Shear cell methods

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In the manufacturing of pharmaceuticals, a large number of processes involve powder transfer and feeding such as putting raw materials into a mixer and filling a powder into the mortar of a tableting machine. Since powder flowability is related to preparation characteristics such as mass and content uniformity, it greatly affects the product quality. It is important to evaluate powder flow properties in the design of formulations, processes and pharmaceutical production equipment. Since shear cells are among the most important methods for measuring powder flow properties and can be performed under a wide variety of stress conditions, parameters useful for predicting various powder behaviors during the manufacturing of pharmaceuticals, such as an angle of critical state line, unconfined yield strength and flow factor, can be determined.

1. Principle

A powder in a hopper, etc. may not immediately flow due to adhesion/coagulation of particles and interference to the mutual motion by complex surface shape when shear stresses are applied from the outside. When a sufficiently large shear stress is attained, the powder suddenly starts to flow. In addition, powder flow under quasi-static conditions such as flow in a bin strongly depends on the consolidation stresses. Consolidation is an operation to apply a load to a powder bed to reduce the bulk volume to change the bulk density or the void fraction of the powder bed. The shear cell methods are the tests to determine the flow properties of a powder in the process of transition from a static state to a non-static state when the powder is sheared by applying a normal stress. For example, a maximum shear stress immediately before fail and a dynamic friction force in a steady-state flow are measured.

Powder flowability under compressed conditions is governed by three factors: the degree of consolidation (bulk density or void fraction, ε), normal stress (σ) and shear stress (τ). A three dimensional representation of the applied normal stress, shear stress and void fraction is called the Roscoe condition diagram (Fig. 1), and the shear cell methods are test methods to obtain the Roscoe condition diagram or yield loci which constitute the Roscoe condition diagram.

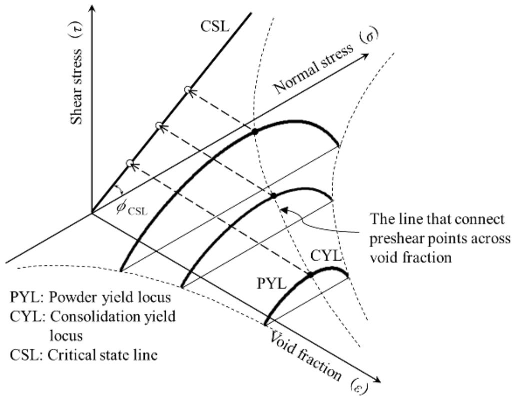

Fig. 1 Roscoe condition diagram

2. Apparatus

The shear cell methods can be performed under constant load or constant volume conditions. In both conditions, apparatuses typically consist of a shear cell, weights or a press machine for applying normal stress to a sample, a mechanism for shearing a sample, and load cells for measuring normal stresses and shear stresses.

2.1. Shear cell

Many shear cells have the structure which can make a shear plane somewhere in a powder bed by shearing a powder filled in a container dividable into two parts up and down (cells) while applying a normal stress. In the constant load conditions, a lid that fits within an upper cell is free to move up and down when a shear stress is applied, and the volume of the powder container changes. In the constant volume conditions, the position of the lid is fixed by pressing the lid with a press machine, etc.

The shear cells are classified into two types according to the motion that provides a shear stress; translational or rotational.

2.1.1. Translational shear cell

In the translational shear cell, one of the upper and lower cells is fixed and the other is moved horizontally (translationally) to apply a shear stress to the powder bed filled in the two cells. The shear plane forms at the boundary between the powder contained in the lower cell and the powder contained in the upper ring cell. Some translational shear cells are cylindrical (Fig. 2), and others are sandwiching a sample between two plates on top and bottom and with no side walls. The representative example of the former is the Jenike shear cell, and that of the latter is a parallel-plate cell.

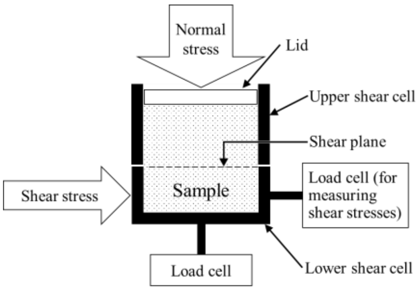

Fig. 2 Example of translational shear cell

2.1.2. Rotational shear cell

In the rotational shear cell, one of the upper and lower cells is fixed, and the other is rotated to apply a shear stress to the powder bed filled in the two cells. Some rotational shear cells are cylindrical and others are annular (Fig. 3). Any rotational shear cells usually have surface features that prevent the powder from sliding at the cell surface. Several blades are radially attached on the side of the shear cell where it contacts the sample, so that the powder is hold in place by the thrusting blades. A shear plane forms in the powder bed directly under the blades when the shear cell is rotated.

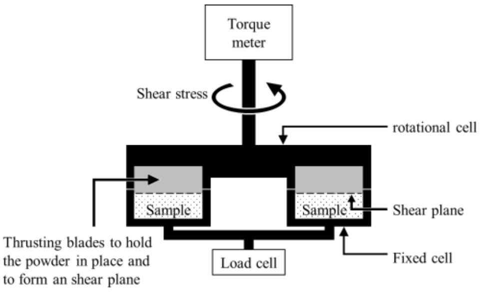

Fig. 3 Example of rotational shear cell

2.2. Other components

Load cells are device using a spring, a piezoelectric element, etc. to detect a load or torque and converts the applied force into an electrical signal. The load cells and weights for applying a normal stress to a sample must be regularly calibrated with standards with measurement traceability.

3. Measurement

A temperature of 20±5°C and a relative humidity of 50 ±10% are recommended as measurement environment. A fresh powder samples should be used for each test. However, this does not apply to samples clearly having no consolidation history or rare samples, if the description of their reuse is recorded. Gently fill the shear cell with a sample of powder using a spatula or a sieve with an opening size larger than the maximum particle size of the sample. At this time, care should be taken not to form cavities in the powder bed. The surface of the filled sample is leveled with a spatula, etc. Under the constant load conditions, first the powder sample is consolidated (preshear) in order to perform a test with a desired constant void fraction during one measurement.

The test procedure under constant load conditions using the Jenike shear cell, etc. is shown in Fig. 4 by a pattern diagram. Prior to a test, the powder sample is sheared with applying a preconsolidation stress (σ_{pre}) until a shear stress reaches a steady value (τ_{pre}) for preshear (Fig. 4 (a) A). Under the constant load conditions, the volume of powder can decrease or increases during the preshear and becomes constant when a steady-state is achieved. In other words, the void fraction of the powder bed where the shear stress became constant under certain normal stress conditions is uniquely determined by the powder flow properties. In the following main test, measurements are performed on the sample having this void fraction. After the shear stress is reduced to zero, the normal stress acting on the sample is decreased from σ_{pre} to a new value (σ_{sx}, $x = 1, 2, 3\cdots$) for the next step of the test procedure (Fig. 4 (a) B). When the shear stress is gradually increased, the maximum shear stress measured immediately before the consolidated powder starts to flow is τ_{sx} ($x = 1, 2, 3\cdots$). The A-B procedure is repeated at 3 to 5 points of σ_{sx} which is less than the normal stress at preshear (σ_{pre}), and the powder yield locus (PYL, Fig. 4 (b)) is obtained by plotting the results.

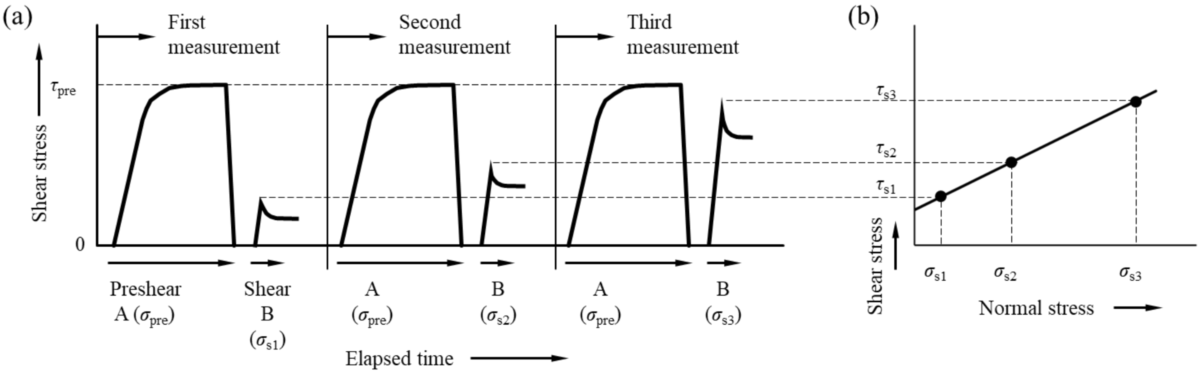

Fig. 4 Plot of shear stress versus time during the test procedure (a) and corresponding powder yield locus (b)

On the other hand, under the constant volume conditions, shear stresses are continuously measured with progressively changing a normal stress while the void fraction is kept at a specified value by a press machine, etc. controlling the lid position. Since the void fraction is constant under the constant volume conditions, the consolidation yield locus (CYL) in Fig. 1 is obtained during the process of consolidation failure. The PYL and CYL share a preshear point and are combined to form a single yield locus (YL).

4. Data analysis

Shear stresses can be measured in both states where a powder is not flowing (static state) and where a powder is flowing (dynamic state).

The approximate line connecting the points (σ_{sx}, τ_{sx}) shown in Fig. 4 (b) represents the relationship between the shear stress and normal stress immediately before the consolidated powder sample starts to fail, namely, the relationship in a static state, and is referred to as a PYL. Further, the point (σ_{pre}, τ_{pre}), where the shear stress τ_{pre} reached constant by applying the preshear normal stress σ_{pre}, is added to Fig. 4 (b) (Fig. 5). This point is measured in a dynamic state and is called a preshear point. Next, two circles having a center on the normal stress axis are drawn: The first is a circle passing through the preshear point and tangential to the PYL (the larger semicircle in Fig. 5). The second is a circle passing through the origin and tangential to the PYL (the smaller semicircle in Fig. 5). The circles having a center on the normal stress axis and tangential to a PYL are called the Mohr stress circles.

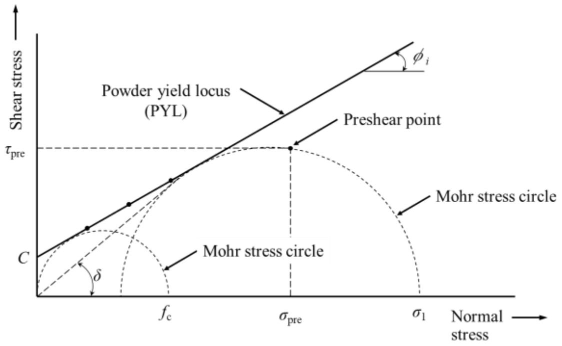

Fig. 5 Graphical representation of various parameters obtainable from PYL

Various parameters that describe powder flowability can be obtained from a PYL and Mohr stress circles.

4.1. Shearing cohesion (*C*)

The failure shear stress at zero normal stress, normally obtained by extrapolation of the PYL. An indication of the intrinsic strength of an unconfined powder.

4.2. Angle of internal friction (φ_i)

The angle formed by the PYL and the σ axis. The inclination (tan φ_i) of the PYL indicates the internal friction between the powder particles under the consolidation conditions measured.

4.3. Effective angle of internal friction (δ)

The angle formed by the straight line which passes through the origin and tangential to the larger Mohr stress circle in Fig. 5, and the σ axis. A relative indication of the internal friction when the powder flow is in a steady-state.

4.4. Flow function (FF)

The ratio (σ_1/f_c: ff_c) of the maximum principal stress (σ_1) of the larger Mohr stress circle and the maximum principal stress (uniaxial collapse stress: f_c) of the smaller Mohr stress circle in Fig. 5 may be used as a quantitative classification indication of powder flowability (Table 1). A regression line obtained from the σ_1-f_c relationship measured under various consolidation conditions for one material, which is called FF, is used for powder flow analysis such as when designing a hopper.

Table 1 General classification of flowability

ff_c	Flowability
<1	Not flowing
1 – 2	Very cohesive, difficult-flowing
2 – 4	Cohesive, slightly difficult-flowing
4 – 10	Easy-flowing
10<	Free-flowing

It should be noted that even a same powder shows different flowability if the degree of consolidation is different because the above parameters can be determined from Fig. 5 where the measurements were performed with a sample having one specified void fraction.

On the other hand, the critical state line (CSL) in Fig. 1 is obtained by projecting the preshear points (black circles in the figure), which are determined from several samples having a different void fraction, onto the σ-τ plane, and is a straight line passing through the origin. Since the CSL shows the normal stress-shear stress relationship in a dynamic state, it reflects the powder flow properties without depending on the type of apparatus used for measurement. The angle formed by the CSL and the σ axis is called the angle of critical state line (φ_{CSL}); the smaller the value, the higher the flowability.

5. Report of results

Measurements under the same conditions are repeated an appropriate number of times according to the variation in the obtained values, and the average value is reported along with the items listed in Table 2.

Table 2 Examples of items to be described in the report of results

Item	Content
General information	Measurement date/time, name of operator, sample name, apparatus used (type, model/manufacturing company) and type of cells, measurement method (constant load method or constant volume method), etc.
Sample-related information	Particle size and particle size distribution, method of particle size measurement, bulk density, water content, conditions for drying, etc.
Measurement conditions	Temperature and relative humidity during measurements, size of cells used, sample volume, preconsolidation conditions, shear rate, etc.
Results	Normal stress and shear stress of each measurement in the main test, σ-τ plot showing yield locus, various parameters obtained by the analysis of angle of critical state line, etc.
Other special notes	Descriptions when the measurement conditions such as the preconsolidation stress and the number of measurements are changed from the normal setting, or when the sample is reused, etc.