascading type to cataracting type. The specific impact energy increases when the motion is of the cascading type, suggesting that a greater amount of  $P_W$  is absorbed by the NiO/Fe<sub>2</sub>O<sub>3</sub> powder, resulting in finer particles. Hence,  $V_s$  reaches a high value of 40 mL when the motion is of the cascading type. Subsequently, the motion shifts from the cataracting type to the rolling type when the number ratio is increased further. Rolling-type motion is ineffective for grinding because the rate of collision of the balls decreases considerably and the balls start colliding against the mill walls. Hence,  $V_s$  decreases slowly when rolling-type motion occurs.

As can be seen from Fig. 2,  $P_{W,n}$  decreases when  $V_s$  and the number ratio increase with  $P_{W,n}$  exhibiting the lowest value at  $V_s$  of 40 mL and number ratio of 360/20. The grinding process is the most efficient when  $P_{W,n}$  is the lowest.

### 3.3 Analysis of $V_s$ and $P_{W,n}$ for different rotation-to-revolution speeds

Figure 4 plots  $V_s$  and  $P_{W,n}$  as functions of the rotation-to-revolution speed,  $v$ ; the rest of the parameters were the ones listed in Table 3. Both  $V_s$  and  $P_{W,n}$  increase with an increase in  $v$ . The value of  $V_s$  peaks at 40 mL when the rotation-to-revolution speed is 300.9 r/min and  $P_{W,n}$  is 2.9 mJ/s. Further,  $P_{W,n}$  reaches its maximum value of 7.5 mJ/s when the rotation-to-revolution speed is 420.4 r/min and  $V_s$  is 30 mL. Subsequently, both  $V_s$  and  $P_{W,n}$  decrease for further increases in the rotation-to-revolution speed.

The changes in  $V_s$  and  $P_{W,n}$  with  $v$  can be interpreted on the basis of the value of  $P_W$  during milling. Based on the DEM, KANO et al [20], MORI et al [16], and MIO et al [12,13] found that  $P_W$  can be calculated using Eq. (6). In this equation,  $v_j$  is the relative velocity corresponding to the collisions between two balls and/or of a ball against the mill wall. Equation (1) can be expressed as Eq. (8) on the basis of Eq. (6).

$$P_W = \frac{1}{t} \sum_{j=1}^n \frac{M v_j^2}{2W} \quad (6)$$

$$M = \frac{2m_1 m_2}{m_1 + m_2} \quad (7)$$

$$\frac{1}{t} \sum_{j=1}^n \frac{M v_j^2}{2W} = P_{W,m} + P_{W,n} \quad (8)$$

where  $W$  is the mass of the sample placed in the mill;  $n$  is the number of collisions of the balls per second in the mill;  $m$  is the mass of a small ball;  $M$  is the reduced mass of balls with diameter of 10 mm and 20 mm;  $t$  is the grinding time.

In this experiment,  $M$  and  $W$  were assumed to be constant. The values of  $P_W$  and  $n$  were affected only by  $v_j$ .  $V_s$  increases with  $P_{W,n}$  in Eq. (8). Cascading-type motion does not occur when  $v$  is very small, owing to the centrifugal force caused by the rotation of the pot being small. In this case,  $v_j$  and  $P_W$  are very small, and hence, the value of  $V_s$  is also very small. Cascading-type motion does occur when  $v$  reaches a critical value, as can be seen from Eq. (8), owing to there being equilibrium between the centrifugal forces acting on the balls because of the rotation of the pot and the revolution of the disk. The motion changes from the cascading type to the cataracting type as  $v$  increases further.  $V_s$  peaks at 39.5 mL, indicating that most of the  $P_W$  is absorbed by the NiO/Fe<sub>2</sub>O<sub>3</sub> powder and that the particle size is smaller when cascading-type motion occurs and eventually changes into cataracting-type motion. Eventually, the motion shifts from the cataracting type into the rolling type when  $v$  increases beyond the critical value. As mentioned previously, rolling-type motion is ineffective for grinding, as it results in a lower number of collisions of the individual balls and leads to collisions against the mill wall. The value of  $v_j$  decreases as the rolling speed increases, and  $P_W$  is mostly determined by  $P_{W,n}$ . Further,  $V_s$  also decreases with an increase in the rolling speed.

It can be seen from Fig. 4 that  $P_{W,n}$  increases with an increase in  $v$ , reaching a peak value of 7.5 mJ/s. Subsequently, its value falls. Cascading-type motion does not occur when  $v < 300.9$  r/min. As both  $v_j$  and  $P_W$  are very small, so is the value of  $P_{W,n}$ . However, cascading-type motion takes place when  $v \geq 300.9$  r/min in Eq. (8). More of  $P_W$  is absorbed by the NiO/Fe<sub>2</sub>O<sub>3</sub> powder, and the particle size is smaller when cascading-type motion occurs. Hence,  $P_{W,n}$  increases slightly. Then, the motion shifts from the cataracting type to the rolling type when  $v \gg 300.9$  r/min. The value of  $v_j$  decreases as the extent of rolling increases, and  $P_W$  is determined mostly by  $P_{W,n}$ . Hence, the value of  $P_{W,n}$

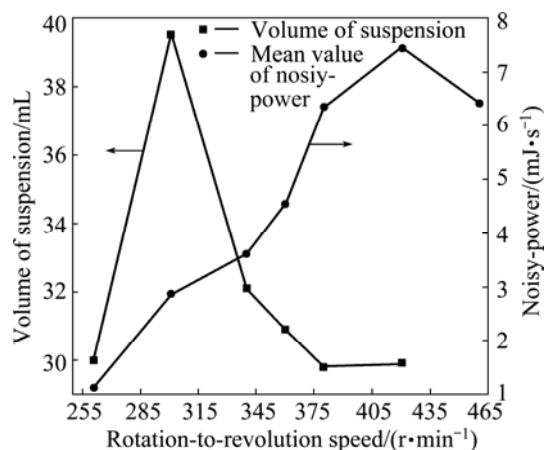


Fig. 4 Relationship among rotation-to-revolution speed,  $V_s$ , and  $P_{W,n}$

is extremely large. It is interesting to note that the value of  $V_s$  peaks at 39.5 mL when  $P_{W,n}$  reaches a low point, and  $V_s$  reaches the lowest value when  $P_{W,n}$  reaches its peak, which is 7.5 mJ/s. The above-described phenomena suggest that the grinding efficiency increases with a decrease in the degree of noisy-power dissipation.

### 3.4 Analysis of $V_s$ and $P_{W,n}$ for different powder-filling ratios

Figure 5 reveals that both  $V_s$  and  $P_{W,n}$  decrease with an increase in the powder-filling ratio when the other parameters listed in Table 3 remain unchanged. This phenomenon can be explained by Eqs. (6) and (8). With an increase in the powder-filling ratio (i.e., with an increase in  $W$  in Eqs. (6) and (8)), the powders completely cover the container walls and the milling balls. This leads to a decrease in the collision frequency ( $n$ ) and  $v_j$ . Thus,  $P_W$  and  $P_{W,n}$  decrease in Eqs. (6) and (8). Cascading-type motion does not occur as the powder-filling ratio is increased. On the other hand, it does occur when the powder-filling ratio is decreased. Figure 5 shows that the volume of the suspension is the highest when the powder-filling ratio is 5.44%; however, it is suggested that the powder-filling ratio is set to be ~10.88% in order to reduce the friction and wear.

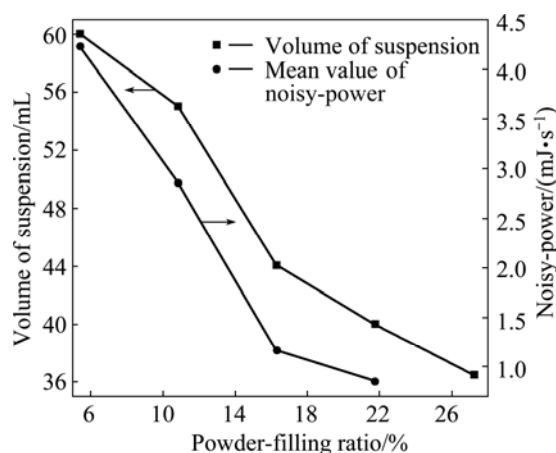


Fig. 5 Relationship among powder-filling ratio,  $V_s$ , and  $P_{W,n}$

Figure 6 shows the volume of the suspension for different number ratios of the balls with diameters of 10 and 20 mm; the power-filling ratio remains the same. The number ratios are 5 and 18, respectively, for curves 1 and 2 in Fig. 6. Figure 6 also shows that  $V_s$  increases with an increase in the number ratio and that  $V_s$  decreases with an increase in the powder-filling ratio. When the powder-filling ratio is increased (i.e.,  $W$  in Eqs. (6) and (8) is increased), the powder completely covers the container walls and the balls, leading to a lower collision frequency ( $n$ ) and  $v_j$ . As a result,  $P_W$  in Eqs. (6) and (8) decreases. Figure 6 also suggests that  $V_s$  increases with an increase in the number ratio. Both  $n$  and  $v_j$  increase

with an increase in the ball number ratio in Eqs. (6) and (8). This causes  $P_{W,n}$  to increase. Further,  $V_s$  increases with the increase in  $P_{W,n}$ . Finally, Fig. 5 also shows that the volume of the suspension is the maximum when the powder-filling ratio is 5.44%; however, as noted previously, it is suggested that the powder-filling ratio is set to be  $\sim 10.88\%$  in order to reduce the friction and wear.

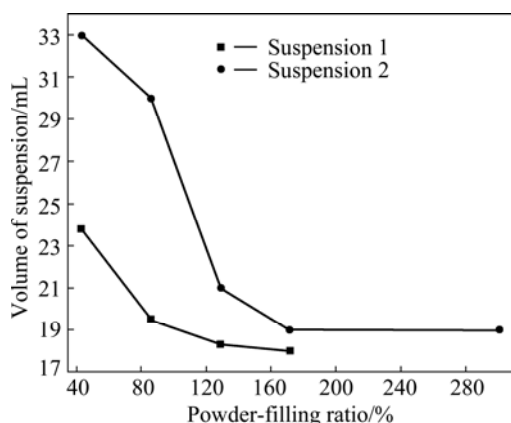


Fig. 6 Relationship between powder-filling ratio and  $V_s$

### 3.5 Analysis of $V_s$ and $P_{W,n}$ for different ball-filling ratios

Figure 7 shows the plots of the experimental values of  $V_s$  and  $P_{W,n}$  as functions of the ball-filling ratio while the other parameters listed in Table 3 were kept constant. It can be seen from the figure that  $V_s$  reaches a maximum value of 44 mL when the ball-filling ratio is 20.53%–23.88%, at which  $P_{W,n}$  is 0.4–0.7 mJ/s. Subsequently, the value of  $V_s$  decreases. The value of  $P_{W,n}$  increases slowly when the ball-filling ratio is smaller than 20.53%. However, it increases significantly when the ball-filling ratio is greater than 23.88%.

This phenomenon can be explained by Eqs. (5), (6), and (8). It was assumed that  $W$  remained constant in this experiment. Initially, the excess NiO/Fe<sub>2</sub>O<sub>3</sub> slurry that covered the container walls and the milling balls leads to

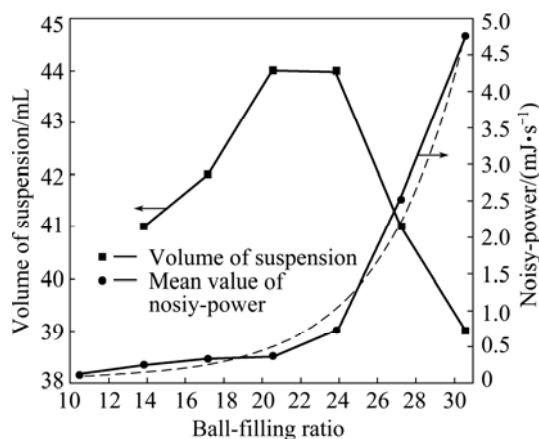


Fig. 7 Relationship among ball-filling ratio,  $V_s$ , and  $P_{W,n}$

lowering of the collision frequency ( $n$ ) and  $v_j$  in Eqs. (6) and (8). The  $\mu$  in Eq. (5) is also very large, while  $v$  is very small, owing to the centrifugal force being small. Hence,  $V_s$  increases with an increase in the ball-filling ratio. Further,  $P_{W,n}$  increases slowly with an increase in  $V_s$ . Therefore, the collision frequency ( $n$ ) and  $v_j$  in Eqs. (6) and (8) increase with an increase in the ball-filling ratio (i.e.,  $M$  increases in Eqs. (6) and (8)), because the extent to which the NiO/Fe<sub>2</sub>O<sub>3</sub> slurry covers the container walls and the balls decreases. As result,  $\mu$ ,  $R$ , and  $\omega$  also decrease in Eq. (5). Cascading-type motion occurs and shifts to the cataracting type with an increase in the ball-filling ratio when the term on the left-hand side in Eq. (5) is smaller than the term on the right-hand side. Thus, the value of  $P_{W,n}$  increases in Eqs. (6) and (8), and  $V_s$  increases with an increase in  $P_{W,n}$ . However, cascading-type motion does not occur when the ball-filling ratio is increased further. This is owing to the fact that the container walls and the balls are no longer covered by the NiO/Fe<sub>2</sub>O<sub>3</sub> slurry and because when the number of balls is high, it leads to lowering of the collision frequency ( $n$ ) and  $v_j$  in Eqs. (6) and (8), while the volume of the mill pot remains constant. The value of  $mg$  in Eq. (5) is also very large.

It can be seen from Fig. 7 that  $P_{W,n}$  increases sharply when the ball-filling ratio  $\geq 23.88\%$ . The dashed line is the nonlinear fitting line that represents the relationship between  $P_{W,n}$  and the ball-filling ratio,  $J$ , in Fig. 7. The relationship between  $P_{W,n}$  and  $J$  can be described empirically by Eq. (9), where the values of the empirical constants,  $P_{W,n}^0$ ,  $A$ , and  $K$  are  $5.59 \times 10^{-5}$ ,  $3.48 \times 10^{-6}$  and  $-4.23$ , respectively, within the experimental range. These values were obtained using the least squares method. By analyzing the experimental results, the constants  $P_{W,n}^0$ ,  $A$ , and  $K$  could be explained as follows: noisy-power dissipation during planetary balling mill operation without loading, per-exponential factors, ball grinding media factors which depend on the material, respectively. The  $R$  and  $R^2$  values for Eq. (9) were 99.4% and 98.1%, respectively.

$$P_{W,n} - P_{W,n}^0 = A \exp\left(\frac{-J}{K}\right) \quad (9)$$

where  $K$  is the ball grinding media factor which depends on the material.

On the basis of the above-described results, it can be surmised that the volume of the suspension increases with a decrease in the extent of noisy-power dissipation. Thus, it can be assumed that the efficiency of planetary ball milling would decrease when the extent of noisy-power dissipation exceeds a specific value, which is determined by Eq. (9). Therefore, Eq. (9) is useful for determining the efficiency of planetary ball milling.

### 3.6 Analysis of $V_s$ and $P_{W,n}$ for different grinding time

Figure 8 shows that  $P_{W,n,t}$ , which is the product of  $P_{W,n}$  and the grinding time, increases with the grinding time as the other milling parameters, listed in Table 3, remain constant. However,  $V_s$  reaches a maximum value of 30 mL and subsequently decreases with an increase in the grinding time, as shown in Fig. 8. This is because the powder grain size is large for grinding time less than 6 h, and the finer powder particles agglomerate when the grinding time is more than 6 h. Thus, the NiO/Fe<sub>2</sub>O<sub>3</sub> powder should be ground for ~6 h.

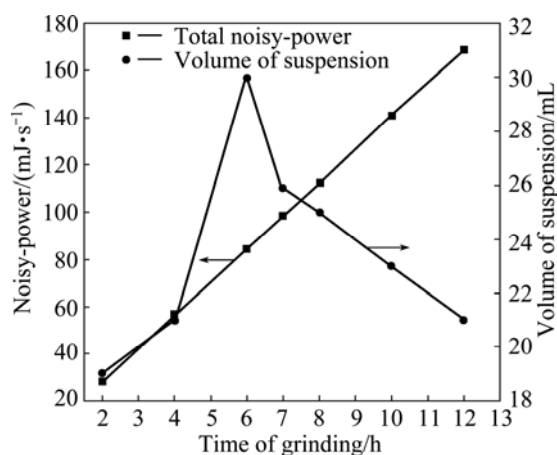


Fig. 8 Relationship among grinding time,  $V_s$ , and  $P_{W,n}$

### 3.7 Analysis of specific surface area of ground powder

Table 4 lists the specific surface area distribution corresponding to milling conditions A to E, that is, for the optimum rotation-to-revolution speed (A), slurry water content (B), ball-filling ratio (C), powder-filling ratio (D), and grinding time (E). It can be seen from the table that the specific surface area of the NiO/Fe<sub>2</sub>O<sub>3</sub> powder increases as the milling conditions are improved, resulting in a smaller mean particle size.

Table 4 Distribution of specific surface area of powder and corresponding mean particle size

Condition	Specific surface area/(m <sup>2</sup> ·g <sup>-1</sup> )	Mean particle size/μm
A	7.758	0.176
B	8.213	0.166
C	9.071	0.150
D	9.392	0.145
E	10.389	0.131

## 4 Conclusions

1) The effects of various grinding parameters on the characteristics of a NiO/Fe<sub>2</sub>O<sub>3</sub> powder were studied using a planetary ball mill. The relationship between the noisy-power dissipation and the grinding parameters was

also investigated. The DEM was employed to explain the obtained results. It was found that the optimum grinding parameters were as follows: a slurry water content of 64.1%–85.47%; a number ratio of balls with diameters of 10 and 20 mm of 360/20; a rotation-to-revolution speed ratio of 300.9 r/min; a powder-filling ratio of approximately 10.88%; a ball-filling ratio of 20.53%–23.88%; and a grinding time of approximately 6 h.

2) It was found that the mean particle size of the ground powder increased with an increase in the extent of noisy-power dissipation owing to collisions, while the milling efficiency and the degree of utilization of the specific impact energy decreased. The relationship between the extent of noisy-power dissipation and the ball-filling ratio was determined using the nonlinear fitting method.

3) The findings of this study should help to improve the efficiency of ball mill grinding of various materials.

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## NiO/Fe<sub>2</sub>O<sub>3</sub> 湿式行星球磨中的噪声能消耗与效率

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**摘 要:** 研究 NiO/Fe<sub>2</sub>O<sub>3</sub> 湿式混合与球磨过程中的噪声能消耗、球磨效率及二者之间的关系。结果表明, 球磨工艺的最佳工艺参数为: 填水率 64.10%~85.47%, 小/大球个数比 360/20, 球磨机转速 300.9 r/min, 填粉率约 10.88%, 填球率 20.53%~23.88%, 球磨时间 6 h。引入离散元法(DEM)分析球磨过程中噪声能消耗与球磨效率之间的关系, 得到噪声能消耗与填球率关系的经验方程。研究表明粉体的平均粒度随着噪声能的消耗而减小, 而球磨效率和球碰撞能的利用率则随着噪声能消耗的增加而减小。

**关键词:** 噪声能消耗; NiO/Fe<sub>2</sub>O<sub>3</sub> 粉体; 行星式球磨; 平均粒度

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