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# Ultra-Fine Grinding Mechanism of Inorganic Powders in a Stirred Ball Mill
## - Examination of Grinding Kinetics of Using Grinding Aids -

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**Abstract**−Recently, in various industrial processes, the need for fine particles, especially submicron-sized particles, has increased in the field of preparing raw powders such as fine ceramics and high value added products. Therefore, the research in fine grinding has gained more importance, especially, in submicron grinding. In the previous paper, a series of wet grinding experiments using inorganic powders by a stirred ball mill were performed. The grinding consumption power was measured, and the grinding rate constant, K, in the grinding kinetics equation was examined, based on a grinding kinetics analysis of experimental specific surface area with particle size distribution of ground products obtained under various grinding conditions. Also the effect of grinding aids on grinding rate constant K was investigated. It was confirmed that the grinding rate constant K, when using grinding aids improved by 1.95% and 25.6% for a 60 wt% and 70wt% slurry concentrate, respectively, when compared with the case of the absence of grinding aids. It was found that grinding aids have an important effect on increasing the grinding rate, especially for a slurry of high concentration.

Key words: Ultra Fine Grinding, Submicron, Inorganic Powder, Stirred Ball Mill, Energy Efficiency, Grinding Mechanism, Grinding Rate Constant, Grinding Aids

## INTRODUCTION

Ultrafine grinding in the submicron range has recently become important due to the development of the new functional materials such as new ceramics and electronic materials for various industrial fields. The importance of stirred media mills increases steadily, because of an increasing demand for fine particles. In many cases, the grinding by a stirred ball mill to submicron range has been achieved commercially. Because of their easy operation, simple construction, high grinding rate and low energy consumption compared with the other fine grinding machines, stirred ball mills have received more and more attention in recent years [Jimbo, 1992; Choi, 1996]. In view of their growing importance, some basic researches of the power characteristics of stirred media mills have been carried out [Jimbo, 1992; Gao et al., 1995; Zheng et al., 1996; Choi, 1996; Kwade et al., 1999; Choi et al., 2000]. Shinohara et al. [1999] investigated the fine grinding characteristics of hard materials by means of the attrition mill, and Gao et al. [1996] found that by the increasing of power the size reduction was accelerated dramatically with only a small change to the energy efficiency of the process. Bernhardt et al. [1999] also showed that the slurry concentration has an important effect on grinding efficiency. Especially, the investigation of determining optimum experimental conditions was very important for increase of grinding efficiency. In addition, Forssberg et al. [1995] investigated that the influence of chemical dispersants on the milling of dolomite using a stirred ball mill and grinding aids in the ultra fine grinding is important from the viewpoint of grinding efficiency. Fuerstenau [1995] reviewed the use of chemical additives for improving the efficiency of both wet and dry comminution, and Hasegawa et al. [2001] studied the effect of liquid additives on dry ultrafine grinding of quartz.

Many investigators have studied the grinding kinetics of various grinding mills [Choi et al., 1996, 2000, 2001]. Kanda et al. [1998] carried out batch grinding tests of gypsum with a ball mill and investigated the influences of the feed size and the ball diameter on the grinding rate constant. Saito et al. [1998] studied the correlation between the rate constant of size reduction, K, and impact energy.

To examine further with the ultra-fine grinding mechanism, this investigation, that used inorganic powders by the stirred media mill, was carried out at various experimental conditions. The specific grinding energy based on the measured power and the increase of specific surface area of ground products is discussed.

Specifically, the effect of grinding aids on grinding rate constant K [Choi et al., 2001] was investigated. The values of the grinding rate constant K with or without grinding aids were compared at various experimental conditions.

## EXPERIMENTAL

### 1. Equipment

The grinding tests were performed in a vertical stirred media mill KMD-1B manufactured by Korea material development Co., LTD. This was the same equipment used in a previous paper [Choi et al., 2000]. Fig. 1 illustrates the dimensions of grinding chamber and the shape of a laboratory-scale stirrer. The net volume of milling chamber is 0.95 *l*. The grinding media alumina ball produced by

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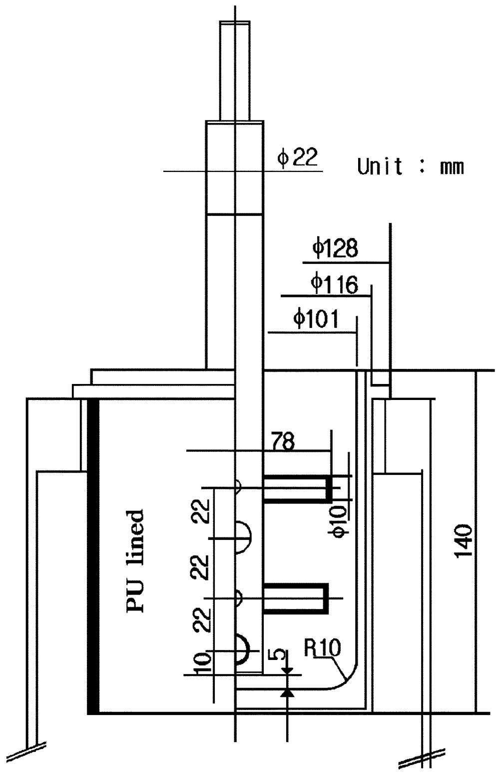


**Fig. 1. Dimensions and shape of grinding mill pot of experimental stirred ball mill.**

Nikkato Co., LTD. Japan, were of 99.9% purity, diameter $\phi$=1.0 mm, and a density of 3.60 g/cm$^3$. The measuring devices of energy consumption consisted of an automatic voltage regulator of GPA 100SS AVR, Sung Sin Electronic Co., LTD. Korea, and the detection circuit was made in our laboratory. The electronic power consumption of the motor for driving the stirrer was measured by an A/D converter (AX5210, Axiom Technology Inc. Taiwan) and Interface card (ACL-8112PG, AD Link Technology Inc. UK) with an on-line PC by turbo C++ software. The particle size distribution of ground products was analyzed by Mastersizer microplus (Malvern Co., LDT. UK), which uses laser diffraction and scattering. The homogenizer US-300T (Nihonseiki kaisha LTD. Japan) was used as a dispersion device. Calcite (S500, $x_{50}$=6.42 µm, density=2.72 g/cm$^3$, $S_{w0}$=1.42 m$^2$/g), talc (TS-1000, $x_{50}$=10.48 µm, density=2.74 g/cm$^3$, $S_{w0}$ =0.57 m$^2$/g) and pyrophyllite (PLW, $x_{50}$=4.07 µm, density=2.73 g/cm$^3$, $S_{w0}$=1.49 m$^2$/g), which are produced by Wang Pyo Chemicals Co., LTD. Korea were used as grinding materials. The grinding aids were produced by JEONG-WEON Chemical Co., Ltd., Korea. The basic material of these aids is poly-acrylic acid, $(CH_2CHCOOH)_n$.

**2. Methods**

The stirrer was rotated at a constant gap of 5 mm between the bottom and the end of stirrer. When the stirrer started rotating, the measuring of grinding consumption power was started with a computer on-line system. The computer program was calibrated by taking 10 measurements by multimeter [Choi et al., 2000]. The total charged weight of the balls for each value of ball filling ratio, J was 590 g for J=0.3, 980 g for J=0.5, 1,380 g for J=0.7, and the weight percent of the slurry concentration was changed between 10.0 and 70.0%. The grinding experiment was carried out on a batch process from which samples were taken from the pot at the determined grinding time interval. The particle size distribution was measured without any dispersion agent and prior to the measurement of particle size distribution; the homogenizer was used for the dispersion of particles for 90 seconds. After 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 effect of experimental parameters such as stirrer speed, ball size, ball filling ratio and slurry concentration on the particle size distribution of the products at different grinding time, a series of experiments were carried out. The grinding consumption power was recorded by Excel software every 2 minutes within the first 30 minutes, then every 5 minutes after that. Table 1 shows the summary of experimental conditions.

**Table 1. The summary of experimental conditions**

| Item | Experimental conditions |
|---|---|
| Rotation speed | 300, 500, 700 [rpm] |
| Ball filling ratio | 0.70 [-] |
| Ball size | 1.0 [mm] |
| Slurry concentration | 10.0-70.0 [wt%] |
| Temperature | Room temperature |
| Material of media | Alumina ($Al_2O_3$) |

In this experiment, two sets of tests were conducted to determine the effect of rotation speed and slurry concentration on the relationship between the specific surface area and the grinding rate constant K. The grinding experiment was carried out with a batch process, from which samples were taken from the pot at the determined grinding time interval. The same methods of addition of grinding aids used in the previous paper [Choi et al., 2000] were used in this experiment. The specific surface area of ground products based on weight was calculated from data of particle size distribution, where the shape of the ground product particles are assumed to be spherical according to the following equation:

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

Here, $S_w$ is specific surface area based on weight, $V_i$ is the relative volumes in size class i, $d_i$ is mean size class diameter and $\rho_p$ is particle density, D[3, 2] is surface weighted, or Sauter, mean diameter.

## RESULTS AND DISCUSSIONS

Fig. 2 shows the frequency of particle size distribution of products obtained at various grinding times. The initial distribution shows the existence of two populations. The median diameter of the coarser modal is about 20 µm, but it shifted from the right side to left during grinding. The second peak of lower frequency can be observed at around 4 µm on the initial distribution. However, these two populations merge into one broad one after 60 minutes. The peak of the finer population is centered at 0.9 µm. Increasing the grinding time does not only produce finer product, but also narrows the particle size distribution by reducing the coarse particle

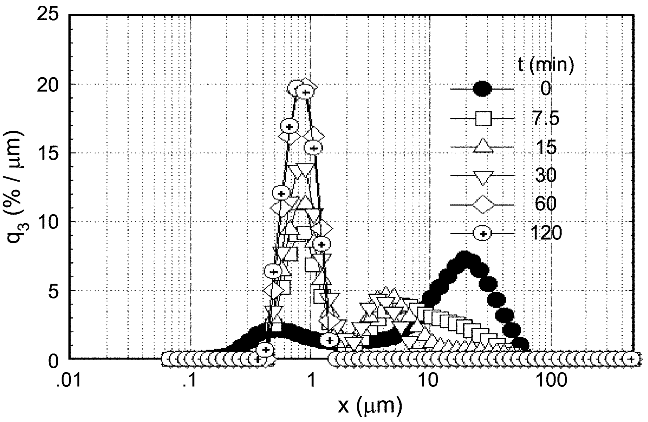


**Fig. 2. Particle size distribution of feed and ground products for experimental conditions (sample: calcite, exp. conds.: n=700 rpm, J=0.7 [-], $d_B$=1.0 mm, $C_s$=60 wt%).**

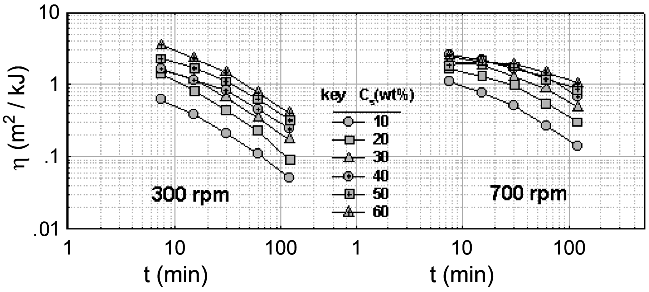


**Fig. 3. The relationship between grinding energy efficiency and grinding time for experimental conditions (sample: calcite, exp. conds.: J=0.7 [-], $d_B$=1.0 mm).**

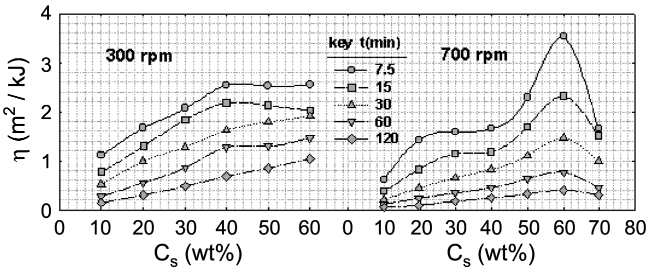


**Fig. 4. The relationship between grinding energy efficiency and slurry concentration for experimental conditions (sample: calcite, exp. conds.: J=0.7 [-], $d_B$=1.0 mm).**

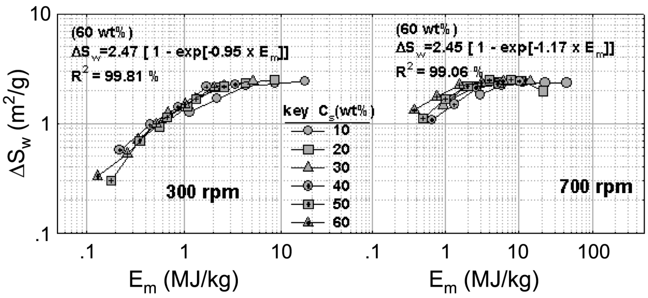


**Fig. 5. The relationship between increase of specific surface area and specific consumption energy for experimental conditions (sample: calcite, exp. conds.: J=0.7 [-], $d_B$=1.0 mm).**

size [Park et al., 1998].

Fig. 3 shows the relationship between grinding time and grinding energy efficiency. The grinding energy efficiency, $\eta$ is decreased as grinding time is increased and it is increased in the higher slurry concentration range. Where, the ground products are in existence at optimum particle range. It is also defined to take unit mass products. The grinding energy efficiency was defined as follows [Soc. Powder Technology, Japan, 1998],

$$\eta=\Delta S_w/E_m \tag{2}$$

where $\Delta S_w$ and $E_m$ were obtained from following Eq., respectively.

$$\Delta S_w=(\phi/\rho_p)(1/x_{vs,p}-1/x_{vs,f}) \tag{3}$$

$$E_m=\int_0^t P dt / W_s \tag{4}$$

where, $\eta$ is grinding energy efficiency, $\rho_p$ is particle density, $x_{vs,p}$, $x_{vs,t}$ are volume-surface diameter of product and feed material, respectively, and $\phi$ is the shape coefficient for specific surface area of particles. For this equation, the shape of particles is assumed to be spherical, $\phi$=6 [Soc. Powder Technology, Japan, 1998].

Fig. 4 shows the relationship between the slurry concentration and the grinding energy efficiency. The optimum experimental conditions are different with the low rotation speed range and the high rotation speed range. The best grinding energy efficiency is found at 60 wt% slurry concentration in this experimental range. In high slurry concentration (70 wt%), the powder is probably poorly wetted and size reduction takes place with nearly dry grinding modalities. Upon increasing the slurry concentration the decrease of in the amount of water deteriorates the flow-ability of the suspension and the efficiency of the size reduction process decreases.

Fig. 5 shows the relationship between the increasing of specific surface area and the specific grinding consumption energy. The increase in the amount of specific surface area is proportional to the specific grinding consumption energy at the initial stage when not using grinding aids, where Rittinger's law is governed by the very low $E_m$. 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 governs only the first stage in this experimental range when using grinding aids. The, specific surface area did not reach the grinding limit when grinding aids were used [Choi, 2000].

Figs. 6-1 and 6-2 show the change of specific surface area with grinding time, using balls with a 1.0 mm diameter and calcite powder. The specific surface area was increased with increase of grinding time. Fig. 6-1 shows the specific surface area $S_w$ as a function of grinding time according to slurry concentration, $C_s$ when operated at 700 rpm stirrer rotation speed and Fig. 6-2 shows the specific surface area $S_w$ as a function of grinding time according to stirrer rotation speed, n when operated in a 40 wt% slurry concentration.

This phenomenon can be expressed by the following Eq. (5):

$$\frac{dS_w}{dt}=K(S_{w\infty}-S_w) \tag{5}$$

Here, K is the grinding rate constant. Eq. (5) can be solved to give Eq. (6) using the initial condition of $S_w=S_{w0}$ for t=0.

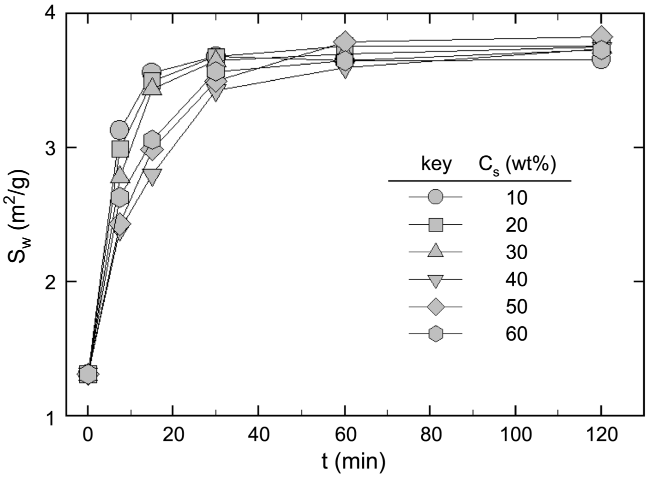


**Fig. 6-1. The relationship between specific surface area and grinding time for various slurry concentrations (sample: calcite, exp. conds.: calcite, n=700, J=0.7 [-], $d_B$=1.0 mm).**

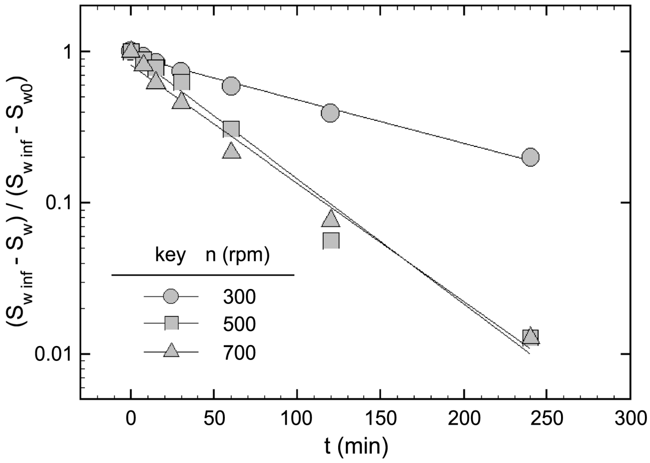


**Fig. 7-1. Change value of specific surface area and grinding time for various rotation speeds (sample: calcite, exp. conds.: J=0.7 [-], $d_B$=1.0 mm, $C_s$=40 wt%).**

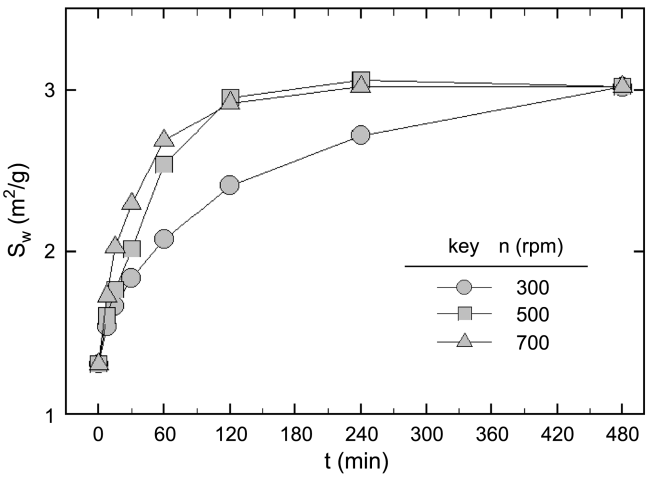


**Fig. 6-2. The relationship between specific surface area and grinding time for various rotation speed (sample: calcite, exp. conds.: calcite, J=0.7 [-], $d_B$=1.0 mm, $C_s$=40 wt%).**

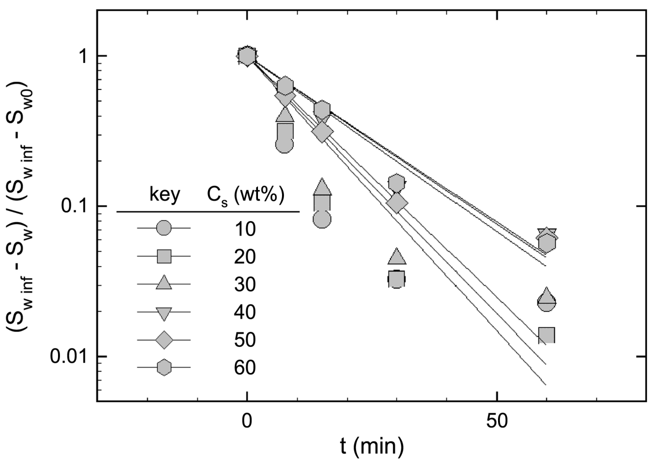


**Fig. 7-2. Change value of specific surface area and grinding time for various slurry concentrations (sample: calcite, exp. conds.: n=700 rpm, J=0.7 [-], $d_B$=1.0 mm).**

$$\frac{S_{w\infty}-S_w}{S_{w\infty}-S_{w0}}=\exp(-Kt) \quad (6)$$

The equation to describe the relationship between the newly created solid surface and the energy consumed by it in the very fine size range was first proposed by Tanaka.

Figs. 7-1 and 7-2 show the relationship between the change of specific surface area and the grinding time by using balls with 1.0 mm diameter and calcite powder. This presents a linear relationship when plotted on semi-log graph paper, and it is possible to calculate the value of K in Eq. (6). These plots indicate that the value of K is dependent on the rotation speed and slurry concentration. This experiment demonstrates this linearity in many cases. Austin et al. [1976] have proved it under certain conditions for the tumbling ball mill, vibration and hammer mills and shredder cutters. The linearity was demonstrated for a small vertical spindle mill used in a batch process without airflow by Austin et al.

Fig. 8 shows the relationship between K and rotation speed n,

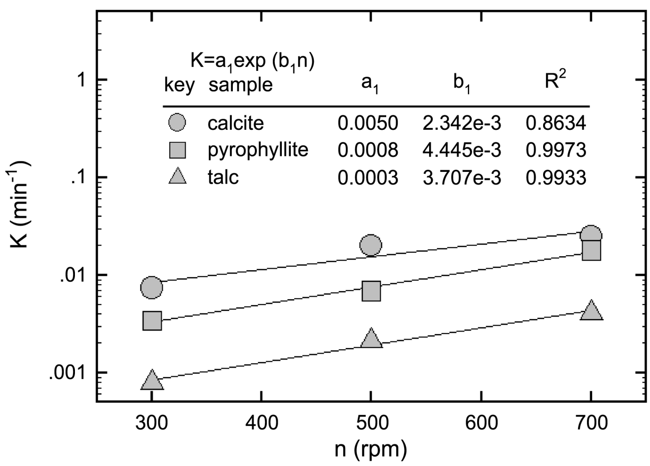


**Fig. 8. The relationship between grinding rate constant K and rotation speed for various test samples (exp. conds.: J=0.7 [-], $d_B$=1.0 mm, $C_s$=40 wt%).**

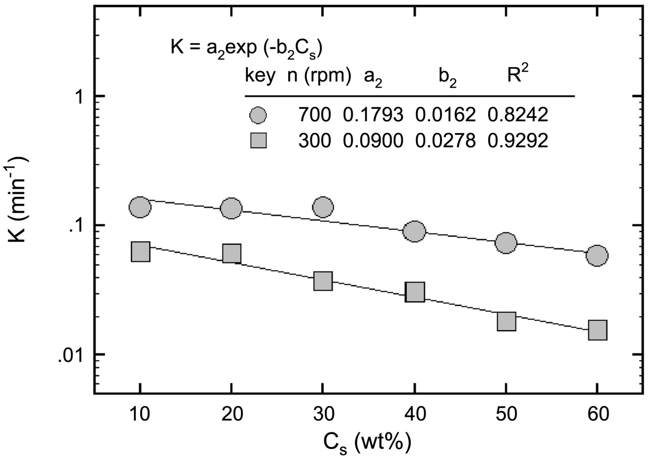


**Fig. 9. The relationship between grinding rate constant K and slurry concentration for various rotation speeds (sample: calcite, exp. conds.: J=0.7 [-], $d_B$=1.0 mm).**

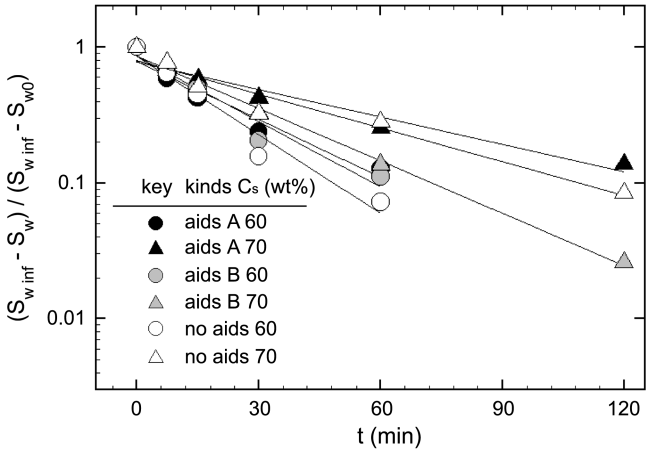


**Fig. 11. Change value of specific surface area and grinding time for experimental conditions (sample: calcite, exp. conds.: n=700 rpm, J=0.7 [-], $d_B$=1.0 mm).**

which was plotted on the semi-log scale with various test samples of calcite, pyrophyllite and talc. K increases as rotation speed increases. This tendency is the same for all of the test samples. All the regression data for each plot show similar results. The result is that the grinding rates are proportional to the mill torque and the latter can be used directly to monitor and analyze the mill performance.

Fig. 9 shows the relationship between K and slurry concentration $C_s$, which was plotted on the semi-log scale with different rotation speeds the lowest a rotation speed of 300 rpm and the highest a rotation speed of 700 rpm K decreases as slurry concentration increases. The regression slopes of the experimental conditions are different between the low rotation speed range and the high rotation speed range. The particle size distributions of ground products are a function of the cumulative energy input only in normal grinding systems, even when the power drawn by the mill varies appreciably with change of milling conditions.

Fig. 10 shows the relationship between the change of specific surface area and the grinding time using balls with a 1.0 mm diameter and calcite powder. It shows the specific surface area $S_w$ as a function of grinding time according to with a parameter of slurry concentration, $C_s$ with or without grinding aids operated at 700 rpm stirrer rotation speed and a 1.2 wt% grinding aids concentration with experimental conditions. It was confirmed that the grinding aids were effective in increasing the specific surface area.

Fig. 11 shows the relationship between the change of specific surface area and the grinding time using balls with a 1.0 mm diameter and calcite powder. This figure presents the linear relationships when plotted on semi-log graph paper, and that it is possible to calculate the value of K in Eq. (6). These plots indicate that the value of K is dependent upon the addition of grinding aids and the slurry concentration.

Fig. 12 shows the relationship between the grinding rate constant K and the slurry concentrations when using grinding aids. The optimum experimental conditions when using grinding aids varied with the slurry concentration. It was confirmed that the grinding rate constant K, when using grinding aids, improved by 1.95% and 25.6% in a 60 wt% and 70 wt% slurry concentration, respectively,

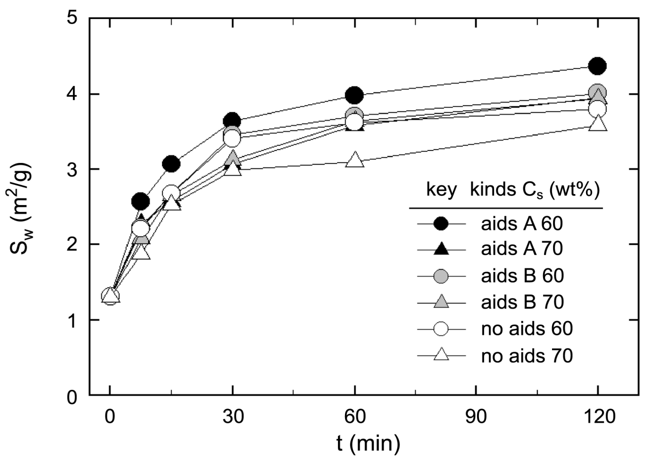


**Fig. 10. The relationship between specific surface area and grinding time for experimental conditions (sample: calcite, exp. conds.: n=700, J=0.7 [-], $d_B$=1.0 mm, $C_{aids}$=1.2 wt%).**

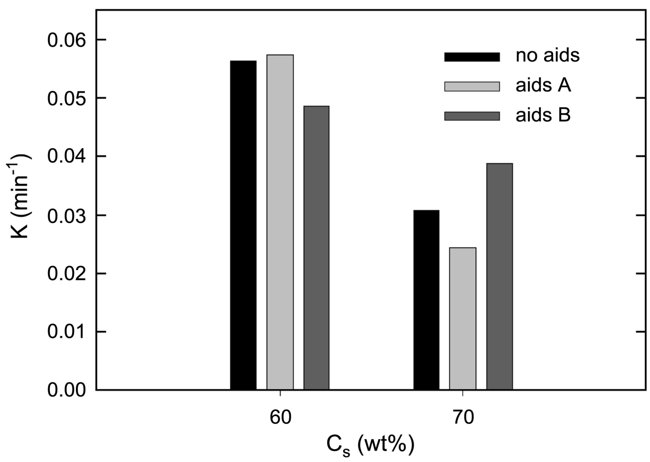


**Fig. 12. The grinding rate constant K and slurry concentration for experimental conditions (sample: calcite, exp. conds.: n=700, J=0.7 [-], $d_B$=1.0 mm, $C_{aids}$=1.2 wt%).**

when compared with the case of the absence of grinding aids. It was found that the grinding aids have an important effect on increasing the grinding rate, especially in high concentration slurries. For wet grinding processes at high solid concentrations, when improving grinding results with the presence of grinding aids, it is always accompanied by increased energy consumption. Grinding aids appear to serve the function of preventing reagglomeration by adsorbing on the external particle surface.

## CONCLUSIONS

A series of wet grinding experiments using calcite, pyrophyllite, and talc powder in a vertical type stirred ball mill have been carried out in order to investigate the influence of the various experimental conditions on the grinding energy efficiency, the particle size and the grinding rate constant K. The main results are summarized as follows:

1. The grinding energy efficiency decreased with an increase in grinding time. The optimum operating conditions for the grinding energy efficiency were obtained within these experimental ranges as follows:

rotation speed: n=700 rpm, ball charge ratio: J=0.7 (-),
ball size: $d_B$=1.0 mm,
sample concentration: $C_s$=60 wt%,
grinding energy efficiency (at the grinding time 30 min): $\eta$=1.46 $m^2/kJ$

2. The grinding rate constant, K increased with an increase in rotation speed. The grinding rate constant, K, was represented for rotation speed, n as follows:

$$K=a_1\exp(b_1 n)$$

: $a_1$=0.0900, $b_1$=0.0278, $R^2$=0.9292 (for 300 rpm)
: $a_1$=0.1793, $b_1$=0.0162, $R^2$=0.8242 (for 700 rpm)

3. The grinding rate constant, K decreased with an increase in slurry concentration, $C_s$. The grinding rate constant, K, was represented for test materials as follows:

$$K=a_1\exp(-b_1 C_s)$$

: $a_1$=0.0050, $b_1=2.342\times10^{-3}$, $R^2$=0.8634 (for calcite)
: $a_1$=0.0008, $b_1=4.445\times10^{-3}$, $R^2$=0.9973 (for pyrophyllite)
: $a_1$=0.0003, $b_1=3.707\times10^{-3}$, $R^2$=0.9933 (for talc)

4. It is confirmed that the grinding rate constant was improved when using grinding aids compared with not using grinding aids. It is also confirmed that the optimum level of grinding aids on an experimental condition exists.

## ACKNOWLEDGMENT

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## NOMENCLATURE

$C_s$ : slurry concentration based on weight [%]
$C_{aids}$ : grinding aids concentration based on weight [%]
D[3, 2]: surface weighted, or Sauter mean diameter [μm]
$d_B$ : grinding ball diameter [mm]
$d_i$ : mean diameter of size class i [μm]
E : grinding consumption energy [MJ]
$E_m$ : specific grinding consumption energy [MJ/kg]
J : ball filling ratio [-]
K : grinding rate constant [$min^{-1}$]
n : rotation speed of stirrer [rpm]
P : power consumed to grinding [W]
$q_3$ : frequency percent under particle size [%]
$S_w$ : specific surface area based on weight [$m^2/g$]
$S_{w\infty}$ : limit specific surface area based on weight [$m^2/g$]
t : grinding time [min]
$V_i$ : the relative volumes in size class i [$m^3$]
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]

### Greek Letters

$\Delta S_w$ : increase amount of specific surface area based on weight [$m^2/g$]
$\eta$ : grinding energy efficiency [$m^2/kJ$]
$\rho_p$ : density of particle [$kg/m^3$]
$\rho_B$ : density of grinding ball [$kg/m^3$]

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