Characteristic Shearing Curves of a Powder Bed
Measured with a Rotary Intrusion Rheometer

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Abstract

A rotary intrusion rheometer with a conical rotor was improved to detect the intruding stress as well as the shearing torque at a cell bottom. The characteristic curves of shearing torque versus a rotor's depth of intrusion in a powder bed, and normal stress versus depth of intrusion were measured simultaneously under various operating conditions. Two experimental coefficients $C$ and $m$ were introduced to characterize the relationship between the shearing torque and the depth of intrusion. The coefficient $m$ indicates the compressibility tendency of the powder bed during rotor intrusion. The friction property of powders is discussed based on the coefficient $C$ measured at $m = 1$ (without the compressive condition).

1. Introduction

To handle powder appropriately, it is very important to understand its dynamic physical characteristics, such as flow and shear. Various methods have been proposed and actually used to clarify these characteristics. Some evaluations involve measuring the angle of repose in a rotating cylindrical vessel, while others measure the velocity of powder outflow from an orifice. A number of new approaches to such analyses have been reported recently: e.g., evaluation of powder fluidity by measuring the transfer resistance of a metal sphere in a vibrating powder bed\(^4\), evaluating change in particle surface physical properties by measuring the frequency transfer characteristic of a vibrating powder bed\(^5\), and evaluating physical properties by measuring a powder's specific frequency absorption characteristics using an acoustic tube, thereby obtaining guidance in sound-absorbing/vibration-proof material design\(^6\).

Until now, powder flow characteristics have been evaluated on the basis of rotor frictional resistance or mechanical stirring of a powder bed. For certain kinds of powders, a liquid viscometer has been used to measure the relation between the friction coefficient and the shear rate. Powder dynamic physical properties have been evaluated on the basis of change in torque over time, as measured with a rotary shear tester using rotors of various shapes\(^7\). Further development of this method made it possible to use a rotary intrusion rheometer with a conical rotor to detect slight powder physical property changes achieved through wax-coating\(^8\).

In the present study, we improved the above-mentioned rotary intrusion rheometer\(^9\) to make possible the simultaneous measurement of changes in the shearing torque of a powder bed and in the normal load applied to the bed bottom by rotor intrusion. Variation curves of torque and intruding load were obtained using this improved rheometer. Using the shape-determining parameters of these mechanical characteristic curves, we then investigated the stress and friction status in a powder bed to evaluate equipment characteristics and powder dynamic physical properties.

2. Experimental Apparatus and Procedure

2.1 Experimental Apparatus

Figure 1 schematically shows the experimental apparatus used to simultaneously measure the shearing torque and intruding load on a powder bed. The main body design is based on the structure of a double-cylinder type rheometer (1). A circular cone with a 60° vertical angle and 44 V-shaped grooves on its circumference is used for the powder-shearing rotor (2). A test cell (3), which is a cylindrical vessel with a 58 mm inside diameter and 95 mm in height, is fixed on the linear head (4) (stroke: 50 mm) via...
a load cell (capacity: 98 N). An electromagnetic vibrator is provided between the load cell and the linear head to adjust the powder bed's initial packing condition by applying torsional vibration at 60 Hz in a direction 20° upward from the horizontal plane. Signal outputs of rotor rotational speed \( N_R \), intrusion depth of rotor \( h \), shearing torque \( T \), and intruding load \( L_1 \) are measured continuously using a personal computer through an A/D converter.

### 2.2 Experiment Procedure

The test cell, filled with a specified amount of sample powder, was set on the load cell mounted on the vibrator, which was then operated at constant acceleration (2G) for pre-packing. When powder bed height stabilized, the vibrator was stopped. As the conical rotor (30 mm in maximum diameter) was then rotated at a specified speed, the linear head was operated to raise the test cell so that the rotor intruded the powder bed. During this operation, the shearing torque, intruding load, and intrusion depth were measured simultaneously. When the intrusion depth exceeded the range within which the rotor could maintain stable rotation and torque becomes excessive, the linear head was stopped. The test cell was then immediately lowered to its original position with the rotor and vibrator still in operation. The vibrator was then stopped, and the test cell was raised again, enabling the rotor to intrude into and shear the powder bed. This series of operations was repeated until measurements for each powder sample stabilized. An average of at least five measurements was taken as shear characteristic data for each sample.

Table 1 gives the sample powders used in the experiment, and their physical property values. We used the apparatus shown in Figure 2(a), Shinto Scientific Co., Ltd.'s Heidon Model 10 with modified sliding surface, to measure the sliding friction coefficient. Measurements were taken as follows: First, the sample powder was allowed to adhere to two-sided adhesion tape applied to one side of a plane weight (35 × 75 mm). The weight was placed gently, with the adhesion tape side down, on sample powder in a tray set on the flat board, which was then inclined gradually. This apparatus is designed so that the board automatically stops inclining at the moment the weight begins to slide. The normal stress imposed by the weight can be varied through a range from 0.76 to 1.23 kPa. At various normal stresses of the weight, sliding friction coefficient \( \mu_s \) was calculated from the tangent of the maximum inclination angle \( \theta_{\text{max}} \) of the flat board the moment it stopped inclining. The above operation was repeated 50 times, and the average of 50 measure-

| Powder material          | Average diameter (µm) | Density (kg/m³) | Compressibility (%) | Angle of repose (º) | Sliding friction coeff. (º) |
|--------------------------|-----------------------|----------------|---------------------|---------------------|-----------------------------|
|                          | True                  | Loose          | Pack*               |                     |                             |
| Calcium carbonate        | 2.38                  | 2600           | 532                 | 33.9                | 0.32                        |
| Cornstarch               | 13.9                  | 1475           | 467                 | 38.3                | 0.717                       |
| PMMA MB-8                | 8.4                   | 1190           | 500                 | 19.0                | 0.619                       |
| Toyoura sand             | 200                   | 2550           | 1590                | 2.45                | 0.679                       |
| Zircon sand              | 130                   | 4660           | 2700                | 10.0                | 0.610                       |

*Powder Tester (Hosokawa Micron Co., Ltd.) **Heidon (Shinto Scientific Co., Ltd.)
plate weight

3mm

tray

rib

plate weight

plate weight

(a) Testing apparatus for sliding friction coefficient \( \mu_s \).

(b) Sliding friction coefficient \( \mu_s \) vs. normal stress.

Fig. 2
Measurement of sliding friction coefficient \( \mu_s \).  
(a) Testing apparatus for sliding friction coefficient \( \mu_s \).  
(b) Sliding friction coefficient \( \mu_s \) vs. normal stress.

Figures 2(b) shows one of the measurement results. \( \mu_s \) is virtually constant regardless of normal stress. Similar results were obtained for other samples, as shown in Table 1. This implies that \( \mu_s \) represents one aspect of powder physical properties.

3. Results and Discussion

3.1 Torque and Intruding Load Characteristic Curves

Torque \( T \) and intruding load \( L_i \) were measured at various rotational speeds \( N_r \) and intrusion rates \( u_i \). Figure 3 shows the torque and intruding load characteristic curves thus obtained. \( T \) and \( L_i \) increase with intrusion depth \( h \), their increase rates differing according to the sample powder, owing to differences in the shear plane area increase among particles with rotor intrusion, as well as differences in the powder bed’s rotor compression characteristics. In other words, differences in these characteristic curves are presumably attributable to differences in powder physical properties such as friction and packing characteristics. The following paragraphs discuss powder physical properties from this viewpoint.

3.2 Shear Characteristic

Infinitesimal area \( d_s \) on an inverted cone of vertical angle \( \alpha \), at arbitrary depth \( h \) in a powder bed can be expressed by the following equation:

\[
d_s = \frac{\tan(\alpha/2)}{\cos(\alpha/2)} \; h \; dhd\theta
\]  

(1)

Therefore, if normal stress \( \sigma \) applied to a shear plane is of uniform distribution, shear torque \( T \), required to rotate a conical rotor at an intrusion depth of \( h \) in powder bed, can be calculated by the following equation, which is obtained by integrating Equation (1) in the domains of \( 0 \leq h \leq h \) and \( 0 \leq \theta \leq 2\pi \).

\[
T = \frac{2\pi \tan^2(\alpha/2)}{3 \cos(\alpha/2)} \sigma \mu h^3
\]  

(2)

where \( \mu \) is the powder bed friction coefficient.

As indicated by Equation (2), \( T \) is proportional to the third power of \( h \). Measured values for \( T \) and \( h \), plotted on logarithmic graph paper, should therefore have a linear relation slope of 3. Figure 4 shows an example of the relation between \( T \) and \( h \).
plotted on a log scale. The slope of the straight line is nearly 3, although it varies somewhat depending on measurement conditions. Normal stress $\sigma$ and the friction coefficient $\mu$ can vary due to interaction among various factors, such as stress distribution changes caused by rotor intrusion, and adhesion resistance generated in the sheared powder bed. Coefficient $C$, containing $\sigma$ and $\mu$, was therefore introduced to define the following equation, on the assumption that $T$ is proportional to the $3m$ power of $h$.

$$T = f(a) Ch^m$$  \hspace{1cm} (3)

where $C$ is $(N \cdot m^{1-3m})$, and

$$f(a) = \frac{2\pi \tan^2(a/2)}{3 \cos(a/2)}$$  \hspace{1cm} (4)

Parameters $C$ and $m$ represent the degree of change in $T$ with variation in $h$. If $C$ and $m$ are large values, $T$ changes greatly. $m$ indicates the degree of deviation from Equation (2). When $m=1$, Equation (3) coincides with Equation (2). $C$ and $m$ were obtained from the relation between $T$ and $h$ using the least squares method, and sample powders' dynamic characteristics were studied on the basis of the $C$ and $m$ values thus obtained, which change depending on measuring conditions.

**Figure 5** shows changes in $C$ and $m$ for calcium carbonate at various rotational speeds $N_T$ and constant intrusion rate $u_i$. Both $C$ and $m$ decrease, and $m$ approaches unity, as $N_T$ increases. This corresponds to the phenomenon in which, as $N_T$ increases, powder bed friction changes from a static to a dynamic state, causing fluidization to progress, which results in lower shearing stress.

**Figure 6** shows changes in $C$ and $m$ for calcium carbonate, at various intrusion rates $u_i$ and constant rotational speed $N_T$. Both $C$ and $m$ increase with $u_i$, increasing sharply when $u_i$ exceeds approximately $1 \times 10^{-4}$ m/s. This is because at such a high $u_i$ value, the powder bed compressive effect becomes apparent due to the imbalance between the powder scraping rate and the rotor scraped powder removal rate. By contrast, at $u_i$ values below $1 \times 10^{-4}$ m/s, i.e., in the region where rotor intrusion is semi-static, changes in $C$ and $m$ are relatively small, $m$ being close to unity, due to the slight compressive effect of the powder bed.

$C$ and $m$ therefore represent powder bed frictional status and degree of compression, $m$ being the index.

### 3.3 Intrusion Characteristics

From Equation (1), infinitesimal force $dF$ applied vertically to infinitesimal area $d_s$ is given by:

$$dF = \frac{\tan(a/2)}{\cos(a/2)} \cdot \frac{dF}{dh} dh d\theta$$  \hspace{1cm} (5)
Assuming that \( \sigma \) is constant, vertical component \( F_v \) of the force the rotor receives from the powder bed at intrusion depth \( h \) is obtained by integrating Equation (5), as follows:

\[
F_v = \pi \tan^2(a/2) \omega h^2
\]  
\( (6) \)

If \( F_v \) is equal to the intruding load of rotor \( L_1 \), the following equation holds:

\[
L_1 = F_v = g(a) \omega h^2
\]  
\( (7) \)

where

\[
g(a) = \pi \tan^2(a/2)
\]  
\( (8) \)

Therefore, \( \sigma \) can be obtained from the linear relation between \( L_1 \) and \( g(a) h^2 \). Figure 7 shows an example of measured \( L_1 \) values plotted against \( g(a) h^2 \). \( L_1 \) and \( g(a) h^2 \) have a linear relation except when \( N_R \) is low, i.e., compressive effect is large. Slope \( \sigma \) of a straight line becomes lower as \( N_R \) increases, due to the change in frictional status, as mentioned previously. When \( N_R \) is low, the relation between \( L_1 \) and \( g(a) h^2 \) yields a descending convex curve, indicating that \( \sigma \) increases with \( h \), reflecting the process in which the powder bed is compacted as the rotor intrudes. Therefore, since change in \( \sigma \) against \( h \) represents powder bed compressive status, \( \sigma \) is expressed by the following equation:

\[
\sigma = kh^n
\]  
\( (9) \)

where \( k \) and \( n \) are constants. In the following paragraphs, the relation between compression index \( m \) mentioned earlier, and \( n \), which represents the degree of dependence of \( \sigma \) on \( h \), is determined.

By substituting Equation (9) into Equation (7), the following equation is obtained:

\[
L_1 = \frac{kg(a) h^{n+2}}{n+2}
\]  
\( (10) \)

\( n \) is obtained by applying Equation (10) to the measured relation between \( L_1 \) and \( h \). Simultaneously, \( m \) is obtained from the measured relation between \( T \) and \( h \). The calculated \( n \) and \( m \) data are then arranged to show the relation between them, as in Figure 8, \( n \) increases with \( m \), confirming that \( m \) expresses definitely the powder bed's compressive status. \( n \) is close to 0 at \( m = 1 \), which indicates that under the measuring conditions satisfying \( m = 1 \), \( \sigma \) is constant; namely, \( \sigma = k = c_0 \) (constant). In other words, normal stress is virtually constant on the shear plane, irrespective of rotor intrusion. This fact leads to the reasonable conclusion that, under the above-mentioned measuring conditions, the rotor can always form a new shear plane as it intrudes, without compressing the powder bed.

When rotor intrusion rate \( \omega \) is high, \( m \) takes a large value due to the large compressive effect. As rotational speed \( N_R \) is increased, however, the powder removal effect increases, making compression less likely to occur, which results in a tendency for \( m \) to decrease. Therefore, the compression or non-compression of a powder bed depends on the balance between \( \omega \) and \( N_R \). There are several different measuring conditions, that is, several combinations of \( \omega \) and \( N_R \), in which \( m \) equals unity. Even if the measuring conditions vary, the coefficient \( C \) obtained at \( m = 1 \) is virtually constant for the same sample powder, although it differs for different sample powders. The following section discusses the relation between the physical properties of powder and \( C_0 \), which is the value of \( C \) obtained under the measuring conditions in which \( m = 1 \).

### 3.4 Relation Between \( C_0 \) and Powder Physical Properties

As mentioned previously, \( C \) represents the frictional characteristic of powder. The relation
between $C_0$ and the angle of repose, a typical index representing powder fluidity, was therefore determined. Figure 9 shows this relation for various sample powders. $C_0$ differs greatly among calcium carbonate, corn starch, and PMMA (Polymethylmethacrylate) MB-8, although the angle of repose is almost the same, which indicates that $C_0$ represents subtle differences in the frictional characteristic and fluidity among different powders.

We therefore sought to calculate the internal friction coefficient from $C_0$. Essentially, $C$ is expressed as the product of normal stress and the friction coefficient. Therefore, the following simplest form of the equation was used to calculate the internal friction coefficient $\mu_0$.

$$C_0 = \mu_0 \sigma_0$$

Normal stress $\sigma_0$ was obtained by plotting $k$ of Equation (10) against $m$, and extrapolating to $m = 1$, as shown in Figure 10. $\mu_0$ was calculated using Equation (11), to identify the relation between $\mu_0$ and sliding friction coefficient $\mu_s$. Figure 11 shows the result. There is a definite correlation between $\mu_0$ and $\mu_s$. In this correlation, $\mu_0$ is always smaller than $\mu_s$, because $\mu_0$ is obtained in the static state, whereas $\mu_s$ is obtained in the dynamic state.

4. Conclusions

The rotary intrusion rheometer was improved to enable simultaneous measurement of shearing torque and intrusion depth made it possible to accurately describe the compressive effect and the change in frictional status under varying measuring conditions.

(2) Under the measuring conditions satisfying $m = 1$, normal stress on the rotor remains constant regardless of rotor intrusion. Coefficient $C$ obtained under such conditions represents such dynamic physical properties of a powder bed as stress and the friction coefficient.

(3) At present, the stress distribution in a powder bed is estimated from the intruding load detected at the sample cell bottom. Our future task is therefore to evaluate intruding load, taking powder bed load transfer characteristics into account. It is also necessary to conduct experiments using different powder bed initial void fractions, and evaluating the results.
Nomenclature

\( C \) : coefficient defined by Eq. (3) \((\text{N} \cdot \text{m}^{1-3\alpha})\)
\( C_0 \) : coefficient at \( m=1 \) \((\text{Pa})\)
\( F \) : force acting vertically on the conical surface \((\text{N})\)
\( F_v \) : vertical component of force \( F \) \((\text{N})\)
\( f(\alpha) \) : function of \( \alpha \) defined by Eq. (4) \((-\text{)}\)
\( g(\alpha) \) : function of \( \alpha \) defined by Eq. (8) \((-\text{)}\)
\( h \) : depth of intrusion \((\text{m})\)
\( k \) : coefficient defined by Eq. (9) \((\text{N} \cdot \text{m}^{-\alpha-3})\)
\( L_n \) : intruding load \((\text{N})\)
\( m \) : coefficient defined by Eq. (3) \((-\text{)}\)
\( N_0 \) : rotational speed of the rotor \((\text{s}^{-1})\)
\( n \) : coefficient defined by Eq. (9) \((-\text{)}\)
\( s \) : area of the shearing zone \((\text{m}^2)\)
\( T \) : torque \((\text{N} \cdot \text{m})\)
\( u_i \) : intrusion rate of the rotor \((\text{m} \cdot \text{s}^{-1})\)
\( \alpha \) : vertical angle of the conical rotor \((\text{rad})\)
\( \theta \) : central angle of the developed conical surface \((\text{rad})\)
\( \theta_s \) : angle of inclination plate \((\text{rad})\)
\( \theta_{\alpha, \text{max}} \) : maximum inclination angle \((\text{rad})\)
\( \mu \) : friction coefficient \((-\text{)}\)
\( \mu_s \) : sliding friction coefficient \((-\text{)}\)
\( \mu_0 \) : friction coefficient defined by Eq. (11) \((-\text{)}\)
\( \sigma \) : normal stress \((\text{Pa})\)
\( \sigma_0 \) : normal stress at \( m=1 \) \((\text{Pa})\)

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