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Ultra-Fine Grinding Mechanism of Inorganic Powders in a Stirred Ball Mill
- The Effect of Grinding Aids -

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Abstract−An experimental investigation on grinding mechanism for calcite used in a stirred ball mill was carried out. The slurry concentration and the amount of grinding aids were chosen as main experimental factors of the grinding process. The effect of grinding aids on particle size distribution and grinding efficiency, defined as the increases of specific surface area per the specific grinding energy, was investigated. It was demonstrated that the grinding rate for calcite could be improved by addition of grinding aids. The grinding energy efficiency by adding a specific grinding aids was improved approximately 45.2% in comparison with and without grinding aids (n=700 rpm, J=0.7, d_B=1.0 mm, C_s=60 wt%).

Key words: Stirred Ball Mill, Grinding Mechanism, Grinding Energy Consumption, Grinding Aids

INTRODUCTION

The improvement of grinding efficiency in the submicron range has recently been very important [Jimbo, 1992; Choi, 1996; Kwade, 1999]. In view of this growing importance, the basic research of power consumption characteristics of stirred ball mill has been conducted. In particular, determining optimum conditions is very important for increasing the grinding efficiency [Choi, 1996].

Paramasivam et al. [1992] studied the effect of the physical properties of liquid additives on dry grinding and they found that the action of additives depends on the nature of the additives used and also on the particle size distribution of the mill contents, which varies with grinding time. Mosquet et al. [1997] studied the dispersing of functional polyethylene oxides agent and found that new hydrophilic polymers bearing a functional group at one chain end adsorb mineral surface. Byron et al. [2000] investigated the stabilization of high ionic strength slurries adding the synergistic effects of a mixed surfactant system. Zheng et al. [1997] examined the effect of additives on stirred ball milling of limestone. Slug characteristics were measured in a fluidized bed with different distributors [Lee et al., 2001].

The effect of grinding aids on grinding has been explained mainly by two kinds of mechanisms. One is based upon the alteration of surface and the mechanical properties of individual particles such as reduction of surface energy and the other is to consider the arrangement of particles and their flow in suspensions [Zheng et al., 1997].

In this study the grinding energy efficiency was examined with various operating factors such as rotation speed and slurry concentration including addition of the grinding aids.

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EXPERIMENTAL

1. Equipment

The grinding tests were performed in a vertical stirred ball mill, KMD-1B, manufactured by Korea Material Development Co., Ltd. as the same equipment used in previous papers [Kim et al., 1998; Choi et al., 2000]. The mill consists of grinding chamber and stirrer. The grinding chamber holds a centrally positioned rotated stirrer system with four arms. The cylindrical pins of 10.0 mm in diameter and 78.0 mm in length are arranged at a right angle to each other. The net inner volume of milling chamber is 0.95 liter.

The alumina ball made by Nikkato Co., Ltd., Japan was used as grinding media: purity 99.9%, diameter ϕ=1.0 mm(−1.41/+0.84), density 3.60 g/cm^3. The measuring devices for consumed energy consisted of the automatic voltage regulator of GPA 100SS AVR (Sung Sin Electronic. Co., Ltd., Korea) and the detection circuit made by our laboratory. The stirrer torque was measured with an A/D converter (AX5210, Axiom Technology Inc., Taiwan) and Interface card (ACL-8112PG, AD Link Technology Inc. UK) with PC on-line by turbo C++ software. The particle size distribution of ground products was analyzed by Mastersizer microplus (Malvern Instruments, Ltd., UK), based on the laser diffraction and scattering method. The homogenizer US-300T (Nihonseiki Kaisha Ltd., Japan) was used as a dispersion device. The grinding materials, calcite (S500, x_{50}=6.42 µm, density=2.72 g/cm^3) was used, which were produced by Wang Pyo Chemicals Co., Ltd., Korea and the grinding aids, JD-4019 and JD-4022 for mixed addition were used, which were produced by JEONG-WEON Chemical Co., Ltd., Korea. The basic material of this aids are poly-acrylic acid, $(CH_2CHCOOH)_n$.

2. Methods

The stirrer was rotated at the constant gap of 5 mm between the bottom and the end of stirrer. When the stirrer started rotating, the measuring of grinding power consumption started with a computer on-line system. The computer program was calibrated with measurement data by multimeter for 10 times [Choi et al., 2000].

Table 1. Amount of grinding aids used

C_s (wt%)	Sample (g)	Water (ml)	Grinding aids (1.2 wt% of sample, g)	
			Dispersant (40 wt%, g)	Diluant (60 wt%, g)
60	246	164	7.38	
			2.95	4.43
65	283	151	8.49	
			3.40	5.09
70	320	137	9.60	
			3.84	5.76
75	363	121	10.89	
			4.36	6.53

The total charged weights of balls for each value of ball filling ratio, J, was 1,380 g for J=0.7. The slurry concentration was changed between 10.0 wt% and 70.0 wt%. The grinding experiment was carried out with a batch process of sampling from the pot at the determined grinding time interval. The amount of grinding aids is 1.2 wt% of charged sample. Table 1 shows the amount of grinding aids for various slurry concentrations.

The slurry of sample including grinding aids of 0.2 wt% was ground during the initial stage of 13 min and then 1.0 wt% was fed into grinding chamber continuously from 14 min to 73 min. During last 2 min, only grinding operation without feeding of grinding aids was done. The particle size distribution of the samples was measured without any dispersion agent. Prior to the measurement of particle size distribution, the homogenizer was used to disperse particles for 90 seconds. At the end of each test, all the media and ground samples were removed from the mill, and the media were separated from the products by sieving. In order to investigate the effects of stirrer rotation speed, ball size, ball filling ratio and slurry concentration on the particle size distribution of the products for different grinding time, a series of experiments were carried out. The grinding consumption power was recorded every 2 minutes within 30 minutes or 5 minutes after that by Excel software.

The grinding consumption power was plotted against grinding time. The regression analysis was also done with the Sigma Plot and the SPSS software [Choi et al., 2000]. Table 2 shows the summary of experimental conditions.

Table 2. Summary of experimental conditions

Item	Experimental conditions
n (rpm)	700 [rpm]
J (-)	0.7 [-]
d_B (mm)	1.0 [mm]
C_s (wt%)	10.0-70.0 [wt%]
Temperature	Room temperature
Material of media	Alumina (Al_2O_3)

The specific surface area of powder is one of the basic properties of the powder and is generally represented by the surface area of total particles contained in a unit mass of powder. If distribution of particle size and shape in a powder sample are known, surface area of the powder can be calculated. The specific surface area of this study was calculated from data of particle size distribution where a particle is assumed to sphere as following equation.

$$S_w = \frac{6\sum\frac{V_i}{d_i}}{\rho_p\sum V_i} = \frac{6}{\rho_p D[3,2]} \quad (1)$$

Here, S_w is specific surface area, V_i is the relative volumes in size class i, d_i is mean size class diameter and ρ_p is particle density and D[3, 2] is surface weighted diameter.

Generally, specific surface area was measured by BET methods. Garcia et al. [2002] reported that the specific surface area obtained by BET method compared with calculated method. The result of comparison of two methods is very similar before agglomeration of particles. In this study, the specific surface area was calculated from particle size distribution due to particle size was not increased in this experimental range.

RESULTS AND DISCUSSION

Table 3 shows the summary of experimental result of the particle size, the increase of specific surface area, the grinding consumption power, the specific grinding consumption energy, and the grinding energy efficiency without grinding aids and with grinding aids for each experimental condition.

1. Change of Particle Size Distribution

Table 3. Summary of results on each experimental conditions

	C_s (wt%)	t (min)	Particle size (μm)			ΔS_w (m^2/g)	P (W)	E_m (MJ/kg)	η (m^2/kJ)
			x_{10}	x_{50}	x_{90}				
Without grinding aids	60	7.5	0.51	0.96	6.34	0.90	205.82	0.37	2.40
		15	0.49	0.78	3.53	1.37	205.08	0.75	1.83
		30	0.46	0.67	0.96	2.10	205.94	1.50	1.40
		60	0.44	0.63	0.89	2.31	207.13	3.02	0.77
		120	0.42	0.61	0.86	2.49	204.92	5.97	0.42
With grinding aids	60	7.5	0.49	0.81	3.88	1.26	192.73	0.35	3.57
		15	0.47	0.71	2.63	1.76	194.14	0.71	2.48
		30	0.44	0.63	0.88	2.33	189.00	1.38	1.68
		60	0.40	0.58	0.82	2.67	182.53	2.67	1.00
		120	0.35	0.53	0.76	3.06	172.49	5.05	0.61

Fig. 1 shows the cumulative particle size distribution of products obtained for various grinding times. Fig. 1(A) is for the case without grinding aids and Fig. 1(B) is for the case with grinding aids. The change of particle size distribution of ground products takes place by way of breakage and attrition as illustrated in Fig. 2.

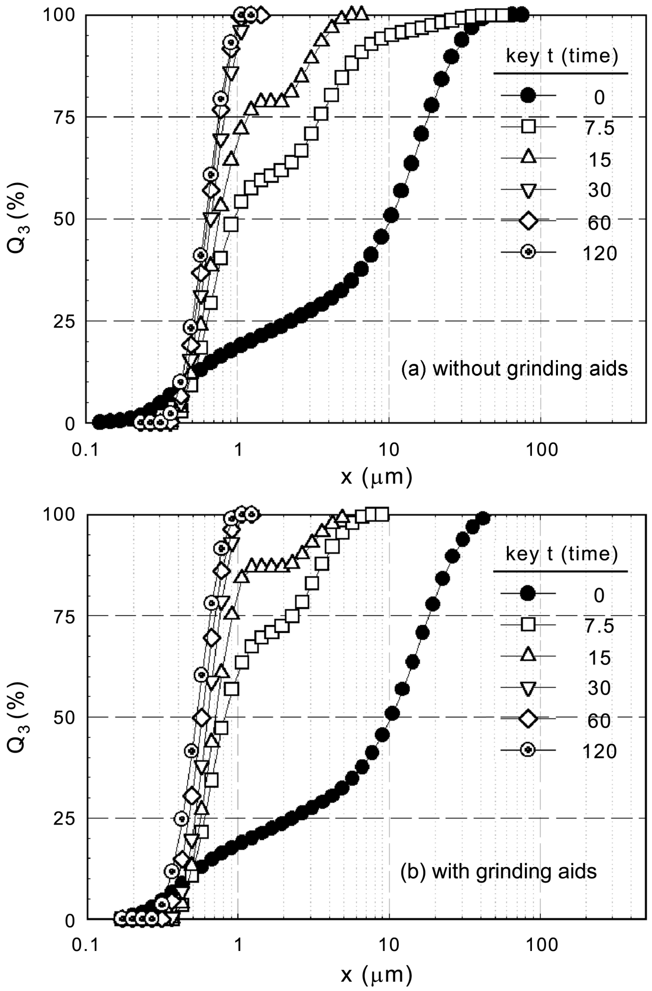

Fig. 1. Cumulative particle size distribution of feed and ground products for experimental conditions: n=700 rpm, J=0.7 (-), d_B=1.0 mm, C_s=60 wt%.
(A) without grinding aids, (B) with grinding aids

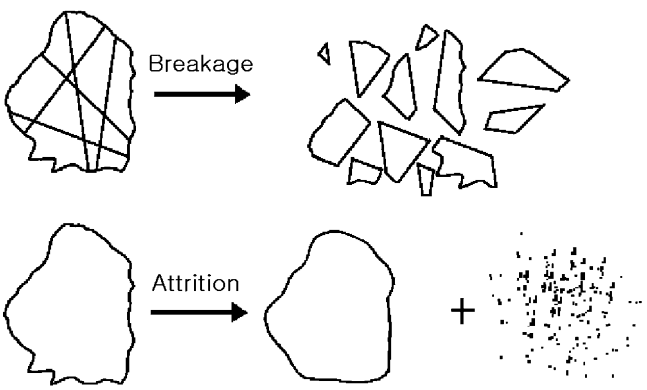

Fig. 2. Pictorial illustration of two ways of breakage and attrition.

Increasing the grinding time does not only produce finer product, but also narrows the particle size distribution by reducing the coarse particles [Park et al., 1998]. The particle size distribution of raw material was broad and bimodal distribution with fine mode of 0.42 μm and coarse mode of 19.31 μm. As the grinding time was increased, the gap of fine modal and coarse modal of bimodal distribution became gradually narrower and the peak of the fine modal became higher, while the peak of the coarse modal became lower with increasing of grinding time from 7.5 min to 30 min. After the grinding time was elapsed enough, here after 60 min, the peak of coarse modal disappeared and the particle size distribution of ground products became narrow and monomodal. In spite of addition or no addition of grinding aids, the change tendency of particle size distribution was similar as shown in Fig. 2. As shown in Table 3, the results such as particle size, increase of surface area based on weight, grinding consumption power, specific grinding consumption energy in the case of with grinding aids were decreased in comparison with those in the case without grinding aids, while the grinding efficiency was increased due to the positive effect of grinding aids. As the mechanism of action of this grinding aids, the following positive effects are mainly considered from various effect of grinding aids [Moothedath et al., 1992; Fuerstenau, 1995; Park et al., 1998]: agglomeration of fine particles, which is caused mainly due to van der Waals force, is prevented or reduced when grinding aids is adsorbed on the particle's surface. In the absence of agglomeration the grinding can proceed to a fine state as the limit of grinding is also shifted towards the finer region as shown in Fig. 3 and Table 3. The energy that is used for breaking agglomerates will now be used for breaking individual particles. The reduction in attractive forces also causes better dispersion of the fine particles, which results in easier flow, reduction or prevention of ball and wall coating.

Fig. 3 shows the relationship between grinding time and median diameter for each experimental condition. Here, C_s and C_{aids} are slurry concentration and concentration of grinding aids, respectively. The median diameter of ground products is decreased with the increase of grinding time in two levels of slurry concentration of 60 wt% and 70 wt% and is smaller when the grinding aids of 1.2 wt%

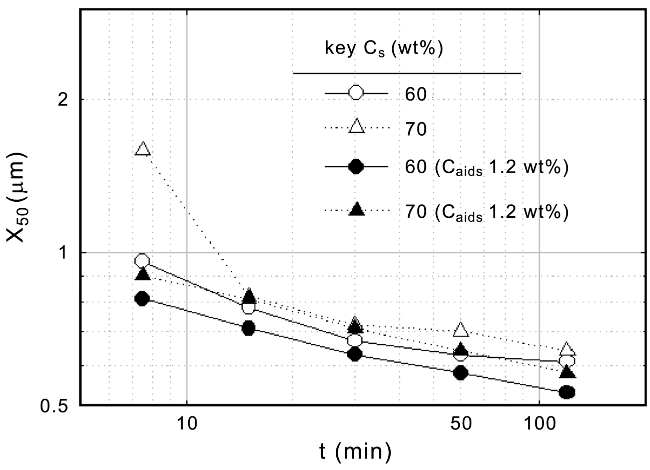

Fig. 3. The relationship between median diameter and grinding for experimental conditions: n=700 rpm, J=0.7 (-), d_B=1.0 mm.

is added in comparison to no addition of grinding aids. The reduction rate of median diameter is larger within grinding time of 30 min and it is decreased gradually with grinding time; and it is considered that the reduction of median diameter may stop after a longer grinding time, here, above 120 min. Furthermore, it is obvious that an optimum slurry concentration (C_s=60 wt%) exists, for which the finest products can be ground with a given experimental conditions as shown in Fig. 3.

Thus the time during which particle size distribution reached to a grinding limit was prolonged with addition of grinding aids in optimum level. This result suggested that grinding aids coat the particles, shielding them from agglomerating forces and thereby preventing the particle from cold welding as pointed out in the action mechanism of grinding aids above.

2. Change of Specific Grinding Consumption Energy

Fig. 4 shows the relationship between the increase of specific surface area and the specific grinding consumption energy. The increase of specific surface area is proportional to the specific grinding consumption energy at the initial stage of without grinding aids, where Rittinger's law governs in the very low E_m. Then it was confirmed that the limit theory of grinding fineness proposed by Tanaka could be applied to the data obtained within this experimental range. However, Rittinger's law governed only the first stage in this experimental range of adding grinding aids. Besides specific surface area did not reach grinding limit when grinding aids were used.

This phenomenon can be expressed by the following Eq. (2):

$$\frac{dS_w}{dE_m}=K(S_{w\infty}-S_w) \tag{2}$$

where, K is proportional constant, E_m is specific grinding consumption energy and $S_{w\infty}$ is specific surface area for t=∞. Eq. (2) can be solved to give Eq. (3) using the initial condition of $S_w=S_{w0}$ for t=0.

$$\frac{S_{w\infty}-S_w}{S_{w\infty}-S_{w0}}=\exp(-KE_m) \tag{3}$$

And Eq. (3) can be rewritten to Eq. (4),

$$\Delta S_w=\Delta S_{w\infty}[1-\exp(-KE_m)] \tag{4}$$

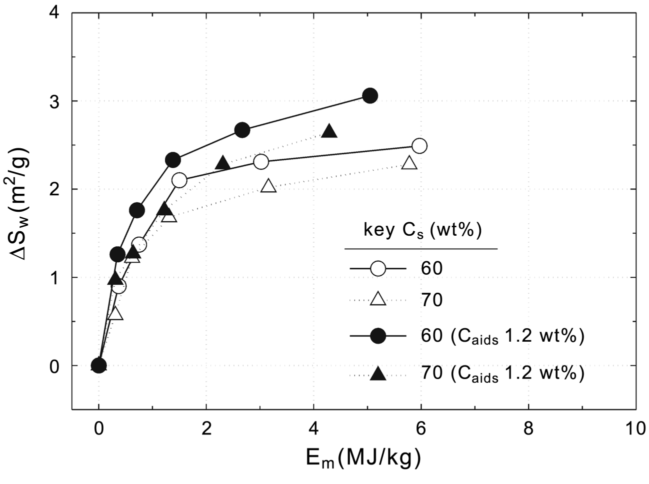

Fig. 4. The relationship between increase of specific surface area and grinding consumption energy for experimental conditions: n=700 rpm, J=0.7 (-), d_B=1.0 mm.

Table 4. Summary of regression analysis of experimental data applied to limit theory by Eq. (4)

	C_s [wt%]	$\Delta S_{w\infty}$ [m²/g]	K [kg/MJ]	R^2 [-]
Without grinding aids	60	2.4534	1.1729	0.9906
	70	2.0784	1.2365	0.9267
With grinding aids	60	2.8887	1.3478	0.9550
	70	2.5694	1.2778	0.9543

where, ΔS_w and $\Delta S_{w\infty}$ are the increased amount of specific surface area based weight for time=t and time=∞, respectively. They are defined as $\Delta S_w=S_w-S_{w0}$ and $\Delta S_{w\infty}=S_{w\infty}-S_{w0}$.

Table 4 shows the results applied to grinding limit theory by Eq. (4). Thus, the values of $\Delta S_{w\infty}$ and K were increased with adding grinding aids in this experiment compared with not adding grinding aids. ΔS_∞ for adding grinding aids was increased 17.7% and 23.6% for 60 wt% and 70 wt% of slurry concentration, respectively. K for adding grinding aids was increased 14.9% and 3.34% for 60 wt% and 70 wt% of slurry concentration, respectively.

3. Change of Grinding Energy Efficiency

Fig. 5 shows the relationship between the grinding time and the grinding energy efficiency. The grinding energy efficiency is decreased as the grinding time is increased, but it is increased with higher concentration range of slurry. This is due to particle friction in the later stage of breakage. The fine grinding is liable to be less efficient than owing to the greater tendency to overgrind in the former.

In this study, the grinding energy efficiency is defined as follows [Soc. Powder Technology, Japan, 1998]:

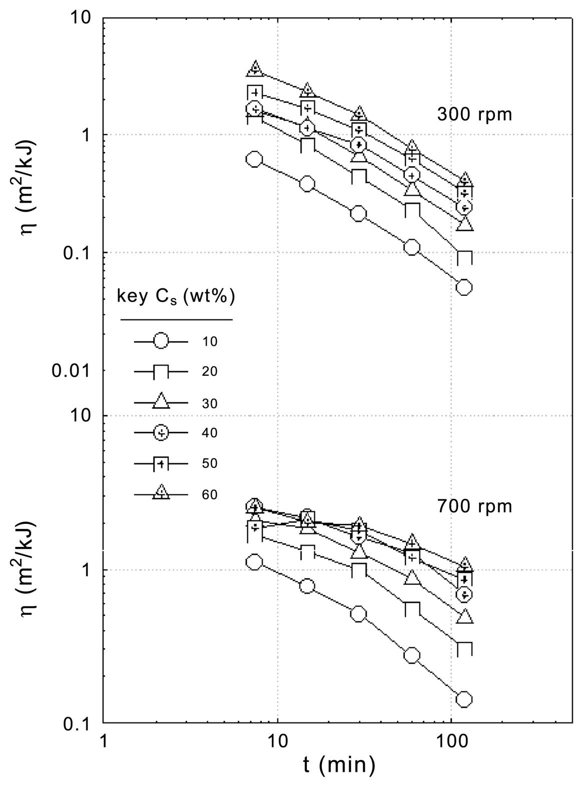

Fig. 5. The relationship between grinding energy efficiency and grinding time for experimental conditions: J=0.7 (-), d_B= 1.0 mm.

$$\eta=\Delta S_w/E_m \tag{5}$$

where ΔS_w and E_m were obtained from following Eq. (6) and Eq. (7), respectively.

$$\Delta S_w=(\phi/\rho_p)(1/x_{vs,p}-1/x_{vs,f}) \tag{6}$$

$$E_m=\int_0^t P(t)dt/W_s \tag{7}$$

where, η is grinding energy efficiency, ρ_p is particle density, $x_{vs,p}$, $x_{vs,f}$ are volume-surface diameter of product and feed material, respectively, and ϕ is the shape factor of specific surface area for particle. Here, the particle shape is assumed as spherical, ϕ=6 [Soc. Powder Technology, Japan, 1998].

Fig. 6 shows the relationship between the slurry concentration and the grinding energy efficiency. The discussion of solid concentration is a very important factor in a wet grinding operation because of its direct influence on the ground product fineness and operating power or energy consumption. The optimum experimental conditions are varied with the low rotation speed range and the high rotation speed range. In this experiment, the best grinding energy efficiency is found at 60 wt% of slurry concentration. The maximum value of grinding energy efficiency can be explained by two effects: in the region of small specific grinding consumption energy (low rotation speed), the number of particles increases by grinding, the stressing conditions become better and the grinding energy efficiency is improved. However, there is a point where the ground particles increase the viscosity of the suspension to such an extent that can hinder the movement of the media. This is the reason for the lower grinding energy efficiency in the region of high specific energy (high rotation speed) [Bernhart et al., 1999].

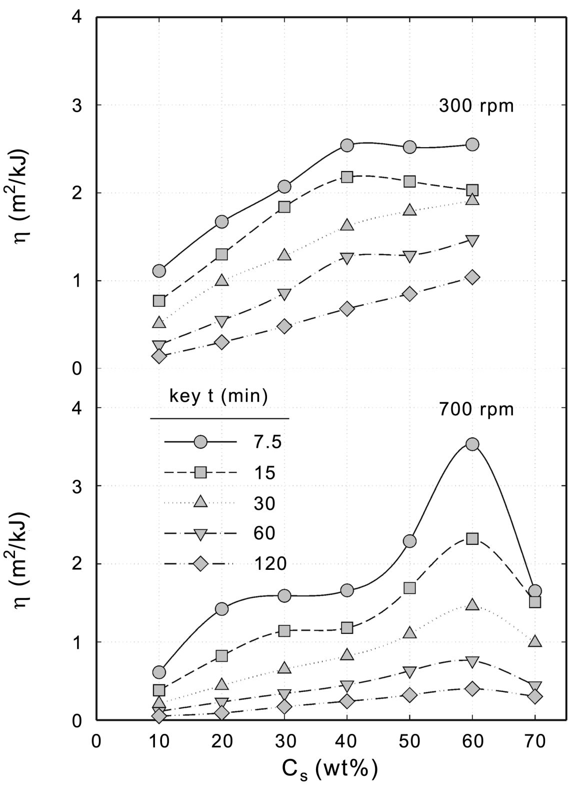

Fig. 6. The relationship between grinding energy efficiency and slurry concentration: J=0.7 (-), d_B=1.0 mm.

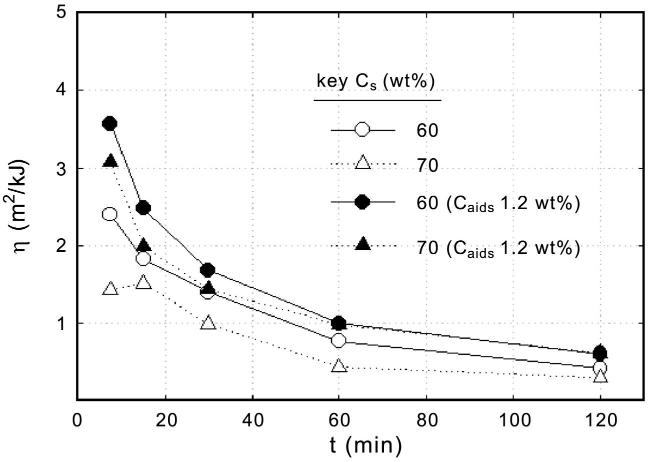

Fig. 7. The relationship between grinding energy efficiency and grinding time for experimental conditions: J=0.7 (-), d_B= 1.0 mm.

It is those nearly unground particles in that stationary region that account for the decrease in product fineness. Previous investigators [Arextion and Piret, 1950] used the concept of energy efficiency, but they did not perform sufficient tests at each particle size to obtain a statistical average applied energy for that size, and hence their result shows a considerable scatter, as would be expected. However, this experiment shows that the change of material affects the grinding energy efficiency.

Fig. 7 shows the relationship between the grinding time and the grinding energy efficiency on each experimental condition. The grinding energy efficiency was decreased with grinding time. In the case of adding grinding aids, the grinding energy efficiency was increased obviously [Bernhart et al., 1999]. In this figure, grinding energy efficiency for adding grinding aids was improved 45.2%, 58.9% for 60 wt%, 70 wt% of slurry concentration, respectively, in comparison with no grinding aids. It was found that the grinding aids have an important effect on grinding energy efficiency [Fuerstenau, 1995]. For wet grinding process at high solid concentrations, when improved grinding results from the presence of grinding aids, it is always accompanied by increased energy consumption. Grinding aids appear to a function of preventing reagglomeration by adsorbing on the external particle surface.

CONCLUSION

Experimental investigations on a grinding mechanism using inorganic powders in a stirred grinding media mill have been carried out. The effects of grinding aids on the particle size distribution and on the grinding efficiency were examined. The followings were found out.

1. The specific surface area based on particle size distribution by grinding aids was increased 22.9% and 15.8% on 60 wt% and 70 wt% of slurry concentration, respectively, compared with not adding grinding aids. When grinding aids were used, the median diameter decreased in a short time.

2. The grinding energy efficiency for adding grinding aids was

improved 45.2% and 58.9% on 60 wt% and 70 wt% of slurry concentration, respectively, compared with not adding grinding aids.

3. All the particles of submicron range were produced in the whole experimental cases of adding grinding aids.

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NOMENCLATURE

C_{aids} : grinding aids concentration based on weight [%]
C_s : slurry concentration based on weight [%]
d_B : grinding ball diameter [mm]
E : grinding consumption energy [MJ]
E_m : specific grinding consumption energy [MJ/kg]
J : ball filling ratio [-]
K : proportional constant [kg/MJ]
n : rotation speed of stirrer [-]
P : grinding consumption power [W]
Q_3 : cumulative particle size distribution based on weight [%]
S_w : specific surface area based on weight [m^2/g]
$S_{w\infty}$: specific surface area based on weigh for $t=\infty$ [m^2/g]
t : grinding time [min or h]
x : particle diameter [μm]
x_{10} : percentile particle diameter for 10% particle diameter [μm]
x_{50} : median diameter [μm]
x_{90} : percentile particle diameter for 90% particle diameter [μm]
$x_{vs,f}$: volume-surface diameter of feed [μm]
$x_{vs,p}$: volume-surface diameter of products [μm]

Greek Letters

η : grinding energy efficiency [m^2/kJ]
ρ_B : density of grinding ball [kg/m^3]
ΔS_w : increase amount of specific surface area based on weight [m^2/g]
$\Delta S_{w\infty}$: increase amount of specific surface area based on weight for $t=\infty$ [m^2/g]

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Retraction: "Ultra-fine grinding mechanism of inorganic powders in a stirred ball mill - The effect of grinding aids" [Korean J. Chem. Eng., 20(3), 554 (2003)]

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The authors hereby submit a formal retraction of the above paper. They have all agreed to the following statement:

Due to an inexcusable fault of the first author, the some figures in the paper published in the "HWAHAK KONGHAK, Vol. 40, No. 4, pp498-506, 2002" are reprinted in the above paper published in the "Korean J. Chem. Eng., Vol. 20, No. 3, pp554-559, 2003" with no reference. As the corresponding author, I (Choi) apologize for the unnecessary imposition on the editors' and reviewers' time and resources on the above manuscript published in the Korean J. of Chemical Engineering. The authors deeply apologize to the editors, the journal publisher, and the scientific community for this blatant breach of the ethical norm.

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Retraction: "Ultra-fine grinding mechanism of inorganic powders in a stirred ball mill - Examination of grinding kinetics of using grinding aids" [Korean J. Chem. Eng., 20(4), 783 (2003)]

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