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GRINDING TIME FOR CONTROL OF THE SIZE FRACTION OF PRODUCTS IN THE ATTRITION MILLING

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Abstract – Grinding tests for garnet were carried out by using an attrition mill under wet processes. Effects of feed filling ratios and a chemical agent (sodium hexametaphosphate, SHP) were investigated on the grinding time of the garnet. The progeny particles obtained were screened into various particle size intervals, which were 100 mesh over, 100/400 mesh and 400 mesh under. In order to estimate the mass fraction of the particles in a given particle size interval, mathematical models were derived from the first-order reaction model, then compared to experimental data. It was observed that variation of the feed filling ratio did not show a significant effect on the mass fraction of the product. The chemical agent was, however, effective so that the mass fraction could be controlled by adjusting the content of SHP.

Key words : Attrition Mill, Sodium Hexametaphosphate, Grinding Time, Mathematical Model, Feed Filling Ratio

INTRODUCTION

Many grinding process models have been suggested for estimation of the size distribution of products. The grinding process model was first based on the differential size-mass balance, then extended by applying matrix algebra, the concept of stages of breakage [Harris, 1966] and a continuous probability formulation [Kelsall et al., 1968; Berube et al., 1979]. Application of this concept to experimental data has been demonstrated successfully [Hwang and Lee, 1991; Kapur and Agrawal, 1970]. The mathematical models suggested in these demonstrations contained two main functions: the breakage function (B) and the selection function (S). These functions have been applied for estimating the grinding behavior of industrial milling processes with both wet and dry conditions. An attrition mill is a highly effective device for the rapid grinding of solid materials. This milling method was developed by U.S. Bureau of Mines in the early 1960s.

Garnet has been used as the grit of abrasive materials, rock cutters and sandpapers. Recently, garnet has been of great use as the abrasive material for manufacturing Braun tubes of televisions and monitors. Since the electric industry has grown, the demand for garnet as the abrasive material has increased. In Korea, though garnet is deposited in Chungchongdo as associated minerals in granite, the garnet material for domestic needs has depended entirely on imports because of the lack of refining and separation techniques. The particle size of the garnet grits ranges between 100 and 400 mesh. Therefore, three size intervals (100 mesh over, 100/400 mesh, 400 mesh under) were considered in order to control the mass fraction of 100/400 mesh. Experimental data were compared to simulated results obtained from the first-order equation. In this report, we have attempted to develop a grinding technique which makes the mass fraction of a given particle size controlled by a chemical grinding agent and a grinding process model giving an optimum grinding time. The effects of slurry densities and grinding agent contents were investigated in relation to the rate of grinding and the variation of the mass fraction at a given size interval.

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BATCH GRINDING MODEL

1. Grinding Model of First-order Equation

A grinding model consists of selection functions leading to the probability of particles to be ground and breakage functions leading to the size distributions after instantaneous grinding. By a standard screening, a mixture of particles with various sizes can be separated into several size intervals, 1...,i,j,... n. These variables take values between 1 for the mass fraction of particles retained on the coarsest screen 1, and n+1 for the fraction of particles passing through the finest screen n. The breakage function B_{ij}, is the probability of particles to distribute from a size interval j to i by grinding and is related to the cumulative mass fraction of each size interval. The selection function S_j, for a size interval j, is the rate of breakage of that size (min^{-1}). The grinding functions S and B of a specific material depend on its physico-mechanical properties and experimental grinding conditions.

If $m_i(t)$ represents the mass fraction of a size interval i at

a milling time of t, the mass balance equation can be written as below.

$$\frac{dm_i}{dt} = -K_i m_i(t) + \sum_{j=1}^{i-1} V_{ij} m_j(t) \qquad (i>j) \tag{1}$$

Here K_i is the breakage rate constant of the size interval i, V_{ij} is assigned to S_jB_{ij}, and $m_i(t)$ is the mass fraction of the size interval i at the milling time of t. With increasing the grinding time, initial particles at each size interval are ground and pass through a smaller size interval. At the milling time t, a part of the particles ground reach the size interval i. The fraction of the particles at the size interval i is, therefore, the sum of the fractions of particles produced from each upper size interval (<i). This grinding phenomenon will simultaneously occur in all size intervals. As a result, the mass fraction of the size interval i at t is obtained by subtracting the cumulative mass fraction of particles passing through the screen i+1 from the cumulative mass fraction of particles on the screen i. Fig. 1(a) shows this material balance. When the number of the size intervals of interest is few, the grinding model can be simplified from the mass balance equations of each size interval, which can be derived from the conventional grinding equation such as Eq. (1).

Fig. 1(b) illustrates that particles at the interval 1 shift to the lower interval by grinding, that is the first-order grinding model having two size intervals. If the size intervals 1 and 2 are supposed to be one size interval in (c) with first-order grinding, the model (a) can be explained by two equations derived from (b) and (c) by the first-order concept. Eqs. (3), (5) and (6) describe the grinding behavior in the mill.

In Fig. 1, a change in the fraction of the size interval 1 can be represented by Eq. (2)

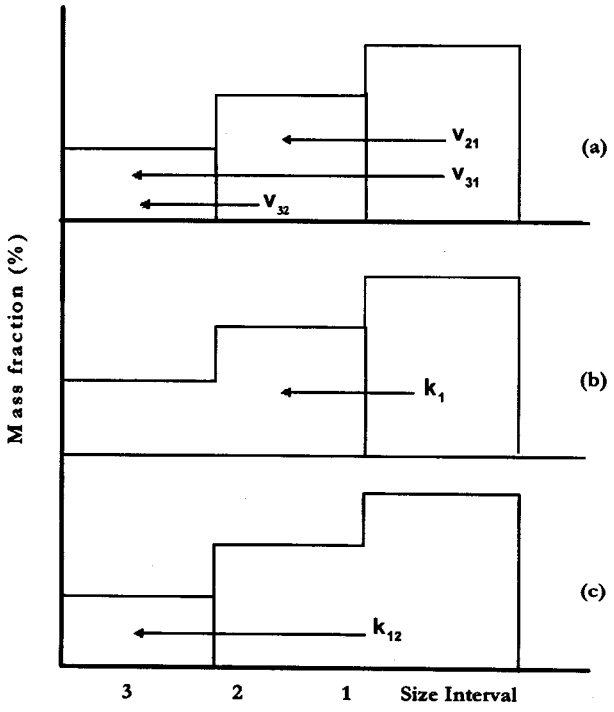

Fig. 1. Illustration of the mass balance for each size interval.

$$\frac{dm_1}{dt} = -K_1 \cdot m_1 \tag{2}$$

Eq. (2) is derived from Eq. (1) by eliminating the Σ term for i=1 for the size interval 1, and the Σ term equals to zero. If K_1 is constant at any grinding time, Eq. (2) is integrated to Eq. (3).

$$m_1(t) = m_1(0)e^{-K_1 t} \tag{3}$$

In Eq. (3), $m_1(t)$ expresses the mass fraction of the size interval 1 at the grinding time of t, $m_1(0)$ is the initial mass fraction of the size interval 1. K_1 is the rate constant for the size interval 1.

If the initial size interval is expanded into a new size interval including the original size intervals 1 and 2, a change of the mass fraction of this new interval can be written by Eq. (4).

$$m_1(t) + m_2(t) = \{m_1(0) + m_2(0)\}e^{-K_{12}t} \tag{4}$$

Here K_{12} is the rate constant for the new interval. If total feed amounts in the mill have a unit that equals to $m_1+m_2+m_3$, the change of the mass fraction in the interval 3, $m_3(t)$, is followed by Eq. (5).

$$m_3(t) = 1 - \{m_1(0) + m_2(0)\}e^{-K_{12}t} \tag{5}$$

From Eqs. (3) and (4), the change of the mass fraction in the interval 2, $m_2(t)$, is represented by Eq. (6).

$$m_2(t) = \{m_1(0) + m_2(0)\}e^{-K_{12}t} - m_1(0) \cdot e^{-K_1 t} \tag{6}$$

2. Determination of Rate Constant

The rate of disappearance, by breakage, of material from a given size interval is given by Eqs. (3) and (5) where K_1 and K_{12} are the breakage rate constants for the given size interval. Milling rates for the garnet powder were determined from cumulative size distribution curves as shown in Fig. 2.

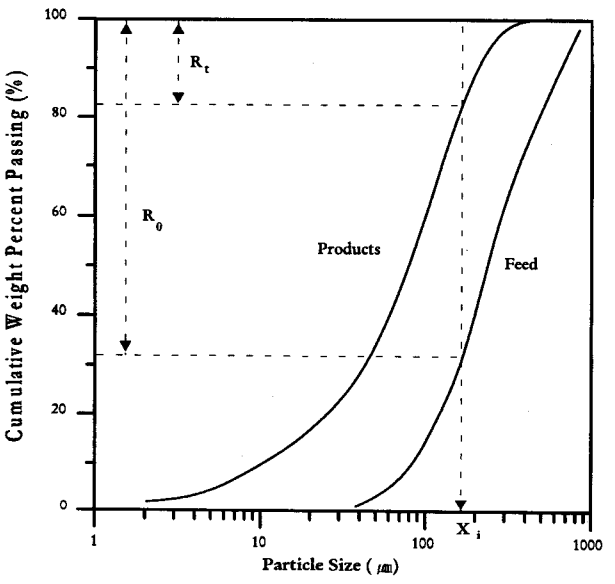

Fig. 2. Mass fractions over 100 mesh, X_i, for feed and products, respectively.

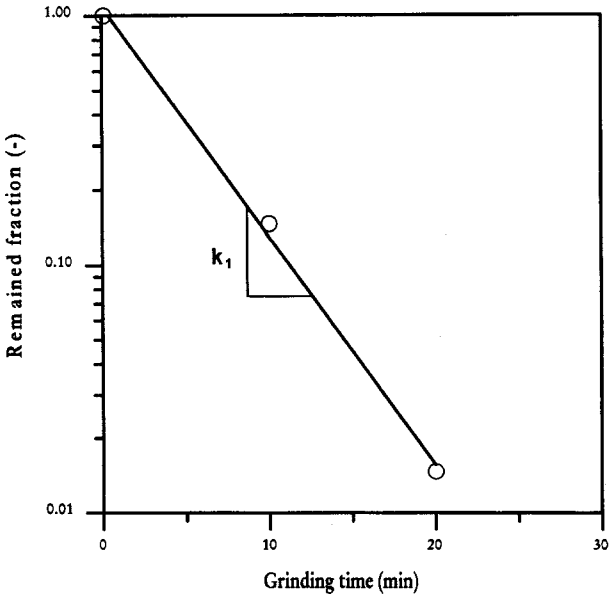

Fig. 3. The rate constant for the size interval of the over 100 mesh.

In this figure, the initial mass fraction of a size interval of x_i (and over) is expressed by R_0. After milling for a given time, the residual mass fraction of the size interval of x_i (and over) is changed from R_0 to R_f. Thus the ratio of R_f/R_0 is obtained from cumulative size distribution curves. By using the semi-log scale, these ratios can be plotted in a line of which slope is the rate constant. The remaining fraction R_f/R_0 of the over 100 mesh in a constant feed filling ratio is depicted in Fig. 3 as an example. The values of the slopes of these lines are rate constants K_1.

EXPRIMENTAL

1. Materials

The raw material used in these experiments was garnet powder recovered as the by-product from raw ore of feldspar. The mean size of garnet feed d_{50} was 250 um, and it was also observed that the amount of 100 mesh undersize was about 27 %. From the chemical analysis, it was found that the sample garnet are formed of two constituent minerals, that is, almandine $Fe_3Al_2(SiO_4)_3$ and spessartine $Mn_3Al_2(SiO_4)_3$.

2. Milling Tests

All grinding experiments were carried out by using a small attrition mill, which consisted of a stainless steel 1,000 cc jar, a cooling jacket, a stainless steel impeller with 4 blades and a motor. The impeller was connected to a 1HP motor to control the rotating speed. Steel balls of 5 mm in diameter were used as the grinding media.

Size distribution analyses were performed using a Laser Diffraction Particle Size Analyzer (SALD-2001, SHIMADZU) in the range of 0.03-700 um. Samples for the size distribution analysis were obtained using a pipette at the center of the jar.

The grinding tests were carried out by varying the feed filling ratio, U, and the chemical agent content, C. U is the ratio of the powder volume to the interstitial volume of balls in the jar. The fraction ball filling J was 0.45, and the slurry density was 45 wt%. J is conventionally expressed as the fraction of mill filled by the ball bed at rest. The feed filling ratios were 0.6, 1.0 and 1.4 and the contents of the chemical agent (sodium hexametaphosphate, SHP) were 0, 0.01, 0.04, 0.16, 0.64 wt% by solid when U was 1.0.

RESULTS

1. Size Distributions of Ground Particles

1-1. Effects of Feed Filling Ratio

Table 1 shows the characteristics of grinding with varying the feed filling ratio when the slurry density is 45 wt% and J is 0.45. As shown in Table 1, the mean size of the progeny particle d_{50} declined with decreasing the feed filling ratio. This is considered to be due to an increase of collision probability between particles and media as the feed filling ratio is lower. Though the hardness of garnet is 6.5-7.5, fine particles were obtained in a relatively short time using the attrition mill. When the feed filling ratio was 0.6, the time reaching the grinding equilibrium was about 80 min. It is assumed that an increase of specific surface area with increasing the grinding time causes an increase of slurry viscosity. The increase of the resistance force of slurry flow in the mill may result in a reduction of grinding effects in wet processes.

1-2. Effects of Grinding Agent Contents

The amount of fine particles increases with increasing the grinding time. If the surface charges of particles are low, the molecular attraction force may cause coagulation of particles resulting in an increase of the slurry viscosity. This coagulation can be prevented by addition of a chemical agent referred to as a grinding agent. A major effect of the grinding agent is to protect particles from the coagulation by changing the surface charges of particles followed by inducing electric repulsion force [James, 1987; Austin et al., 1984], thereby improving the slurry stability. As a result of increased stability, the slurry viscosity decreases.

In these experiment, sodium hexametaphosphate $[(NaPO_3)_6]$ was used for the grinding agent. Phosphate anion PO_3^- in liquid are strongly chemisorbed onto the surface of the garnet particle, and owing to their adsorption they change the charge of the particle. On the other hand, Na^+ ions are assumed to have a number of water molecules orientationally bound to them.

Table 1. Mean size and weight fraction of products with variation of the feed filling ratio

Grinding time (min)	Mean size, d_{50} (μm)			Mass fraction of 100/400 mesh (-)		
	U=0.6	U=1.0	U=1.4	U=0.6	U=1.0	U=1.4
0		250			0.27	
10	40.9	78.5	101.4	0.40	0.54	0.45
20	17.6	28.8	51.3	0.22	0.36	0.52
40	7.7	12.7	23.8	0.04	0.13	0.34
80	3.8	7.8	10.2	0.01	0.025	0.05
160	3.6	4.4	5.3	0	0.001	0.005
320	3.5	3.1	3.3	0	0	0

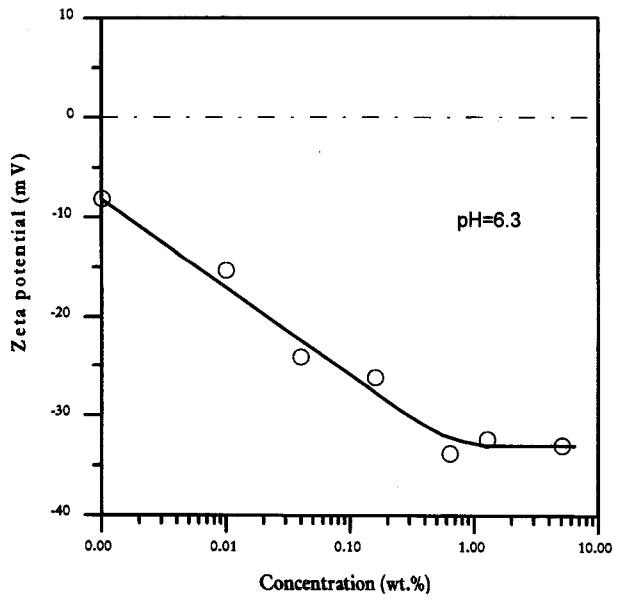

Fig. 4. Zeta potential of garnet as a function of SHP concentrations.

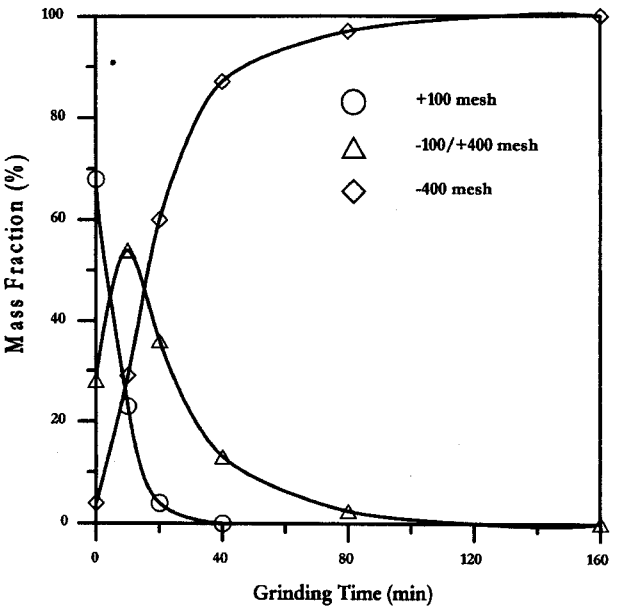

Fig. 5. Variations of mass fraction for each size interval with grinding time.

Table 2. Mean size and weight fraction of products with variation of SHP contents

Grinding time	Mean size (μm)				Mass fraction of 100/400 mesh (-)			
(min)	c=0.01	c=0.04	c=0.16	c=0.64	c=0.01	c=0.04	c=0.16	c=0.64
0	250				0.27			
10	82.3	70.2	49.2	37.2	0.50	0.71	0.53	0.40
20	40.5	43.5	22.5	9.5	0.42	0.52	0.33	0.08
40	18.6	15.3	16.5	7.4	0.24	0.18	0.20	0
80	8.8	8.5	8.0	6.4	0.05	0.04	0.05	0
160	4.6	4.5	5.5	4.2	0	0	0	0
320	3.4	3.5	3.6	2.7	0	0	0	0

Table 3. Grinding rate constant K_1, K_{12} and its ratio K_1/K_{12} for different conditions

		K_1	K_{12}	K_1/K_{12}
c=0	U=0.6	0.21	0.069	3.04
	U=1.0	0.13	0.042	2.99
	U=1.4	0.09	0.025	3.78
U=1.0	c=0.01	0.11	0.032	3.30
	c=0.04	0.18	0.035	5.11
	c=0.16	0.21	0.045	4.67
	c=0.64	0.24	0.095	2.49

In other words, it is supposed that the garnet particle has large negative charge with addition of SHP. Fig. 4 shows the zeta-potential charge of the garnet particle as a function of SHP concentration at pH 6.3. The zeta-potential charge has a large change in the range of 0.6-1.0 wt%.

The rate constants K_1 and K_{12} vary with the values of S_j by adding SHP, thereby changing the mass fraction in the wet grinding process. Table 2 shows mean sizes and weight fractions of products with varying SHP contents. Compared to d_{50} when U is 1.0 as shown in Table 1, the effect of SHP on the rate of size reduction was remarkable at the initial stage of milling, but not significant after 160 min.

2. Mass Fractions

Fig. 5 shows the mass fractions for each size interval at the feed filling ratio U=1.0 without adding SHP. As particles were ground, the fraction of the over 100 mesh decreased and disappeared after 30 min. The fraction of 400 mesh under, however, increased over 99 % after 100 min. For the fraction of 100/400 mesh, the highest value of the fraction was obtained at about 10 min. This indicates that the fraction of 100/400 mesh increases with an increase of the rate constant ratio K_1/K_{12}. The grinding rate constants K_1, K_{12} and the ratio K_1/K_{12} listed in Table 3 were obtained from the size distribution curves of progeny particles in different conditions. The variation of the 100/400 mesh fraction was shown in Fig. 6 as a function of grinding time at different feed filling ratios. The grinding time t_{opt} showing a maximum fraction of 100/400 mesh was obtained from Eq. (7) which was derived by differentiating Eq. (6) with boundary conditions.

$$t_{opt} = \frac{\ln K_1 - \ln K_{12} + \ln\{1 + m_2(0)/m_1(0)\}}{K_1 - K_{12}} \quad (7)$$

It was not observed in Fig. 6 that the maximum fraction of 100/400 mesh was affected by the feed filling ratio. This result is attributed to a similarity in the rate constant ratios as shown in Table 3. Changes in the rate constant with the feed filling ratio may be caused by changes in the impact frequencies of particles to the grinding media in the mill. The rate constant K_1 showed a relatively linear increase with increasing the SHP content. It is assumed that the addition of SHP is more effective not on coarse particles but on fine particles produced by grinding. These particles may not coagulate together, because the grinding agent SHP generates a repulsion force between particles.

Fig. 7 represents the optimal grinding time to obtain the maximum fraction of the 100/400 mesh. The fraction of the

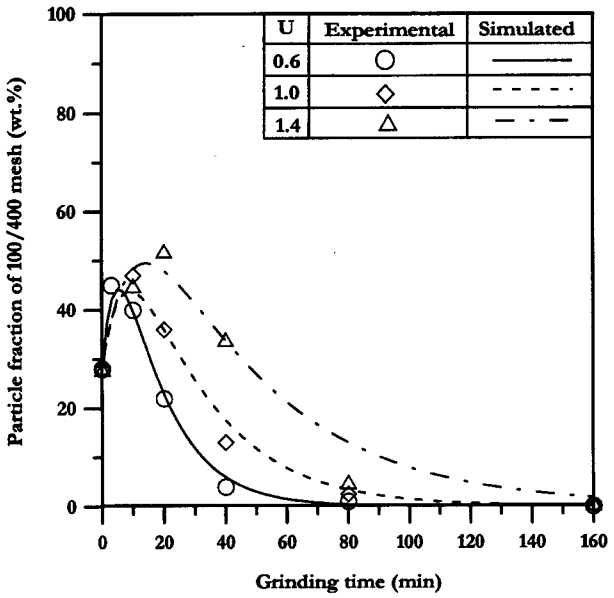

Fig. 6. Mass fraction of 100/400 mesh of products for different feed filling ratios.

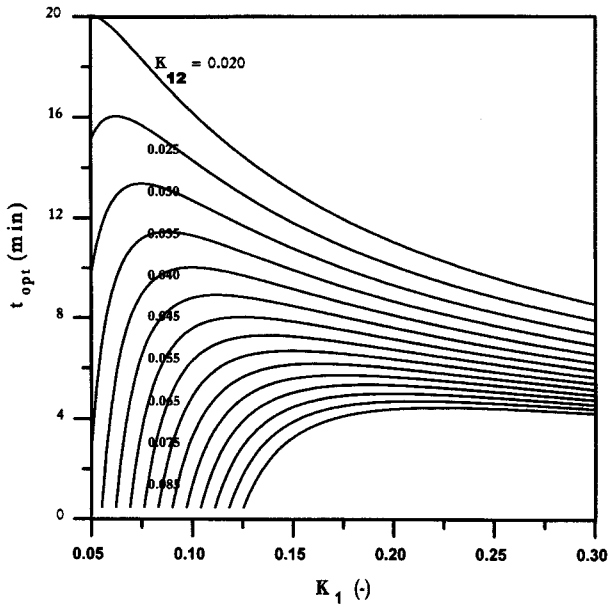

Fig. 7. Grinding time for the maximum mass fraction of 100/400 mesh estimated from Eq. (8).

100/400 mesh is shown in Fig. 8 as a function of the grinding agent SHP contents. When the SHP content was 0.04 wt%, the fraction of 100/400 mesh showed the highest value of about 70 %. As can be seen in Table 3, K_1/K_{12} was a maximum value at the SHP content of 0.04 wt%. This result leads to the fact that the size fraction of progeny particles is controlled by the grinding agent in the wet milling.

CONCLUSIONS

In this study, grinding tests for garnet were carried out by using an attrition mill under wet conditions. Effects of feed filling ratios and chemical agent (sodium hexametaphosphate,

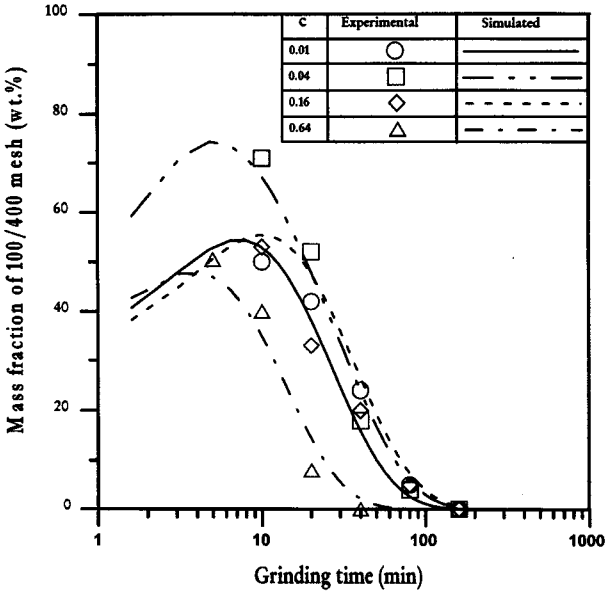

Fig. 8. Comparison of experimental results with those simulated by Eq. (6).

SHP) contents on the products ground were investigated. The results are as follows:

1. Variation of the mass fraction of the size interval followed first-order equations.
2. As the feed filling ratio increased, the time to reach a maximum of the mass fraction of 100/400 mesh increased, but the maximum mass fractions obtained were not changed significantly. This is considered to be due to an increase of collision probability between particles and media as the feed filling ratio is lower. When the feed filling ratio was 1.0, the maximum of the mass fraction of 100/400 mesh reached about 54 % at about 10 min.
3. The rate constant at each size interval was different with contents of SHP. The mass fraction of 100/400 mesh showed a maximum at the SHP content of 0.04 wt%. It was considered that the grinding agent affected on the behavior of fine particles, but the rate constant of grinding had a larger effect on coarse particles.
4. At the grinding condition to obtain the maximum mass fraction of 100/400 mesh, the rate constant K_1 showed a large increase, while the rate constant K_{12} had a small change. This condition was achieved by adjusting the SHP content, and the feed filling ratio was not a crucial factor.

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