Measurement of the Dynamic Physical Properties of Solid Particles by a Rotary Shear Tester with a Conical Rotor†

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Abstract

The transitional torque characteristics of a particle bed have been measured by using a rotary shear tester which is basically a Couette type rheometer. The torque measured on the rotor shaft under constant operating conditions showed the maximum value to be at the initial period. It then decreased exponentially with changes in the packed state of the particle bed around the shearing surface during rotation. The magnitude of the decay coefficient $k$ of the torque indicated some of the dynamic physical properties of the solid particles, such as flowability and surface condition. It was found that the conical rotor was suitable for the more sensitive and accurate measurement of the change in the torque. A dimensionless parameter $k/N_R$, where $N_R$ is the rotor speed, was introduced and compared with the other flowability indices. It was experimentally shown that this parameter was more sensitive in detecting slight changes in the physical properties of the particles during mixing and coating operations. The coating processes in two different types of mixers and the coating state of the binary mixture were evaluated based on this parameter.

1. Introduction

The flowability characteristics of powder materials are influenced not only by the primary properties of the particles, such as diameter, true density, and physicochemical structure, etc., but also by the inter/exterrelationship among the particles, such as the state of packing and void, size distribution, cohesion force, surface conditions, internal/external friction coefficient, how external forces act thereupon, etc. In addition, the evaluation of flowability may vary according to the method of measurement.

A number of testing methods have been proposed and used to evaluate the flowability. One is the measurement of the internal friction coefficient by methods based on triaxial compression, single shear, ring shear, or by using a rotational double cylinder. Other methods are based on the measurement of the cohesive force, the angle of sliding friction and the flowability index, the latter including measurements of the angle of repose, the spatula angle, and compressibility, etc. Among others, the rotational double cylinder method has been the subject of study as a relatively convenient method for determining the quasi-static and dynamic frictional characteristics and the rheological properties of solid particles.

In this paper, the design of the rotor of the rotary shear tester for the powder bed is studied to find the most sensitive shape for detecting the characteristics of the transitional shearing torque with good reproducibility. A decay coefficient of the torque curve as a function of the shearing rate is proposed as a new characteristic parameter to express a dynamic physical property of solid particles. The transitional torque curves for several kinds of solid particles were evaluated based on this parameter.

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particles and a mixture of an inorganic particle and a wax powder are measured by using a conical rotor. It has been shown that the new parameter is almost equivalent to the compressibility and is more sensitive to the detection of slight changes in the physical properties of a mixture.

2. Experimental Equipment and Method

Figure 1 shows a schematic diagram of the rotary shear tester used in this work. The tester consists of a cylindrical vessel (ID: 12.5 cm) and round cornered bottom), a rotor placed coaxially within the vessel, and an adjustable speed motor (from 0 to a maximum of 11.7 rps). Three different shapes of rotors were tested, namely, a cylinder, a disk and an inverted cone, with 2 mm-pitched V-shaped cuts on the external surface of each one of them, as shown in the same figure. The rotational torque was measured by a strain gauge type torque-pickup (capacity: 4.9 Nm) placed between the rotor and the drive shaft.

Table 1 shows the physical properties of the materials used. The bulk density $\rho_b$ was obtained by tapping the powder in a cylindrical vessel of 100 cm$^3$ under a 5 mm stroke at 0.8 Hz for 5 minutes. The compressibility $C$ was also obtained by the tapping method, the angle of repose $\phi_r$ by an injection angle method, and the sliding friction coefficient $\mu_s$ by an inclining plate method.

In the experiments, a vessel containing a certain amount of the testing material is mounted on an electromagnetic vibrator and vibrated with an intensity of 2.1 G at 60 Hz. Under these conditions, in which the powder bed appears to be in a fluidized state, the vessel is jacked up to an extent such that the rotor penetrates the bed a prescribed distance. The cylindrical rotor was inserted to a depth of $h = 65$ mm, the conical rotor down to 37 mm, and the disk rotor a depth of 10 mm.

Once the rotor has been properly placed, the vibration intensity is decreased to 1.35 G at 60 Hz and maintained at this value for 5 minutes with the purpose of setting the conditions for the initial packing state of the powder bed. After this preliminary conditioning, the bed is left at rest for 2 to 3 minutes. Then, the rotor is set in motion at a prescribed speed, and the changing transitional torque is measured continuously until the steady state torque is reached.

Table 1  Physical properties of solid particles tested in this work

| Material             | Symbol | Average diameter $d_p$ [μm] | Density (true) $\rho$ [kg/m$^3$] | Density (bulk) $\rho_b$ [kg/m$^3$] | Compressibility $C$ [-] | Angle of repose $\phi_r$ [deg] | Coeff. of friction $\mu_s$ [-] |
|----------------------|--------|----------------------------|----------------------------------|----------------------------------|------------------------|-----------------------------|-----------------------------|
| Tuyoura sand         | TS     | 200                        | 2550                            | 1630                             | 2.45                   | 34.1                        | 0.679                       |
| Zircon sand          | Zr     | 130                        | 4650                            | 2950                             | 3.39                   | 33.0                        | 0.610                       |
| Silicon carbide #46* | SIC 46 | 360                        | 3240                            | 1680                             | 3.63                   | 33.8                        | 0.651                       |
| Silicon carbide #80* | SIC 80 | 259                        | 3240                            | 1744                             | 9.98                   | 33.2                        | 0.644                       |
| Silicon carbide #120*| SIC 120| 130                        | 3240                            | 1652                             | 10.29                  | 37.0                        | 0.641                       |
| Silicon carbide #180*| SIC 180| 80                         | 3240                            | 1611                             | 15.83                  | 37.2                        | 0.670                       |
| Silicon carbide #320*| SIC 320| 57                         | 3240                            | 1537                             | 17.50                  | 39.1                        | 0.678                       |
| Mica #2000           | Mica   | 4.7                        | 2865                            | 485                              | 49.28                  | 43.0                        | 0.831                       |
| Alumina powder       | Al$_2$O$_3$ | 3.8              | 3980                            | 1767                             | 28.92                  | 46.9                        | 0.827                       |
| Calcium carbonate    | CaCO$_3$ | 2.5               | 2700                            | 1072                             | 33.77                  | 46.9                        | 0.832                       |
| Talc                 | Talc   | 1 ~ 10                     | 3050                            | 509                              | 35.95                  | 46.8                        | 0.797                       |

*) JIS R 6111, **) sliding friction
3. Experimental Results and Discussion

3.1 Torque curve and rotor shape

The characteristic curves of the change in the torque with time for two different powders and for the three types of rotors are shown in Fig. 2. When using the conical rotor in silicon carbide (\#120), the flowability of which is relatively good, the torque showed the maximum value $T_1$ to be at the start and then gradually decreased until reaching the steady torque $T_S$. When the disk rotor was used, the maximum value of the torque was several times larger than that of the conical rotor, and it took a long time to reach the steady value. Besides, the disk rotor exhibited an irregular torque curve and a poor reproducibility of $T_S$. As for the cylindrical rotor, which has been widely used, the torque showed no clear difference between $T_1$ and $T_S$ and no clear peak either. Rather, the torque increased with time and, in some cases, it was difficult to find a definite value of $T_S$. The reasons for this behavior may be found in the changing of the local packing state of the particles bed close to the rotor during rotation. Observing the motion of the particles by using colored tracer particles, it was found that, unlike the cone or disk, the movable regions in the particles bed were formed only at and near the surface of the rotor. On the other hand, the cylindrical rotor was surrounded by flowable regions developing a funnel form.

For calcium carbonate, which is cohesive and has poor flowability although changes in the torque showed a behavior similar to that in silicon carbide, the steady torque of both disk and cylindrical rotors showed conspicuous cyclic changes such that no definite value could be appropriately assigned to $T_S$. On the other hand, using a conical rotor, the torque decayed relatively smoothly, and after the experiment it was found that there was no consolidation of the powder bed at or near the rotor.

These results proved the conical rotor to be the most suitable shape for the measurement of the transitional torque.

3.2 The decay coefficient of torque

Assuming that the torque has its maximum value $T_1$ at time $t=0$, and decays continuously until it reaches the steady torque $T_S$, the following expression was used to fit the experimental data,

$$T = (T_1 - T_S) \cdot \exp(-kt) + T_S$$  (1)

The exponent $k$ can thus be evaluated from the slope of a semilog plot of torque $T$ versus time $t$.

Figure 2 shows the relationship between the torque ratio $(T - T_S)/(T_1 - T_S)$ and the time $t$ for coarse and fine materials using the conical

![Fig. 2](image1)

**Fig. 2** Effect of rotor shape on the torque curve

![Fig. 3](image2)

**Fig. 3** Semilogarithmic plotting of the experimental data
Fig. 4 Decay coefficient of torque

rotor at different rotational speed, $N_R$. It is seen that, in the case of the coarse powder, $T$ approximately follows Eq. (1). However, the decreases of $T$ in the fine powder take place in two or more steps. For both materials, the slope $k$ of the curve at the initial stage increases with the rotational speed $N_R$.

The relationship of $k$ and $N_R$ for a number of different powders is shown in Fig. 4. It was found that $k$ is directly proportional to $N_R$ and the ratio, $k/N_R$, represents a peculiar value for each material tested. The value of $k/N_R$ turned out to be large for relatively large-size particles, i.e., particles which can be easily replaced and rearranged the region of the surface of the rotor so that the torque can reach a steady state in a relatively short time. On the other hand, the value of $k/N_R$ is small for fine powders which are more difficult to disperse into single particles because of their tendency to cohere and form agglomerates. Therefore, the parameter $k/N_R$ gives information about the flowability and packing characteristics of a powder material. $k$ and $N_R$ also showed a linear relationship when the disk rotor was used, but in some cases the ratio $k/N_R$ failed to show clear differences among different materials. For this reason, only the results corresponding to the conical rotor will be discussed here.

3. 3 Comparison of $k/N_R$ with other characteristic values

Average particle diameter $\bar{d}_p$: Figure 5 shows the relationship between the value of $k/N_R$ and the average particles diameter $\bar{d}_p$ of silicon carbide SiC. $k/N_R$ increases with $\bar{d}_p$. It is clear that the flowability of powders increases as the particle size increases. However, $k/N_R$ is not more sensitive to particle size for large sized particles (more than 250 μm), as shown in the figure.

Coefficient of sliding friction $\mu_S$: The relationship between $k/N_R$ and the friction coefficient $\mu_S$ (measured by a tilting plate method) for various powders is shown in Fig. 6. For
large particle sizes, as \( \mu_S \) increases \( k/N_R \) decreases, that is the flowability becomes lower. It is also seen that in experiments with fine powders, although there was no appreciable change in \( \mu_S \), \( k/N_R \) showed significant differences. Since \( k/N_R \) exhibits a wide variation range despite the narrow variation range of \( \mu_S \), \( k/N_R \) is considered to be effective for detecting minute changes in physical properties.

**Compressibility C**: The relationship between the parameter \( k/N_R \) and the compressibility \( C \) (measured by a tapping method in a 100 m\( \text{m} \) cylindrical vessel) is shown in Fig. 7. As for silicon carbide, \( C \) increases with a decrease in particle diameter, but the changes in \( C \) were within a limited range compared with those of \( k/N_R \). For fine powders, such as calcium carbonate, alumina, talc and mica, \( C \) was so likely to be influenced by the measuring conditions that \( C \) varied within a wide range; however, \( k/N_R \) was obtained with a relatively good reproducibility.

4. **Binary Powder Mixtures and \( k/N_R \)**

The parameter \( k/N_R \) was used to characterize the mixtures of inorganic powders with small amounts of a synthetic wax having a relatively low melting point. In the mixing process, the coarse inorganic powders are coated by the fine wax powders. As a result, the physical properties and surface condition of the powders are modified. Using the subscripts \( O \) and \( M \) to designate before and after mixing, respectively, a dimensionless parameter, \( K \) is defined as,

\[
K = \frac{(k/N_R)_M}{(k/N_R)_O}
\]

\( K \) is used to account for the changes in physical properties that occurred throughout the mixing process.

4. 1 **Influences of mixer type and operating conditions**

A V-type mixer with an effective capacity of 5 \( \text{L} \), and a high speed stirred type mixer (1.5 \( \text{L} \)) with a device to vibrate the vessel were used. The coating wax used consisted of glycerolmonostearate in powder form \( (d_p = 390 \mu\text{m}, \text{m.p.} = 60.5 \pm 2.5\, ^\circ\text{C}) \).

4. 1. 1 **V-type mixer**

It is known that in this type of mixer, the time required for complete mixing is about 6.5 minutes for relatively flowable powders.

**Figure 8** shows the variation of \( K \) with time for three different mixtures (the rotational speed of the mixer vessel for all runs: 1 rps). In the mixing of 1.5 g of wax with 1.5 kg of zircon sand (having the highest density amongst the materials tested), \( K \) decreased sharply during the first 5 minutes, and thereafter it maintained a constant value of about 0.43 (see • in the figure). With Toyoura and (0.3 g of wax mixed with 1.4 kg), \( K \) showed no noticeable change even after 30 minutes of mixing (see ○ in the figure). A similar trend was shown by a glass powder, the physical properties of which were almost equal to those of the Toyoura sand. In the mixtures of silicon
carbide (1.5 g of wax mixed with 1.0 kg), $K$ showed an intermediate behavior between those of zircon sand and Toyoura sand (see $\triangle$ in the figure). These results suggest that the shearing effect between particles resulting from their circulating flow in the mixer vessel varies depending on the density and friction coefficient of the material. During mixing, the temperature of the powder bed was constant (20 ~ 25°C), and the wax remained in a granular form even after 30 minutes of mixing.

To examine the effect of the melting of wax on $K$, the mixed samples were placed in a thermostatic vessel for 2 to 3 hours at a temperature above the melting point of the wax. In these conditions, the melted wax adhered onto the particles surface. After cooling, $K$ was measured again. The symbols $\diamond$ and $\bullet$ in Fig. 8 represent zircon sand and Toyoura sand, respectively. $K$ showed a further decrease in the zircon sand but only a slight decrease in the Toyoura sand. It was presumed that the degree of adhesion of the wax differed between the two samples.

4. 1. 2 High speed stirred type mixer

The mixing vessel used here was vibrated at a frequency of 33.3 Hz in order to improve the flowability of the powder bed and to eliminate the stagnant zone in the mixer. It has been shown that the mixing of coarse particles was complete in the first period of 3 ~ 4 minutes. The final degree of mixing was found to be approximately equal to the degree of mixing attainable in a V-type mixer.

The wax powder was added while the vessel was vibrating, and the mixture was stirred by a knife-like impeller rotating at 16.7 rps.

Figure 9 shows the variations in the mixing torque $T$, the parameter $k/N_R$ and the temperature of the mixture with time.

When using zircon sand (1.5 kg), the torque decreased immediately after the addition of the wax (0.25 g) attaining a steady-state value in about 3 minutes. The temperature of the mixture rose to close to the melting point of the wax in about 27 minutes. As a result of the increased adhesion by the softened wax, the torque increased slightly. On the other hand, the decreasing tendency of $k/N_R$ with time continued for up to 25 minutes.

When Toyoura sand (1.4 kg) was mixed with the wax (0.5 g), the temperature also rose markedly from the beginning, but the torque did not change until 16 minutes after the beginning of mixing. At this point, the torque decreased sharply and became steady. $k/N_R$ decreased from the beginning of mixing, at room temperature, until the temperature reached the melting point and then attained a constant value of steady state.

According to these experimental results, the mixing mechanism of the binary powders with a coating process is estimated. In the first stage, a rearrangement of the relative positions of sand and wax particles takes place, and as mixing proceeds, the particles of wax are ground and melt as a result of the heat generated by interparticle friction. The melted wax adheres to the surface of the inorganic particles which thus become coated. It was observed that the color of the mixture changed from yellowish brown to dark brown, which proves that the coating process is occurring within the mixer. The compressibility $C$ was almost the same before and after coating, as shown in Fig. 7 enclosed within dotted lines. On the other hand, $k/N_R$ showed clearer changes, thus reflecting the differences in flowability caused by the modification of the surface condition of the particles.
Figure 10 shows the relationship between $K$ and the mixing time $\theta_M$. $K$ decreased gradually for each material in about 10 minutes. In every case, the value of $K$ was smaller than that obtained with the V-type mixer. These results show that the shearing force exerted by the impeller and the interparticle friction effect enhance a remarkable change in the surface condition.

4. 2 Effect of the amount of wax on the parameter $K$

The effect of the amount of coating agent on the parameter $K$ was investigated by adding different amounts of wax to silicon carbide ($\#120$) in the high speed stirred type mixer. In the experiments, 1 kg of silicon carbide was mixed within the weight range of wax of 0.1 to 3.0 g, at a vibration frequency of 33.3 Hz and an impeller velocity of 16.7 rps, during a period of 40 minutes. The mixture was maintained at a temperature above the melting point of the wax and then gradually cooled to the room temperature. $K$ decreased as the amount of wax increased and attained a constant value for concentrations of wax greater than 1.5 g/kg., as shown in Fig. 11.

The cosine of the contact angle $\beta$, which was determined by a penetration rate method using acetone, is also shown in the same figure. It is seen that the changes of $\cos\beta$ and those of $K$ show the same trend. In this figure, the decay coefficient of torque measured by using a cylindrical rotor$^6$ have also been plotted. The results for the mixtures of glass: oleic-acid and glass: capric-acid show that $K$ decreases as the amount of the oleic/capric-acid is increased. This trend is in agreement with the results of the present experiments.

5. Conclusion

The dynamic physical properties of solid particles were evaluated in a rotary shear tester by measuring the changes in the torque with time. Three types of rotors were compared, and the conical rotor was found to be the most sensitive in detecting the characteristics of the transitional torque, the decay of which was approximated by an exponential function. A dimensionless decay coefficient $k/N_R$ intimately related to the flowability of the powder, was proved to be effective in detecting minute changes in the physical properties of the solid particles. This parameter was also shown to be useful in the following up of the coating process of inorganic powders by a wax. It also made clear the influence of the mixer type, i.e. difference in mixing mechanisms, on the coating state of the mixture. Furthermore, the change in $k/N_R$ was related to the change in the contact angle of the solid particles.

Nomenclature

| Symbol | Description                  | Unit       |
|--------|------------------------------|------------|
| $C$    | compressibility              | $[-]$      |
| $d_p$  | average particles diameter   | $[\mu m]$  |
| $K$    | coefficient defined by Eq. (2)| $[-]$     |
| $k$    | rate constant                | $[s^{-1}]$ |
| $N_R$  | rotational speed of rotor    | [rps]      |
| $N_V$  | frequency of mixer vessel    | [Hz]       |
| $T$    | agitating torque             | [Nm]       |
$T_i$: maximum torque [Nm]
$T_s$: torque at steady state [Nm]
$t$: time [s]

$\beta$: contact angle [deg]
$\theta_m$: mixing time [s]
$\mu_s$: coefficient of sliding friction [-]
$\rho$: true density of particle [kg/m$^3$]
$\rho_b$: bulk density of particles bed [kg/m$^3$]
$\phi_r$: angle of repose [deg]

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