A New Method for Measurement of Powder Characteristics Based on Reentrainment Phenomena†

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Abstract

A new method has been developed to measure and evaluate the operational powder characteristics for fine particle processing based on the reentrainment phenomena. Experiments on particle reentrainment were carried out using an accelerated air flow for 21 different test powders. Both the reentrainment fluxes, which were measured by an electrostatic method, and the average air velocities were automatically sampled by a computer. The sampled data were processed to obtain the reentrainment profiles as a function of the average air velocity. The cumulative reentrainment efficiencies were also obtained and represented as a function of the wall shear stress.

It was found that fine particles having tendencies to form large aggregates were reentrained mainly in the early stage of the measurement when the flow velocity was as low as $5 \text{ m}\cdot\text{s}^{-1}$. Also, the mass ratio of the reentrained large aggregates to the total particles was used to obtain information on the particle-particle (cohesion) or particle-wall interactions (adhesion). On the other hand, the cumulative reentrainment efficiency-curves must be applied to determine the operational conditions controlling the amount of adhered particles in various aerosol processes.

1. Introduction

The quality and functions of industrial products using fine particles can be enhanced with the use of smaller particles. However the reduction in particle size increases adhesion between particles or between particles and wall surfaces and this reduces the operational qualities. Compared to the wet process, the reduction of adhesion in the dry process is difficult and especially with aerosol operation, the effect of air flow in addition to the matter of adhesion complicates the problem. For example, when fine particles deposit on inner walls of equipment by turbulent diffusion, the primary particles retain and form aggregate particles there owing to the effect of their adhesion and when the separation strength due to fluid resistance becomes stronger than the adhesion strength of the aggregate particles, reentrainment is induced¹⁾. This

phenomenon is a problem which relates to almost all aerosol operations such as dust collection, dispersion, classification, pneumatic conveying, etc. The reentrainment mechanism, at first sight, may seem to be very simple but when an actual attempt is made to obtain the time course of reentrainment as a function of the adhesion strength distribution of the aggregate particles or the separation strength within the turbulence boundary layer, even an approximate analysis is difficult. Therefore, studies in the past on reentrainment mainly centered on clarifying its mechanisms and on making models¹⁻¹⁰⁾. However the estimation of the various reentrainment phenomena for each powder using such studies alone is difficult and how powder characteristics are to be evaluated remains an issue for future clarification.

Obtaining characteristics relating to adhesion strength is possible by measuring the adhesion strength of primary particles by a centrifugal method or by measuring the stress through various destructive tests of the powder layer. However, reentrainment

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is controlled by the balance between the adhesion strength of particles and the separation strength caused by an air flow, and studies must be made on the separation strength. When particle reentrainment is analyzed using a stress model, the separation strength can be equated to the product of wall shear stress caused by an air flow and the dimensionless form factor relating to reentrainment¹⁰⁾ but it is impossible to estimate theoretically the values of form factors for each powder. However, it is possible to obtain practical operational powder characteristics including form factors by measuring the wall shear stress which corresponds to reentrainment.

In the present study, fine particles deposited on a wall surface were reentrained by a continuous increase of the air velocity and the reentrainment phenomena for each type of powder were discussed using adhesion and aggregation properties. Furthermore, basic studies were performed in order to apply the relation between reentrainment and wall shear stress as a new method for measuring operational powder characteristics.

2. Test equipment and measuring method

Figure. 1 shows the structural outline of the test equipment. This equipment reentrains small amounts of powder attached to wall surfaces by an air flow. From the point of practical application, the equipment was made compact and data were automatically analyzed by digital processing. The amount of powder required for one measurement was less than several milligrams and the required measuring time was about 15 minutes.

Clean compressed air was used for particle reentrainment, and the electromagnetic valve for the air flow was controlled by a personal computer to increase the flow velocity at a constant rate. The average air velocity in the test section was obtained from the output signal of the mass flowmeter using the personal computer. For arithmetic processing, the output signal was calibrated by a volumetric type gas meter in advance and compensation calculation was incorporated. Aerosol discharged through the test section was evacuated into a reentrainment particle detector and the reentrained amount per unit time and area was obtained. Namely, the particle reentrainment flux could be obtained from the value of minute electric current generated when reentrained particles collided with the detection tube¹¹⁾. Since the sensitivity of the electrostatic detection method is high, the method is especially suitable for detection

Fig. 1 Experimental apparatus

of low concentration aerosol particle flow. A short stainless steel tube (JIS SUS 304), 70 mm long with an inner diameter of 6 mm, was used as a detection tube. Since stainless steel has a hard oxide surface film, it is superior in electrostatic stability¹²⁾. Also, to avoid deviation of the standard values of the generated electric current due to changes in the collision velocity of reentrained particles, it was necessary to maintain a constant average air velocity inside the detection tube. The test section of this equipment and the detector are not connected by piping and the setting was made so that the suction flow rate into the detector was greater than the maximum flow rate of compressed air passing through the test section. The minute electric current generated at the detector was amplified and then converted to voltage and automatically sampled as digital signals. The sampling cycle was 0.1 second and sampling was taken at 8000 points per one measurement (not less than 50 points while the average flow velocity increases up to $1 \text{ m} \cdot \text{s}^{-1}$). The minimum digital input unit of the generated electric current was 0.05 pA and a maximum of 100 pA could be measured. The background in a room in normal conditions was not more than 0.05 pA.

The test section consisted of a rectangular channel (width 8 mm, height 1 mm, total length 135 mm) made of stainless steel with a glass window on top. The test piece on which particles were deposited was installed behind a 100 mm fore-flow region at the same height as that of the inner surface of the channel. The selection of the test piece material was optional but here, a general purpose stainless steel (JIS SUS 304) was used with a normal machined surface finish (center line average roughness of $0.1 \sim 0.2 \text{ } \mu\text{m}$). As shown in **Table 1**, the test powders were the micron order powders for industrial testing (JIS Z8901) and also included a wide selection of others such as alumina abrasives, metals, and plastics. Among the powders, inorganic oxides which do not change in quality in an oven (at 120°C) were

Table 1 Test powders used

No.	Material	D_{p50} (μm)	$\rho_p \times 10^{-3}$ ($\text{kg} \cdot \text{m}^{-3}$)	$W/A \times 10^3$ ($\text{kg} \cdot \text{m}^{-2}$)
1	Quartz powder (JIS Z8901-3)	7	2.7	1.7 ~ 4.6
2	Talc (JIS Z8901-4)	8	2.8	1.7 ~ 2.3
3	Talc (JIS Z8901-9)	4	2.8	1.7 ~ 4.0
4	Fly ash (JIS Z8901-5)	15	2.2	3.4 ~ 11.7
5	Fly ash (JIS Z8901-10)	5	2.2	3.4 ~ 8.6
6	Kanto loam (JIS Z8901-8)	7	3.0	1.1 ~ 5.7
7	Kanto loam (JIS Z8901-11)	2	3.0	2.3 ~ 4.6
8	Calcium bicarbonate (JIS Z8901-16)	4	2.8	1.7 ~ 4.0
9	Calcium bicarbonate (JIS Z8901-17)	2	2.8	1.1 ~ 2.3
10	Alumina (APPIE standard powder no.1)	3	4.0	1.7 ~ 3.4
11	Alumina (APPIE standard powder no.2)	5	4.0	1.7 ~ 4.0
12	Alumina (CB-A05; spherical particle)	3	4.0	1.7 ~ 2.9
13	Alumina (WA #320)	44	4.0	5.7 ~ 17.2
14	Alumina (WA #1000)	17	4.0	3.4 ~ 10.9
15	Alumina (WA #8000)	1	4.0	2.9 ~ 4.0
16	Antimony trioxide	7	5.2	5.7 ~ 13.2
17	Antimony trioxide (fine)	4	5.2	0.4 ~ 11.5
18	Copper	20	8.9	1.7 ~ 3.4
19	Toner	12	1.2	0.6 ~ 1.7
20	Polymethyl methacrylate (PMMA)	7	1.1	1.7 ~ 4.0
21	Polytetrafluoroethylene (PTFE)	30	2.2	2.9 ~ 12.1

dried for not less than 12 hours before their use. Deposition of particles on the test piece was carried out with the help of a sieve. With consideration to screening a suitable amount of micron order particles by normal vibrations, the openings were set at $74 \mu\text{m}$ (200 mesh). A test piece was cleaned with ethanol and air dried, then particles were dropped under gravity onto the center of the test piece (width 6 mm by length 30 mm). The reentrainment test was then conducted immediately.

3. Experimental results and discussion

3.1 Characteristics of equipment

The flow near the wall surface inside the channel is especially important since it is directly related to the particle reentrainment. In general, flow near the wall surface is analyzed by using friction velocity u^* defined by the following equation.

$$u^* = \sqrt{\tau_w / \rho} \quad (1)$$

where τ_w is the wall shear stress and ρ is the air density. The wall shear stress is proportional to the separation strength relating to particle reentrainment¹⁰⁾, and it is necessary to obtain the channel flow characteristics of the equipment (relation be-

tween wall shear stress and average air velocity) in advance. The wall shear stress τ_w can be obtained using the following equation.

$$\tau_w = \Delta p D_e / (4L) \quad (2)$$

where Δp is the pressure loss in section L , and D_e is the hydraulic diameter of the channel. Pressure taps were attached at the mounting position of the test piece and pressure loss was measured by a diaphragm type differential pressure gauge. As shown by the solid line in **Fig. 2**, the wall shear stress is divided into two zones and both can be approximated by a function of the average air velocity. The obtained functions were incorporated in the program and used for data analysis. For reference, the results of the Hagen-Poiseuille equation and Blasius empirical formula applied respectively to laminar flow and turbulent flow zones are shown by the two broken lines.

3.2 Observations of particles on a test piece

Figure 3 shows the particles deposited on the test piece. As for Kanto loam (JIS Z8901-8) having small mass median diameter, large aggregates in an insular pattern are conspicuous. On the other hand, in alumina WA #320 having large mass median dia-

Fig. 2 Characteristic of flow

meter, almost all of them are primary particles, not aggregates.

Figures 4 and 5 give the progressive process of reentrainment of fine particles of Kanto loam (JIS Z8901-8) and fly ash (JIS Z8901-5) as typical examples. The basic reentrainment tendency of fine particles is similar and at low air velocity, relatively large aggregate particles are vigorously reentrained from insular aggregates and when the average air velocity reaches $20 \sim 30 \text{ m} \cdot \text{s}^{-1}$, hardly any large aggregates are seen on the test piece. With the rise of air velocity, successive reentrainments of small aggregate particles continue. Reentrainment of primary particles or very minute aggregate particles becomes difficult and even when the average air velocity exceeds $100 \text{ m} \cdot \text{s}^{-1}$, an extremely small residue of particles remain. However, there is a difference in the degree of reentrainment of aggregate particles depending on the type of the powder. Furthermore, in cases where hardly any aggregate particles are on the test piece from the beginning and only relatively large primary particles exist, such as with alumina WA #320, reentrainment progresses in a relatively mild manner.

(1) Kanto loam (Z8901-8)
 $D_{p50} = 7 \mu\text{m}$, $W/A = 5.7 \times 10^{-3} \text{ kg} \cdot \text{m}^{-2}$

(3) Copper
 $D_{p50} = 20 \mu\text{m}$, $W/A = 2.9 \times 10^{-3} \text{ kg} \cdot \text{m}^{-2}$

(2) Fly ash (Z8901-5)
 $D_{p50} = 15 \mu\text{m}$, $W/A = 1.17 \times 10^{-2} \text{ kg} \cdot \text{m}^{-2}$

(4) Alumina (WA #320)
 $D_{p50} = 44 \mu\text{m}$, $W/A = 1.21 \times 10^{-2} \text{ kg} \cdot \text{m}^{-2}$

0 500 μm

Fig. 3 Photomicrographs of test powder adhering to the test piece

Fig. 4 Photomicrographs of particles remaining on the surface (Kanto loam, JIS Z8901-8)

3.3 Measurement results of generated electric current

Taking the reentrainment test of alumina abrasives as an example, Fig. 6 shows the output results of generated current with elapsed time when reentrained particles collide with the detection tube. In the bottom graph of the figure, the time course of the average air velocity is shown. Pulse current corresponding to momentary reentrainment is detected, but actually, since multiple particles collide simultaneously inside the detection tube, a condition in which pulse currents are cumulative is measured. Therefore, the generated current corresponds to the particle reentrainment flux on the test piece and reentrainment tendency can be derived from the overall increase and decrease of the generated current. Furthermore, information on reentrainment can be obtained from the momentary variation of the generated current. This is to say, generated current with a large variation indicates an interrupted or

Fig. 5 Photomicrographs of particles remaining on the surface (Fly ash, JIS Z8901-5)

uneven reentrainment while conversely, a small variation in the generated current indicates that a continuous and uniform reentrainment is occurring. Basically, the generated current becomes larger because particles are more readily reentrained with an increase in flow rate but when reentrainment continues further, the amount of particles remaining on the test piece is reduced and therefore, the current gradually becomes smaller. As will be explained next, it was found that the generated current displayed a number of patterns depending on the characteristics of the powder.

Figure 6 shows the results of a reentrainment test of particles having the same quality (alumina, WA) but with three different mass median diameters ($D_{p50} = 1, 17, 44 \mu\text{m}$). With WA #8000 which has a small mass median diameter ($D_{p50} = 1 \mu\text{m}$), the generated current varies greatly at low air velocity but when this condition ends with the air velocity becoming higher, a minute current continues to be

Fig. 6 Generated currents and average air velocity as a function of time elapsed

generated. As described in section 3. 2, the generated current at low air velocity corresponds to a vigorous reentrainment of relatively large aggregate particles. The generated current when the air velocity is high corresponds to the reentrainment of small aggregate particles but here, the quantity is extremely small. Even with WA #1000 having a slightly larger mass median diameter ($D_{p50} = 17\mu\text{m}$), generation of current at low air velocity is observed but compared with WA #8000, the value is small. However when the air velocity becomes higher, the current again starts to increase and eventually, decreases slowly. At this time, a similar increasing and decreasing tendency is observed in the lower limit value of the generated current at each moment, this is because a pulse form current keeps on being generated and indicates that small aggregate particles

or primary particles are continuously reentrained. With WA #320 ($D_{p50} = 44\mu\text{m}$) which has an even larger mass median diameter, there are hardly any aggregate particles from the beginning (refer to Fig. 3 (4)), and generation of current corresponding to the reentrainment of primary particles is clearly observed. In this way, the difference in primary particle diameters affects the aggregating characteristics and it was confirmed that the adhesion and aggregating state of powder on a test piece affect the time course of the generated current.

Now, when time integration of the generated current is performed, a electric charge corresponding to reentrainment quantity is obtained and when this electric charge is made dimensionless as in the following equation, the cumulative reentrainment efficiency η can be obtained.

$$\eta = \frac{\int_0^t Idt}{\int_0^{t_e} Idt} \quad (3)$$

where t_e is the time up to the completion of the measurement. With regard to the alumina abrasive mentioned above, the output results of the relation between cumulative reentrainment efficiency and average air velocity are shown in Fig. 7. In this test, the condition of dynamic reentrainment in which the average air velocity continues to increase was measured and strictly speaking, the effect due to the delay of the reentrainment is included in the cumulative reentrainment efficiency η^{8-10} . The value of η for the respective powders increases in accordance with the increase in average air velocity but a difference in the increasing tendency is observed with different mass median diameters. Here, the ratio of reentrained particles at low air velocity against total particles is defined as η_L . η_L marked

Fig. 7 Relationship between cumulative reentrainment efficiency η and average air velocity $\bar{u}$

by $\bigcirc$ in **Fig. 7** is the cumulative reentrainment efficiency at the inflection point. A notable difference is seen in η_L with $\eta_L = 0.95$ for WA #8000 ($D_{p50} = 1 \mu\text{m}$), $\eta_L = 0.25$ for WA #1000 ($D_{p50} = 17 \mu\text{m}$), and $\eta_L = 0.05$ for WA #320 ($D_{p50} = 44 \mu\text{m}$). In general, it is considered that reentrainment of particles having smaller mass median diameters becomes more difficult, but this should be applied only to the reentrainment of primary particles from a wall surface. It must be considered that as particle diameters become smaller, large aggregate particles are more readily formed. It is hinted that unless this is considered, results contrary to predictions may be obtained. Also, it can be predicted that the initial state may vary depending on the adhered amount of powder on the test piece but within the range of the adhered amount shown in **Table 1**, no apparent effect was observed. It is thought that this is because the effect of expansion of the area occupied by the powder on the test piece is greater than the effect of the powder being stacked in the vertical direction when the powder amount is increased. Of course, when the adhered amount becomes so great that the surface of the test piece can no longer be seen, the adhered amount will greatly affect the reentrainment. Furthermore, the reentrainment of relatively large aggregate particles is seen at low air velocities of not more than $5 \text{ m}\cdot\text{s}^{-1}$ and reentrainment of small aggregate particles or primary particles starts to progress when the air velocity exceeds $10 \text{ m}\cdot\text{s}^{-1}$. As shown in **Fig. 2**, the flow at the test section changes greatly at a velocity of $19 \text{ m}\cdot\text{s}^{-1}$ but no notable change in the reentrainment is seen at this velocity in **Fig. 7**. Therefore, these reentrainment results are not characteristic of the equipment and can be applied to evaluate the operational characteristics of powder.

Next, the derivative of cumulative reentrainment efficiency η by $\bar{u}$ is shown in **Fig. 8** against the average air velocity $\bar{u}$. This figure corresponds to the relation shown in **Fig. 6**. Here, the entire reentrainment profile which was hidden by the momentary generated current changes and which could not be observed in **Fig. 6** can be clearly seen.

3.4 Results relating to reentrainment of various powders

The reentrainment tendency of powder with differing mass median diameters was studied in the previous chapter and here, the reentrainment of various powders with a narrow range of mass median diameters of $4 \sim 7 \mu\text{m}$ is studied. **Figure 9** shows

Fig. 8 Reentrainment efficiency distributions as a function of average air velocity

the generated currents of 5 kinds of powders as a function of the elapsed time. Depending on the kind of powder, the generated current value differs greatly and therefore, a simple comparison of the particle reentrainment is impossible but differences can be seen in the time course of the generated currents. **Figure 10** shows $d\eta/d\bar{u}$ obtained from the cumulative reentrainment efficiency η of Eq. (3). With Kanto loam (JIS Z8901-8) and calcium bicarbonate (JIS Z8901-16), it is seen that reentrainment of aggregate particles at low air velocity is dominant while the proportion of aggregate particles at low air velocity diminishes in the order of talc (JIS Z8901-9), fly ash (JIS Z8901-10), and alumina (APPIE standard no. 2). These are typical results and these profiles are roughly simulated with other fine particles. Next, the output results of the cumulative reentrainment efficiency η are shown in **Fig. 11**. Here, to eliminate the effect of the size of the test channel and to apply to industrial equipment, the wall shear stress τ_w instead of the average air velocity $\bar{u}$ was used for the abscissa. The respective curves show the characteristics relating to reentrainment. It is seen that when special conditions to prevent all fine particles in the order of microns from adhering to the wall are required, the wall shear stress must be made

Fig. 9 Generated currents as a function of time elapsed ($\alpha \cong 0.17 \text{ m}\cdot\text{s}^{-2}$).

extremely large. However, it is important to control the adhered amount in general powder processes and the characteristic curve shown with the cumulative reentrainment efficiency η is effective in selecting such conditions. For example, to reduce the adhered amount of powder to $1/n$ of the actual amount, shear stress corresponding to η_1 must be selected as the new condition instead of η_0 . The following relation is seen between η_1 and η_0 .

$$\eta_1 = 1 - (1 - \eta_0)/n \quad (4)$$

Fig. 10 Reentrainment efficiency distributions as a function of average air velocity

In this test, reentrainment of aggregate particles was mainly targeted and **Fig. 12** summarizes the proportion η_L of reentrainment of large aggregate particles at low air velocities for all tested powders. A certain degree of scatter is seen but the characteristics of each powder are displayed and it can be considered that η_L is a characteristic including both aggregating and reentraining properties. This shows that as η_L approaches unity, reentrainment at low air velocity becomes easier. This is to say that with

Fig. 11 Relationship between cumulative reentrainment efficiency η and wall shear stress τ_w

such powder, the adhesion properties between particles are strong and large aggregate particles are readily formed. Conversely, $\eta_L \approx 0$ corresponds to a

powder for which the adhesion between aggregate and wall occurs more strongly than the adhesion between aggregate and aggregate. In particular, those with extremely high aggregating properties ($\eta_L \approx 1$), and on the other hand, those with low aggregating properties ($\eta_L \approx 0$) have fairly good reproducibilities.

Finally, a study was carried out on the applicability of the electrostatic detection method for the respective powders. Figure 13 shows the electric charge per unit mass of tested powders q/W_r . There are powders having a positive polarity and others having a negative polarity. It is thought that Fig. 13 illustrates a kind of an electrostatic series. The test powders include metals and plastics in addition to inorganic oxides, and q/W_r reaches a value of the order of $10^{-5} \sim 10^{-2} \text{ C} \cdot \text{kg}^{-1}$. It was possible to detect all of these sufficiently.

The adhesion state of particles to the wall differs depending on how the particles are made to deposit on the test piece and this should affect the reentrainment. Here, a most convenient screening method was adopted and it was possible to evaluate the difference in aggregate conditions by mass median diameter, and it is thought that this is a practical method for processes in which gently deposited aggregate particles are reentrained. However, it is necessary to review the deposition method of the

Fig. 12 Mass ratio of reentrained large aggregates to total particles (cf. Table 1)

particles in accordance with the process applied. Namely, depositing conditions such as dispersion efficiency, particle inertia and wall surface material as well as the ambient conditions such as temperature, humidity which are as close as possible to those in the actual process should be selected. It is not so difficult to take measurements using an deposition method which fulfills these conditions and data an-

alysis can be applied successfully using the method which we have described in this paper. Depending on the deposition method, it may be possible to grasp the mutual relation between particles (cohesion), and between particles and wall surface (adhesion) more clearly. In this regard, further detailed studies are scheduled for the future.

Fig. 13 Characteristics of particle electrification (cf. Table 1)

4. Conclusion

Using methods to increase the air velocity successively, reentrainment tests of various powders were conducted and the following conclusions were reached.

- (1) It is possible to obtain reentrainment profiles from the generated current values by the electrostatic method and it was found that reentrainment could be generally divided into an uneven reentrainment of the relatively large aggregate particles at low air velocity and a uniform reentrainment of the small aggregate particles or primary particles at high air velocity.
- (2) Aggregating properties become stronger as the size of the mass median diameter becomes smaller with reentrainment at low air velocity being dominant but depending on the type of

powder, great differences are observed in aggregating and reentraining properties.

- (3) It is possible to obtain the electric charge corresponding to the reentrainment quantity from time integration of generated current and it is possible to obtain the cumulative reentrainment efficiency by making the generated current dimensionless using the total electric charge corresponding to the total reentrainment quantity. This relation between the cumulative reentrainment efficiency and the wall shear stress gives a powder characteristic curve relating to reentrainment and by using this curve, conditions of the air flow for controlling the powder amount adhering to the wall surface can be selected.

Nomenclature

A	: area covered by deposited particles	(m ²)
D_e	: hydraulic diameter of rectangular channel	(m)
D_{p50}	: mass median diameter	(m)
I	: generated electric current	(A)
L	: distance between pressure taps	(m)
n	: variable in Eq. (4)	(—)
Δp	: pressure loss	(Pa)
q	: total charge	(C)
t	: time	(s)
t_e	: measuring time	(s)
$\bar{u}$	: average air velocity	(m•s ⁻¹)
u^*	: friction velocity	(m•s ⁻¹)
W	: mass of deposited particles	(kg)
W_r	: mass of reentrained particles	(kg)
α	: air acceleration	(m•s ⁻²)
η, η_0, η_1	: cumulative reentrainment efficiency	(—)
η_L	: mass ratio of large aggregates to total particles	(—)
ρ	: air density	(kg•m ⁻³)
ρ_p	: particle density	(kg•m ⁻³)
τ_w	: wall shear stress	(N•m ⁻²)

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