Flow Properties of Powders and Bulk Solids

Dietmar Schulze[1]

In order to compare and optimize powders regarding flowability and to design powder handling equipment like silos, feeders, and flow promoting devices, it is necessary to know the mechanical properties – the so-called flow properties. In the present paper it is outlined which physical parameters describe the flow properties of a powder or a bulk solid, and how these parameters are determined experimentally.

1 Introduction

Knowledge of the flow properties of a powder or a bulk solid is necessary to design silos and other bulk solid handling equipment so that no flow problems (flow obstructions, segregation, irregular flow, flooding, etc.) occur. Furthermore, quantitative information regarding flowability of bulk products is required, e.g. as part of comparative tests (e.g. effect of flow agents or other additions on flow behaviour) and quality control. The flow properties depend on several parameters, e.g.,

- particle size distribution,
- particle shape,
- chemical composition of the particles,
- moisture,
- temperature.

It is not possible to determine theoretically the flow behaviour of bulk solids in dependence of all of these parameters. Even if this were possible, the expense for the determination of all parameters of influence would be very high. Thus it is necessary, and also simpler, to determine the flow properties in appropriate testing devices.

The present paper deals with all kind of particulate solids, which are also called bulk solids, powders, or granulates. In the following the general expression “bulk solid” is used for all these products.

2 Stresses in bulk solids

Figure 1 shows a bulk solid element in a container (assumptions: infinite filling height, frictionless internal walls). In the vertical direction, positive normal stress ($\sigma_v > 0$, compressive stress) is exerted on the bulk solid.

If the bulk solid were to behave like a Newtonian fluid, the stresses in the horizontal and vertical direction (and in all other directions) would be of equal magnitude. In reality the behaviour of a bulk solid is quite different from that of a fluid, so that the assumption of analogies is often misleading.

Within the bulk solid (Figure 1) the horizontal stress, σ_h, is a result of the vertical stress, σ_v, where the resulting horizontal stress is less than the vertical stress exerted on the bulk solid from the top. The ratio of horizontal stress to vertical stress is the stress ratio, K (also known as λ).

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[1] Prof. Dr.-Ing. Dietmar Schulze, Ostfalia University of Applied Sciences, Wolfenbüttel, Germany.
E-Mail: mail@dietmar-schulze.de, Internet: www.dietmar-schulze.de

$$K = \sigma_{\mathrm{h}} / \sigma_{\mathrm{v}} \tag{1}$$

Typical values of *K* are between 0.3 and 0.6 [15].

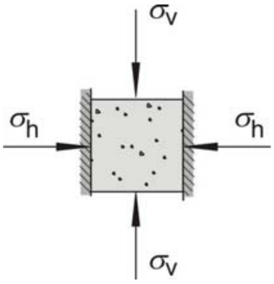

Figure 1: Element of bulk solid

It follows that – in analogy to solids – in a bulk solid different stresses can be found in different cutting planes. Stresses in cutting planes other than the vertical and the horizontal can be analyzed using a simple equilibrium of forces:

No shear stresses τ are exerted on the top or bottom surface of the bulk solid element in Figure 1; i.e., the shear stresses in these planes are equal to zero. No shear stresses are acting at the lateral walls, since the lateral walls were assumed as frictionless. Thus only the normal stresses shown are acting on the bulk solid from outside. Using a simple equilibrium of forces at a volume element with triangular cross-section cut from the bulk solid element shown in Figure 1 (Figure 2, on the left), the normal stress, σ_α, and the shear stress, τ_α, acting on a plane inclined by an arbitrary angle α, can be calculated. After some mathematical transformations, which need not be considered here, it follows that:

$$\sigma_{\alpha} = \frac{\sigma_{\mathrm{v}} + \sigma_{\mathrm{h}}}{2} + \frac{\sigma_{\mathrm{v}} - \sigma_{\mathrm{h}}}{2} \cos(2\alpha) \tag{2}$$

$$\tau_{\alpha} = \frac{\sigma_{\mathrm{v}} - \sigma_{\mathrm{h}}}{2} \sin(2\alpha) \tag{3}$$

The pair of values (σ_α, τ_α), which are to be calculated according to equations (2) and (3) for all possible angles α, can be plotted in a σ,τ-diagram (normal stress, shear stress - diagram); see Figure 2 on the right. If one joins all plotted pairs of values, a circle emerges; i.e., all calculated pairs of values form a circle in the σ,τ-diagram.

This circle is called “the Mohr stress circle”. Its centre is located at $\sigma_{\mathrm{m}} = (\sigma_{\mathrm{v}}+\sigma_{\mathrm{h}})/2$ and $\tau_{\mathrm{m}} = 0$. The radius of the circle is $\sigma_{\mathrm{r}} = (\sigma_{\mathrm{v}}-\sigma_{\mathrm{h}})/2$. The Mohr stress circle represents the stresses on all cutting planes at arbitrary inclination angles α, i.e., in all possible cutting planes within a bulk solid element.

Since the centre of the Mohr stress circle is always located on the σ-axis, each Mohr stress circle has two points of intersection with the σ-axis. The normal stresses defined through these points of intersection are called the principal stresses, whereby the larger principal stress – the major principal stress – is designated as σ_1 and the smaller principal stress – the minor principal stress – is designated as σ_2. If both principal stresses are given, the Mohr stress circle is well defined.

In the example of Figure 1 both the horizontal and the vertical plane are free from shear stresses ($\tau = 0$) and are thus principal stress planes. In this case the vertical stress, σ_{v}, which is greater than the horizontal stress, σ_{h}, is the major principal stress, σ_1, and the horizontal stress, σ_{h}, is the minor principal stress, σ_2.

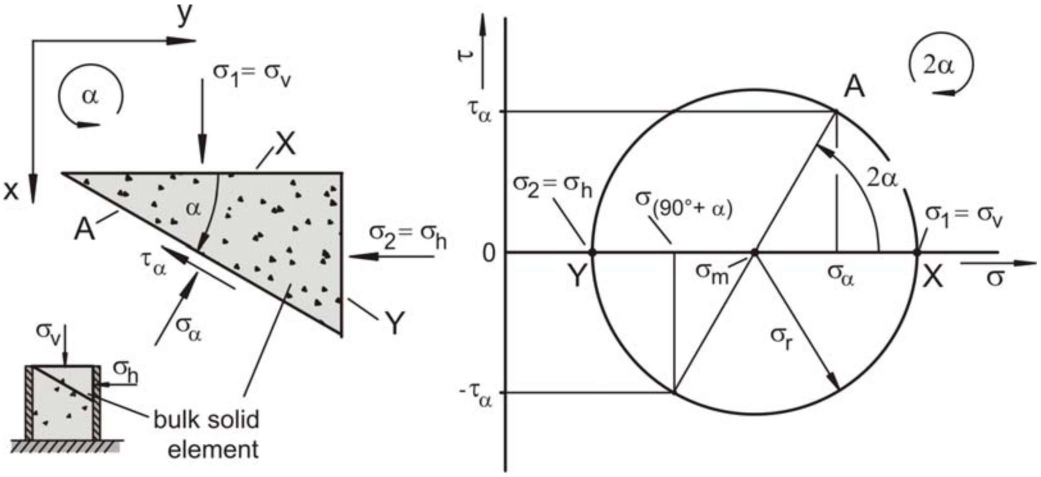

Figure 2: Force equilibrium on an element of bulk solid, the Mohr stress circle

An important qualitative result of the above analysis is that shear stresses can occur in bulk solids at rest. This is impossible for a Newtonian fluid at rest (in contrast to Newtonian fluids, bulk solids can have sloped surfaces even at rest). Therefore, a representation of the stresses (fluids: pressures) in different cutting planes of a Newtonian fluid at rest in a σ,τ-diagram would yield a stress circle with the radius zero (equation (3) with $\sigma_h = \sigma_v$ yields $\tau_\alpha = 0$).

From the explanation above it follows that the state of stress in a bulk solid cannot be completely described by only a single numerical value. Depending on the actual load acting on a bulk solid element, the corresponding Mohr stress circle can have a smaller or a larger radius, a centre at a lesser or greater normal stress, and hence also different principal stresses, σ_1 and σ_2. In principle, at a given major principal stress, σ_1, stress circles with different values for the lowest principal stress, σ_2, are imaginable. Therefore, a stress circle is defined clearly only if at least two numerical values are given, i.e., σ_1 and σ_2.

In summary, the following can be stated with regard to the stresses acting in bulk solids:

- A bulk solid can transmit shear stresses even if it is at rest.
- In different cutting planes different stresses are acting.
- Stress conditions can be represented with Mohr stress circles.

3 Adhesive forces

The flowability of a bulk solid depends on the adhesive forces between individual particles. Different mechanisms create adhesive forces [5]. With fine-grained, dry bulk solids, adhesive forces due to van der Waals interactions play the essential role. With moist bulk solids, liquid bridges between the particles usually are most important. Liquid bridges are formed by small regions of liquid in the contact area of particles, in which due to surface tension effects a low capillary pressure prevails.

Both types of adhesive forces described above are dependent on the distance between particles and on particle size.

Some bulk solids continue to gain strength if stored at rest under compressive stress for a longer time interval. This effect is called time consolidation. The reasons for time consolidation are also to be found in the effects of adhesive forces. Possible mechanisms are:

- Solids bridges due to solid crystallizing when drying moist bulk solids, where the moisture is a solution of a solid and a solvent [5] (e.g. sand and salt water).
- Solid bridges from the particle material itself, e.g. after some material at the contact points has been dissolved by moisture [5] (e.g. crystal sugars with slight dampness).

- Bridges due to sintering during storage of the bulk solid at temperatures not much lower than the melting temperature [5]. This can appear e.g. at ambient temperature during the storage of plastics with low melting points.
- Plastic deformation at the particle contacts, which leads to an increase in the adhesive forces through approach of the particles and enlargement of the contact areas.
- Chemical processes (chemical reactions at the particle contacts).
- Biological processes (e.g. due to fungal growth on biologically active ingredients).

Whether a bulk solid flows well or poorly depends on the relationship of the adhesive forces to the other forces acting on the bulk solid. It can be shown that the influence of adhesive forces on flow behaviour increases with decreasing particle size. Thus, as a rule, a bulk solid flows more poorly with decreasing particle size. Fine-grained bulk solids with moderate or poor flow behaviour due to adhesive forces are called cohesive bulk solids.

If particles are pressed against each other by external forces, the compressive force acting between the particles increases. Thereby large stresses prevail (locally) at the particles' contact points, because the contact points are very small. This leads to plastic deformation of the particles in the contact area, so that the contact areas increase and the particles approach each other. Thereby the adhesive forces increase. Thus a compressive force acting from outside on a bulk solid element can increase the adhesive forces. This mechanism is used e.g. in the production of tablets or briquettes.

The dependence of the adhesive forces between the particles on external forces exerted on a bulk solid is characteristic of bulk solids, especially for cohesive bulk solids. Therefore an evaluation of bulk solids behaviour must always take into consideration the forces or stresses previously acting on the bulk solid, the stress history. The stress history includes, for example, the consolidation stress exerted on a bulk solid, leading to certain adhesive forces and hence to a certain strength of the bulk solid (e.g., the strength of a tablet is dependent on the maximum consolidation stress at tabletting).

4 Flowability

4.1 Uniaxial compression test

The phrase “good flow behaviour” usually means that a bulk solid flows easily, i.e., it does not consolidate much and flows out of a silo or a hopper due to the force of gravity alone and no flow promoting devices are required. Products are “poorly flowing” if they experience flow obstructions or consolidate during storage or transport. In contrast to these qualitative statements, a quantitative statement on flowability is possible only if one uses an objective characteristic value that takes into account those physical characteristics of the bulk solid that are responsible for its flow behaviour.

“Flowing” means that a bulk solid is deformed plastically due to the loads acting on it (e.g. failure of a previously consolidated bulk solid sample). The magnitude of the load necessary for flow is a measure of flowability. This will be demonstrated first with the uniaxial compression test. Figure 3 shows a hollow cylinder filled with a fine-grained bulk solid (cross-sectional area A; internal wall of the hollow cylinder assumed as frictionless). The bulk solid is loaded by the stress σ_1 – the consolidation stress – in the vertical direction. The more the volume of the bulk solid specimen is reduced, the more compressible the bulk solid is.

In addition to the increase in bulk density from consolidation stress, one will observe also an increase in strength of the bulk solid specimen. Hence, the bulk solid is both consolidated and compressed through the effect of the consolidation stress.

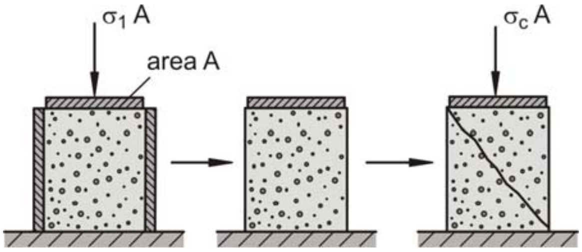

Figure 3: Uniaxial compression test

After consolidation, the bulk solid specimen is relieved of the consolidation stress, σ_1, and the hollow cylinder is removed. If subsequently the consolidated cylindrical bulk solid specimen is loaded with an increasing vertical compressive stress, the specimen will break (fail) at a certain stress. The stress causing failure is called compressive strength or unconfined yield strength, σ_c (another common designation is f_c).

In bulk solids technology one calls the failure "incipient flow", because at failure the consolidated bulk solid specimen starts to flow. Thereby the bulk solid dilates somewhat in the region of the surface of the fracture, since the distances between individual particles increase. Therefore incipient flow is plastic deformation with decrease of bulk density. Since the bulk solid fails only at a sufficiently large vertical stress, which is equal to the compressive strength, there must exist a material-specific yield limit for the bulk solid. Only when this yield limit is reached does the bulk solid start to flow.

The yield limits of many materials (e.g. metals) are material-dependent and are listed in tables. However, the yield limit of a bulk solid is dependent also on its stress history, i.e., previous consolidation: The greater the consolidation stress, σ_1, the greater the bulk density, ρ_b, and unconfined yield strength, σ_c.

Uniaxial compression tests (Figure 3) conducted at different consolidation stresses, σ_1, leads to different pairs of values (σ_c, σ_1) and (ρ_b, σ_1). Plotting these pairs of values as points in a σ_c,σ_1-diagram and a ρ_b,σ_1-diagram, respectively, and drawing in each diagram a curve through these points, usually results in curves like those for product A in Figure 4, where bulk density, ρ_b, and unconfined yield strength, σ_c, typically increase with consolidation stress, σ_1. Very rarely a progressive slope like in the left part of curve B is observed. The curve $\sigma_c(\sigma_1)$ is called the flow function.

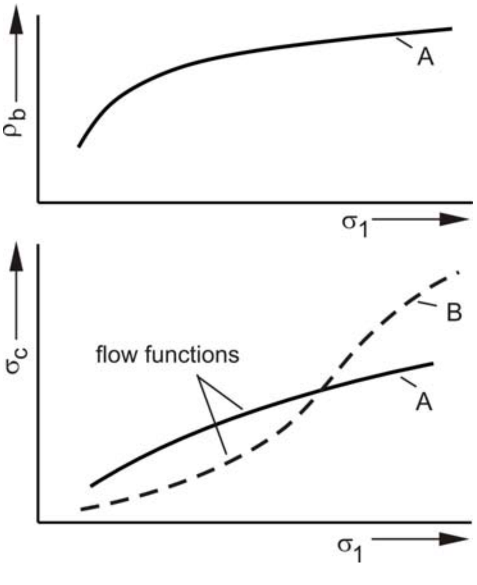

Figure 4: Bulk density, ρ_b, and unconfined yield strength, σ_c, vs. consolidation stress, σ_1

4.2 Time consolidation (caking)

Some bulk solids increase in strength if they are stored for a longer time at rest under a compressive stress (e.g. in a silo or an intermediate bulk container). This effect is called time consolidation or caking. Time consolidation can be determined with the test shown in Figure 3, in order, e.g., to simulate long-term storage in a silo. For this one loads the specimen with consolidation stress, σ_1, not only for a short moment, but for a defined period of time, t_1. Then the unconfined yield strength is determined following the principle explained above (Figure 3).

Figure 5 shows the flow function $\sigma_c(\sigma_1)$ of product A as previously shown in Figure 4 (unconfined yield strength without influence of time consolidation, i.e., for a storage period $t = 0$). Additionally, examples of curves $\sigma_c(\sigma_1)$ for storage periods $t > 0$ (curves A_1, A_2) are drawn. The curves $\sigma_c(\sigma_1)$ for the storage periods $t > 0$ are called time flow functions. Here each curve emerges from the connection of several pairs of values (σ_c, σ_1), which were measured at identical storage periods, t, but at different consolidation stresses, σ_1.

For the example of bulk solid A, the unconfined yield strength, σ_c, increases with increasing storage time. This result is true for many bulk solids, but not for all. There are bulk solids which undergo no or only very slight consolidation over time; i.e., σ_c does not increase, or increases only very slightly with increasing storage period, t (e.g. dry quartz sand). Other bulk solids undergo a large increase in unconfined yield strength after storage periods of only a few hours, whereas after longer storage periods their unconfined yield strength does not increase further. These differences are due to the different physical, chemical, or biological effects that are the causes of consolidation over time, e.g. chemical processes, crystallizations between the particles, enlargement of the contact areas through plastic deformation, capillary condensation, or biological processes such as fungal growth (see Section 3).

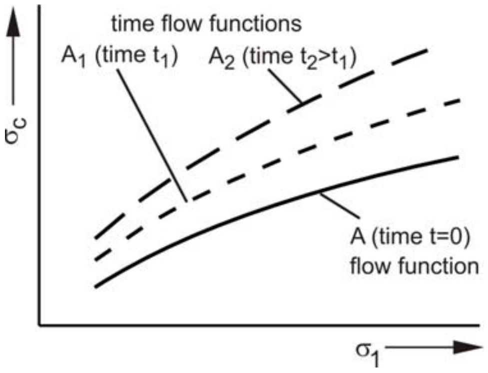

Figure 5: Flow function and time flow functions for two different storage times t_1 and $t_2 > t_1$.

With measurement of time consolidation, a “time-lapse effect” is not realizable; i.e., one must store a bulk solid specimen at the consolidation stress, σ_1, for exactly that period of time for which one would like to get data on time consolidation. Without such a test no quantitative statement can be made regarding time consolidation.

4.3 Yield limit and Mohr stress circles

The uniaxial compression test presented in Figure 3 is shown below in a σ,τ-diagram (Figure 6). If one neglects the force of gravity of the bulk solid specimen and assumes that no friction is acting between the wall of the hollow cylinder and the bulk solid, both vertical stress, σ_v, as well as horizontal stress, σ_h, are constant within the entire bulk solid specimen. Therefore at each position in the bulk solid sample the state of stress, which can be represented by a Mohr stress circle, is identical.

During consolidation the vertical normal stress, σ_1, acts on the top of the bulk solid specimen. Perpendicular to the vertical stress the lesser, horizontal stress prevails according to stress ratio K (see Section 2). Neither at top nor at bottom of the specimen, nor at the internal wall of the hollow cylinder, which is assumed as frictionless, will shear stresses be found; i.e., $\tau = 0$. The pairs of values (σ, τ) for vertical and horizontal cutting planes within the bulk solid specimen are plotted in the σ,τ-diagram (Figure 6). Both points are located on the σ-axis because $\tau = 0$. The Mohr stress circle A, which describes the stresses in the bulk solid sample at consolidation, is thus well defined (because each stress circle has exactly two intersections with the σ-axis).

Since in the vertical plane the shear stress is zero, the vertical stress is identical with the major principal stress, σ_1 (see Section 2: Principal stresses are the normal stresses in those planes in which the shear stresses are equal to zero). The major principal stress, σ_1, is equal to the vertical stress, σ_v, and the minor principal stress, σ_2, is equal to the horizontal stress, σ_h.

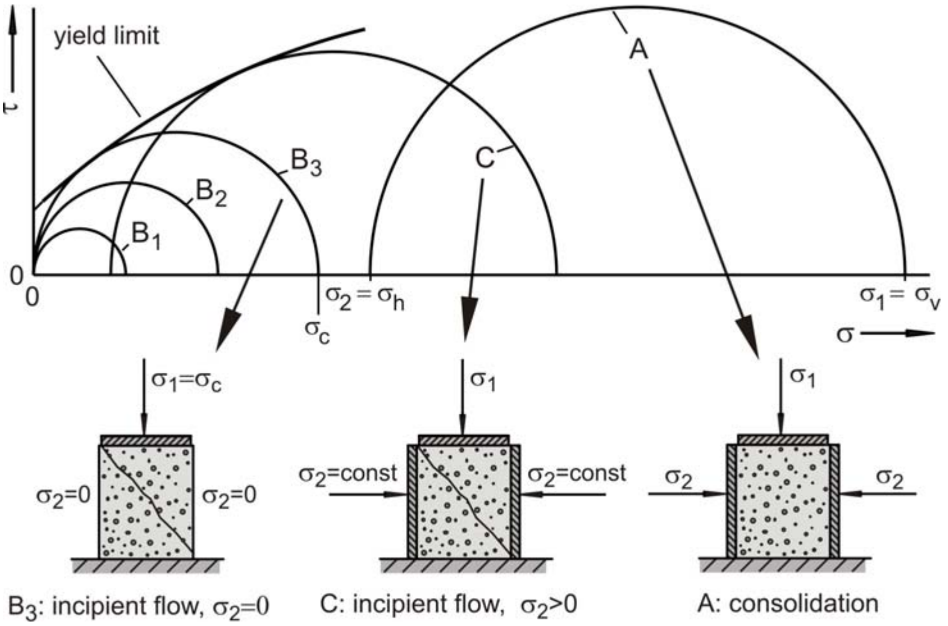

Figure 6: Measurement of unconfined yield strength in a σ,τ-diagram

In the second part of the test shown in Figure 3, the specimen is loaded with increasing vertical stress after it has been relieved of the consolidation stress and the hollow cylinder has been removed. The vertical stress and horizontal stress are principal stresses. The horizontal stress is independent of the vertical load equal to zero, since the lateral surface of the specimen is uncovered and not loaded.

During the increasing vertical load in the second part of the test, the stress states at different load steps are represented by stress circles with increasing diameter (stress circles B_1, B_2, B_3 in Figure 6). The lesser principal stress, which is equal to the horizontal stress, is equal to zero at all stress circles.

At failure of the specimen the Mohr stress circle B_3 represents the stresses in the bulk solid sample. Since the load corresponding to this Mohr stress circle causes incipient flow of the specimen, the yield limit of the bulk solid must have been attained in one cutting plane of the specimen. Thus, Mohr stress circle B_3 must reach the yield limit in the σ,τ-diagram. In Figure 6 a possible yield limit is shown. The real course of the yield limit can not be determined with only the uniaxial compression test.

The Mohr stress circles B_1 and B_2, which are completely below the yield limit, cause only an elastic deformation of the bulk solid specimen, but no failure and/or flow. Stress circles larger than stress circle B_3, and thus partly above the yield limit, are not possible: The specimen would already be flowing when the Mohr stress circle reaches the yield limit (failure), so that no larger load could be exerted on the specimen.

If, during the second part of the experiment shown in Figure 3 (measurement of compressive strength), one were to apply also a constant horizontal stress $\sigma_h > 0$ on the specimen (in addition to the vertical stress, σ_v), one would likewise find stress circles that indicate failure of the specimen and reach the yield limit (e.g. stress circle C in Figure 6). Thus the yield limit is the envelope of all stress circles that indicate failure of a bulk solid sample.

4.4 Numerical characterization of flowability

Flowability of a bulk solid is characterized mainly by its unconfined yield strength, σ_c, in dependence on consolidation stress, σ_1, and storage period, *t*. Usually the ratio ff_c of consolidation stress, σ_1, to unconfined yield strength, σ_c, is used to characterize flowability numerically:

$$ff_c = \sigma_1 / \sigma_c \tag{4}$$

The larger ff_c is, i.e., the smaller the ratio of the unconfined yield strength, σ_c, to the consolidation stress, σ_1, the better a bulk solid flows. Similar to the classification used by Jenike [1], one can define flow behaviour as follows:

$ff_c < 1$	not flowing
$1 < ff_c < 2$	very cohesive
$2 < ff_c < 4$	cohesive
$4 < ff_c < 10$	easy-flowing
$10 < ff_c$	free-flowing

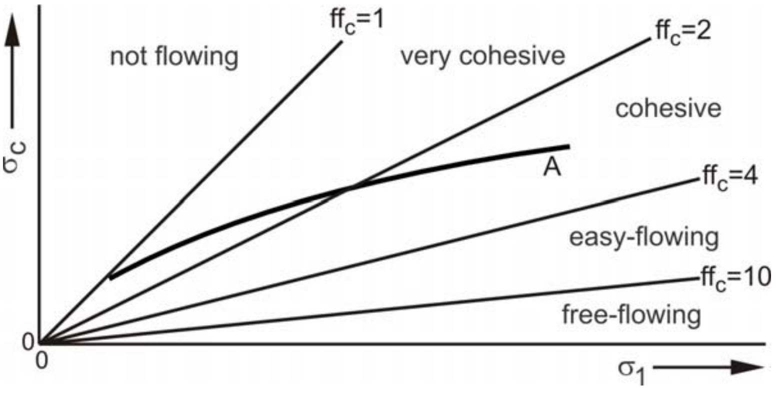

Figure 7: Flow function and lines of constant flowability

In Figure 7 the flow function A taken from the σ_c,σ_1-diagram in Figure 4 are shown. Additionally, the boundaries of the ranges of the classifications listed above are shown as straight lines, each representing a constant value of flowability, ff_c. This diagram clearly shows that the flowability, ff_c, of a specific bulk solid is dependent on the consolidation stress, σ_1 (in most cases ff_c increases with σ_1 as with bulk solid A). Therefore, with each consolidation stress at which σ_c and thus ff_c were determined, one obtains a different value of flowability: The flowability of a bulk solid depends on the stress level (= consolidation stress); thus for most bulk solids one will obtain a larger value of flowability (= better flowability) at a greater consolidation stress. For most bulk solids one will find a (possibly extremely low) consolidation stress at which the bulk solid flows poorly. Because of the dependence of flowability on consolidation stress, it is not possible (unfortunately!) to describe the flowability of a bulk solid with only one numerical value.

With the results of time consolidation tests, flowability can be determined with Eq. (4), using the unconfined yield strength, σ_c, which was measured after the corresponding storage period. If the bulk solid shows a time consolidation effect, one will measure an increasing unconfined yield strength with increasing storage period, so that from Eq. (4) lesser flowability will follow. This is logical: If a bulk solid gains strength with an increasing period of storage at rest at a certain consolidation stress, it will be more difficult to get this bulk solid to flow; i.e., its flowability decreases with increasing storage period.

In Figure 5 flow function A and two time flow functions are shown. The flow function represents unconfined yield strength, σ_c, in dependence on consolidation stress, σ_1, without influence of a storage period, i.e., for the storage period $t = 0$. A time flow function represents the unconfined yield strength which emerges after storage at the consolidation stress over a period of time, t. The flow function and time flow functions from Figure 5 are shown in Figure 8.a along with the boundaries of the ranges, which follow from the classification of flowability as outlined above. It can be seen that flowabilities, ff_c, measured at identical consolidation stress, but after different consolidation periods, decrease with increasing consolidation time (Figure 8.b). For the consolidation stress σ_{Example} chosen as an example, one obtains measurement points in areas of decreasing flowability when increasing the consolidation period t (see arrow in Figure 8.a).

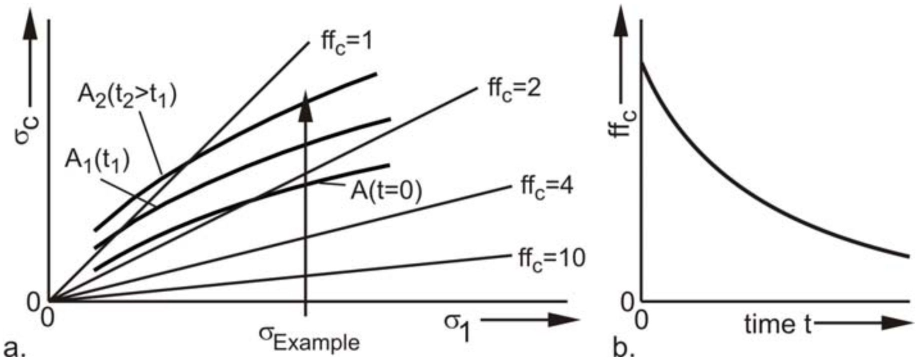

Figure 8: Influence of storage time on flowability

From the dependence of flowability, ff_c, on consolidation stress, σ_1, it follows that one can compare the flow behaviour of several bulk solids quantitatively using ff_c only if all measurements have been performed at identical consolidation stresses. Otherwise totally different (incorrect) statements might result. This shows how important it is to test a bulk solid at defined and known conditions (e.g. known consolidation stress).

5 Practical determination of flow properties

In the previous section the flow behaviour has been explained in a simplified way by using the uniaxial compression test as a model. The use of the uniaxial compression test with fine-grained, cohesive bulk solids is problematic, because one obtains unconfined yield strength values that are too low [7], and preparation of the hollow cylinder to obtain frictionless walls is very time-consuming. In addition, further important parameters (e.g. internal friction and wall friction) cannot be determined with this test. It is, however, an appropriate measurement technique for the measurement of the time consolidation of coarse-grained bulk solids.

In order to measure the flow properties of fine-grained bulk solids, in advanced bulk solids technology so-called shear testers are used. In the following first the principle of shear testing is outlined. Afterwards, the translational shear tester introduced by Jenike around 1960

(Jenike shear tester; the first shear tester especially designed for bulk solids) [1,2,5,16] and the Schulze ring shear tester [2,9–11,14,17] will be described.

5.1 Shear test procedure (yield locus)

The goal of a shear test is to measure the yield limit of a consolidated bulk solid. The yield limit is called yield locus in bulk solids technology. For a shear test, a bulk solid specimen is loaded vertically by a normal stress, σ (Figure 9.a). Then a shear deformation is applied on the specimen by moving the top platen with a constant velocity, v. This results in a horizontal shear stress, τ (Figure 9.b). With increasing shear stress the resultant force, F_R, acting on the bulk solid specimen, increases.

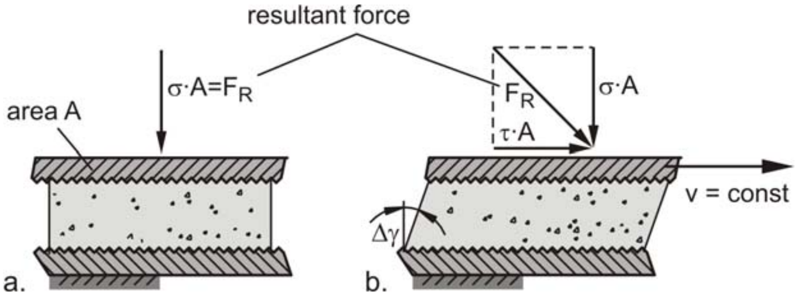

Figure 9: Bulk solid specimen: a. initial loading with normal stress σ; b. shear deformation (velocity v = const)

When a point of a yield locus is measured, in analogy to the uniaxial compression test, two steps are necessary: First the bulk solid specimen is consolidated, what is called “preshear”. Subsequently a point of the yield limit is measured. This step is called “shear” or “shear to failure”.

For preshear the bulk solid specimen is loaded in the vertical direction by a well-defined normal stress, $\sigma = \sigma_{pre}$. Then the specimen is sheared. At the beginning of preshear the shear stress τ increases with time (as shown in the left diagram in Figure 10). With time the curve of shear stress vs. time becomes flatter, and finally the shear stress remains constant even though the specimen is sheared further. The constant shear stress is called τ_{pre}. After constant shear stress has been attained, neither shear resistance (and strength) nor bulk density increase further. Thus the bulk solid specimen is sheared at constant normal stress, σ, constant shear stress, τ, and constant bulk density, ρ_b. Thus flow, or plastic deformation, occurs at constant bulk density. This type of flow, attained at preshear, is called steady-state flow. The state of the bulk solid after steady-state flow is attained is called “critically consolidated with respect to normal stress, σ_{pre}”. The characteristic stress for this consolidation – the major principal stress σ_1 – will be considered later.

The bulk density, ρ_b, and the shear stress, τ_{pre}, attained at steady-state flow are characteristic for the applied normal stress at preshear, σ_{pre}. In principle, an identical state of consolidation, characterized by the same bulk density, ρ_b, and the same shear stress, τ_{pre}, will be attained with other specimens of the same bulk solid presheared under the same normal stress, σ_{pre}.

After the bulk solid specimen has been consolidated by the preshear procedure, the shear deformation is reversed until the shear stress, τ, is reduced to zero. The pair of values of normal stress and shear stress at steady-state flow (σ_{pre}, τ_{pre}) is plotted in a normal stress - shear stress diagram (σ,τ-diagram, Figure 10, right). Point (σ_{pre}, τ_{pre}) is called the “preshear point”.

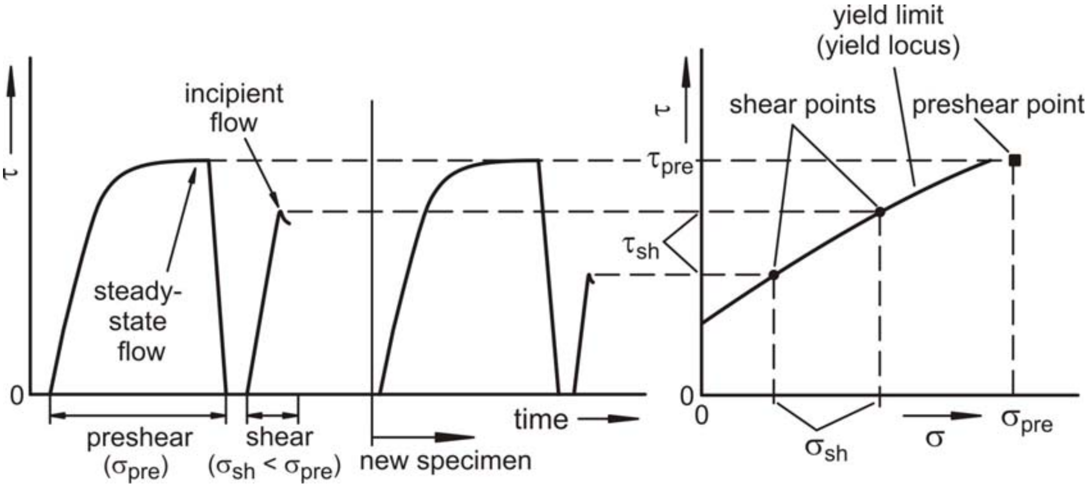

Figure 10: Plot of shear stress vs. time; yield locus

After preshear the bulk solid specimen in the shear cell is defined as a critically consolidated specimen. The second step of the test procedure – shear or shear to failure – is discussed next.

For shear to failure the normal stress acting on the specimen is decreased to a value σ_{sh}, which is less than the normal stress at preshear, σ_{pre}. Had the specimen been presheared under the lower normal stress, σ_{sh}, and not under σ_{pre}, its bulk density and strength would have been less. Since the specimen was presheared under the greater normal load, σ_{pre}, it was consolidated more than it would have been with the lower normal load, σ_{sh}.

If the consolidated specimen is sheared under the normal stress $\sigma_{sh} < \sigma_{pre}$, it will start to flow (fail) when a sufficiently large shear force, or shear stress, is attained. At that point particles start to move against each other. The material will start to dilate (decrease in bulk density) and shear resistance and thus shear stress will decrease (Figure 10). The maximum shear stress characterizes incipient flow. The corresponding pair of values (σ_{sh}, τ_{sh}) is a point of the yield limit of the consolidated specimen in the σ,τ-diagram (Figure 10, right). Such a point is called a “shear point” or a “point of incipient flow”.

In order to measure the course of the yield locus, several of the tests described above must be performed, where the specimens first must be consolidated at identical normal stress, σ_{pre} (preshear). Then the specimens are sheared (to failure) under different normal stresses, $\sigma_{sh} < \sigma_{pre}$. As outlined above, by preshearing at identical normal stress, σ_{pre}, each specimen reaches the same state of consolidation. Each test yields the same preshear point (σ_{pre}, τ_{pre}), and one individual shear point (σ_{sh}, τ_{sh}) in accordance with the different normal stresses, σ_{sh}, applied at shear. The yield locus follows from a curve plotted through all measured shear points (Figure 10, right).

5.2 Jenike shear tester

Around 1960 Jenike [1] published his fundamental work on silo and bulk solids technology and introduced the Jenike Shear Tester, a translational shear tester. This tester was the first one designed for the purposes of powder technology (e.g. measurement at small stresses), and still today shear testers are compared to the Jenike Shear Tester.

The shear cell of the Jenike shear tester consists of a bottom ring (also called mould ring), a ring of the same diameter (so-called upper ring) lying above the bottom ring, and a lid (Figure 11). The lid is loaded centrally with a normal force, F_N. The upper part of the shear cell is displaced horizontally against the fixed bottom ring by a motor driven stem which pushes against a bracket fixed to the lid. The force F_S – the shear force – exerted by the stem is measured. Due to the displacement of the upper ring and the lid against the bottom ring, the bulk solid undergoes a shear deformation. The normal stress, σ, and shear stress, τ, acting in the horizontal plane between upper ring and bottom ring are determined by dividing normal force, F_N, and shear force, F_S, by the cross-sectional area of the shear cell, A.

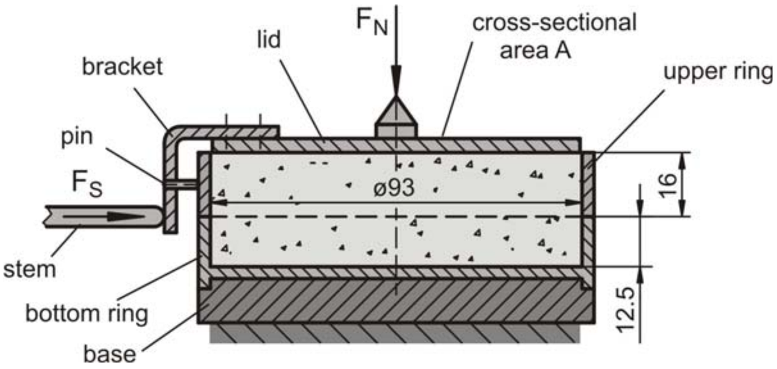

Figure 11: Shear cell of the Jenike shear tester [1,16]

For the measurement of a point of a yield locus, the shear cell is filled with the bulk solid specimen. After a manual preconsolidation [16] the specimen is presheared and then sheared to failure as outlined in the previous section. For the next point of the yield locus, a new bulk solid specimen has to be prepared and sheared.

Although the Jenike Shear Tester is internationally recognized, from today's point of view a disadvantage might be the time required for a test (one to two hours per yield locus; depending on the powder and the operator's skill) during which the operator has to be present. In addition, the manual preconsolidation of each specimen can be a source of measurement errors, and due to the limited shear displacement (maximum: twice the thickness of the wall of the upper ring) materials requiring too much deformation to attain steady-state flow can hardly be tested.

5.3 Ring shear tester

Ring shear testers (rotational shear testers) have been used in soil mechanics since the 1930s [4]. In the 1960s Walker designed a ring shear tester for bulk solids [3], where lower stresses than in soil mechanics are of interest. In the following decades different ring shear testers have been built and investigated at several universities (e.g. [6,8]. In 1992 a ring shear tester (type RST-01.01) [9,10] was developed by the author, followed by a computer-controlled version in 1997 (type RST-01.pc). It is connected to a personal computer running a control software. With this control software yield loci, wall yield loci, time consolidation, etc. can be measured automatically. A smaller computer-controlled ring shear tester (type RST-XS) has been available since 2002. This tester enables use of small specimen volumes (3.5 ml, 9 ml, 30 ml, and 70 ml).

Figure 12 shows the principle of the shear cell of a ring shear tester (series RST-01) [2,9,10,17]. The ring-shaped (annular) bottom ring of the shear cell contains the bulk solid specimen. The (annular) lid is placed on top of the bulk solid specimen. The lid is fixed at a crossbeam.

A normal force, F_N, is exerted to the crossbeam in the rotational axis of the shear cell and transmitted through the lid to the bulk solid specimen. Thus a normal stress σ is applied to the bulk solid specimen. The counterbalance force, F_A , also acts in the centre of the crossbeam. F_A is directed upward and is created by counterweights. F_A counteracts the gravity forces of the lid, the hanger, and the crossbeam.

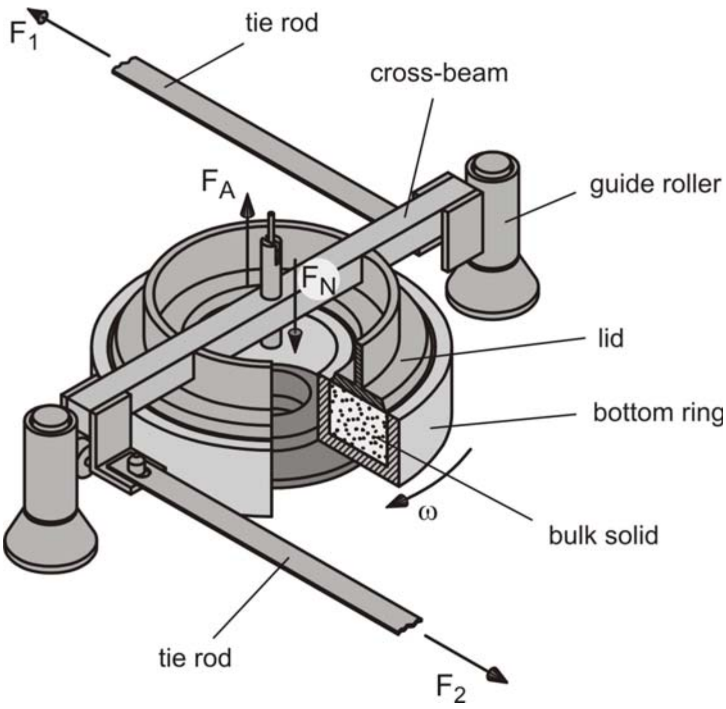

Figure 12: Shear cell of a ring shear tester (here: Schulze ring shear tester type RST-01) [2,9,10,17]

To shear the bulk solid, the lid and the bottom ring of the shear cell must rotate relative to each other. This is accomplished by rotating the bottom ring in the direction of the arrow ω (ω is the angular velocity), whereas the lid and the crossbeam are prevented from rotation by two tie-rods connected to the crossbeam. Each of the tie-rods is fixed at a load beam, so that the forces, F_1 and F_2, acting in the tie rods can be measured.

The bottom of the shear cell and the lower side of the lid are rough in order to prevent the bulk solid from sliding relative to these surfaces. Therefore, rotation of the bottom ring relative to the lid creates a shear deformation within the bulk solid. Through this shearing the bulk solid is deformed and thus a shear stress τ prevails. The forces acting in the tie rods (F_1 and F_2) are directly proportional to the shear stress τ acting in the bulk solid.

In addition to the shear force (F_1, F_2), the ring shear tester also measures the vertical position of the lid. If a bulk solid is compressible, its bulk density, ρ_b, will increase more or less, depending on the normal load applied. If the vertical position of the lid relative to the bottom ring is measured, one can calculate the volume of the bulk solid specimen. If the mass of the specimen is also determined through weighing, bulk density can be calculated.

The principle of the ring shear tester described above is different from other ring shear testers in a few important details, e.g. the lid, which lies on the bulk solid sample, similar to the lid of the Jenike shear cell, and is not held horizontally by a bearing as with older ring shear testers. This yields a more homogeneous stress distribution across the specimen, and the absence of any bearing friction increases the accuracy of the measurement. Additionally, the masses of the lid and all parts connected to it are small, so that tests at low normal stresses are possible, and the shear cell including the lid and the bulk solid specimen can be taken from the tester without disturbing the sample, e.g. for time consolidation tests using a time consolidation bench.

The test procedure (Figure 13) is quite similar to the test procedure recommended for the Jenike Tester (preshear and shear, see above), although the test procedure for the ring shear tester is less time consuming, easier to perform, and, hence, less influenced by the person who runs the test. This in combination with the design outlined in the last paragraph results in the good reproducibility compared to other testers [18].

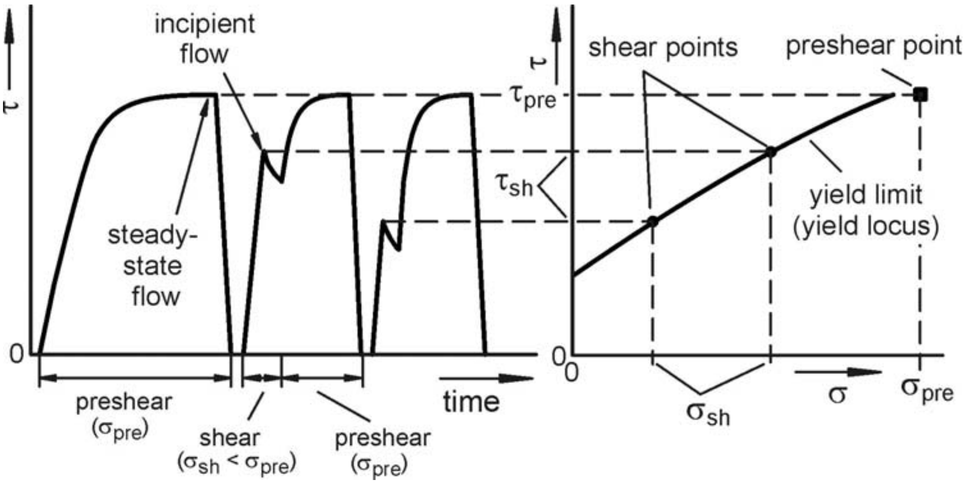

Figure 13: Shear test procedure of a ring shear tester

With the ring shear tester usually a complete yield locus is measured with one specimen (in contrast to the Jenike Tester where only one point can be measured with one specimen). In order to measure another point of the yield limit with the same specimen, after shear (shear to failure) the normal stress is increased again to σ_{pre}, which is the normal stress applied at first preshear. Then the specimen is presheared again under this normal stress until steady-state flow is attained. Thus, the specimen is again critically consolidated. After the specimen is relieved from shear stress (backward rotation of the shear cell until $\tau = 0$), the normal stress is reduced to another value of $\sigma_{sh} < \sigma_{pre}$ (Figure 13), and the specimen is then sheared again, thus obtaining another point of the yield limit in the σ,τ-diagram. After shear, the specimen is again presheared, then sheared, and so on, until a sufficient number of points of the yield limit are known and the yield locus can be drawn.

5.4 Yield locus

The parameters which describe the flow properties can be determined from the yield locus (Figure 14). The relevant consolidation stress σ_1 is equal to the major principal stress of the Mohr stress circle which is tangential to the yield locus and intersects at the point of steady state flow (σ_{pre}, τ_{pre}). This stress circle represents the stresses in the sample at the end of the consolidation procedure (stresses at steady state flow). It corresponds to the stress circle at the end of consolidation at the uniaxial compression test (Figure 3). The unconfined yield strength, σ_c, results from the stress circle which is tangential to the yield locus and which runs through the origin (minor principal stress $\sigma_2 = 0$). This stress circle represents a similar stress state as the one which prevails in the second step of the uniaxial compression test (stress circle B_3, Figure 4). In contrast to the uniaxial compression test the unconfined yield strength, σ_c, has to be determined on basis of the yield locus and does not follow directly from the measurement.

Please note that the analogy between the uniaxial compression test and the shear test is used here for the explanation of the yield locus. In reality, the stress circles at uniaxial compression and at steady state flow are not exactly the same, and a uniaxial compression test usually results in a smaller unconfined yield strength than a shear test [2,7,12,13,20].

A straight line through the origin of the σ,τ-diagram, tangent to the greater Mohr circle (steady-state flow), is the effective yield locus as defined by Jenike [1] (broken line in Figure 14). It encloses the σ-axis with the angle φ_e (effective angle of internal friction). Because the largest Mohr stress circle indicates a state of steady-state flow, the angle φ_e can be regarded as a measure of the internal friction at steady-state flow. This angle is required for silo design according to Jenike's theory.

If several yield loci are measured at different stress levels, i.e., with different normal stresses at preshear, σ_{pre}, each yield locus represents another state of consolidation and another bulk density. The above mentioned flow properties (unconfined yield strength, effective angle of internal friction) can be indicated as a function of the consolidation stress, σ_1, similar to Figure 4 where bulk density and unconfined yield strength are plotted vs. the consolidation stress.

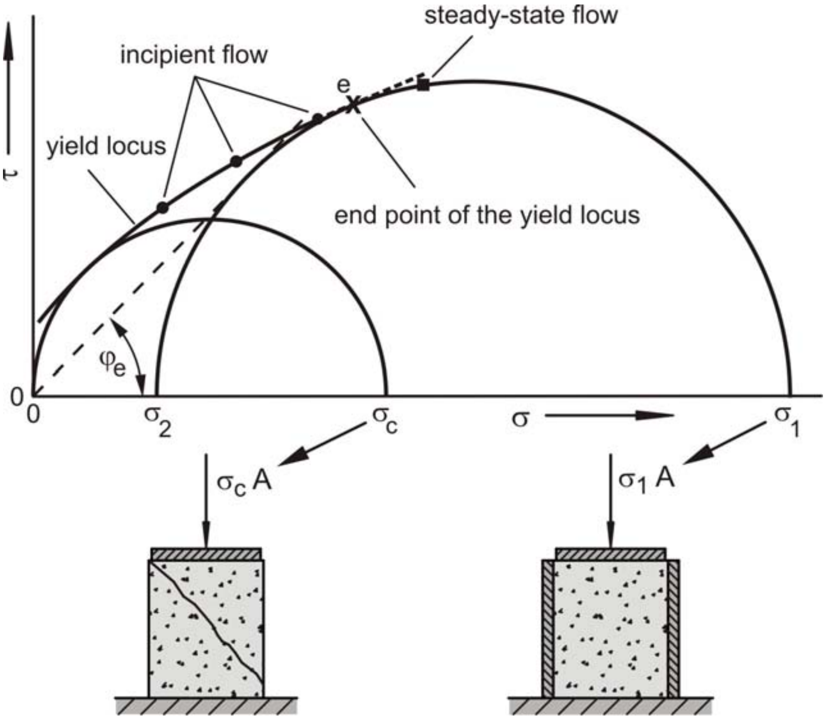

Figure 14: Yield locus, analogy to uniaxial compression test

5.5 Time consolidation

The effect of time consolidation has been outlined in Section 4.2. Before time consolidation can be measured, a yield locus must be determined at the same consolidation stress.

The time consolidation, which describes the increase of the unconfined yield strength with time during storage at rest, is measured with a shear tester similar to the measurement of a yield locus. First a bulk solid specimen is presheared (consolidated). After preshear the specimen is stored for a period, *t*, under the vertically acting normal stress, σ, which is selected to be equal to the consolidation stress, σ_1, of the corresponding yield locus. This ensures that during the consolidation period the same major principal stress (= consolidation stress, σ_1) acts on the specimen as during steady-state flow at preshear.

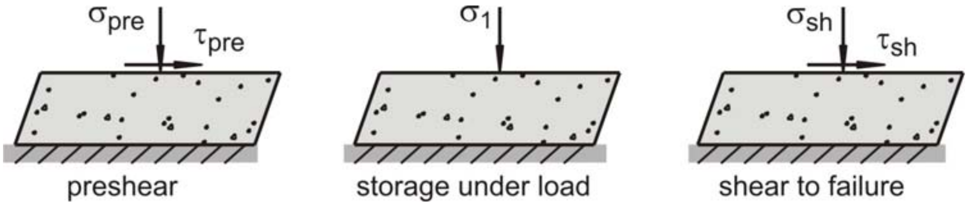

Figure 15: Time consolidation test

After the time consolidation period t the specimen is sheared to failure. For this, a vertical normal load, $\sigma_{sh} < \sigma_1$, is selected. As with shear without time consolidation (measurement of a point of a yield locus), so also after time consolidation will one observe a shear stress maximum. If consolidation time affects the bulk solid under consideration, after the consolidation period the shear stress maximum will be larger than it would have been without a consolidation period between preshear and shear (Figure 16).

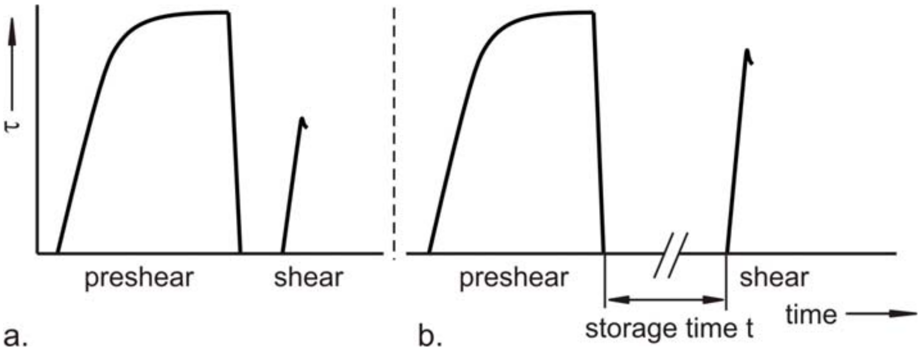

Figure 16: Shear stress vs. time at shear; with (a) and without (b) time consolidation

The maximum shear stress, τ, is a point of a yield limit, which is valid for the applied storage period, t, and called a “time yield locus”. Figure 17 shows a yield locus and two time yield loci obtained for different consolidation periods, t_1 and t_2. The yield locus can also be regarded as a time yield locus for $t = 0$.

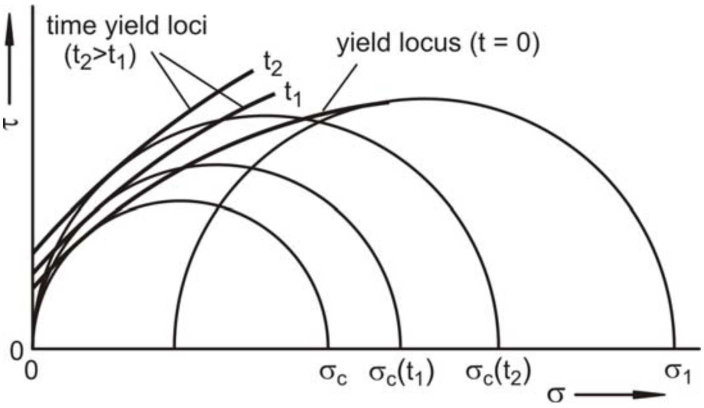

Figure 17: Yield locus and time yield loci

With the measured shear points a time yield locus can be approximated similarly to the approximation of a yield locus. Compared to the yield locus, the time yield locus is shifted towards greater shear stresses, τ (if the bulk solid shows an increase of strength with time). The unconfined yield strength, σ_c, is determined in the same way as for a yield locus by drawing a Mohr stress circle through the origin and tangent to the time yield locus. In Figure 17 the values of the unconfined yield strength for the consolidation periods, t_1 and t_2, are designated as $\sigma_c(t_1)$ and $\sigma_c(t_2)$.

Time yield loci can be determined for different storage periods (consolidation periods). Each time yield locus is valid for only one consolidation period and one consolidation stress. If the strength of the bulk solid increases over time, the time yield loci will be shifted towards larger values of τ as the consolidation period, t, increases (see Figure 17, $t_2 > t_1$)

5.6 Wall friction

Wall friction is the friction between a bulk solid and the surface of a solid, e.g. the wall of a silo or a bin. The coefficient of wall friction or the wall friction angle, respectively, is important both for silo design for flow and silo design for strength, but also for the design of chutes and other equipment, where the bulk solid will flow across a solid surface. Knowing the wall friction angle, it is possible to decide whether or not the polishing of the wall surface or the use of a liner would have advantages in the flow of the bulk solid.

The principle of a wall friction test, where the kinematic angle of wall friction is determined, is shown in Figure 18. The bulk solid specimen is subjected to a vertical normal stress. The normal stress acting between bulk solid specimen and wall material is called the wall normal stress, σ_w.

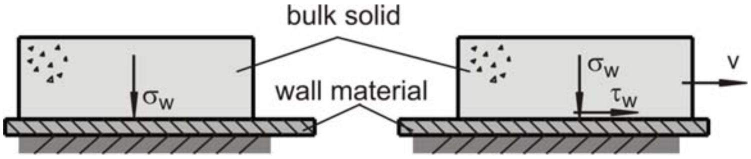

Figure 18: Principle of a wall friction test

The bulk solid specimen is then shifted relative to the wall material surface with a constant velocity, *v*. This process is called shear (similar to the yield locus test). The shear stress acting between bulk solid specimen and wall material is measured.

It is usual to measure wall friction at incrementally decreasing wall normal stresses [1,16]. Thus one begins with the greatest wall normal stress (σ_{w1} in Figure 19). At the beginning of the shear process the wall shear stress, τ_w, increases. With time, the increase of the wall shear stress becomes less until finally a constant wall shear stress, τ_{w1}, is attained (steady-state shear stress). The constant wall shear stress, τ_{w1}, is characteristic for the applied wall normal stress, σ_{w1}. After the steady-state condition is attained, the normal load is reduced. With each decrease in wall normal stress, wall shear stress, τ_w, also decreases (Figure 19). After a certain time, a steady-state shear stress is again attained. In this way values of steady-state wall friction at several wall normal stresses are measured.

The pair of values of wall normal stress and constant wall shear stress (σ_w, τ_w) describes the kinematic wall friction at the wall normal stress, σ_w, and is used for the evaluation of the test. All pairs of values of wall normal stress and steady-state wall shear stress are plotted in a σ_w,τ_w-diagram (Figure 19, right). The curve (or line) running through the measured points is called the wall yield locus.

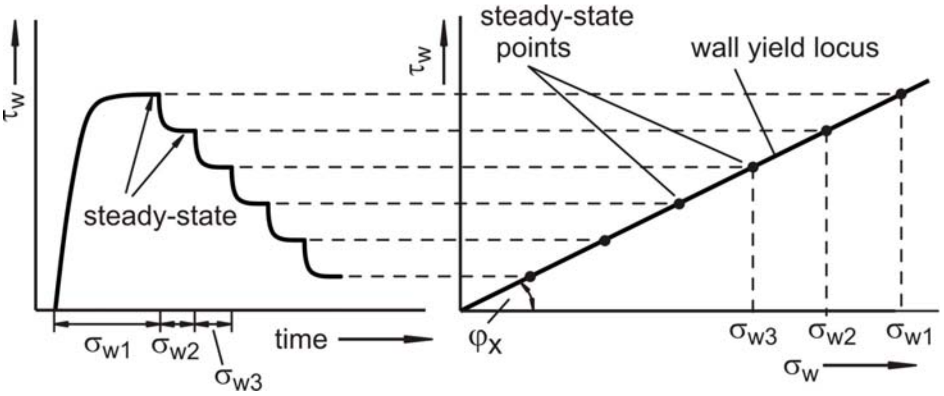

Figure 19: Course of wall shear stress in a wall friction test; wall yield locus

The wall yield locus is a yield limit like the yield locus. The wall yield locus describes the wall shear stress, τ_w, necessary to shift a bulk solid continuously across a wall surface under a certain wall normal stress, σ_w. Since the wall yield locus is based on the shear stresses measured at steady-state conditions, it describes the kinematic friction of the bulk solid. Thus the wall yield locus could more exactly be called a kinematic wall yield locus [16].

To quantify wall friction, the wall friction angle, φ_x, or the coefficient of wall friction, μ, are used. The larger the wall friction angle or coefficient of wall friction, the greater is wall friction. The coefficient of wall friction, μ, is the ratio of wall shear stress, τ_w, to wall normal stress, σ_w. The wall friction angle, φ_x, is the slope of a line running through the origin of the σ_w,τ_w-diagram and a point of the wall yield locus.

If the wall yield locus is a straight line running through the origin (Figure 19), the ratio of wall shear stress, τ_w, to wall normal stress, σ_w, has the same value for each point of the wall yield locus. Thus one obtains the identical wall friction coefficient, μ, and the identical wall friction angle, φ_x, for each point of the wall yield locus. In this case wall friction is independent of wall normal stress.

The wall yield locus shown in Figure 20 is curved and does not run through the origin. In this case one finds a different wall friction coefficient and wall friction angle for each point of the wall yield locus. Thus the wall friction coefficient and the wall friction angle are dependent on wall normal stress, σ_w. This can be seen by the wall friction angles, φ_{x1} and φ_{x2}, which follow for the wall normal stresses, σ_{w1} and σ_{w2}. A wall yield locus intersecting the τ-axis at $\tau_{ad} > 0$ is typical for materials tending to adhere at walls (e.g. like moist clay). The shear stress, τ_{ad}, at the point of intersection is called adhesion.

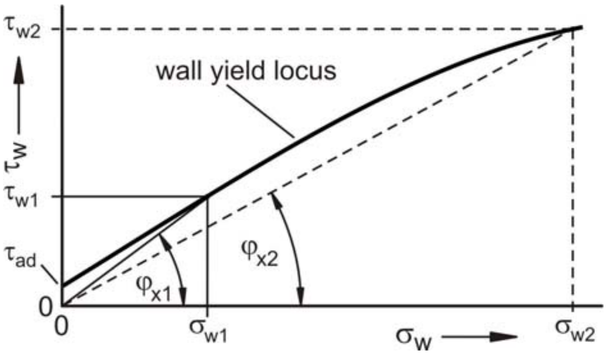

Figure 20: Wall yield locus; wall friction angle is dependent on the wall normal stress.

Wall friction can be measured with the shear testers described above. The setup of the Jenike shear tester for a wall friction test is shown in Figure 21. The bottom ring of the shear cell is replaced by a sample of wall material (e.g. stainless steel, coated steel). The wall normal stress is then adjusted by the normal force, F_N, and the shear force, F_S, is measured following the procedure outlined above.

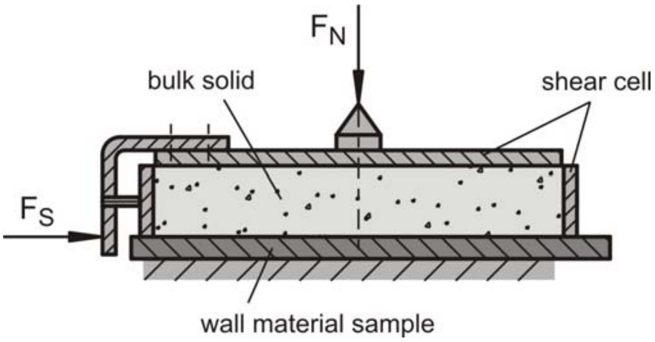

Figure 21: Measurement of wall friction with the Jenike shear tester [1]

Figure 22 shows the setup of the wall friction shear cell of the Schulze ring shear tester [2,9,10,17]. The annular bottom ring contains the sample of the wall material. On top of the wall material sample is the bulk solid specimen, which is covered with the annular lid of the shear cell. The lid is connected to the crossbeam. Except for the geometry of bottom ring and lid, the setup is similar to the setup of the shear cell for flow properties testing (Section 5.3).

To measure wall friction the shear cell is rotated slowly in the direction of arrow ω, while the lid is prevented from rotating by the two tie rods. The forces acting on the tie rods, F_1 and F_2, are measured. The layer of bulk solid located between the lid and the surface of the wall material sample is prevented from rotating by the lid, which has a rough underside. Thus the bulk solid is shifted across the surface of the wall material sample while it is subjected to the normal stress, σ_w. The wall shear stress, τ_w, is calculated from the F_1 and F_2.

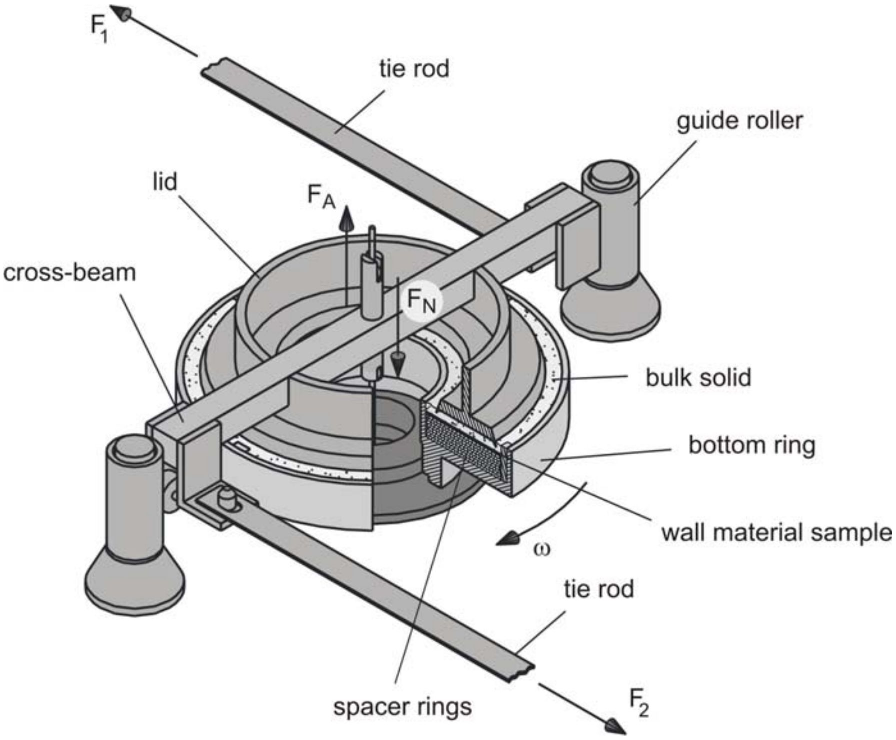

Figure 22: Setup of the shear cell for wall friction tests (Schulze ring shear tester) [2,9,10,17]

6 Further measurement methods and devices for the determination of bulk solid properties

Several empirical methods are used for the assessment of bulk solid properties, e.g. the determination of the angle of repose, α_M (Figure 23). Different results are obtained even with this simple test procedure: So a conical heap (a) will yield a different angle of repose α_M compared to a wedge-shaped heap. If the bulk solid flows out of a container with a central outlet (b), then the angle of repose α_M will be even higher. In a rotating drum another (smaller) angle of repose will prevail.

Examples of further simple test methods are (overview see [2,12,13,20,21]):

- Determination of the time required for a sample of bulk solid to flow out of a model silo.
- Determination of the compressibility by measuring loose density and tap density (Hausner ratio).

- The Carr index determined from compressibility, different angles of repose, and a particle size distribution [19].

The results of these so-called simple tests depend on particular boundary conditions (e.g. in the case of determining the angle of repose, the conditions at the free surface of the heap) which give no information about the behaviour of a bulk solid under the stresses which are present in a typical application, e.g. in a container or in a hopper.

- The results depend on the test devices, therefore the physical figures are not device-independent.
- The influences which are important for the design of silos (storage time, stress level) are not included or only measured qualitatively.
- Many of the simple test methods cannot reasonably be applied to cohesive bulk solids with poor flowability (flour, zinc oxide).
- The preparation of the bulk solid influences the measured results (e.g. the thorough stirring of bulk solids which easily fluidise leads to smaller angles of repose).

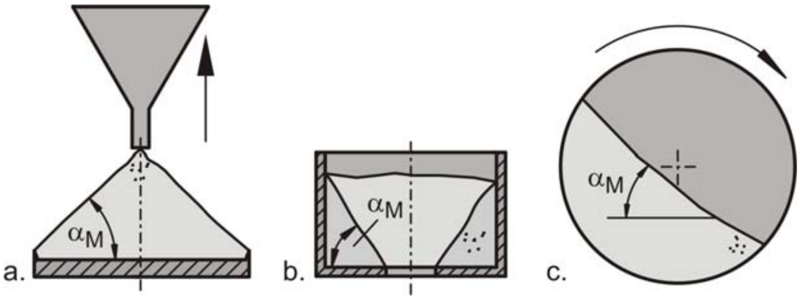

Figure 23: Methods to measure the angle of repose

Because of the weaknesses in these simple test methods mentioned above, test devices which allow the defined preparation of the sample of bulk solid must be preferred (e.g. preshear until the onset of steady state flow as a defined consolidation procedure). Only this way it is possible to determine the relevant flow properties (unconfined yield strength, internal friction angle, wall friction angle, bulk density, time consolidation dependent on the consolidation stress) independent from the devices used [12,13].

7 Summary

Consolidated bulk solids have yield limits like other materials. These yield limits, which can be measured with shear testers, are called yield loci. They depend on the consolidation which has previously taken place and, sometimes, also on the storage time. If a bulk solid is to be set in motion, e.g. to flow out of a silo, the stresses acting on the bulk solid must be large enough to ensure that the corresponding Mohr stress circle touches the yield locus of the consolidated bulk solid.

The flow properties which can be obtained from the measured yield loci are exactly defined physical figures. Unconfined yield strength (dependent on consolidation stress and storage time), bulk density, and wall friction angle are the most important flow properties for the design of storage and conveying systems as well as for comparative tests, quality control, and product development.

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