Review

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Review on testers for measuring flow properties of bulk solids

(based on an IFPRI-Report 1999)

Jörg Schwedes

Abstract The author was asked by the International Fine Particle Research Institue (IFPRI) to write a critical review on shear testers for IFPRI-members. The review was delivered to IFPRI in summer 1999. Following the contract with IFPRI it was not allowed to publish the review elsewhere for at least two years. Granular Matter invited the author to submit the review in the original form; during the refereeing process (besides other changes) it turned out that some remarks should be added here for clarification: - Since 1999, the author is not aware of a really new device for testing bulk solid properties, which could lead to a change of the general comments and conclusions provided in the review.

– It was argued, that the review is referring too much to the work of Jenike, while the works of Johanson and Peschl were not adequately cited. Both are excellent engineers with a lot of experience, but their basic ideas are not available in published form, and if, they are not set in relation to alternative approaches so that an objective comparison in detail would be a future research issue rather than a topic in this report.

– A discussion on the influence of electrostatic charges was missing. There hardly is an influence, since the particles are in continuous contact. Only with non-conducting plastic particles electrostatic charges could cause problems. But no relevant experiments and results are known. In closed systems, the effect of electric charges is thus mainly neglected, but it is clear that electrostatic forces are eminent in flows with a free surface – an issue not addressed in this review.

Keywords Shear tester, Powder, Bulk solid, Flowability, Flow function, Silo, Friction, Flow properties

1 Introduction

Many ideas, methods and testers exist to measure the flowability of bulk solids. The primary intent is the characterization of bulk solid's flow properties, but most often the measured data are used to design equipment for storage, transportation or general handling of bulk solids. Flowability testing is also needed to compare the flowability of similar or competing bulk solids, to determine whether a product fulfills the requirement of quality control, to model processes with the finite element method or to judge any other process in which the strength or flowability of bulk solids plays an important role. Many testers are available which measure some value of flowability, only some of these shall be mentioned in this introduction: Jenike's shear cell, annular shear cells, triaxial tester, true biaxial shear tester, Johanson Indicizers, torsional cell, uniaxial tester, Oedometer, Lambdameter, Jenike and Johanson's Quality Control Tester, Hosokawa tester and others.

The scope of this report is as follows: First, flow properties shall be defined. Secondly, applications are mentioned. Here the properties which are needed for design are described. In the main chapter the known testers for flow properties measurement are listed and described. Following, the suitability of these testers to actually measure the required properties is discussed. Finally, a comparison with regard to applications will be made. It is beyond the scope of this report to describe all testers very detailed or to compare all of them one by one.

2 Flow function

The flow function was first introduced by Jenike and measured with help of Jenike's shear cell [1]. Therefore, a short explanation of the flow function and the relevant measurement procedure will be given here. A very detailed description was given by the Working Party on the Mechanics of Particulate Solids within the European Federation of Chemical Engineering [2] after running comparative shear tests with the same fine calcite powder in more than 20 laboratories around the world and discussing the results [30]. For all who have to run shear tests it is strongly recommended to start with that report on the "Standard Shear Testing Technique" [2].

2.1 Jenike's shear tester

The most important part of Jenike's shear tester is the shear cell (Fig. 1). It consists of a base (A), a ring (B) resting on top of the base and a lid (C). Base and ring are filled with a sample of the bulk solid. The proce-

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Jörg Schwedes
Institut für Mechanische Verfahrenstechnik,
TU Braunschweig, Volkmaroder, Straße 4/5,
D-38104 Braunschweig, Germany
e-mail: j.schwedes@tu-bs.de

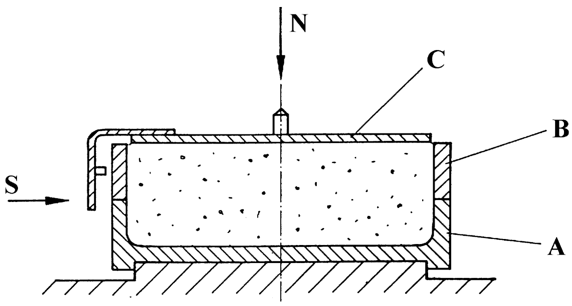

Fig. 1. Jenike's shear cell (A: base, B: ring; C: lid)

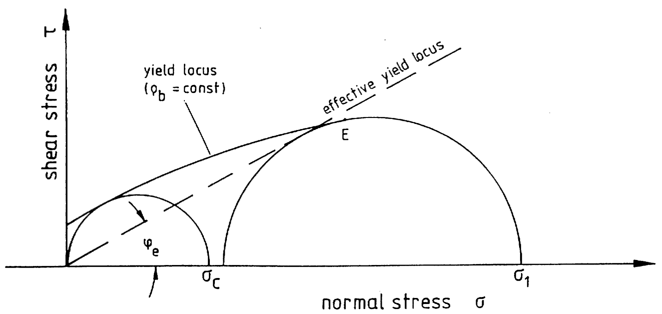

Fig. 2. Yield locus and effective yield locus

dure to fill the ring and to preconsolidate the sample in a reproducible manner is described in many papers, besides others in the report mentioned above [2]. A vertical force is applied to the lid. A horizontal shearing force is applied on a bracket attached to the lid. Running shear tests with identically preconsolidated samples under different normal loads results in maximum shear forces S for every normal load N. Division of N and S by the cross-sectional area of the shear cell leads to the normal stress σ and the shear stress τ, respectively. Figure 2 shows a σ, τ-diagram. The curve represents the maximum shear stress τ the sample can support under a certain normal stress σ and is called the yield locus. The parameter of a yield locus is the bulk density ρ_b at preconsolidation.

With higher preconsolidation loads, the bulk density ρ_b increases and the yield loci move upwards. Each yield locus terminates at point E in direction of increasing normal stresses σ. Point E characterizes the steady state flow, which is the flow with no change in stresses and bulk density. Two Mohr stress circles are shown. The major principal stresses of the two Mohr stress circles are characteristic of a yield locus, σ_1 is the major principal stress at steady state flow, called major consolidation stress, and σ_c is the unconfined yield strength. Each yield locus gives one pair of values of the unconfined yield strength σ_c and the major consolidation stress σ_1. Plotting σ_c versus σ_1 leads to the flow function (see later, Fig. 9). The angle φ_e between the σ-axis and the tangent to the larger Mohr circle, called effective yield locus, is a measure for the inner friction at steady state flow and is very important in the design of silos for flow.

To get the yield locus of Fig. 2, the bulk solid sample is sheared in two steps [1–4]: In the first, often called "preshear", the sample is sheared under a normal stress σ_{sf} until steady state flow with $\tau = \tau_{sf} =$ const. prevails, thus leading to the larger Mohr stress circle in Fig. 2. After steady state flow has been obtained, which is indicated by a constant shear stress τ_{sf}, the shear stress is reduced to zero, the normal stress is reduced to $\sigma < \sigma_{sf}$ and the second step of the shear process, often called "shear", is performed by applying again a shear force on the bracket (attached to the lid C in Fig. 1). The maximum in the shear stress, shear strain-path gives one value of the yield locus. To get further points the procedure has to be repeated with new samples, the same normal stress σ_{sf} during "preshear", but with different normal stresses $\sigma < \sigma_{sf}$ during "shear". The procedure is shown schematically in Fig. 3. To get further yield loci the normal force during preconsolidation and "preshear" has to be increased or decreased and the procedure has to be repeated.

2.2 Uniaxial Test

Very often a theoretical experiment is used to show the relationship between σ_1 and σ_c, Fig. 4. A sample is filled into a cylinder with frictionless walls and is consolidated under a normal stress $\sigma_{1,c}$ leading to a bulk density ρ_b. After removing the cylinder, the sample is loaded with an increasing normal stress up to the point of failure. The stress at failure is the unconfined yield strength σ_c. Contrary to results of shear tests steady state flow cannot be reached during consolidation, that is, the Mohr stress circle will be smaller, Fig. 5 [3,4]. As a result, bulk density ρ_b and unconfined yield strength σ_c will also be smaller compared to the yield locus gained with shear tests [4,5].

2.3 Biaxial shear tester

A tester in which both methods of consolidation, either steady state flow (Figs. 1 to 3) or uniaxial compression (Fig. 4), can be realized in the biaxial shear tester [5–9] (Fig. 6). The sample is constrained in lateral x- and y-direction by four steel plates. Vertical deformations of the sample are restricted by rigid top and bottom plates. The sample can be loaded by the four lateral plates which are linked by guides so that the horizontal cross-section of the sample may take different rectangular shapes. In deforming the sample, the stresses σ_x and σ_y can be applied independently of each other in x- and y-directions. To avoid friction between the plates and the sample the plates are covered with a thin rubber membrane. Silicon grease is applied between the steel plates and the rubber membrane. Since there are no shear stresses on the boundary surfaces of the sample, σ_x and σ_y are principal stresses. With the biaxial shear tester the measurement of both stresses and strains is possible.

With the biaxial shear tester experiments were carried out to investigate the influence of the stress history and the influence of different consolidation procedures on the unconfined yield strength [5,6,8,9]. In order to obtain a yield locus corresponding to Fig. 2, the minor principal stress σ_2 in the y-direction (Fig. 6) is kept constant during a test (Fig. 7). In x-direction a positive strain rate $\dot{\varepsilon}_1$ is applied resulting in an increasing major principal stress

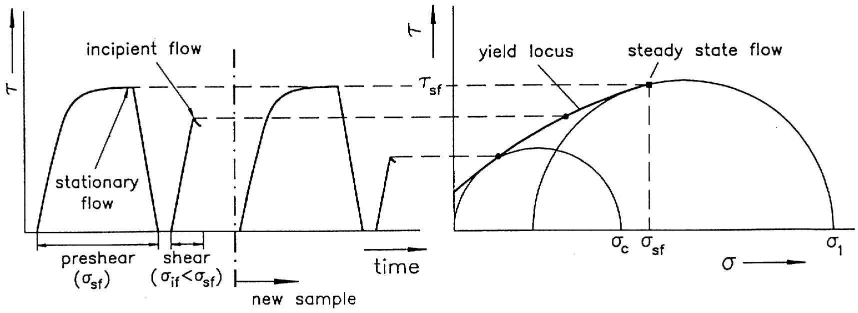

Fig. 3. Procedure for Jenike's shear tester to get a yield locus

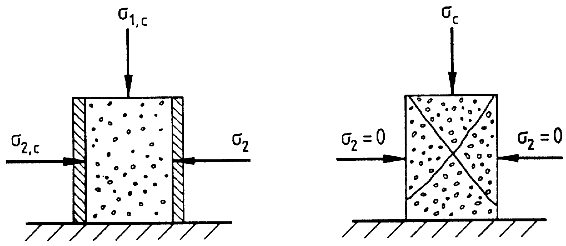

Fig. 4. Unconfined yield strength σ_c after uniaxial consolidation $\sigma_{1,c}$

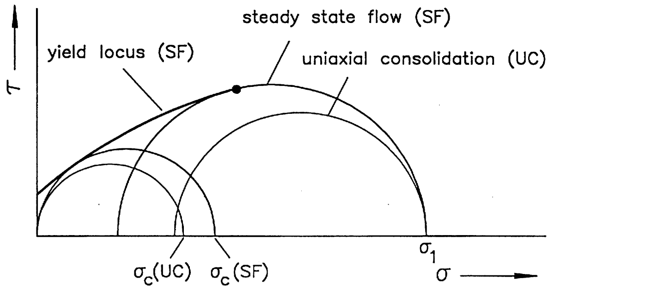

Fig. 5. Mohr stress circles for steady state flow (SF) and uniaxial consolidation (UC)

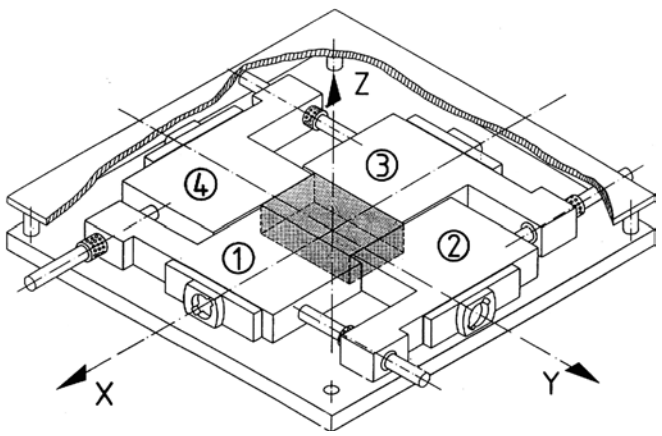

Fig. 6. True biaxial shear tester

σ_1. σ_1 is increased continuously up to the point of steady state flow with constant values of σ_1, σ_2 and ρ_b. After this "preshear" the state of stress is reduced to a smaller σ_2 value ($\sigma_2 = \sigma_{2,b}$ in Fig. 8) and the second step of the shear process is performed by applying again a positive strain rate $\dot{\varepsilon}_1$ in x-direction resulting in increasing $\sigma_{1,b}$-

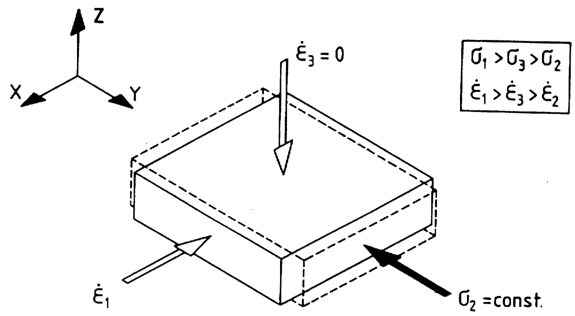

Fig. 7. Shear of a sample at constant σ_2 in the biaxial shear tester

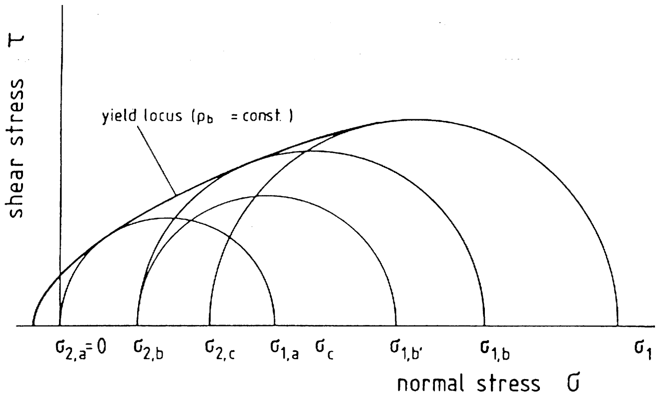

Fig. 8. Yield locus gained with the biaxial shear tester

values. $\sigma_{2,b}$ and the maximum value of $\sigma_{1,b}$ define the Mohr stress circle of failure, thus belonging to the yield locus. Furthermore, Mohr stress circles of failure can be obtained by repeating the procedure with identical values of σ_2 and σ_1 during "preshear" and "shear" with different $\sigma_{2,b} < \sigma_2$. By setting $\sigma_{2,b} = 0$, the unconfined yield strength σ_c can be measured directly (Fig. 8).

Comparative tests with the same fine limestone powder were performed with Jenike's shear tester and the biaxial shear tester, following the described procedures with "preshear" and "shear". In Fig. 9 the flow function is given, which is the plot of unconfined yield strength σ_c versus major consolidating stress σ_1 at steady state flow. Although two different kinds of shear testers were used, the measurements are in agreement [5].

For investigation on the influence of different consolidating procedures, in analogy with the uniaxial test of

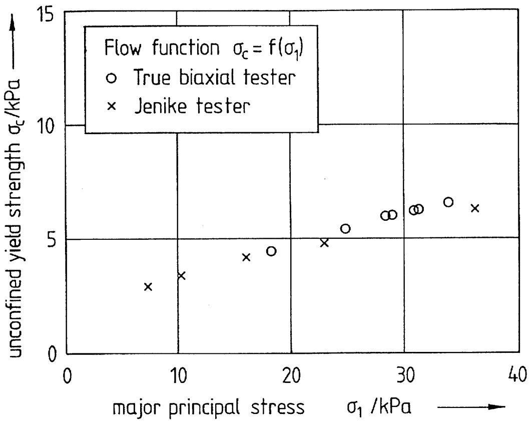

Fig. 9. Flow function; unconfined yield strength σ_c versus major principal stress at steady state flow σ_1 (limestone: $x_{50} = 4.8\ \mu m$)

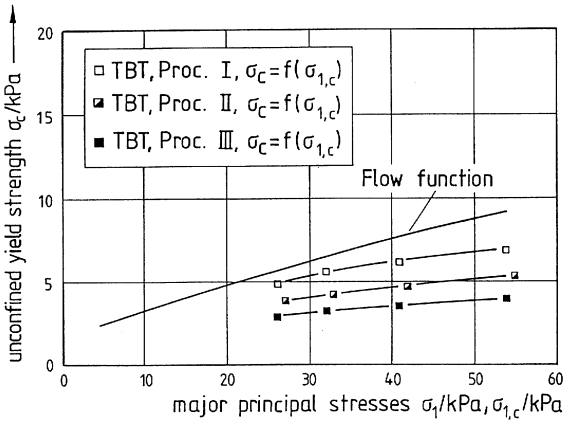

Fig. 11. Flow function from Fig. 9 and unconfined yield strength σ_c versus major principal stress at consolidation $\sigma_{1,c}$

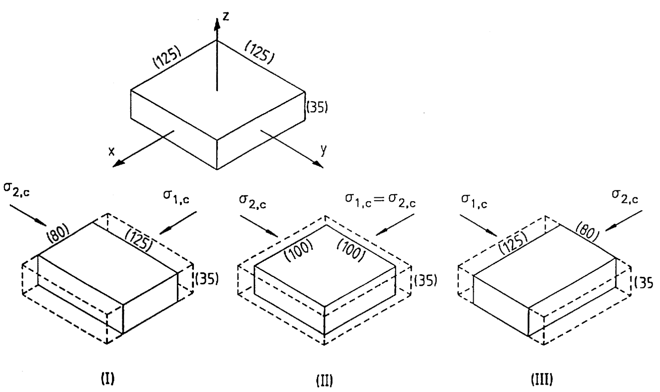

Fig. 10. Sample consolidation in the biaxial shear tester

Fig. 4, samples were consolidated in the biaxial shear tester from a low bulk density to a selected higher bulk density before the shear test was started. The higher bulk density ρ_b could be obtained in different ways. Figure 10 demonstrates three different possibilities (I, II, III) of consolidating the sample in order to get the same sample volume and, hence, the same bulk density. In the case of procedure I the x-axis and in the case of procedure III the y-axis coincide with the direction of the major principal stress $\sigma_{1,c}$ at consolidation. In the case of procedure II, the major principal stress $\sigma_{1,c}$ is acting in both directions. After consolidation the samples were sheared in the following way: σ_2 in y-direction was kept constant at $\sigma_2 = 0$ and σ_1 was increased up to the point of failure, leading to the unconfined yield strength. The results are plotted in Fig. 11 as σ_c versus $\sigma_{1,c}$, with $\sigma_{1,c}$ being the major principal stress at consolidation. The functions $\sigma_c = f(\sigma_{1,c})$ corresponding to procedures I, II and III are below the flow function $\sigma_c = f(\sigma_1)$, being identical with the results already shown in Fig. 9.

It can be clearly seen that the three functions $\sigma_c = f(\sigma_{1,c})$ underestimate the unconfined yield strength σ_c which a bulk solid sample gains after steady state flow (flow function). Procedure I is identical to the procedure given in Fig. 4, realized in uniaxial testers [10,11], see also chapter 4.5. As described above in Fig. 5 the unconfined strength after uniaxial consolidation is smaller than that achieved by the consolidation with steady state flow. Figure 11 clearly confirms this finding. The function $\sigma_c = f(\sigma_{1,c})$ of procedure II leads to an even smaller unconfined yield strength. Steady state flow, uniaxial consolidation (procedure I) and the consolidation procedure II thus produce different strengths although identical major principal stresses σ_1 were used during consolidation and ρ_b was the same. Consolidation procedures I and III are basically identical. Only the direction of measuring the unconfined yield strength is different. Since the results deviate strongly, a remarkable anisotropic effect exists. Thus, the strength of a bulk solid sample depends on its "stress history" and even, if this stress history is identical, it can show anisotropic behaviour. Since both effects are important to understand results of shear tests and of all other flow properties testers, both effects will be discussed in the following chapters in more detail.

2.4
Influence of stress history

The discussion of the results presented in Fig. 11 has clearly shown that a strong influence of the stress history exists, i.e. the method or procedure how a bulk solid sample has been consolidated to a definite state. To demonstrate this influence further results of tests with the biaxial shear tester will be presented [7,8,12–14]. The way of conducting experiments in the biaxial shear tester ensures that no shear strains or shear stresses can develop on x-, y- and z-planes. Therefore, all measured normal stresses and normal strains must be principal stresses and principal strains. This means that the experimental results can be fully described in the principal space. The line linking all measured states in the principal strain space is referred to as "strain path". Similarly, a "stress path" is defined in the principal stress space. As the biaxial shear tester allows deformations in the x-y-plane only, the strain path must lie completely in the $\varepsilon_y - \varepsilon_z$-plane. The stress path, however, is a general three dimensional curve in the $\sigma_x - \sigma_y - \sigma_z$-plane. Nevertheless, for convenience the diagrams will be limited to two dimensions.

Experiments with a linear strain path belong to the simplest deformations possible in the biaxial shear tester.

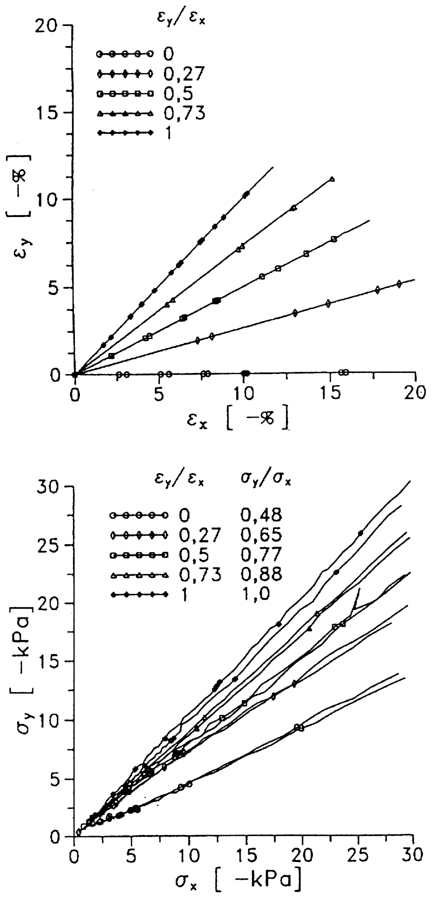

Fig. 12. Consolidation under proportional deformation

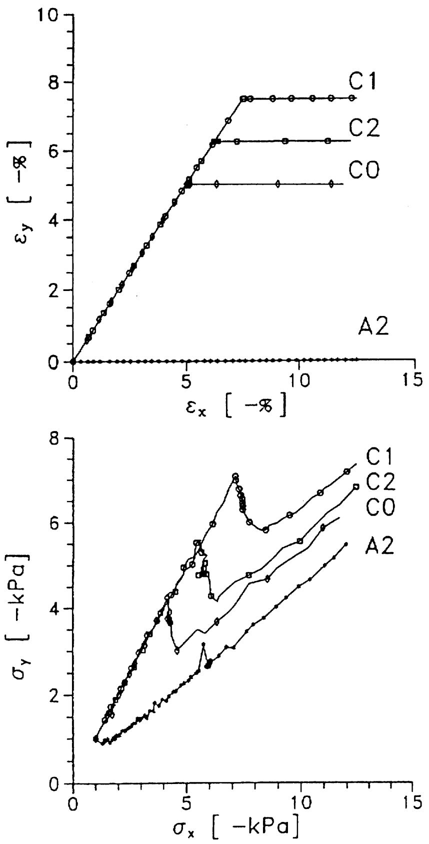

Fig. 13. Asymptotic behaviour

The experiment starts with $\varepsilon = 0$ and progresses with a constant deformation rate in x- and y-directions. As the ratio of $\dot{\varepsilon}_{\mathrm{y}}/\dot{\varepsilon}_{\mathrm{x}}$ remains constant, these strain paths are referred to as proportional strain paths. For proportional strain paths the ratio $\dot{\varepsilon}_{\mathrm{y}}/\dot{\varepsilon}_{\mathrm{x}}$ equals the ratio $\varepsilon_{\mathrm{y}}/\varepsilon_{\mathrm{x}}$, therefore, in Fig. 12 the $\varepsilon_{\mathrm{y}}/\varepsilon_{\mathrm{x}}$-ratio is used to characterize the depicted strain paths. The resulting stress path is also linear and proportional. For each strain path the measured stress paths from two independent experiments are shown. One can clearly see that a distinct direction of the stress path can be correlated to each direction of the strain path. These paths are therefore called "associated stress and strain path", their directions "associated directions".

In Fig. 13 the results from experiments are shown, in which the strain path shows a sharp bend. In the first part of the experiment the sample was consolidated with $\varepsilon_{\mathrm{y}}/\varepsilon_{\mathrm{x}} = 1.0$. After 10 to 15% of volume change the y-plates where stopped and deformation commenced only in the x-direction (curves C0... C2). The measured stress paths are made up of two parts as well. Initially the stress paths follow the associated direction already known from Fig. 12. After the change in the respective strain path, however, σ_{y} decreases rapidly and the stress paths finally evolve parallel to the stress path A2, which is associated to the strain path with $\varepsilon_{\mathrm{y}}/\varepsilon_{\mathrm{x}} = 0$. The ordering of the stress paths C0... C2 with respect to A2 follows the amount of volume change during the first part of the strain path. This behaviour is called "asymptotic behaviour".

An experiment leading to steady state flow has to guarantee a deformation at constant volume. In the biaxial shear tester this requires $\dot{\varepsilon}_{\mathrm{y}}/\dot{\varepsilon}_{\mathrm{x}} = \varepsilon_{\mathrm{y}}/\varepsilon_{\mathrm{x}} = -1$, and this deformation is called "pure shearing". To perform a pure shearing test, the sample always has to be consolidated before it can be subjected to pure shearing. Figure 14 shows the stress and strain paths of six experiments with pure shear loading. Starting from the same initial bulk density the samples have been subjected to consolidation up to two stress levels under three initial strain path directions. One can see that the stress paths initially follow the directions known from Fig. 12. As soon as the shear part of the strain path begins the stresses decrease down to some stationary level. Subsequent shearing results in scatter around this point but no significant change in stress.

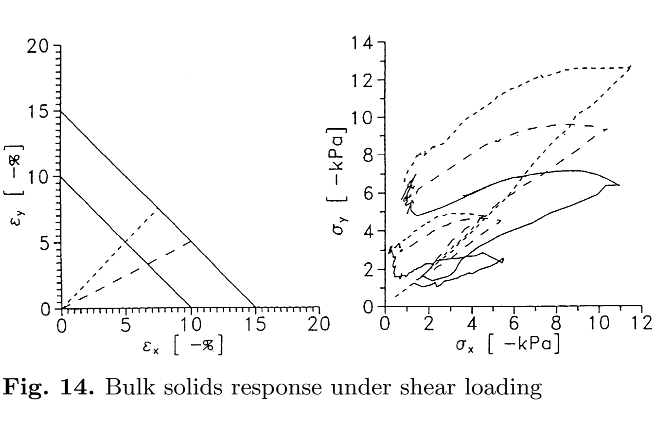

Fig. 14. Bulk solids response under shear loading

This behaviour – deformation without change in volume and stress – is considered steady state flow. From Fig. 14 one can also see the influence of the loading history. While six different consolidation paths have been used, only two levels of steady state flow arise. Stress paths, which belong to strain paths with the same level of volumetric strain, end at the same steady state stress level. Further experiments have shown that all steady state stresses lie on a line through the origin, the "critical state line".

In steady state flow the sample deforms at constant bulk density and constant stresses. As can be seen in Fig. 14, the final state (stresses σ_x and σ_y) is only dependent on the bulk density and is independent of the stress history, i.e. independent of the strain and stress paths. Thus, in steady state flow the sample has lost its memory of the stress history, whereas in all other stress states prior to steady state flow the state of stress depends on the stress and/or strain history.

2.5 Anisotropy

When discussing the consolidation procedures I and III and their influences on the unconfined yield strength in Figs. 10 and 11, it became clear that the yield strength of all samples being consolidated to the same bulk density depends on the direction of stress application. If the direction of the major consolidation stress $\sigma_{1,c}$ is identical to the direction of stress application during the subsequent failure experiment (as in the uniaxial test of Fig. 4), the strength is highest. Figure 14 shows that a sample is loosing its memory of the stress history in steady state flow. However, this does not imply that it behaves isotropically in the following failure test, and typically the anisotropic behaviour after steady state flow is most distinct. This can be explained with the stress ratio of minor to major principal stress σ_2/σ_1 being smallest after steady state flow consolidation (see Fig. 5). Only when all three principal stresses are identical (iso-or hydrostatic consolidation), an isotropic behaviour might be expected. But isostatic consolidation is a rare event in industrial applications.

First Molerus [15] and later Saraber et al. [16] reported on the anisotropic behaviour of cohesive bulk solids. For their investigations Saraber et al. used a Jenike shear tester and a biaxial tester. Following the procedure explained in chapters 2.1 and 2.3 they performed shear tests in two steps, "preshear" to steady state flow and subsequent "shear" to failure. Thus, they got yield loci. In addition to the normal procedure they performed tests, in which after "preshear" they rotated the Jenike shear cell by angles up to 180° before shearing the sample up to failure. In Fig. 15 the measured unconfined yield strength is plotted versus the angle of rotation α. There is little influence for angles $\alpha < 30°$ but for higher angles the strength is decreasing significantly. Both effects – the small influence at small angles and the significant one at higher angles – are important for the application of measured properties (see chapter 3.1). In the tests of Saraber et al. [16] with the biaxial tester only angles of $\alpha = 0$ and $\alpha = 90°$ could be realized. The results confirm the findings of Fig. 15: If the major principal stress during "shear" is perpendicular ($\alpha = 90°$) to the one at "preshear" (steady state flow), the unconfined yield strength is significantly smaller compared to the procedure where both directions coincide ($\alpha = 0°$).

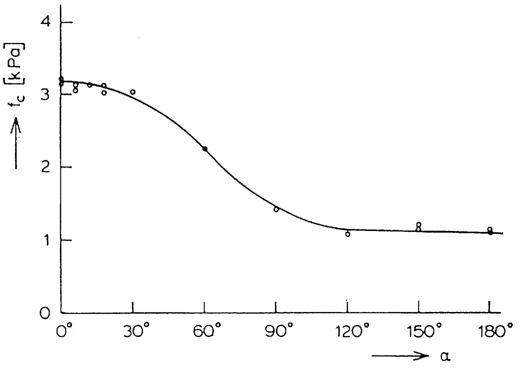

Fig. 15. Unconfined yield strength σ_c as a function of α (Jenike's shear tester) [16]

2.6 Conclusions

The reported measuring procedures for Jenike's shear tester argued that "preshear" up to steady state flow has to be performed as a check for the correct preconsolidation [2]. This preconsolidation is supported by a twisting motion of the cover of the shear cell under a normal stress σ. Thus, shear stresses are induced in the sample acting tangentially. These combined normal and shear stresses might locally lead to steady state flow, but the complete cross-section of the shear cell is not in an identical steady state flow condition, identical with respect to direction. This is because the directions of the major principal stresses during the twisting motion are different at different positions within the cell. Also it is not sure if steady state flow really is obtained at all positions. Thus, the first step of a shear test - "preshear" to steady state flow – is not a check for a correct preconsolidation, but a necessary step to get steady state flow across the complete cross-section and in the same direction. Only this "preshear" guarantees a reproducible and a clearly defined consolidation where all influences of the stress history are eliminated.

The flow function (relationship unconfined yield strength σ_c and major consolidation stress σ_1 at steady state flow) can only be determined with testers in which both stress states can be realized and both stresses σ_1 and σ_c act in the same direction – at least nearly. Steady state flow can be achieved in Jenike's tester, in ring shear testers, in a torsional shear cell, in biaxial shear testers and in a very specialized triaxial cell [5]. The unconfined yield strength σ_c can be determined by running tests in Jenike's tester, in ring shear testers, in uniaxial testers and in biaxial shear testers. Therefore, only Jenike's tester, ring shear testers and biaxial shear testers can guarantee the measurement of flow functions $\sigma_c = f(\sigma_1)$ without further assumptions. All other procedures to get a dependence of the unconfined yield strength σ_c on the major principal stress at consolidation $\sigma_{1,c}$ (without reaching steady state flow) lead to smaller unconfined yield strengths. Those relationships can only be used as estimates of the flow function. But, when using these estimates for some applications (i.e. design of silos for flow, chapter 3.1) it has to be kept in mind that the predictions for the bulk solids behaviour are on the "unsafe side" (Figs. 11 and 15).

3 Application of measured flow properties

In the following it will be shown which flow properties have to be known for special applications and which testers are suited to measure these properties.

3.1 Design of silos for flow

The best known and the most applied method to design silos for flow is the method developed by Jenike [17]. He distinguished two flow patterns, mass flow and funnel flow, the border lines of which depend on the inclination of the hopper, the angle φ_e of the effective yield locus (Fig. 2) and the wall friction angle φ_w between the bulk solid and the hopper wall. The angle φ_e can only be measured if steady state flow is achieved in the tester. The wall friction angle φ_w can easily be tested with Jenike's shear tester, but also with other direct shear testers (also see chapter 4.7).

The most severe problems in the design of silos for flow are doming and piping. Jenike's procedure to avoid doming starts from steady state flow in the outlet area. After stopping the flow (outlet closed) and restarting it the flow criterion for doming can only be applied, if the flow function is known. As stated before, the flow function can only be measured without further assumptions with Jenike's shear tester, ring shear testers or biaxial shear testers. Biaxial shear testers are very complicated and cannot be proposed in its present form for application in the design of silos for flow.

Some bulk solids gain strength, when stored under pressure without movement (see also chapter 4.8). Principally this time consolidation can be tested with all testers. Time consolidation can most easily – with regard to time and equipment – be tested with Jenike's shear tester and a new version of a ring shear tester [18]. Again, only these testers measure the time flow functions which have to be known for applying the doming and piping criteria.

Piping can occur directly after filling the silo or after a longer period of satisfactory flow, for example due to time consolidation. In the latter case, flow function and time flow functions have to be known to apply the flow-no flow criteria. In the first case, the stresses in the silo after filling have to be known, and these are different from those during flow.

The anisotropic behaviour of bulk solids mentioned in chapter 2.5 is of no influence in the design of silos for flow. With the help of Figs. 6 to 9 it was explained that steady state flow was achieved with σ_1 (at steady state flow) acting in x-direction. The unconfined yield strength was also measured with the major principal stress acting in x-direction. During steady state flow in a hopper, the major principal stress is horizontal in the hopper axis. In a stable dome above the outlet, the unconfined yield strength

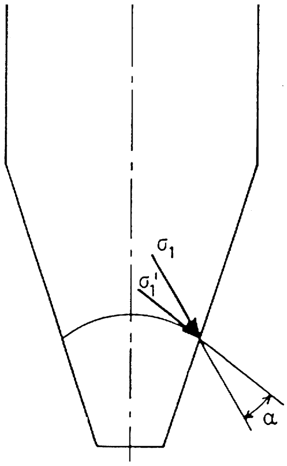

Fig. 16. Possible directions of σ_1 during steady state flow and σ_1' at doming in a hopper [16]

also acts horizontally in the hopper axis, i.e. in the same direction as the major principal stress during steady state flow. Directly at the wall the inclination of the major principal stress σ_1 during steady state flow differs by only a small angle α from the inclination of the surface supporting a stable dome of bulk solid with the unconfined yield strength σ_c, Fig. 16 [16]. Following the discussion of Fig. 15 on the influence of this angle on the strength σ_c it can be concluded that the flow function reflects reality in the hopper area since in Fig. 16 $\alpha < 30°$.

3.2 Design of silos for strength

For the structural design of silos, the stresses acting between the stored bulk solid and the silo walls have to be known. Since 1895 Janssen's equation has been used to calculate stresses in the bin section. Janssen derived his equation by considering the force equilibrium on a slice element of thickness dz (Fig. 17). His equation

$$\sigma_v = \frac{g \cdot \rho_b \cdot A}{\lambda \cdot \mu \cdot U} \cdot \left(1 - e^{-\frac{\lambda \cdot \mu \cdot U}{A} \cdot z}\right)$$

$$\sigma_{v,\max} = \frac{g \cdot \rho_b \cdot A}{\lambda \cdot \mu \cdot U}$$

is still the basis for many national and international codes and recommendations [19]. The equation contains geome-

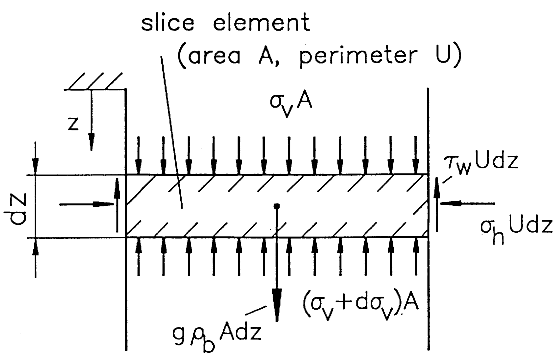

Fig. 17. Force equilibrium on a slice element due to Janssen

trical terms (cross-section A, perimeter U), the gravity constant g, the bulk solid density ρ_b, the coefficient of wall friction $\mu = \tan\varphi_w$ and the horizontal stress ratio λ (= k in the English literature):

$$\lambda = \frac{\sigma_h}{\sigma_v}$$

For ρ_b the maximum possible bulk solid density being a function of the largest σ_1 value in the silo has to be used. The coefficient of wall friction μ can be measured with shear testers, if the tests are carried out at the appropriate stress level and if the results are correctly interpreted [20]. It should be mentioned that the value of the angle used for the mass flow-funnel flow decision is generally not identical with the one needed in the design of silos for strength (also see chapter 4.7).

It is far more difficult to get reliable values for the parameter λ. In Janssen's equation and all following applications, λ is defined as the ratio of the horizontal stress σ_h at the silo wall to the mean vertical stress σ_v. Thus, a locally acting stress is related to the mean value of all stresses acting on a cross-section, or two stresses acting on different areas are related. In research work and codes, several different instructions to calculate λ are suggested. From the large number of different recommendations, it can be seen that there is still an uncertainty in calculating λ.

A step forward to a reliable determination of λ is the recommendation by the scheduled Euro code [21] to measure λ in an uniaxial compression test, using a modified Oedometer. An Oedometer is a standard tester in soil mechanics to measure the settling behaviour of a soil under a vertical stress σ_v. Such a modified Oedometer, called Lambdameter, was proposed by Kwade et al. [22] (Fig. 18). The horizontal stress σ_h can be measured with the help of strain gauges, lined over the entire perimeter of the ring. For further details see [22]. A large number of tests were performed to investigate influences such as filling procedures, influence of side wall friction, influence of friction at lid and bottom, duration of the test, minimum stress level and others. Forty-one bulk solids having angles φ_e of the effective yield locus between 20 and 57° were tested in the Lambdameter. The results are summarized in Fig. 19, where λ is plotted versus φ_e. For comparison, the proposals by Koenen and Kezdy and the recommendation of the German code DIN 1055, part 6, are plotted in the graph. It can be concluded that none of the three is in line with the measured values and that especially with high values of φ_e great differences exist between the measured and the recommended λ values.

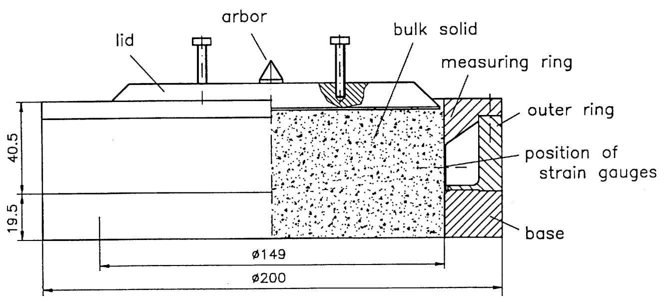

Fig. 18. Lambdameter

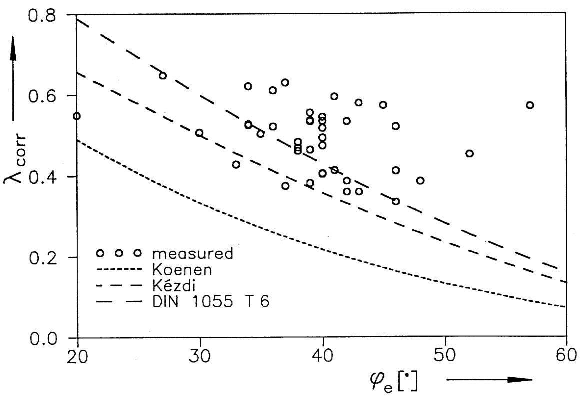

Fig. 19. Horizontal stress ratio λ versus angle φ_e of effective yield locus (41 bulk solids)

The described problem in getting reliable λ values for design results from the fact that no simple, theoretical model exists which combines known bulk solid properties such as φ_e, φ_w or others with application in a satisfactory manner. As long as this relationship is not known, the direct measurement in a special designed tester like the Lambdameter is the best solution.

3.3 Calibration of constitutive models

Eibl and others have shown that the Finite Element Method can be used with success to model stresses in silos [23]. To apply this method, a constitutive model has to be used. The models of Lade [24] and Kolymbas [25] may be mentioned as examples. Each constitutive model contains parameters which have to be identified from calibration tests. The most important requirement for this calibration test is that the complete state of stress and the complete state of strain can be measured in the equivalent testers. From the testers mentioned, this requirement can only be fulfilled by the true biaxial shear tester and by very special triaxial cells [5]. Lade himself and also Eibl used results from triaxial tests for calibration. Feise and Schwedes [12, 13,26] could show the advantages of using the true biaxial shear tester.

3.4 Quality control, qualitative comparison, flowability

In chapter 3.1 it was shown that the knowledge of the flow function, the time flow functions, the angle φ_e of the effective yield locus and the wall friction angle φ_w are necessary to design a silo properly. Having only estimates of the flow function (see Fig. 11) uncertainties remain and assumptions are necessary to get reliable flow. These assumptions are hard to check.

Very often testing of bulk solids is not done with respect to silo design. Typical other questions are:

– A special bulk solid has poor flow properties and these should be improved by adding small amounts of a flow aid. Which is the best kind and concentration of a flow aid?
– A bulk solid having a low melting point has sufficient flow properties at room temperature. Up to what temperatures is a satisfactory handling possible?

– The flow properties of a continuously produced bulk solid vary. Which deviations can be accepted?

For solving those problems, it is not necessary to measure the flow function completely and accurately.

What is a "good" or a "poor" flowability or a "sufficient" or "insufficient" flowability? The words "good" and "sufficient" express that no problems have to be expected when handling the bulk solid. Poor and insufficient imply the opposite. To characterize flowability Jenike [1,17] proposed to use the ratio of the major principal stress σ_1 at steady state flow to the unconfined yield strength σ_c:

$ff_c = \sigma_1/\sigma_c$

His classification was extended by Tomas [27] to the following:

		ff_c	$<$	1	hardened
1	$<$	ff_c	$<$	2	very cohesive
2	$<$	ff_c	$<$	4	cohesive
4	$<$	ff_c	$<$	10	easy flowing
10	$<$	ff_c			free flowing

In Fig. 20 the flow functions of two bulk solids A and B are plotted [3,4]. Flow function A represents a typical, often to find, degressive increase of the unconfined yield strength σ_c with an increasing major consolidation stress σ_1. Sometimes, but less often, a progressive increase as in flow function B can be observed. Also plotted in Fig. 20 are the areas of different flowability following the above mentioned classification of ff_c-values. Looking at flow functions the ratio ff_c and therefore also the classification depends on the major consolidation stress σ_1. Thus, the flowability of different bulk solids or bulk solids with different amounts of an additive must be compared at identical σ_1-values with help of ff_c. Published ff_c-values should always include the σ_1-values. By comparing the flowabilities of bulk solids A and B in Fig. 20 it can be deduced that at low major consolidation stresses bulk solid B has the better flowability and that the opposite is true for higher major consolidation stresses.

With help of Fig. 11 it was shown that the flow function can only be obtained, if steady state flow is achieved during consolidation and if the directions of the major principal stresses during consolidation and failure coincide. If one or both of these assumptions are not fulfilled, only estimates of the flow function can be obtained, which are on the unsafe side for the design of silos for flow.

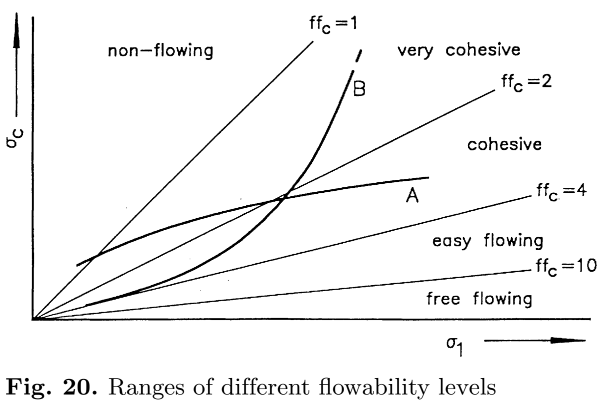

Fig. 20. Ranges of different flowability levels

When only comparing the flowability of different bulk solids qualitatively, i.e. without the aim of a quantitative design or statement, also estimates of the flow function can be used. But also for these estimates all the above holds true. The flowability depends also on the applied stress.

In chapter 4 testers for measuring flow properties will be listed, described and discussed regarding their advantages and disadvantages. In all testers the tests are performed in two steps: consolidation and failure. During consolidation a stress history will be imposed on the bulk solid sample, which has an effect on the subsequent failure experiment. Therefore, it can only be expected that two different testers yield the same results, if the stress history and the stress level during the consolidation step are identical.

After describing available testers in chapter 4 and discussing special bulk solid properties in chapter 5 the mentioned testers will be discussed again in chapter 6 regarding their useful application in solving industrial problems and fulfilling the demands of quality control.

4
Testers for measuring flow properties

Figure 21 gives a survey of possible shear principles and names some testers [28]. It can be distinguished between direct and indirect shear testers. The design defines the location of the shear zone of direct shear testers, whereas in indirect shear testers the shear zone develops unhindered according to the applied state of stress. In direct shear testers the major principal stress rotates during the test. In indirect shear testers the directions of the principal stresses are fixed and remain constant during the test. The already described shear tester by Jenike (Fig. 1) is a direct shear tester, whereas the also mentioned true biaxial shear tester (Fig. 6) is an indirect shear tester.

Shear testers are not only used in powder technology but also in soil mechanics. As an engineering discipline

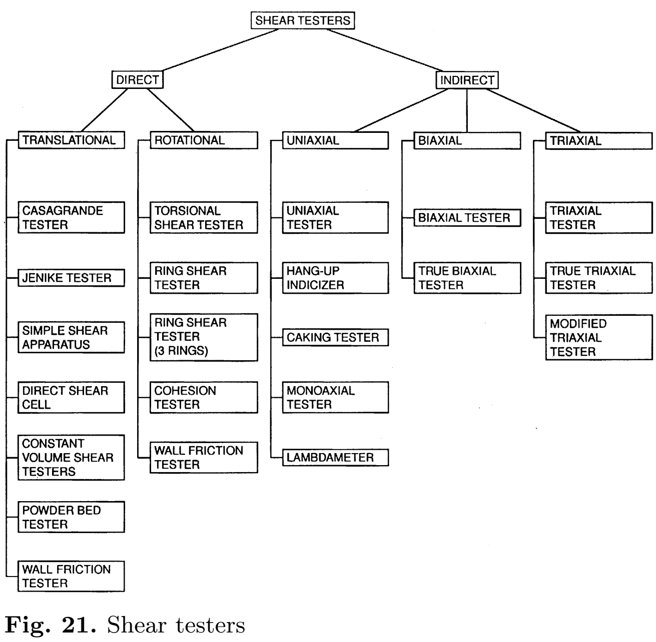

Fig. 21. Shear testers

soil mechanics are a lot older than powder technology. Most of the principles of shear testers have their origin in soil mechanics. But there are two differences in the application. In soil mechanics the deformation of bulk solids should be within elastic limits so that failure cannot occur. Contrary to this in powder technology for most cases the state of failure must be achieved. The other difference is the stress magnitude which in soil mechanics are significantly higher than in powder technology. Figure 21 mentions shear testers used in soil mechanics and in powder technology.

Not all testers mentioned in Fig. 21 will be described in detail in this report because they are either only usable for soil mechanics questions or they can today be regarded as ineffective for application. The Casagrande tester, the simple shear apparatus, the ring shear tester with 3 rings and the biaxial tester belong to this group. There are other testers or test principles which do not fit in a survey like Fig. 21, but which, nevertheless, are worth to be mentioned and have to be discussed with regard to their potentials for application: Angle of repose, avalanching behaviour, tensile strength, penetration test, Hosokawa tester, Jenike & Johanson Quality Control Tester,..... More information on those testers not described here in detail can be found in the literature [28].

To understand the behaviour of a bulk solid in a shear tester the yield locus – as shown in Fig. 2 – has to be discussed again, see Fig. 22. The significance of the endpoint of a yield locus is that the bulk solid sample having reached this state of stress is yielding at a steady state, i.e. without any further change in stresses and volume. Since there are no volume changes samples reaching the yield locus at the endpoint are called "critically consolidated". Performing shear tests with samples of the same initial bulk density but under higher normal stresses the sample has to be compacted during the shear process. This leads to a point of steady state flow of a yield locus having a higher bulk density, i.e. to a yield locus lying above the one in Fig. 22. With respect to the normal stress during the test this sample has been "underconsolidated". Accordingly, samples are "overconsolidated", if they start to dilate when reaching the yield limit. Tests with those samples determine further points of a yield locus. In chapter 2.1 it was explained that a shear test is performed in two steps: "preshear" under a normal stress σ_{sf} to steady state flow and

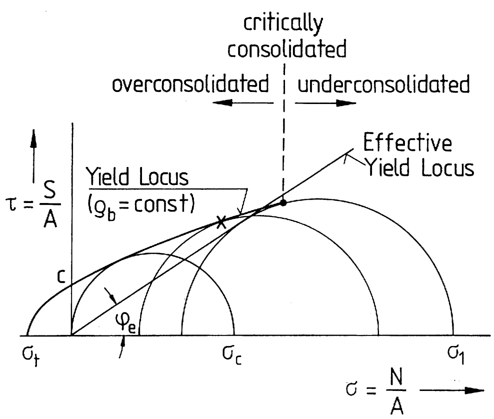

Fig. 22. Yield locus and effective yield locus

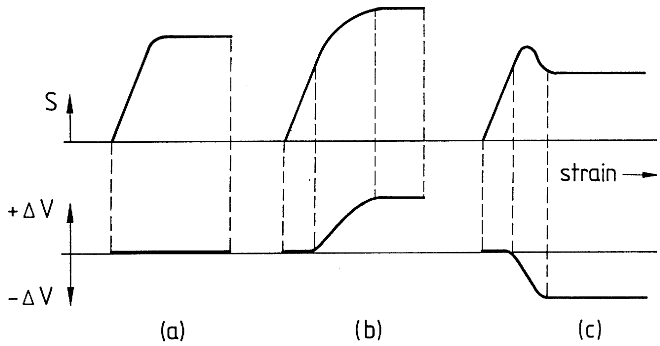

Fig. 23. Shear force S and volume change ΔV versus strain

"shear" under a normal stress $\sigma < \sigma_{sf}$. With respect to the normal stress σ during "shear" the sample is overconsolidated and has to dilate when reaching the yield locus.

The equivalent curves of shear force S and volume change ΔV (positive for a decrease in volume) against shear strain are plotted in Fig. 23 for the three states of consolidation mentioned above. "a" corresponds to a critically consolidated sample which shows no volume change and a constant shear force when yielding. When shearing an underconsolidated sample (case "b") the relative increase in shear force will reduce after a linear part. This is in line with a decrease in volume until again steady state flow at a higher bulk density is obtained. When shearing an overconsolidated sample (case "c") the sample will start to dilate when the yield limit is reached. Dilation means an increase in volume and a decrease in bulk density and strength. Therefore, the shear force, which is necessary for yield, decreases until again steady state flow is reached.

4.1 Jenike's shear tester: success and criticism

Jenike introduced his tester together with his theory on the flow of bulk solids in silos and his method for designing silos for flow [1,17]. The procedure of performing shear tests and to get yield loci, as already shortly explained, is very often described in the literature and shall not be repeated here. For those who want to get familiar with the correct procedure to run shear tests with Jenike's shear tester, it is recommended to read the "Standard Shear Testing Technique" [2], a report of the EFCE (European Federation of Chemical Engineering) Working Party on the Mechanics of Particulate Solids, which was published after running comparative shear tests with the same powder in Jenikes shear tester in more than 20 laboratories [internal report of the working party]. Also an ASTM-Standard is in preparation and will be available soon [29]. To check your ability in performing shear tests correctly a limestone sample CRM-116 can be obtained from BCR (Community Bureau of Reference of the European Union). For this bulk solid certified yield loci were measured with Jenike's shear tester [30,31], being delivered together with a representative sample.

Undoubtedly, Jenike's shear tester has served very successfully as an engineering tool for the design of silos for flow and it still belongs to the few testers being able to

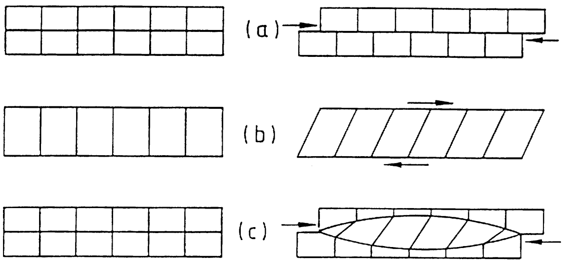

Fig. 24. Shear of a bulk solid's sample

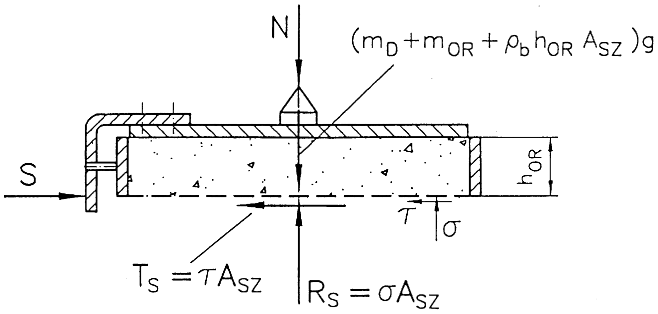

Fig. 25. Forces on ring, lid and upper half of bulk solids sample in Jenike's shear cell

measure the flow function (chapter 2). It never claimed to be a scientific tool for studying the stress-, strain-behaviour of bulk solids, for the following reasons: If a bulk solids sample is sheared in a direct shear tester, two ideal cases can be distinguished (Fig. 24 [32,33]). "a" is the case of pure Coulomb-friction without volume change; "b" is a shear process, where the shearing takes place homogeneously throughout the sample with possible volume changes. The available direct shear testers behave neither as in case "a" nor as in case "b". The shear process in Jenike's shear tester is shown for comparison in Fig. 24c. Inside the lens-shaped shearing zone case "b" is nearly realized; outside this zone the bulk solid is moving as in case "a". Two disadvantages regarding research work follow from this shear process:

- It is assumed, that the horizontal plane coincides with a slip plane. Only in this case are the measured σ, τ-values tangential points of the Mohr stress circles to the yield locus (x in Fig. 22). This cannot be proved in Jenike's shear tester and also not in other direct shear testers (besides the simple shear apparatus).
- The shear process does not take place homogeneously throughout the sample. The volume and the dimensions of the shear zone are unknown. Thus strain measurements are impossible. Only volume changes can be registered qualitatively.

A direct shear tester, in which the homogeneous deformation of Fig. 22b can be realized, is the simple shear apparatus [32,33]. Comparative tests with the same bulk solid by using the simple shear apparatus and Jenike's shear tester have shown that the assumption of coincidence of the horizontal plane with a slip plane is not absolutely correct, but of little and negligible small influence, when e.g. a hopper has to be designed for flow [32,33]. The disadvantage of the simple shear apparatus is the high amount of time and electronic and mechanical equipment which is needed for running shear tests. Thus this tester cannot be recommended, neither as an engineering nor as a research tool. For the latter case true biaxial shear testers are a lot more advantageous and enable more different test procedures.

When performing tests with Jenike's shear testers to get yield loci strain measurements are not necessary, but it has to be assured that the applied normal force N and the measured shear force S are evenly distributed across the cross-section A of the cell to get normal stresses $\sigma = N/A$ and shear stresses $\tau = S/A$. The even distribution is obtained if the shear force acts in the height of the shear zone and if the ring and the base of the shear cell do not touch. In Fig. 25 [34] the forces acting on ring (including the sample), cover and lid are plotted. From force- and momentum-equilibria it can be deduced that normal and shear stresses are evenly distributed across the shear plane. To be sure that ring and base do not touch a small combined twisting and lifting motion of the ring should be performed after preconsolidation of the sample with the normal load for "preshear" acting on the cover.

The following disadvantages of Jenike's shear tester are often mentioned:

- It requires a high level of training and skill.
- More time is needed than with other testers.
- No measurements at small normal stresses are possible.
- The maximum strain is small and sometimes not sufficient.

It is correct that training and skills are necessary to perform tests and to interpret the results correctly. However, it is most critical to obtain steady state flow at the first step of a shear test, the "preshear". Plotting shear stress versus strain or time gives the best information and has to be interpreted correctly. Reading the "Standard Shear Testing Technique" [2] helps but is no guarantee.

The statement regarding too much time is only partly true. If a hopper is to be designed than time is needed to get the necessary information. If the need is only for quality control or product development, it is also possible to use Jenike's shear tester (or ring shear testers) with a simpler procedure. An estimate of a yield locus can be derived by running only one test (preshear and shear) and a repetition test (see chapter 6.2).

With the help of Fig. 3 it was explained, how a yield locus is obtained by running shear tests. To get a yield locus, at least three tests with "preshear" under identical normal stresses σ_{sf} and "shear" under three different normal stresses $\sigma < \sigma_{sf}$ are necessary. Several proposals can be found in the literature to get a yield locus with Jenike's shear tester or similar translational shear testers in only one test: Pitchumani et al. [35] reported on tests with the certified limestone CRM-116 where they compared the conventional procedure with "preshear" and "shear", as shown in Fig. 3, with a modified technique. During shear they stopped the shear as soon as the maximum was reached, retracted the stem for the shear application, reduced the normal stress, sheared again to the maximum, etc. Thus, they were able to get up to 5 shear points in

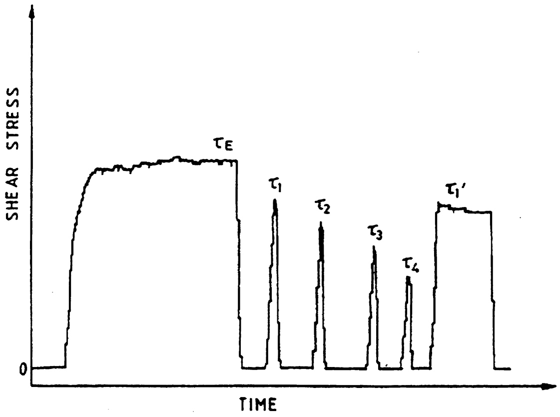

Fig. 26. Modified shear procedure to get a yield locus in Jenike's shear tester with only one test [35]

one single test, as shown in Fig. 26. The last shearing action, leading to shear stress τ_1', was performed under the same normal stress σ_1 as used in the first shearing action leading to shear stress τ_1. The idea of this test procedure is the assumption that a bulk solid sample deforms only elastically, before the yield locus is reached. Reaching the maximum shear stress is an indicator of beginning plastic deformation (with a volume increase). Therefore, if the shear process is stopped at the maximum, no significant dilation is expected to have taken place.

The assumption that no volume change occurs before the yield limit is reached, is in line with the explanation to Fig. 23, but it is nevertheless only a theoretical assumption. In reality volume changes already occur before the maximum shear stress is reached. In Fig. 27 [36] the shear of an overconsolidated limestone powder (5 μm mean particle size, similar to the sample which was used by Pitchumani et al. [35]) is shown. The relative shear stress – relative with respect to the shear stress at steady state flow – and the increase in sample height is plotted versus time (i.e. versus strain). It can be seen clearly that a volume increase already starts before the maximum shear stress is reached. Thus, the bulk density and therefore the strength of the sample are reduced and the sample cannot be used again for getting other points of the same yield locus. It has to be suspected that especially the shear stress corresponding to the smallest normal stress will be underestimated in Pitchumani's experiment. The consequence is that the derived unconfined yield strength σ_c gets smaller and the design e.g. of a hopper is on the unsafe side.

Tsunakawa and Aoki [37], Haaker et al. [38,39], Puri et al. [40,41] and others [42,43] have tried to use the concept of a "constant volume shear test" to measure a yield locus in one single test. In such a test not the normal stress, but the volume of the sample has to be kept constant. When the sample wants to dilate it will be hindered by an increasing normal stress. In Fig. 27 the normal stress would have to increase as soon as the sample starts to dilate and that is before the maximum stress is reached. Thus the maxima in shear stress are reached at higher normal stresses – compared to the conventional procedure -, the yield locus lies at smaller τ-values, especially at small normal stresses, and the unconfined yield strength will be underestimated. Puri et al. [40], being aware of the dilatant behaviour, start the procedure with steady state flow and then lift the cover of their "direct shear cell" at a constant small speed. Due to this the normal stress and the corresponding shear stress are decreasing in a curve which they claim to be identical with the yield locus. What is measured will depend on the speed of lifting the cover and again due to the volume increase the strength of the sample is decreasing with the result that especially at small normal stresses the measured shear stresses will be smaller compared to the ones measured in the conventional procedure.

Besides the fact, that the "constant volume shear tester" and "the direct shear cell" are a lot more complicated than Jenike's shear tester, it is to be suspected that even with a lot of experience and the help of comparative measurements both testers will underestimate the unconfined yield strength. The only way to get more than one point of a yield locus in one single shear test is the sequence of preshear, shear, preshear, shear etc., but this procedure is restricted due to the small maximum strain of translational direct shear testers. It is not restricted in ring shear testers, as will be shown in chapter 4.2.

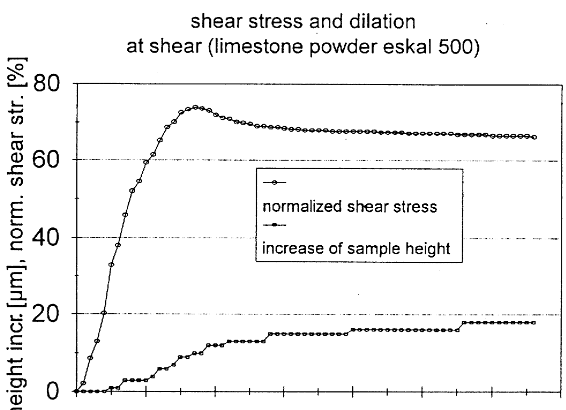

Fig. 27. Shear stress and increase in height when shearing an overconsolidated sample

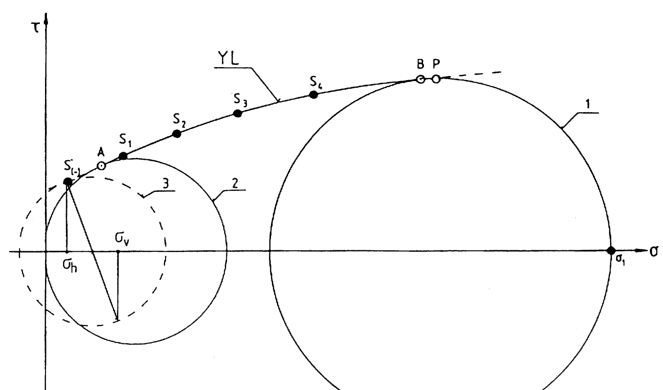

Fig. 28. Yield locus showing valid shear points due to SSTT [2]

It is often argued, also in the "Standard Shear Testing Technique" [2], that

> "points to the left of point A (Fig. 28) are invalid because they represent a state where tensile stresses can occur in the shear cell...... If a Mohr circle 3 is drawn through point S which is tangential to the extrapolated yield locus, part of that circle will be to the left of the origin indicating negative normal stresses, i.e. tensile stresses. The Jenike Shear Cell is not adapted to such tensile stresses which, if present, would tend to cause the particulate solid to separate from the wall, leaving a gap between the filling and cell base and/or ring ..."

Also plotted in Fig. 28 are the normal stresses acting in horizontal and vertical direction, σ_h and σ_v. Both are positive, i.e. they are compressive stresses, and a gap between sample and base or ring cannot develop. Thus, the above mentioned argument is invalid. The real reason (for not using σ, τ-values close to the τ-axis) is the possibility of the cover of the shear cell to tilt and to loose contact to the sample [34]. If that happens the even distribution of normal and shear stresses in the shear plane is not any longer fulfilled and the measured σ, τ-value is invalid. In Fig. 29 the stresses acting on cover and bracket (including pin) of Jenike's shear cell are drawn [34]. A_x and A_y are the normal and friction forces acting between pin and ring. $m_D \cdot g$ is the weight of cover and bracket and R and T are the normal and shear forces between cover and sample. From momentum equilibrium with respect to point A an equation can be derived including forces R and T. If R and T are set equal to zero – cover is lifted -, an equation can be derived relating the shear stress τ in the shear plane to the normal stress σ in the shear plane (s. Fig. 25). For typical dimensions of a Jenike shear cell made from aluminium and for a sample with a bulk density of 1000 kg/m^3 Schulze [34] derived

$$\tau = 5.75(\sigma - 230 \text{ Pa})$$

with σ and τ in [Pa]. Only for smaller τ-values tilting is prohibited. If the smallest normal stress σ for "shear" is set to 20% of the normal stress σ_{sf} at steady state flow (as proposed by the Standard Shear Testing Technique [2]), a minimum major consolidation stress σ_1 of about 2.3 kPa is obtained for the data mentioned above [34]. As a consequence no points of the flow function (Fig. 9) to the left of $\sigma_1 = 2.3$ kPa will be measured with Jenike's shear tester.

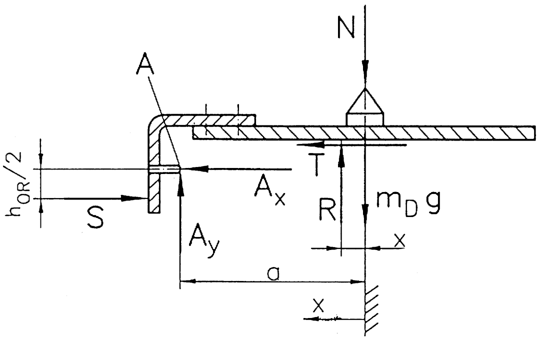

Fig. 29. Forces on the lid of Jenike's shear cell

If the cohesion c or the tensile strength σ_t (Fig. 22) could be measured accurately, the yield locus at small σ-values could be constructed by interpolation instead of by extrapolation. In chapters 2.4 and 2.5 it was reported that the stress-strain behaviour of a bulk solid sample strongly depends on the stress history and that it behaves anisotropically. The anisotropic behaviour is only of no influence with regards to application, if the directions of the major principal stresses during compaction (or steady state flow) and subsequent failure coincide. In the known testers for the direct measurement of tensile strength σ_t and cohesion c (see chapter 4.6) this condition is not fulfilled and smaller values for σ_t and c will be measured, smaller compared to conditions where the mentioned coincidence is guaranteed and the sample had reached steady state flow before. Thus, it is not advisable to use the measured values for the construction of a yield locus and the subsequent derivation of the unconfined yield strength σ_c, needed for the flow function.

Wall friction and time consolidation are very important parameters when designing equipment for bulk solid handling, storage and transportation. Both influences can easily be measured with Jenike's shear tester (see chapter 4.7 and 4.8). This is another advantage of this tester. A disadvantage is the small maximum strain creating problems when testing very elastic bulk solids and bulk solids with coarse particles or high humidity content (see chapter 5.1, 5.2 and 5.4).

4.2 Ring and torsional shear testers

The direct shear testers with translational displacement, as described in chapter 4.1, have only a limited shear strain, at Jenike's shear tester e.g. a maximum of 4 to 5 mm. To be sure, that within this short strain "preshear" with steady state flow and "shear" with a peak shear stress are obtained, the sample has to be preconsolidated by a twisting motion, see e.g. the "Standard Shear Testing Technique" [2] and original publications of Jenike [1]. To perform this preconsolidation correctly the already mentioned training and skill is necessary. This preconsolidation is not necessary in direct shear testers with

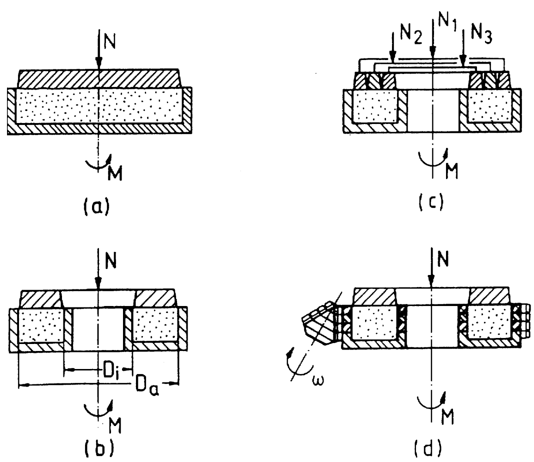

Fig. 30. Rotational shear testers

rotational displacement due to their unlimited shear strain. Four versions of these testers are shown in Fig. 30. The cross-section of the sample is either circular [44–46] or annular [18, 47–50, others]. The shear process is induced by rotation around the vertical axis. The covers of all testers shown are roughened or are equipped with bars to be sure that the shear process takes place within the bulk solid and not between bulk solid and cover. In most testers the base is rotating and the shear moment acting on the cover is measured.

In the tester with circular cross-section (a), called torsional shear tester, there is no shear in the centre. Therefore, ring shear testers are used. An influence of the ratio D_i/D_a (Fig. 30 b) on the results might be expected as can be seen in Fig. 31. Upon an angle α of rotation the strain in the middle of the ring is s, s + Δs at the outer wall and s − Δs at the inner wall. If an overconsolidated sample is sheared and the maximum value of shear stress has just been reached in the middle, it is beyond the maximum on the outer wall but has not quite reached the maximum at the inner wall. The total shear moment is measured thus resulting in a mean value of shear stress which might be less than the maximum value. The determination of steady state flow is without problems, since it is obtained and maintained on all radii after some time.

To investigate the influence of D_i/D_a either ring shear testers with different values of D_i/D_a [50] have to be used or the cover consists of three concentric rings, which do not touch each other (Fig. 30 c) [51, 52]. Another complicated method of running a ring shear test is shown in Fig. 30 d [53]. The shear cell consists of several rings which rotate at different speeds thus producing a more homogenious deformation with respect to height. The testers shown in Fig. 30 c and d are very complicated. The tester with the three rings above each other (d) enables no additional information compared to the simpler tester (b) and also the tester with the three concentric rings as cover did not prove to be superior to the simpler tester. Thus, both testers are not recommended or described any further neither as an engineering nor as a research tool.

The first ring shear tester used in powder technology was the one of Walker [48]. Many copies of this tester were built by others with small, but not significant changes [49–55, others]. It was also tried to compare test results gained with Jenike's shear tester and ring shear testers. In doing this some researchers took the results of Jenike's shear tester as "a standard" and tried to run the ring shear test in such a way to get similar results. The agreement was only partly sufficient. Thus ring shear testers were not used very often. The situation changed after Schulze built a new version of a ring shear tester [18, 34, 56–58].

The shear tester of Schulze is shown in Fig. 32. The ring shear cell (standard: D_i = 100 mm; D_a = 200 mm) contains the sample. An annular lid attached to a crossbeam lies on top of the sample. The shear cell is driven in the direction of arrow ω. The lid is prevented from rotating by two tie rods which are connected to the crossbeam. Due to the relative displacement of the shear cell to the lid the sample is sheared. From F_1 and F_2 the shear stress acting in the sample is calculated. To prevent the lid from moving horizontally, two guiding rollers are installed. In combination with the rollers at the end of the crossbeam the guiding rollers realize a nearly frictionless guide. Besides the tie rods and the guiding rollers no additional bearing is necessary for the lid. Thus, the movement of the lid is unhindered in a similar way as the cover in Jenike's shear tester. This is the main difference to formerly built ring shear testers and a big advantage. The normal force is exerted on the sample by a weight hanger connected to the crossbeam. Furthermore, with a counterweight system F_A a very small normal stress σ on the sample can be achieved. Compared to previous built ring shear testers the new tester is a very light construction, yielding smaller friction and inertia forces. As a result, more accurate measurements are possible especially at low normal stresses.

The test procedure with the ring shear tester is equivalent to that described in chapter 2.1 and used with Jenike's shear tester (chapter 4.1). The bulk solid is sheared in two steps: "preshear" under normal stress σ_{sf} up to steady state flow at τ_{sf} = const. and subsequent "shear" under a normal stress $\sigma < \sigma_{sf}$ up to a peak shear stress τ. Using Jenike's shear tester, for each measurement ("preshear" and "shear"), a new sample of the bulk solid has to be used. Opposite to this, the ring shear tester allows the measurement of a complete yield locus with only one sample. After measuring the first point of a yield locus (first "preshear"

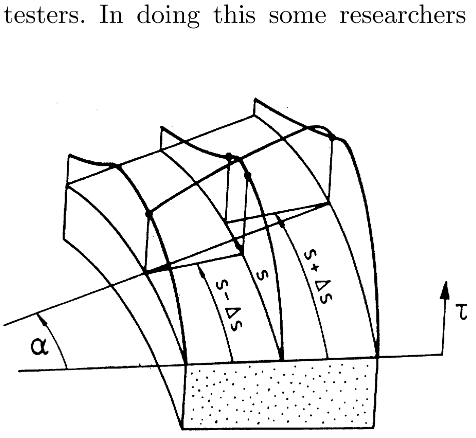

Fig. 31. Shear stress versus shear strain of an overconsolidated sample in a ring shear tester

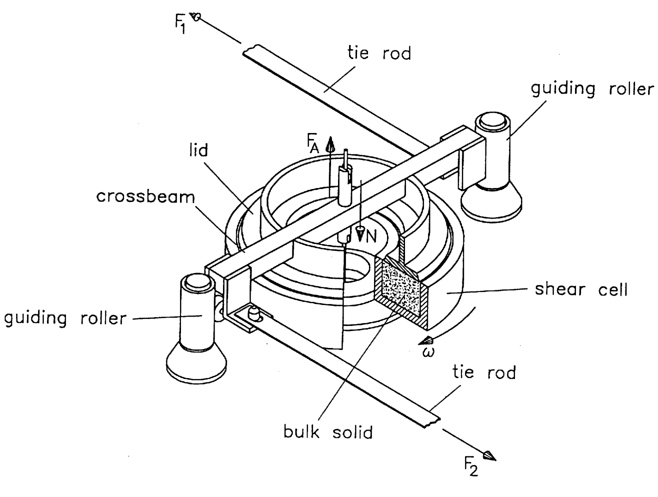

Fig. 32. New ring shear tester of Schulze

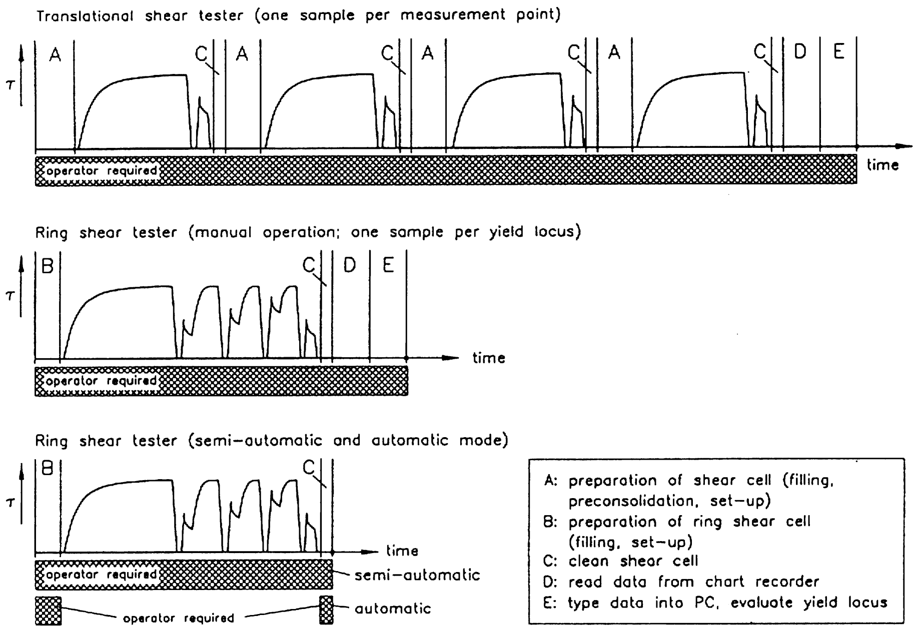

Fig. 33. Time required for the measurement of a yield locus (qualitatively)

and "shear") the normal stress is increased again to σ_{sf}, the sample is sheared again to steady state flow (τ_{sf}) and a further point of the yield locus can be gained under another normal stress $\sigma < \sigma_{sf}$, etc......

Today an automatic version of the new ring shear tester exists [58]. The normal loading system makes use of the approved "hanger and weight system" thus obtaining constant normal stresses during the different steps of the shear process. This is a big advantage over other systems in which a regulated piston tries to maintain constant normal stresses on the cover of the shear cell which can move downwards or upwards due to compression or dilation. The automatic ring shear tester can be operated semi-automatically or full automatically. In automatic mode the measurement is fully controlled by a PC. The software recognizes steady state flow at "preshear" and the shear stress peak at "shear". In the semiautomatic mode the operator has to judge whether "steady state flow" or a "peak stress" has been attained. Figure 33 [58] qualitatively compares the times required for getting one yield locus, when a translational shear tester (e.g. Jenike-type), a manually operated ring shear tester or a ring shear tester in automatic operation is used.

The biggest advantage of ring shear testers over translational shear testers is the unlimited strain. No preconsolidation of the sample is necessary. Thus the results gained with a manually operated ring shear tester are less operator-dependent and those of an automatic one are not operator-dependent. With the help of Fig. 31 it was argued that there might be an influence of the ratio D_i/D_a (inner to outer diameter) on the results and that it might not be possible to measure the peak shear stress accurately. This would be a disadvantage of ring shear testers. But many comparative tests have shown that, when plotting the flow function in a σ_c, σ_1-graph, a good agreement could be found for results gained with Jenike's shear tester and ring shear testers [5,18,55–61]. Two reasons can be named for the good agreement. At the end of "preshear" under a normal stress σ_{sf} steady state flow with a constant shear stress τ_{sf} on all radii is achieved. After "preshear" the shear cell is driven backwards thus releasing the tie rods. Forces F_1 and F_2 and also the shear moment are reduced to zero. But the normal stress σ_{sf} is still acting on the sample. During the backward motion of the shear cell a small elastic displacement between ring and lid can be imagined. This displacement creates a smaller strain at the inner radius and a bigger one at the outer radius. Although the total shear moment between lid and sample is zero after the backward motion a negative shear stress at the outer radius and a positive at the inner radius are possible as a result of the different strain [61]. For the "shear" process the normal stress is reduced to $\sigma < \sigma_{sf}$. This reduction also reduces the magnitude of the negative shear stress at the outer radius and the positive shear stress at the inner radius. In the subsequent "shear" the shear stress at the outer radius starts with a negative value and needs a longer strain to reach the peak shear stress than the shear stress at the inner radius which started positive. It can be argued that due to the different strains and to the different starting values the peak shear stresses are reached simultaneously [61]. The second, but more important argument for the negligible small influence of the different strains on the inner and outer radius is the very small strain necessary to shear an overconsolidated sample. The angle of rotation is smaller than 1° for the certified BCR-limestone CRM-116. Additionally Münz [52] and Gebhard [50] have reported on measurements with ring shear testers of different D_i/D_a-ratios. They got identical results for $D_i/D_a \geq 0.5$.

Another advantage of the ring shear tester is the possibility to get reliable results at very low normal stresses. This is important for cohesive as well as for free-flowing bulk solids. For cohesive bulk solids the extrapolation of the yield locus into the low stress region can be made safer or can be avoided. Testing free-flowing bulk solids with flowability-values of $ff_c > 10$ differences in the flowability

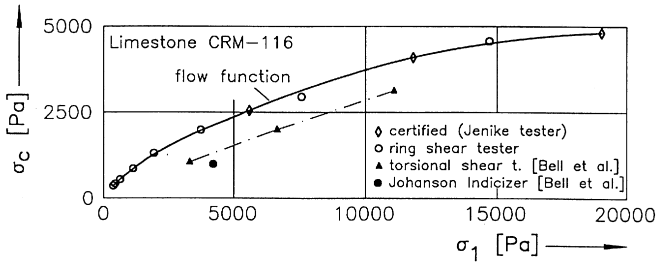

Fig. 34. Flow function of limestone CRM-116 (certified data [30], results of Bell et al. [62,63], ring shear test results [58])

can only be detected if measurements at low normal stresses are possible [34].

The flow function of the BCR Limestone CRM-116 measured with 3 different testers is shown in Fig. 34 [58]. There is good agreement between the certified results, using Jenike's shear tester, and the results of the new ring shear tester. It can be clearly seen that points of the flow function at σ_1-values $<$ 500 Pa can be gained using the ring shear tester. Also plotted in the graph are results from tests using the torsional shear tester of Peschl [58]. The equivalent tests using this torsional shear tester were performed by Bell et al. [62,63]. At least for the used limestone it has to be concluded that results of the torsional shear tester underestimate the strength of the bulk solid.

The torsional shear tester (rotational shear tester with circular cross-section, Fig. 30 a) was also investigated by others and results are gained from comparative studies, mainly in comparison to results from translational shear testers (Jenike-type, direct shear cell) [63–65]. The findings are not always in agreement. Sometimes the results are identical or at least similar. Sometimes they differ a lot and, if they differ, the results from the torsional shear tester always underestimate the strength, thus they predict a better flowability than the bulk solid really has. Why there is an agreement for some bulk solids and a disagreement for others could not be explained yet. A reason for a disagreement can be found in the already mentioned fact (see Fig. 30 a) that in the center of a rotating circular cell no shear stress can be exerted on the sample. That is of no influence on the "preshear". Steady state flow with a constant τ_{sf} across the complete cross-section is guaranteed. But there remains a problem with the subsequent "shear". The argument with the negative and positive shear stresses after "preshear", used to explain the good agreement between results from ring shear testers and Jenike's shear tester, can also be used. But perhaps there is no sufficient compensation between positive and negative shear stresses and different strains. The mentioned result of Münz [52] and Gebhard [50], that only for D_i/D_a-ratios $\geq$ 0.5 an agreement can be expected, is another indication to be cautious, when using data of the torsional shear tester for a flow function.

The advantage of the torsional shear tester is its simplicity and the possibility to use the tester in an automatic mode. Thus the results will be operator-independent. The shear cell can be very small. Thus only small sample amounts are necessary. Therefore, the torsional shear tester is best qualified for comparative measurements and quality control. If e.g. the optimum amount of a flow aid has to be found to improve the flowability of a pharmaceutical powder, the torsional shear tester is best suited and a reliable answer can be gotten quickly by using only small samples. But care has to be taken if results are used to get an estimate of the flow function with the aim to design a silo for flow.

4.3 Triaxial testers

Triaxial testers belong to the indirect shear testers in which the principal stresses in three dimensions are measured or applied. Figure 35 shows two triaxial testers as used in soil mechanics. (a) is the normal triaxial tester and (b) the true triaxial tester. In the normal triaxial tester the sample of cylindrical shape is covered by a rubber membrane and is placed in the vertical direction between two movable stamps. In the horizontal direction it is stressed by water pressure $\sigma_2 = \sigma_3$. By moving the stamps in the vertical direction towards each other the stresses σ_1 will increase until failure is obtained. After failure further measurements are not possible. Thus overconsolidated samples can only be tested up to the point of maximum shear stress. The failure takes place along slip planes which can be observed. Since the principal stresses are known Mohr stress circles can be drawn. The envelope to the Mohr stress circles representing failure defines the yield limit in a σ, τ-graph. The mentioned procedure with increasing σ_1 is called a compression test. If the stress σ_1 is decreased ($\sigma_1 < \sigma_2$), while σ_2 stays constant, again failure occurs, when the Mohr stress circle with the principal stresses $\sigma_2 > \sigma_1$ touches the yield limit. This procedure yields an extension test.

The procedure of running a test is relatively simple. This tester is the standard shear tester in soil mechanics. Typical diameters D of the sample are 3.6 cm (saturated soils) to 10 cm (dry coarse bulk solids). The height H of the sample has to be such, that the failure can take place unhindered by the two stamps (H/D >2). For testing the sample has to be placed into a rubber membrane. This preparation is done outside the apparatus. The rubber membrane has to be prestressed and therefore the tester in its standard form is not applicable for the low stress region being of interest in powder technology.

Haaker and Rademacher [66] have used a modified triaxial tester (Fig. 36) for the use at lower stresses. The

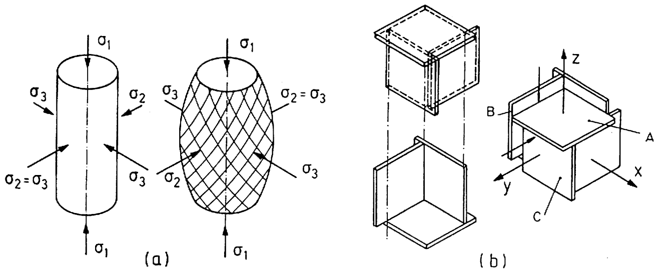

Fig. 35. Triaxial shear tester (a: standard; b: "true")

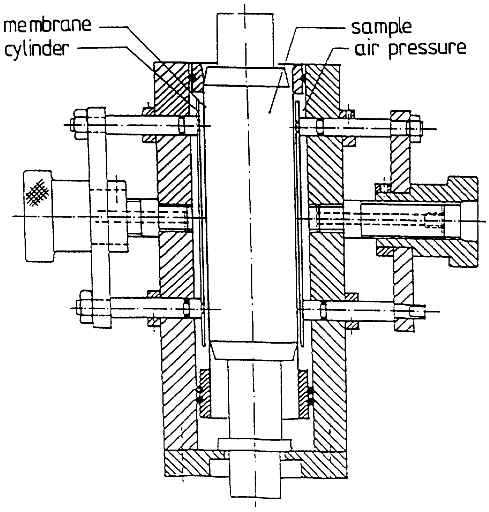

Fig. 36. Modified triaxial shear tester [66]

sample is covered by a very thin rubber membrane and during the filling procedure by an additional perforated metallic cylinder. After preconsolidation this perforated cylinder is removed and the sample is stressed horizontally by a constant air pressure. Then the stamps are moved towards each other up to failure. The necessary stress is measured. They also performed tests with Jenike's shear tester. The agreement was satisfactory.

In the true triaxial tester (Fig. 35b) the six walls, being the boundaries of the sample, are arranged in such a way, that deformations in x-, y- and z-directions are possible at the same time, independent and at different rates, e.g. plate A can be moved relative to plate B in z-direction. Plate C is moved in z-direction identically but can be moved independently in y-direction relative to plate A. Again the sample has to be placed in a rubber membrane, which has to be prestressed in order to handle the sample before it is placed in the tester. Thus, only tests under high stresses give reliable results. Only two such testers are known from soil mechanics with only a few results published [67,68].

Puri [69] has built a cubical triaxial tester with six flexible membranes as the boundaries of the sample, capable of measuring the complete states of stress and strain. This tester is an extension of the biaxial tester used in Porsgrunn and Delft (see chapter 4.4). He has performed hydrostatic triaxial compression tests up to 190 kPa and conventional triaxial compression tests with the minor principal stresses $\sigma_2 = \sigma_3 = 34.5$ kPa. It is to hope that he will be able to also investigate bulk solids in the low stress region.

The advantage of a true triaxial tester is the complete determination of the state of stress and the state of strain, because all three principal stresses and strains are measured. Many different strain paths – different strains in x-, y- and z-direction – can be applied and the stress-response can be measured. Or stresses are given and the resulting strains are measured. Another advantage (compared to the true biaxial shear tester of Fig. 6) is the fact, that it can be checked if the intermediate principal stress has an influence on the yield limit and the stress-, strain-behaviour or not as assumed in the Mohr-Coulomb criterion.

4.4 Biaxial testers

The biaxial shear tester as used at the Technical University of Braunschweig was already introduced and explained in chapter 2.3. It has rigid walls and rubber membranes including a grease between the rubber membranes and the six rigid walls, which ensures negligible small shear stresses. Thus the measured normal stresses are principal stresses and the complete state of stress is known. Another type of biaxial tester, the flexible wall biaxial tester, is used at the Telemark Institute of Technology of Porsgrunn [70,71] and at the Technical University of Delft [72, 73]. Both realize the identical idea, first used by Arthur [74]. Figure 37 shows a schematic top view of the Delft tester. The walls of the sample holder are membranes. These membranes are in fact cubical balloons which can be pressurized. The pressure in the balloons equals the normal stress on the boundary of the sample if its face remains flat. The walls can be moved inwards and outwards with stepper-motors. Optical sensors in the balloons measure the deformation of the membranes. This makes it possible to use the tester both as a stress-controlled and as a strain-controlled tester. The stresses and strains in x- and y-direction can be controlled and measured. The stress in z-direction is only measured. The tester is placed on a table and closed with a top lid. The sample is covered by a top and bottom membrane, which prevent shear stresses to occur since they deform together with the sample. The height of the sample is 80 mm. The minimum size of the cross-section is 75×75 mm^2 and the maximum size is 135×135 mm^2. For comparison the minimum and maximum dimensions of the Braunschweig biaxial shear tester are $35 \times 60 \times 60$ mm^3 and $35 \times 130 \times 130$ mm^3 [5–9,12].

A typical test in the Porsgrunn- and Delft-tester to get one point of the flow function is shown in Fig. 38 [73]:

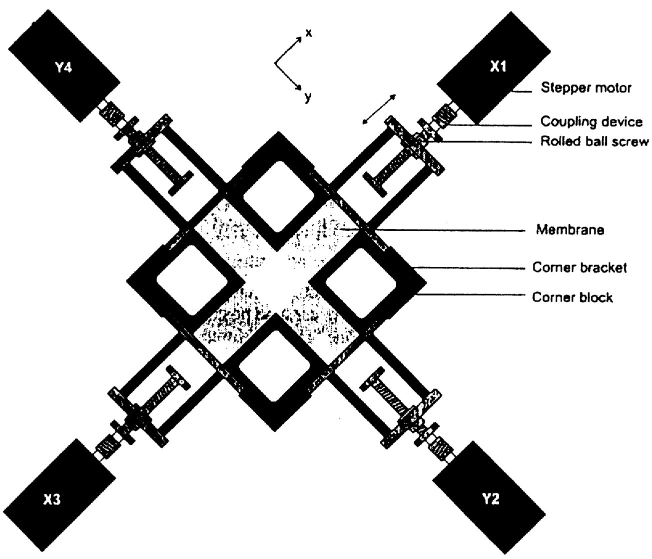

Fig. 37. Flexible wall biaxial shear tester [73]

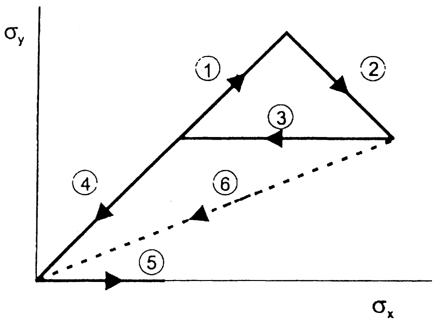

Fig. 38. Stress path to be followed to measure a point of the flow function in a biaxial shear tester [73]

First the sample is biaxially compressed ① followed by "preshear" to steady state flow at $\sigma_x = \sigma_1$ (σ_1= major consolidation stress) ②. The stress path back to the origin can be along ③ and ④ or along ⑥. When the stresses are zero again, the second part of the shear test ("shear") will be performed, in the present case (Fig. 38) with $\sigma_y = 0$. σ_x is increasing along ⑤ up to the maximum thus yielding the unconfined yield strength σ_c. Thus one point of the flow function $\sigma_c = c_c(\sigma_1)$ is obtained. The stress paths ① and ② of this stress-controlled test correspond to the stress paths ① and ② in Fig. 14 of a strain-controlled test in the Braunschweig biaxial shear tester.

With the help of Fig. 9 (chapter 2.3) it was shown that the flow functions measured with the true biaxial shear tester of Braunschweig and with Jenike's shear tester agree well [5,6]. An identical good agreement was obtained by Maltby who used the biaxial tester of Porsgrunn [11, 70]. A different and for the author of this report astonishing result was gained with the flexible wall biaxial shear tester of Delft [72,73]. For the same bulk solid, which was also used in Porsgrunn (the BCR limestone CRM-116), a flow function lying above the certified one was obtained. Therefore both institutes, the one at Delft and the one at Braunschweig, decided to exchange their young researchers in order to perform identical tests in both testers and to then compare and discuss the results.

At least one hour and up to one day is necessary to perform one satisfactory test in the true biaxial shear tester of Braunschweig. For the testers in Porsgrunn and Delft less time might be needed. But compared to the times needed to get a complete flow function with Jenike's shear tester or ring shear testers the biaxial testers are very time-consuming and cannot be recommended as an engineering tool for the design of silos or for quality control. If wall friction and time consolidation, being of great influence in daily engineering problems, have to be measured also, the biaxial testers are even more disadvantageous as an engineering tool.

Biaxial testers are excellent research tools. As was already described in chapters 2.3 to 2.5 they are capable of determining many influences on the stress-, strain-behaviour of bulk solids. Many straining situations can be applied to the sample and the stress-response will be measured. Vice versa different stresses can be applied and it can be detected how the sample deforms. Uniaxial, biaxial and many other consolidations can be realized or simulated. Anisotropy, relaxation, creeping and other time-dependent effects can be measured. Constitutive models can be calibrated and shear testers can be checked by performing identical tests.

4.5 Uniaxial testers

In uniaxial testers it is tried to verify the theoretical experiment already mentioned in chapter 2.2 and described with the help of Fig. 4: A sample is filled into a cylinder and consolidated without wall friction by a normal consolidating stress $\sigma_{1,c}$ leading to bulk density ρ_b. After removing the cylinder the sample is loaded again with an increasing normal stress up to the point of failure, leading to the unconfined yield strength σ_c. With the help of comparative tests in the true biaxial shear tester (chapter 2.3) it was also demonstrated that the dependence between σ_c and $\sigma_{1,c}$ cannot be regarded as a flow function. The curve $\sigma_c = \sigma_c(\sigma_{1,c})$ lies below the flow function $\sigma_c = \sigma_c(\sigma_1)$ with σ_1 as the major principal stress at steady state flow (see procedure I in Fig. 11).

The different consolidation situations in the uniaxial tester (consolidation stress $\sigma_{1,c}$) and at steady state flow (major consolidation stress σ_1) are shown again in Fig. 39. The left hand drawing represents the situation in the uniaxial tester with the horizontal stress $\sigma_{3,c}$ and the strain ε_1 only in the direction of the consolidating stress $\sigma_{1,c}$. The right hand drawing represents the situation at steady state flow which is not possible during uniaxial consolidation. A dilation is possible in the horizontal direction. Steady state flow means plastic deformation at constant stresses and constant volume. Therefore the expansion ε_3 and the compression ε_1 have to be identical. The horizontal stress σ_3 at steady state flow is smaller than the horizontal stress $\sigma_{3,c}$ after consolidation. Thus, the Mohr stress circle, representing the consolidation ($\sigma_{1,c}$ and $\sigma_{3,c}$), is smaller and as a consequence the sample has gained less strength and the unconfined yield strength will become smaller (see also Fig. 5).

Despite the fact that the uniaxial tester underestimates the unconfined yield strength it is a very useful tester, because it is a simple tester and a test can be performed quickly. Testers were proposed by Williams et al. [75], Gerritsen [10,76], Enstad et al. [11,70], Runge [77, 78], von Rijsinge [79] and others and also many laboratories are using different uniaxial testers without publishing

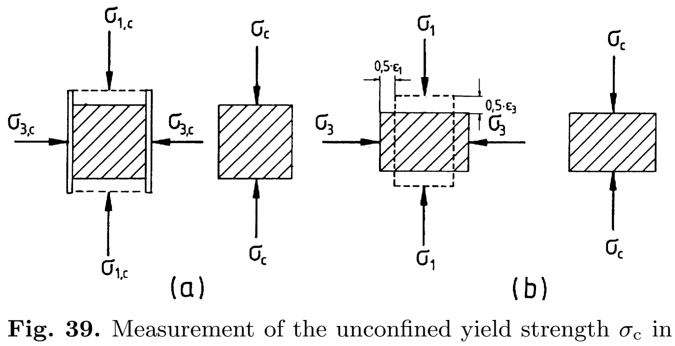

Fig. 39. Measurement of the unconfined yield strength σ_c in a biaxial tester after steady state flow σ_1 (b) or uniaxial consolidation $\sigma_{1,c}$ (a)

their methods and their results. In all testers it is tried to reduce the influence of the wall friction on the homogeneity of the compacted sample. Williams et al. used a mould splitted vertically so that the two halves can be moved apart. It was made up of thirteen sections, so that moulds of different height could be investigated. The sample was filled into the mould in small increments. Each increment was compacted separately thus resulting in a homogeneous compaction with respect to height. When the sample was formed the mould side plates were unlocked and the two halves were removed horizontally without disturbing the sample. The unconfined yield strength was determined by applying a vertical stress by means of a constant strain rate device.

A completely different approach was used by Gerritsen [10,76]. Figure 40a shows the experimental set-up with a mould having a low height to diameter ratio. The mould is filled with the bulk solid to be tested. A thin aluminium disc is placed on the centre of the surface of the sample. An airbag serves to achieve an even stress distribution at the surface. The consolidation stress $\sigma_{1,c}$ is exerted on the sample through the bag and applied to the bag by a weight on a flat plate resting on the bag. After consolidation the weight, the air bag and the mould are removed. Most of the bulk solid is then cut away with a knife to leave only the central part being marked by the aluminium disc (Fig. 40b). The unconfined yield strength of the cylindrical test sample is then measured by placing a polythene flask on the top plate. The weight is gradually increased by filling the flask with water until the sample fails. Gerritsen calculated critical outlet dimensions for arching not to occur and compared these dimensions with measured critical outlet dimensions of an experimental hopper. It was shown that the calculated critical outlet dimensions were sometimes too small to prevent arching. This finding is in line with the above statement according to which uniaxial testers always underestimate the unconfined yield strength.

The uniaxial tester developed by Enstad et al. [11, 70] is shown in principle in Fig. 41. The sample is confined in a slightly conical die and consolidated by means of a piston. A flexible membrane is stretched between the outer periphery of the piston and the inner periphery of the lower part of the confining die. A layer of lubricant will ensure that there is an absolute minimum of friction between the flexible membrane and the die wall. After the desired level of compaction stress has been reached, the compaction stress is reduced to a minimum value and the die is pulled up, allowing the sample to stand by itself. The unconfined yield strength is measured by moving the piston downwards once more. Enstad et al. [11] compared their uniaxial tester results with results from tests in their biaxial tester and Jenike's shear tester. As expected the uniaxial tester underestimates the flow function.

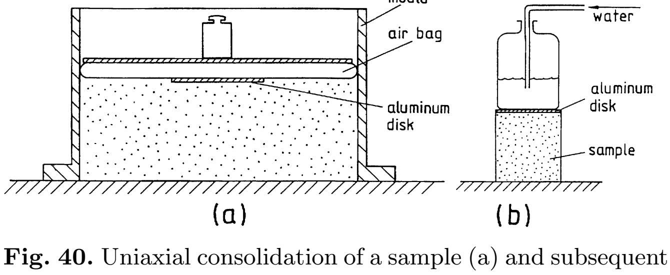

Fig. 40. Uniaxial consolidation of a sample (a) and subsequent measurement of the unconfined yield strength (b)

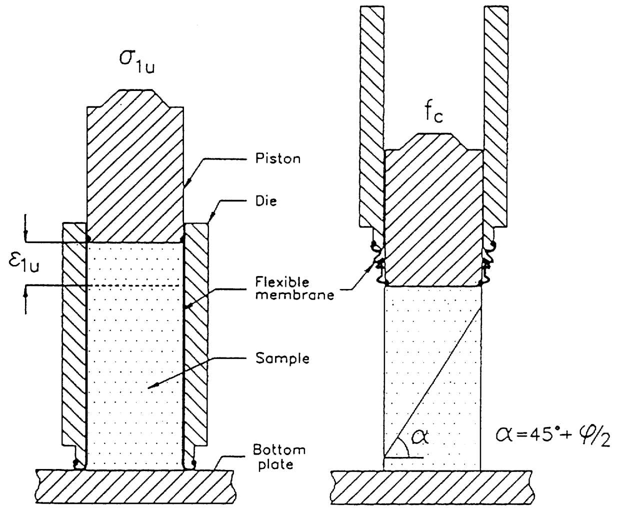

Fig. 41. Uniaxial tester "Postec" [70]

Runge [77,78] used an uniaxial tester to measure the time consolidation behaviour of plastic granules at elevated temperatures. Due to the particle size of 4 mm tests with Jenike's shear tester were not possible. The increase in strength with temperature and time was enormous. Due to the simple test and the easy way to manufacture diametrically split cylindrical cells many cells could be built and many tests could be performed. For investigating the time consolidation at elevated temperatures weights were placed on top of the cells and the cells were stored in an oven for the preselected times.

With the aim to measure caking properties Akzo [79] is using a uniaxial tester shown in Fig. 42. The tester consists of an inner cylinder, closed at the top, with an outer cylinder resting on a pin in the inner cylinder. After filling the bulk solid to be tested in the outer cylinder,

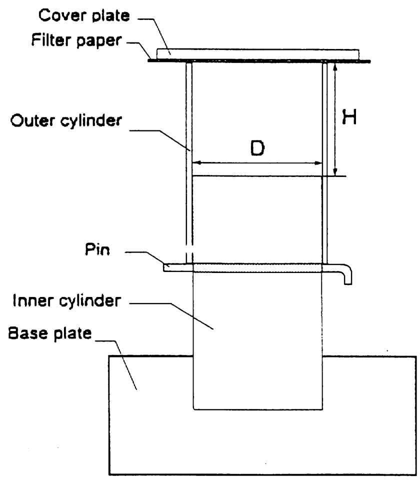

Fig. 42. Caking tester "Akzo" [79]

this cylinder is closed at the top by a filter paper and a perforated cover plate. After putting weights on the cover plate, the outer cylinder is held in a fixed position and the pin is carefully removed. The outer cylinder is released, allowing the sample to be compacted under the consolidation weights. The air included in the sample can escape through the filter paper. At the end of the consolidation period the weights and the cover plate are removed and the outer cylinder is forced to slide down until it clears the sample completely. Alternatively a splittable cylinder with 4 parts can be used. Afterwards the unconfined yield strength will be measured by carefully putting weights on the sample.

In conclusion the uniaxial tester can be regarded as a simple tester. Due to its simplicity the results cannot be very accurate. It underestimates the unconfined yield strength and overestimates the flowability. The consolidation by a vertical force only does not guarantee an homogeneous compaction, as it will be achieved at "preshear" in shear testers due to steady state flow. Thus some scatter in the results have to be expected. The uniaxial tester can only be used when cohesive bulk solids are tested which guarantee a stable sample after consolidation and removing the cylinder. For the same reason no tests are possible in the low stress region. Advantageous is its use for time consolidation measurements of coarse particles where other shear testers cannot be used.

The Lambdameter, already mentioned and described in chapter 3.2, also belongs to the uniaxial testers, because the state of compaction is the same as in the uniaxial tester. But the purpose of performing tests in a Lambdameter is different. With the Lambdameter or similar testers the horizontal stress ratio λ (= k in the English literature) defined as the ratio of horizontal reaction-stress σ_h to the applied vertical compaction stress σ_v shall be determined. In [22] it is reported on a large number of tests which were performed to investigate all possible influences on the measured λ-values. One result of testing 41 bulk solids was already shown in Fig. 18 as a λ, φ_e- plot with φ_e as the angle of the effective yield locus, characterizing internal friction at steady state flow.

4.6 Other testers

The state of stress in the testers mentioned in chapters 4.1 to 4.5 is known and – with only some minor exceptions – the stresses are evenly distributed across the cross-section or the shear plane in the first step of a shear test - "shear" to steady state flow or consolidation as in uniaxial testers – as well as in the second step – "shear" to failure. Thus, the results of those testers can be used to get the flow function or at least an estimate of a flow function (torsional shear tester and uniaxial testers). The testers mentioned and described in this chapter claim to give reliable and reproducible results. Since the state of stress in these testers is not exactly known, at least not in the second step of a shear test, the results of those tests cannot be used without further assumptions to get a flow function and thus their results cannot be recommended for silo design.

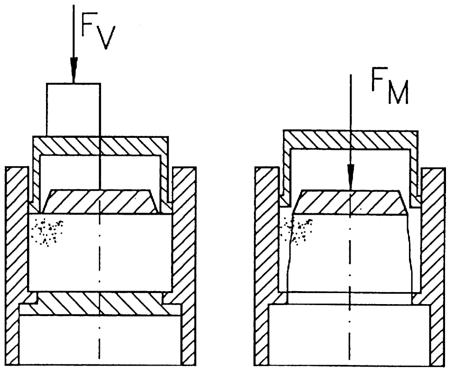

Fig. 43. Johanson Hang – up Indicizer®

4.6.1 Johanson indicizer and similar testers

The principle of Johanson's Hang-up Indicizer [80,81] is shown in Fig. 43. A cylindrical specimen is compressed in axial direction via a piston consisting of two concentric areas. Subsequently the lower piston is removed and the inner part of the upper piston pushes on the sample until failure occurs. From failure force F a strength σ_c an be computed. Because of the cylinder wall friction the vertical stress σ_v during consolidation (consolidation stress $\sigma_{1,c}$) decreases downwards in a mode, which depends on bulk solid and wall properties (horizontal stress ratio λ and coefficient of wall friction $\mu = \tan\varphi_w$). Neither the stress distribution nor the average stress is known. During the measurement of the strength the vertical stress decreases downwards towards zero (free surface) and the sample is subjected along its height to a variable stress. Consequently the calculated strength is an "average value", which results from various consolidation and stress levels that are not clearly defined. Besides others the "average value" depends on the wall friction and on the height/diameter – ratio [82]. The latter was shown by experiments [72]. After the first paper on the Hang-up Indicizer [80] many comments were published [82–84], which are worth to be read.

Comparative tests, using the Hang-up Indicizer and Jenike's shear tester, were performed by Bell at al. [62,63] and Marjonovic et al. [85,86] and the results were included by Schulze [58] in his figure comparing the results gained with his ring shear tester with the certified flow function of the BCR limestone CRM-116, fig. 34. The comparative tests clearly show that the unconfined yield strengths σ_c, gained with the Hang-up Indicizer, are likely to be lower in comparison with Jenike's shear tester and the ring shear tester. Even if the stresses were homogeneous and known during consolidation and failure this was to be expected since no steady state flow can be achieved during consolidation.

According to Johanson [72,82] the unconfined yield strength σ_c can be calculated from

$$\sigma_c \approx 2.2 \cdot \tau_s \approx \frac{2.2F}{\pi H \frac{D_u + D_e}{2}}$$

F is the measured failure force, H is the height of the sample and D_u and D_e are the diameters of the inner upper piston and the lower piston. As far as known, Johanson has never published any concept of the state of

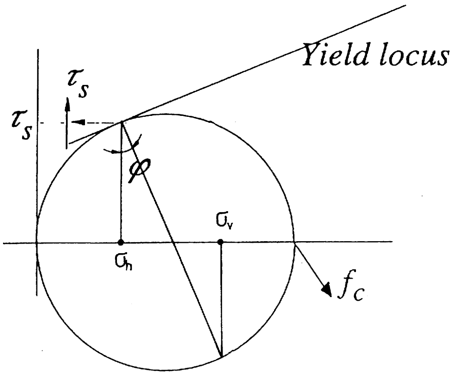

Fig. 44. Unconfined yield strength σ_c (=f_c) from the shear test at failure τ_s according to Johanson [72]

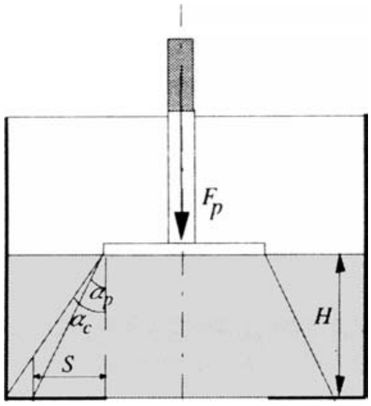

Fig. 46. Failure in the caking tester of van der Kraan [72]

stress in the Hang-up Indicizer at failure. In an attempt to close this gap van der Kraan [72] reconstructed Johansons ideas. τ_s is the average shear stress at failure acting on the sample's cylindrical surface in vertical direction. To get the factor of 2.2 an angle of internal friction of $\varphi = 25°$ has to be assumed (Fig. 44 [72]). From this analysis it seems that Johanson assumed the same angle of friction for all bulk solids. This is in line with a horizontal stress ratio $\lambda = \sigma_h/\sigma_v = 0.4$ (λ = k in the English literature), often suggested by Johanson in other publications [87]. Figure 44 probably presents the assumptions made by Johanson. The shear stress τ_s is vertical and the normal stress σ_h horizontal. The only unconfined sample surface in the tester is located at the bottom of the sample being the only area where the unconfined yield strength will act. Thus the direction of the unconfined yield strength σ_c must be horizontal and not in the direction as it is suggested by Fig. 44. This shows that the probable state of stress that Johanson uses to calculate σ_c does not represent reality, but is a simplification of a more complex state of stress [72].

Similar testers were proposed by van der Kraan [72, 88] and Bates [89] and were used e.g. also by Bell et al. [62]. Figure 45 shows a schematic view of the Bates tester. The main difference to the Hang-up Indicizer is the small H/D-ratio, which, similar to Gerritsen's experiment (Fig. 40), guarantees a uniform consolidation. The wall friction influence is reduced. Due to van der Kraan [72] for H/D-ratios smaller than 0.3 the ratio between the force measured at the bottom of the tester and the force applied at the top of the sample is larger than 0.8. Van der Kraan [72] performed many tests in which he varied the H/D-ratio, the D_o/D-ratio and the D_p/D_o-ratio (Fig. 45). He could show that the major part of the applied force F_p during the failure experiment is transferred through a conical part of the sample with angle α_p (Fig. 46). An angle of $\alpha_p = 30°$, used in soil mechanics [90], seemed to be a good assumption. In conclusion, van der Kraan found that the tester is most sensitive, when the ratio $D_p/D_o \leq 1$. As long as the angle α_p is smaller than the angle α_c, following from geometrical data (Fig. 46), there is no influence of the wall of the tester on the failure experiment and the measured unconfined yield strength σ_c is not dependent on the diameter D_o, as reported by Bates [89], who also assumed a vertical shear plane, which due to van der Kraan is not correct.

Many processes involving the initiation of flow in bulk solids are related to compaction of the powder under action of their own weight. Testers in which the sample is uniaxially compacted are more valid to describe this situation than shear testers, which start with steady state flow not occurring in the mentioned processes. Thus the test procedure used by Bates and van der Kraan gives reliable answers in the optimized form of van der Kraan if these processes have to be judged. As van der Kraan said very distinctly, the tester is a good device for quality control and cake testing, but not suitable for the determination of silo geometries.

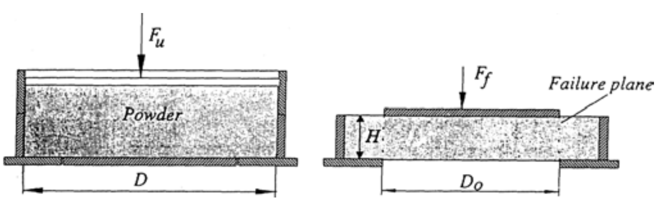

Fig. 45. Uniaxial Direct Shear Tester according to Bates [72, 89]

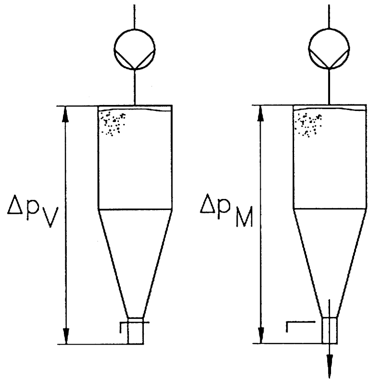

Fig. 47. Quality control tester

4.6.2
Jenike & Johanson quality control tester

The Jenike & Johanson Quality Control Tester is shown schematically in Fig. 47 [3,4,91]. A silo model made from Plexiglas is filled with the bulk solid. The outlet is closed with a porous slide gate. The bulk solid in the model is compacted by an air stream flowing from top to bottom. The pressure drop Δp_V across the bulk solid column is measured and considered as the consolidation pressure. In order to measure the strength of the bulk solid, the silo model is again subjected to an air pressure after opening the slide gate. The air pressure is gradually increased until flow of the bulk solid is initiated. The maximum pressure drop Δp_M is judged as a measure of bulk solids strength.

Ploof and Carson [91] report on comparative measurements with this tester and Jenike's shear tester. They have investigated four bulk solids and plotted $\Delta p_M = f(\Delta p_V)$ (Quality Control Tester) and $\sigma_c = f(\sigma_1)$ (Jenike's shear tester) in the same plot. For two bulk solids they got an astonishing good correlation, for the other two not. Here the results with the Quality Control Tester exceeded the shear test data by a factor of more than 2. Plotting of the results of both testers in one graph was done without any justification and it will be hardly possible to explain that there can or must be a coincidence. The pressure drops Δp_V and Δp_M are integral values being influenced by the different situations along the height of the column (different local air velocities and hence different local pressure drops at least in the hopper), whereas the dependence of the unconfined yield strength σ_c on the major principle stress σ_1 reflects the situation at only one height in the hopper. Thus, only qualitative statements can be expected when using this tester.

4.6.3
Penetration test

Knight and Johnson [92] introduced a penetration test, shown principally in Fig. 48. After compaction of a bulk solid sample by a consolidation force F_V, the force F_M is measured for penetrating a metallic cone, having a half-angle of 5°, into the compacted sample. The tests show that the penetration force is increasing parabolically (power of two) with the penetration depth d. The penetration tests were stopped at a given depth. Due to the proportionality $F \sim d^2$ an unconfined compression strength was defined as

$$f_c = a_{fc} \cdot F/d^2$$

a_{fc} was regarded to be a function of internal and wall friction. Comparative tests were performed with the torsional shear tester of Peschl. The results of tests with five bulk solids – actually only two were really different – were plotted in a σ_c versus σ_1- plot (as in Fig. 47). With a factor of proportionality of $a_{fc} = 1.4$ they got a good agreement. As long as the proportionality factor cannot be explained or derived, the coincidence in the results can only be regarded as accidental. Results of shear tests represent failure, whereas the used unconfined compressive strength f_c does not represent failure. It really is neither a strength nor a stress. Only by the measured proportionality of $F \sim d^2$ a quantity was obtained having the dimension of a stress.

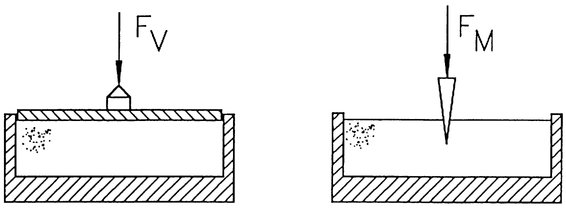

Fig. 48. Penetration test

The authors do not suggest that "penetrometry could replace the use of shear cells when absolute values are required, as in hopper design, but it could be used to supplement shear cell measurements in the quantification of time-induced caking". This seems to be a very optimistic evaluation of the potentials of the penetration test. Personal experience with a similar tester used in industry has shown that the scatter from results of identical tests is big – also mentioned by the authors [92] - and that even a qualitative correlation of time consolidation tendencies can hardly be detected.

4.6.4
Hosokawa tester

The Hosokawa Tester [93] verifies an idea of R.L. Carr [94] who 1965 introduced a scale of 0–100 to characterize the flowability of bulk solids (100: free flowing). In the tester five quantities are measured: the angle of repose, the angle of spatula, a coefficient of compressibility, a cohesion index and an index of uniformity. The measurement of the angle of repose in easy (chapter 4.6.10). The angle of spatula is obtained when a plate impeded in the bulk solid mass is taken upwards: it is the angle of repose at the rectangular plate. The coefficient of compressibility follows from the difference in maximum bulk solid density (after tapping) and minimum bulk density (loosest packing) divided by the maximum bulk density. The cohesion index follows from sieving with three sieves of 74, 149 and 250 μm meshsize for a definite time. With the relative amounts of the bulk solid on the sieves an arbitrarily chosen cohesion index is defined. Finally the index of uniformity characterizes the width of the particle size distribution. For each of the five quantities numbers are appointed to. The sum characterizes the flowability (90–100 : very good; 80–90 pretty good;....; 20–39 : poor; 0–19 very poor).

No qualitative conclusions can be derived from the so defined flowability. It can only be used as a very rough classification of bulk solids behaviour. Regarding the operation of a silo e.g. it is stated, that no flow promoting devices are necessary if the number exceeds 80, that flow promoting devices might be necessary, if the number is between 60 and 80, and that they are necessary for numbers <60.

4.6.5
Powder bed tester

Like the Hosokawa Tester the Powder Bed Tester [95] is able to measure more than one quantity. The set consists of a set of different testers: a parallel plates-type shear tester according to papers of Hirota and Oshima [96,97], a shear cell similar to Jenike's shear cell, a ring shear cell of Walker-type and a tensile strength tester following the Warren Spring idea (see chapter 4.6.8). The description of the four different testers is not very detailed. Thus it cannot be judged, if all the details mentioned in chapters 4.1 and 4.2, which guarantee satisfactory tests in translational and ring shear testers, are fulfilled. For getting a complete yield locus the manual recommends that the tensile strength is measured with the tensile strength tester. It was already mentioned at the end of chapter 4.1 and it will be further explained in chapter 4.6.8 that this type of tensile strength measurement underestimates the tensile strength needed to complete the yield locus in the negative σ-region. The reason is an anisotropic effect between the direction of the consolidation stress (vertical) and the direction of tensile strength measurement (horizontal).

The idea of the parallel plates-type shear tester [95] is shown in Fig. 49. The first step of the shear test – consolidation – is the same as in uniaxial tests. The second step – shear – is similar to that in all shear testers: shear under a normal load up to the maximum shear load. If the maximum shear stress is plotted versus the respective normal stress, a dependence comparable to a yield locus is obtained with the exception that the endpoint and thus the major consolidation stress σ_1 are unknown. In order to get the same yield locus in a Jenike-type shear tester with a silicone powder (5.5 µm diameter) the uniaxial consolidation stress had to be increased up to 24.5 kPa in the parallel plates-type shear tester, while the normal stress at steady state flow in the Jenike test was only $\sigma_v = 4.9$ kPa, yielding a major consolidation stress of 8.4 kPa.

It might be interesting and it seems worthwhile, to compare the results of an uniaxial tester - $\sigma_c = f(\sigma_{1,c})$ - with results of the parallel plates-type shear tester. Since the directions of consolidation stress and unconfined yield stress are identical in the uniaxial tester, but are inclined to each other in the parallel plates-type shear tester, it is to expect that the unconfined yield strength measured in the uniaxial tester exceeds that of the parallel plates-type shear tester. Nevertheless, the parallel plates-type shear tester has the advantage, that measurements are possible at low consolidation stresses, that bulk solids can be tested having only a small or no cohesion and that measurements can be performed if only a small amount of the bulk solid is available.

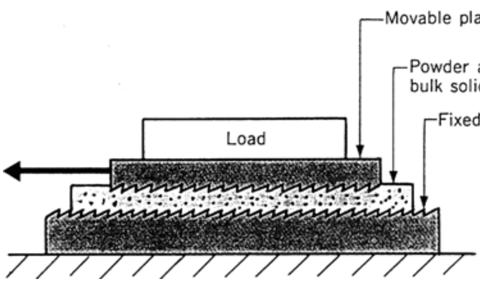

Fig. 49. Powder bed tester [95]

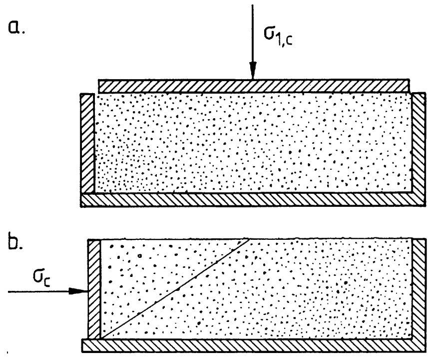

Fig. 50. Monoaxial shear tester

4.6.6 Monoaxial shear tester

The monoaxial shear tester, as proposed by Peschl [98] is shown in Fig. 50. Like in uniaxial tests the sample is consolidated in vertical direction by a consolidation stress $\sigma_{1,c}$. The state of stress is well known and sufficiently homogeneous. After consolidation the lid is removed and the sample is stressed horizontally with an increasing stress up to failure. The equivalent state of stress in the sample is not homogeneous (free surface at the top) and not known. Another disadvantage of this procedure is the direction of stress application, being perpendicular to the one at consolidation. Due to the anisotropic behaviour of uniaxially consolidated samples, a smaller unconfined yield strength will be measured, compared to the results of uniaxial tests as described in chapter 4.5. The test realized in the monoaxial shear tester is similar to the procedure III used in chapter 2.3, Figs. 10 and 11, to describe the anisotropic behaviour of consolidated bulk solids.

4.6.7 Cohesion tester

Cohesion of a bulk solid is its resistance to shear (shear strength) in the absence of a normal stress acting on the plane of failure. Looking at the yield loci of Figs. 2, 3 and others it is the intersection of a yield locus with the ordinate (τ-axis). The method of getting yield loci by performing shear tests was described in chapter 2.1. With the shear tests mentioned, especially those in chapter 4.1, only failure points at positive normal stresses can be obtained. Thus, the yield locus has to be extrapolated towards a normal stress equal to zero to get the cohesive strength.

To avoid the extrapolation a cohesion tester was built at Warren Spring Laboratory in Stevenage, UK. A principle used in soil mechanics was applied, but at a much lower stress level. The Warren Spring tester was further refined at the University of Bradford and got the name Warren Spring-Bradford cohesion tester (WSBCT) [99]. This tester is also offered by AJAX [100]. The main part of the apparatus consists of a vaned paddle made up of a thin strip of metal around a central tube from the surface of which eight spokes project (Fig. 51). After insertion into a compacted bulk solid sample the paddle rotates an annular ring of bulk solid when a torque is applied via a calibrated spring. When the torque is increased to a value

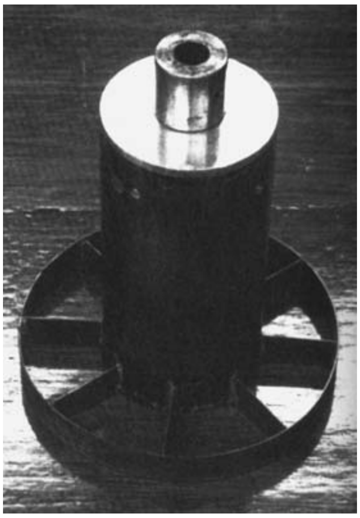

Fig. 51. Vaned paddle of the Warren Spring – Bradford cohesion tester [99]

which is sufficient to cause the annular ring of bulk solid to rotate, it is assumed that the maximum shear resistance is reached simultaneously on each sliding surface. The procedure of performing the test almost ensures that the normal stress acting on the plane of failure is zero. Thus a cohesion is measured. But it has to be questioned if such a cohesion belongs to a yield locus.

For two reasons it has to be doubted. The first step of a shear test is "preshear" with steady state flow (Fig. 3). The first step of the cohesion test is uniaxial consolidation, like in uniaxial testers, with no strain in lateral direction. As a result the Mohr stress circle will be smaller compared to the one at steady state flow (Figs. 4 and 5). As was already explained in chapter 2.2 and lateron in chapter 4.5 that the bulk solid density ρ_b and also the strength will be smaller. This affects the unconfined yield strength as well as the shear strength, being the cohesion. The second reason is the influence of anisotropy. In chapter 2.5 it was explained that bulk solids can behave anisotropically and that this behaviour is only of no influence if the directions of the major principal stresses during consolidation (or steady state flow) and subsequent failure coincide. This is almost obtained in shear tests when following the mentioned "preshear"- and "shear" – steps, but not in the procedure performed in the cohesion tester. During consolidation the major principal stress acts vertically, but at failure it is inclined at an angle of $(\pi/4+\varphi/2)$ to the vertical (φ = angle of internal friction).

Orband and Geldart [99] compare cohesion values which they got directly with their tester or by extrapolation of yield loci after running tests with Jenike's shear tester. The scatter is big. They also state that the bulk solid densities they got from shear tests were higher than the ones in the cohesion tester. This is in line with the explanation in the foregoing paragraph. For the purpose of comparison they propose that bulk densities corresponding to the respective cohesion should be equal. How they really compare the results, does not follow from the paper [99]. Marjanovic et al. [86] also used the Warren Spring-Bradford cohesion tester to enable an interpolation instead of an extrapolation of yield loci to get the unconfined yield strength σ_c. The σ_c-values they got by extrapolation exceeded the ones they got by interpolation, again a proof for the fact, that results of a cohesion tester like the WSBCT cannot be used for a better determination of yield loci.

The WSBCT is a simple apparatus and tests can be performed relatively fast. Thus it has advantages for quality control or qualitative comparisons. A "cohesion" is measured belonging to a special preconsolidation without steady state flow. Since no steady state flow is achieved in preconsolidation, an influence of the "stress history" (chapter 2.4) on the failure experiment remains. Thus the measured cohesion can only be used quantitatively if stress history and failure in the application are identical to the ones in the cohesion tester.

A similar device is realized in a tester offered by Brabender [3,4,101]. Compared to the WSBCT it has the disadvantage, that not a ring of the bulk solid sample is sheared, but a disc of bulk solid. A disc has nearly no shear in the centre and the assumption of getting maximum shear resistance simultaneously cannot be constituted. Additionally, the friction on the perimeter of the disc cannot be neglected and influences the test result in an unknown amount.

4.6.8 Tensile strength tester

In 1965 Ashton et al. [102] proposed an equation to characterize yield loci:

$$\left(\frac{\tau}{c}\right)^n = \frac{\sigma}{\sigma_t} + 1$$

σ, τ are points of a yield locus, c is the cohesion (intersection with the τ-axis), σ_t the tensile strength (intersection with the σ-axis) and n is a parameter, having values between $1 \leq n \leq 2$, describing flowability (1 = free flowing; 2 = very cohesive). The tensile strength σ_t has to be measured to predict parameter n. At Warren Spring Laboratory they used a tester [103], known as the Warren Spring tensile strength tester (Fig. 52). One half of a diametrically splitted cell (similar dimensions as the base of Jenike's shear cell, Fig. 1) is movable in horizontal direction. The consolidation of the sample is uniaxial and similar to the Warren Spring-Bradford cohesion tester described in chapter 4.6.7. To get tensile failure the movable part of the cell is pulled by a wire in horizontal direction without a normal stress acting on the sample. The force at failure divided by the vertical cross-section of the sample

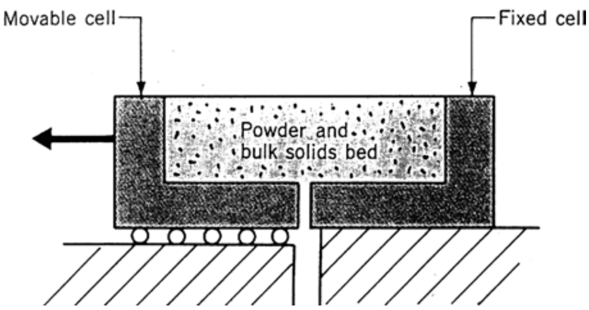

Fig. 52. Warren Spring tensile strength tester

yields a tensile strength. The force is transferred to the sample via wire and wall friction at the bottom and the side walls of the cell. Since bulk solids are not very elastic (high modulus of elasticity), it has to be assumed, that the tensile stress is not evenly distributed along the height of the sample being highest at the bottom. As a result the measured tensile strength (force divided by area) depends on sample height. Measurements at different heights and extrapolation of the results towards zero height might help in getting reliable values.

The principle of the Warren Spring tensile strength tester was used by many others [32,95,104,105, others]. Main differences can only be seen in the way the tensile force is applied: by a prestressed spring, by a constant strain rate device and a force transducer, by adding water in a polythene flask hanging on the wire which is guided by a pulley from the horizontal into the vertical direction or by simply tilting the whole device up to the angle α, where the weight of the movable part times sin α equals the maximum tensile force. As in the case of the cohesion tester (chapter 4.6.7) it has to be doubted, that the measured tensile strength is the correct one for the determination of a yield locus. The consolidation does not result in steady state flow and the directions of major principal stress at consolidation and failure are perpendicular to each other and not identical.

The Warren Spring equation mentioned at the begin of this chapter is often mentioned in the literature. The authors of the equation [102] also offer a physical explanation for the relationship by considering adhesive and repulsive forces at particle contacts. But as far as the author of this report knows the equation was never used for practical applications and the index n is not longer used to characterize flowability. The usefulness of the Warren Spring tensile strength tester can be judged in a similar way as the cohesion tester (chapter 4.6.7). It is a simple tester and tests can be performed relatively fast. Thus it can be used for quality control and comparative measurements. The results can be used quantitatively only if consolidation and failure are identical in test and application.

Besides the well known Warren Spring tensile strength tester other methods were proposed to measure the tensile strength of bulk solids. Schmidt and Walter [106] as well as Schweiger and Zimmermann [107] used a device shown schematically in Fig. 53. The sample container (58 mm diameter) containing a thin layer of the bulk solids sample is moved upwards towards the measuring device, a watch glass covered by white petrolatum as a sticking agent to the particles of the sample. After a short contact time, the container is moved downwards again at a small speed of 0.1 mm/min. The force needed to separate the bulk solid layers is registered (accuracy of the load cell: 0.01 mN). By measuring the contact area a tensile strength can be derived. Figure 54 shows schematically the measured force versus time. "A" represents the weight force of the measuring device. When the watch glass comes into contact with the sample the force decreases until the device lies completely on the sample surface. During the downward motion of the sample container the measured force increases continuously up to failure at "C". "B" stands for the weight force of the device with the adhering bulk solid. Thus the distance "B"-"C" corresponds to the tensile force. With this tester measurements of the tensile strength of loosely packed samples (without any additional consolidation) were performed. Also measurements on preconsolidated samples can be imagined (not yet done). The advantage of this device is the coincidence of the directions of the major principal stress at consolidation and of the tensile stress at failure and a more even distribution of tensile stresses in the cross-section, compared to the Warren Spring tensile strength tester.

In addition to the Warren Spring type tester [108] Kono et al. [109] used the gas flow through a bulk solid sample for consolidation – in direction of gravity – and failure – in opposite direction -. For characterizing the states of consolidation and failure the pressure drops Δp_c and Δp_f are taken. Δp_f is set equal to the tensile strength σ_t. Values of tensile strengths are plotted versus porosity. Results from tests with a Warren Spring type tester are plotted in the same graph. While the Warren Spring type tester can be used only at room temperature and for rela-

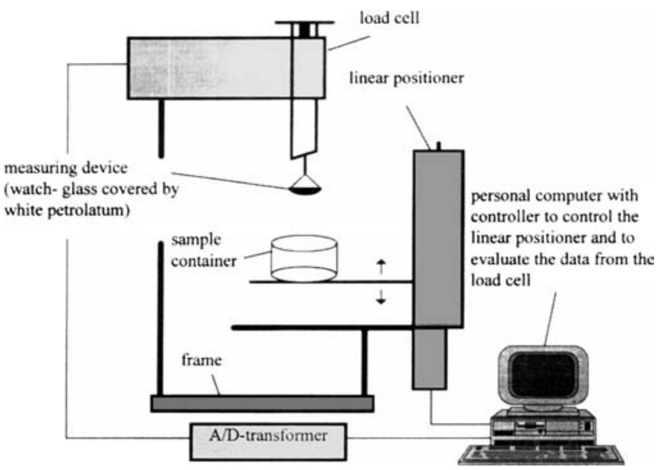

Fig. 53. Tensile strength tester [107]

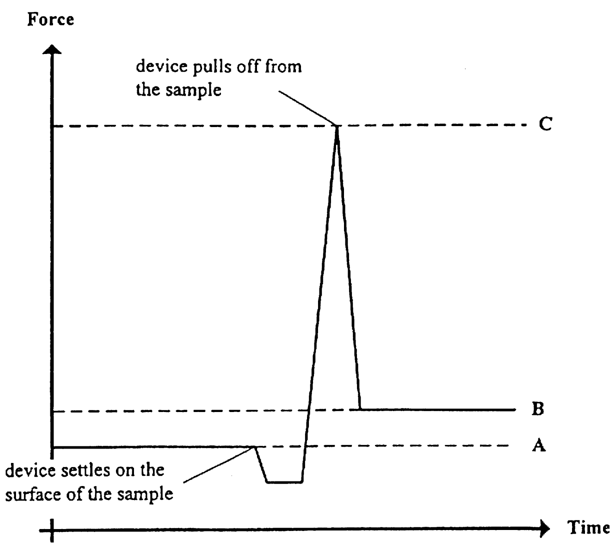

Fig. 54. Force versus time plot of a test in the tensile strength tester of Fig. 53

tively dense packed samples the new method enables measurements at elevated temperatures and for more loosely packed samples. The problem of tensile strength-measurements at elevated temperatures was also investigated by Pilz [110]. He used a Jenike type shear cell – made out of ceramic materials (S_iS_iC) -, performed tests up to 1000°C and tried to get an idea of tensile strength by extrapolating the yield loci into the tensile stress region. The scatter of his results is rather big.

Tensile stresses can also be investigated in fluidized bed experiments [111,112]. If gas flows through a sample of a cohesive bulk solid the pressure drop increases, reaches a peak value and settles to a constant value which equals the weight of the sample divided by its cross-section. According to Valverde et al. [112] the tensile strength σ_t and the consolidation stress σ_c can be derived from such a plot, Fig. 55. By changing the bed height they got σ_c-values from 20 to 180 Pa and measured σ_t-values from < 1 to 16 Pa. It would be interesting to compare results from this method with results from the method discribed in Fig. 53. This would give an idea on the validity of the assumptions made in the tests leading to Fig. 55.

4.6.9 Avalanching behaviour

The angle of repose (see also chapter 4.6.10) is often used to characterize flowability. Independent of the specific method the procedure is always similar. Bulk solid is flowing onto a flat surface and is forming a pile. As soon as the pile is formed fresh bulk solid slides erratically along the pile surface. It slides discontinuously in the form of avalanches. The way avalanches are formed, their sizes and their frequencies seem to be strongly influenced by the flowability of the bulk solid. The dynamic avalanching behaviour of bulk solids was investigated by many authors e.g. [113–117]. As a result of the studies a tester was developed, called Aero Flow [118], the principle of which will be described in the following [114], Fig. 56:

> "The equipment (Fig. 56a) consists of a transparent disc with a port at the front to enable one to insert the powder into the system. A light is shone through the disc onto an array of photocells behind the disc. As the disc rotates slowly the powder builds up to a potential avalanche with the state of the powder in the disc being recorded by the photocells as indicated in Figs. 56(b) and (c). As the point of instability is reached the powder avalanches changing the signal as shown in Fig. 56(c). A series of signals such as those in Fig. 56(c) can be used to create the fractal fingerprint typical of the avalanching behaviour of the powder. The time between the initiation of successive avalanches is used to characterize the avalanching behaviour".

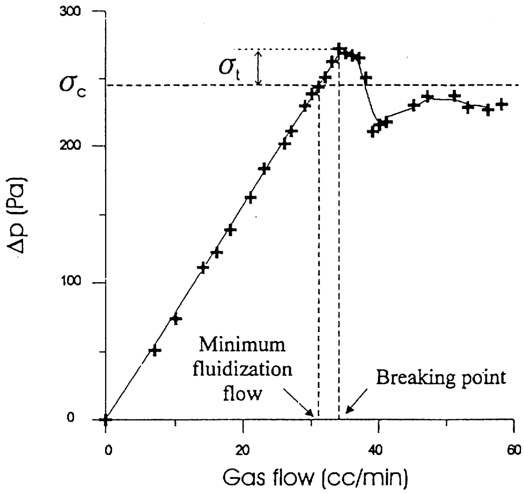

Fig. 55. Pressure drop versus gas velocity in a fluidization experiment [112]

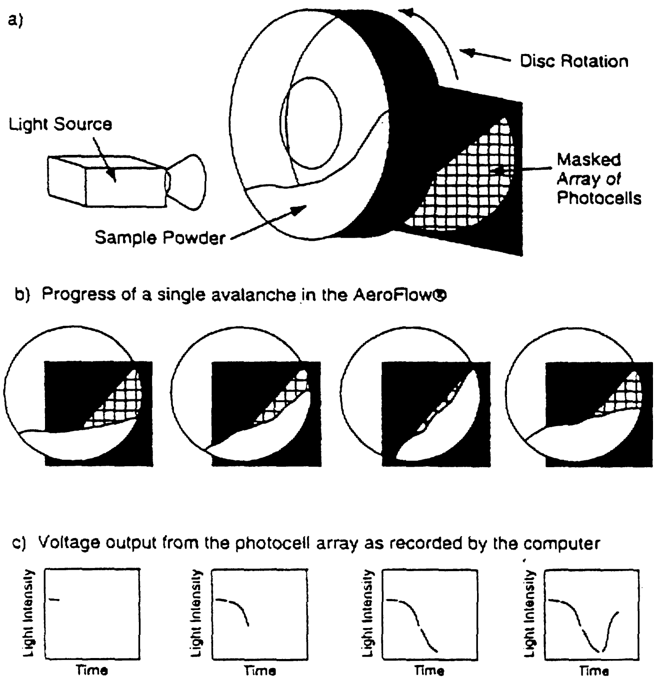

Fig. 56. Rotating disc avalanching equipment [114]

The points of time T_i, where avalanching occurs, is recorded and the times T_{n+1} are plotted versus the times T_n. Figure 57 shows a typical result for a good flowing bulk solid (Fig. 57a) and a poor flowing bulk solid (Fig. 57b). In the graph of the good flowing bulk solid the data points collect near the origin and the scatter is significantly smaller than in the graph of the poor flowing bulk solid. The center of the data points and the mean deviation is used to characterize flowability.

The test does not need an expensive sample preparation, is performed quickly and can be repeated many times. Thus it might be suitable for quality control and especially for comparative measurements, e.g. for comparing different bulk solids or a bulk solid having poor flow properties to which a flow agent shall be added to improve flowability.

4.6.10 Other testers and test principles

Other testers and test principles or procedures exist to measure some value of flowability, but the application of those values, at least in a quantitative and often also in a qualitative mode, is restricted to very special problems. In the following only some methods will be mentioned without claiming completeness. The angle of repose was often mentioned. Only little problems occur when handling free flowing bulk solids. Having cohesive bulk solids the reproducibility gets worse and the measured angle can be influenced by a consolidation; angles exceeding 90° can be produced.

The angle of repose can be measured by many methods. 9 different methods are mentioned in [119], only some of them are shown in Fig. 58. As can be deduced from this figure different results have to be expected even with this simple kind of measurement equipment: A conical heap (a) will yield a different angle of repose α compared to a wedge-shaped heap. If the bulk solid flows out of a container with a central outlet (b), the angle of repose will be higher and will depend on the outlet diameter. Another (smaller) angle α prevails in a rotating drum and it can be distinguished between a static and a dynamic angle. Since no real problems can be solved by only knowing the angle of repose, no further details will be given here. When designing silos, which are filled centrally, the knowledge of the angle of repose allows an estimate of the amount of bulk solid which can be stored in the silo. For those who are interested in further details regarding the angle of repose it is referred to the literature and national standards and codes.

Two simple methods will be mentioned in the following and after describing them it will be explained how little information can be got from results of those experiments: A low container with a bottom plate having many outlets with increasing diameters is filled with the bulk solid while the outlets are closed. After opening the outlets the bulk solid will flow only through the larger outlets. Stables arches have been formed over the smaller outlets. A critical diameter can be gained as a measure of flowability.

In a second test the time is measured that a bulk solid sample needs to be discharged from a funnel. It is assumed that the flowability is increased with a decreasing discharge time. Both methods can be combined and varied in various ways. In all these possible combinations quantities are measured, which are considerably influenced by the material and the geometry of the funnel/container. The significance of these influences is not known and can hardly be predicted. Thus no quantities can be derived, which only depend on the bulk solid.

However, those empirical methods should not be condemned at all. In special applications they can be used, but only if a correlation has been found between the special application and many empirical tests. Often it is attempted to transfer the positive experience of this single correlation to another application. This is not recommended. Again many tests will be necessary to get an equivalent good correlation. But those empirical correlations can only be used for quality control or comparative tests with different bulk solids with regard to the same application.

4.7
Wall friction

To perform a wall friction test with Jenike's shear tester base A of the shear cell (Fig. 1) has to be replaced with the specimen of the wall material (Fig. 59). The test procedure is described in the Standard Shear Testing Technique (SSTT) [2]. Contrary to the determination of a yield locus with Jenike's shear tester it is possible to determine several points of a wall yield locus in one test if constant shear force values are obtained after short shear strains (Fig. 60). The angle between the wall yield locus and the abscissa is the wall friction angle φ_w. Sometimes shear stress – shear strain plots like those shown in Fig. 61 are obtained. "a" is typical for a bulk solid showing slip-stick

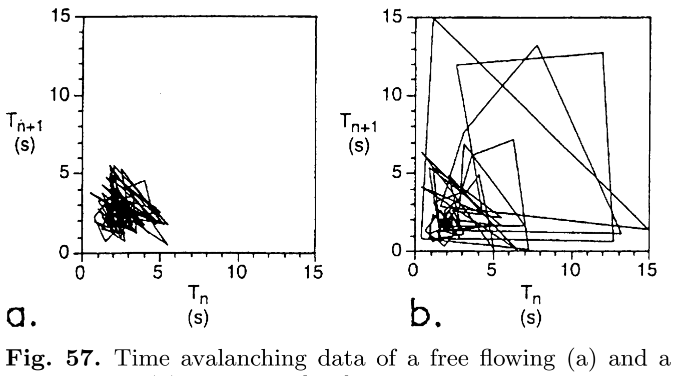

Fig. 57. Time avalanching data of a free flowing (a) and a poor flowing (b) bulk solid [114]

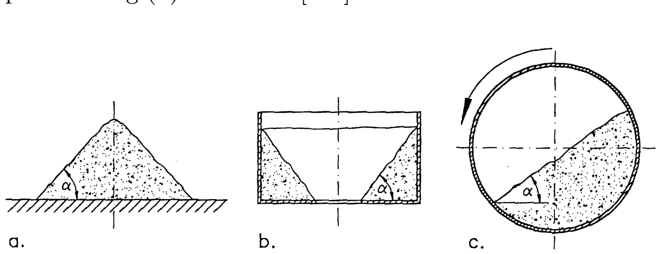

Fig. 58. Angles of repose

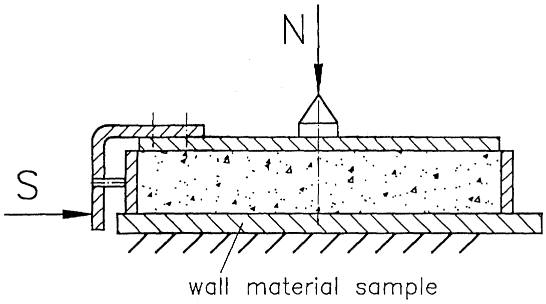

Fig. 59. Determination of wall friction with Jenike's shear tester

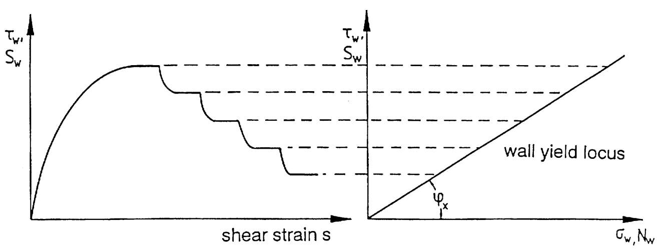

Fig. 60. Determination of the wall yield locus and the angle of wall friction φ_w

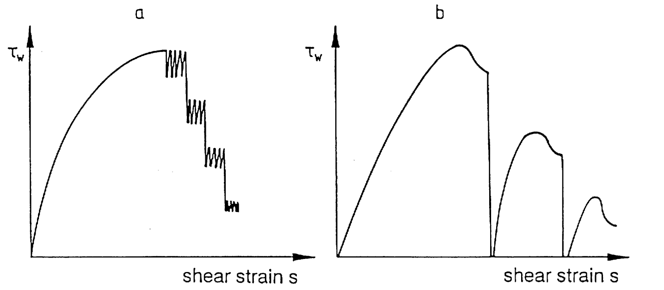

Fig. 61. Typical wall shear stress τ_w – shear strain s – plots

behaviour. When designing a mass flow hopper the larger value has to be taken. The plots in Fig. 61 (b) are typical for plastic coatings on the wall specimen. Here it is sufficient to use the steady state value when designing a mass flow hopper. The wall friction angle φ_w is identical to the angle of inclination of the wall yield locus (Fig. 60). If the wall yield locus is not a straight line through the origin in a $\sigma_\mathrm{w}, \tau_\mathrm{w}$-graph, φ_w is dependent on σ_w and can be determined as a function of normal stress σ_w:

$$\tan \varphi_w = \tau_w / \sigma_w$$

The wall yield locus is often a straight line for metallic surfaces. For plastic coatings the wall friction angle often depends on the normal stress σ_w with the tendency that φ_w decreases with increasing σ_w-values. It is very easy to measure the wall friction angle with a shear tester. It is therefore recommended that a separate measurement of φ_w should be performed for each application instead of taking values from the literature.

All direct shear testers mentioned in Fig. 21, except for the simple shear apparatus, can principally be used for wall friction. Best suited are Jenike's shear tester and ring shear testers. Behres et al. [120] report on comparative measurements with Jenike's shear tester and the new ring shear tester of Schulze. They considered three different variants (Fig. 62): In variant 1 the sample of the wall material is fixed to the lid, in variant 2 the wall material specimen is placed on an intermediate layer in the bottom ring. This experimental set-up resembles that of wall friction measurements in Jenike's shear tester, where the bulk solid is sheared relative to the wall specimen underneath. A disadvantage of variant 1 is that the sample may segregate during the shear process with the result that only the coarser particles having lower wall friction angles are in contact with the wall specimen. But since the fine particles are of importance for the wall friction [121], there is the risk of the measured wall friction angles being too small. Variant 1 was therefore not realized in [120].

Comparative tests (Jenike's tester and ring shear tester, variant 2) have shown that higher wall shear stresses are measured in the ring shear tester, variant 2. It could be shown that this is due to friction between the stationary bulk solid sample and the rotating side walls of the ring or between the stationary part of the bulk solid sample and a rotating part of the bulk solid sample adhering to the wall and resting in the corner to the wall specimen. To avoid an influence of the shear cell wall variant 3 in Fig. 62 was designed. The lid was not changed, but the

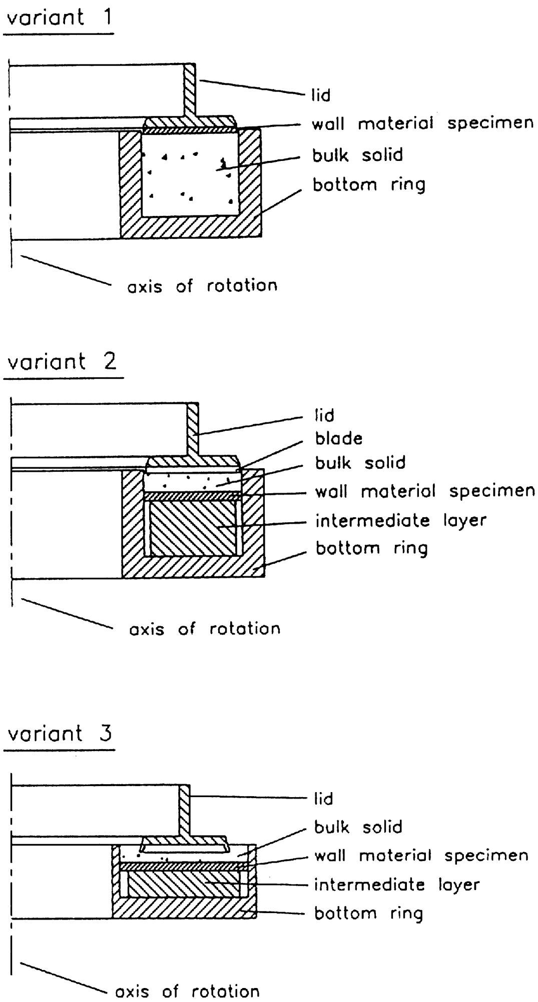

Fig. 62. 3 variants to measure wall friction in a ring shear tester

bottom ring is wider. Thus, a gap of around 10 mm between the lid and the ring is formed. To prevent a lateral overflow of the bulk solid from the area underneath the lid, the lid received additional lateral boundary walls at the level of the blades. Results from tests with variant 3 agree well with those from Jenike's tester, not only the mean values, but also the standard deviations of at least 4 repetative tests.

Another method for measuring wall friction was presented by Haaker [122,123], Fig. 63. A flat belt conveyor with a rough belt withdraws the bulk solid from a silo. A horizontal fixture, with the wall specimen attached to its underside, rests on the moving bulk solid. The normal stress on the wall specimen is exerted by dead weights through a weight hanger. The sample plate is attached to a force transducer, which measures the horizontal shear force. This device also permits measurement of wear of the wall specimen as a function of time or displacement.

Since the wall friction angle is an important quantity in silo design, it is of interest whether and how the wall friction angle is changing with time. Figure 64 (Haaker's design [122]) shows that the coefficient of wall friction $\mu_w = \tan \varphi_w$ may change in various ways. The

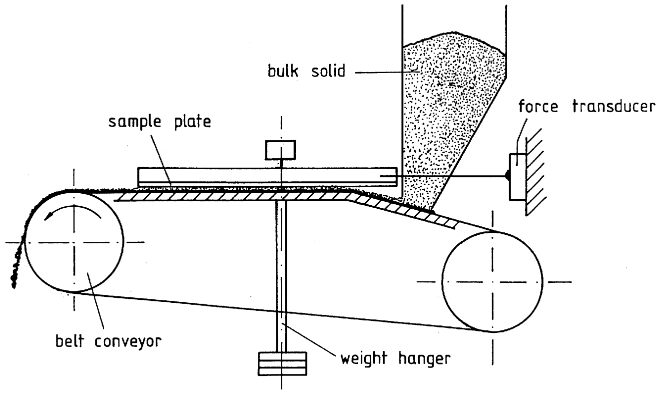

Fig. 63. Wall friction tester of Haaker [122]

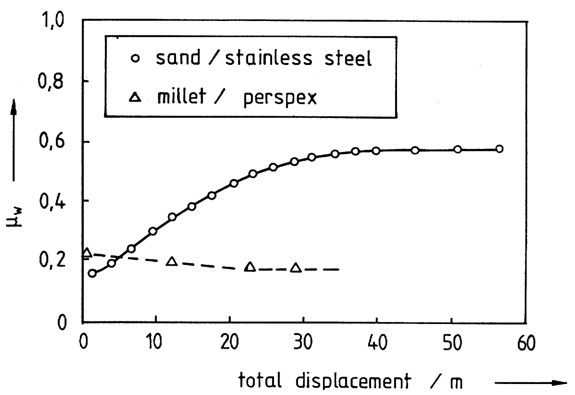

Fig. 64. Wall friction coefficient : μ_w versus total displacement

combination sand/stainlees steel shows an increase, while the combination millet/perspex shows a decrease in the wall friction coefficient. Wear tests (wear of the wall specimen) and attrition tests (attrition of the bulk solid) can easily be performed in a ring shear tester due to its unlimited strain. The expense is lower compared to Haaker's tester. A machine where attrition can be tested is also offered by AJAX [124].

Scott and Keys [125] had to measure wall friction of different coal fractions with particle sizes of up to 30 mm. Following the condition that the cell size should be at least 10 times the largest particle size they used a cell size of 308 mm diameter and a maximum height of 80 mm. They built an inverted test cell with the wall specimen at the top in a fixed position. The bulk solid sample is pressed from underneath against the wall specimen thus allowing very low normal stresses. Comparative tests, using bulk solids with particles sizes <4 mm, in the large wall friction testing machine and in a Jenike type shear tester show that for similar combinations of bulk solid and wall specimen the wall yield loci are essentially the same.

In most wall friction testers the shear force is applied at a constant strain rate in horizontal direction (translational testers) or circumferential direction (rotational testers). As an alternative translational testers can also be tilted up to an angle α against the horizontal direction, at which the sum of the weights of shear cell, shear cover and content and additional weights times sin α equals the friction force between the bulk solid and the wall specimen [85,126]. Those tests are easy to perform, but allow only the measurement of peak values. Plots as shown in Fig. 61 cannot be achieved. The determination of a wall yield locus in only one test, as shown in Fig. 60, is also not possible.

The correct evaluation of wall friction tests depends on the application [20]. The process engineer considers the flow inside and out of the silo. For his calculation he has to take the largest wall friction angle to guarantee mass flow. The stresses in the outlet area influence the flow pattern. In this area the stresses are smallest. The civil engineer has to design the strength of the wall. In doing this he has to consider the largest stresses, prevailing in the vertical part of the silo. The stresses increase as the wall friction angle decreases. Therefore the civil engineer has to take the smallest wall friction angle for his calculation.

Only if the wall friction angle is independent of the normal stress and is not influenced by other parameters, the process and the civil engineer can use identical values. This identity is a theoretical case which does not exist in reality. There is always a scatter in experimental results. A similar scatter exists in the silo as well. Every engineer uses safety margins in his design to be sure to be on the safe side. For the process engineer the safe side is the largest measured wall friction angle whereas the smallest measured value is the safe side for the civil engineer. Figure 65 shows the results of wall friction measurements of wheat flour against two different plastic coatings [20]. The shear stress τ_w is plotted versus the normal stress σ_w. For both coatings a range is indicated in which the results lie. The wall friction coefficient $\mu = \tan\varphi_w$ is defined as the ratio τ_w/σ_w. It can be seen from the plot that these values depend strongly on the normal stress σ_w.

For the mass flow decision the process engineer uses the larger value at a smaller stress whereas the civil engineer uses the smaller value at a larger stress. The table shows the values at a stress of 1 kPa which might occur near the outlet and at a stress of 5 kPa which might prevail in the vertical part. The wall friction angle near the outlet has values of $\varphi_{w,\max} = 33°$ and 25°. They are 14° and 11° larger than the smallest values at high stresses, $\varphi_{w,\min} = 19°$ and 14°.

Wall specimen	$\varphi_{w,\max}$	$\varphi_{w,\min}$
Plastic coating 1	33°	19°
Plastic coating 2	25°	14°
German Code 1055, part 6	–	14°

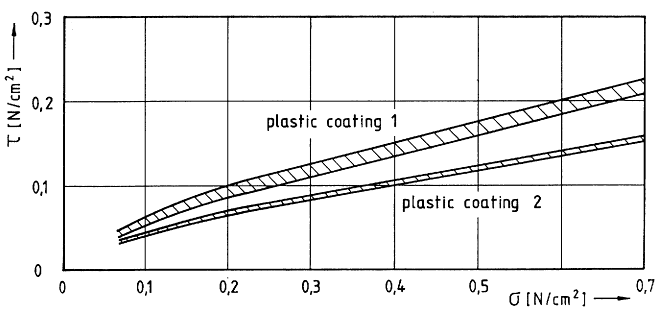

Fig. 65. Wall friction of wheat flour against plastic coatings

This last value is identical to that value which is included in the German Code 1055, part 6 [127] for the design of silos for strength. The table shows that it is possible to determine wall friction angles with a shear tester, which can be used for the structural design even of very large silos. It is only important to measure the friction angle at the equivalent stresses and to interpret the results correctly with regard to the application.

4.8 Time consolidation

Many bulk solids, especially those with small particle sizes, gain strength when stored under pressure without movement (storage at rest). This time consolidation effect can be measured quantitatively in shear testers which enable the correct determination of yield loci. First the procedure will be explained with help of Jenike's shear tester and later on it will be discussed in which other testers the time consolidation can be tested satisfactory.

To perform time consolidation tests the sample must be prepared in the same way as explained for getting yield loci. In "preshear" the sample is sheared up to steady state flow (constant shear stress τ, Fig. 66). After removing the shear pin, the sample will not be "sheared" directly under a smaller normal stress (Fig. 66a), but will be transferred to a consolidation bench. At the consolidation bench the sample will be stressed by a normal stress being identical to the major consolidation stress σ_1 of the Mohr stress circle of steady state flow (Fig. 2). To protect the sample from external influences, especially humidity effects (drying or moistening), the shear cell is covered and sealed. Several samples should be prepared in this way but consolidated for different times t. After time consolidation the samples are "sheared" in the usual way (plots b in Fig. 66). If the peak shear stress after time consolidation (b) is larger than without time consolidation (a), the strength of the bulk solid is time-dependent; it gains strength. The yield loci with a time effect, called time yield loci, are moved towards larger τ-values in a σ, τ-graph (Fig. 67). Normally it is sufficient to only measure one point of a time yield locus and to draw the time yield locus parallel to the yield locus. For more precise evaluations it is recommended to measure two or three points of a time yield locus.

The unconfined yield strength $\sigma_{c,t}$ after time t is calculated from the major principal stress of the Mohr stress

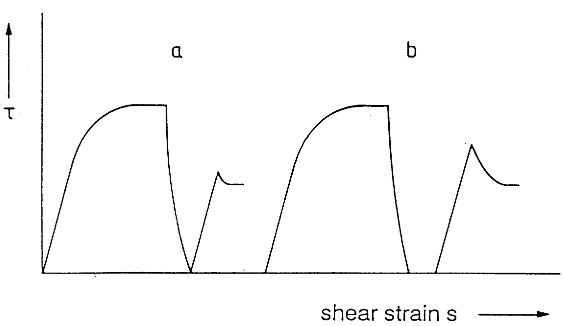

Fig. 66. Shear stress τ- shear strain s – plots a: without time consolidation b: with time consolidation

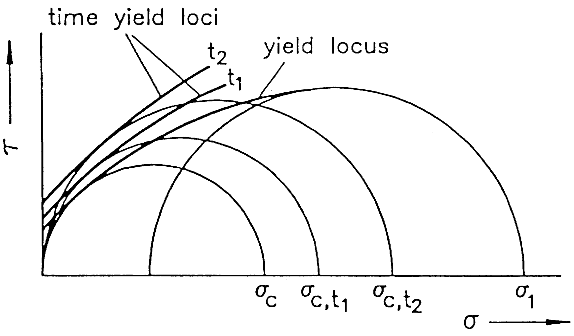

Fig. 67. Yield locus and time yield loci

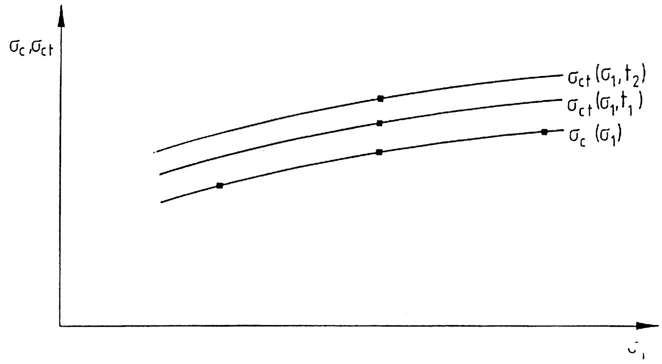

Fig. 68. Flow function and time flow functions

circle being tangential to the time yield locus and going through the origin. Knowing the unconfined yield strength $\sigma_{c,t}$, time flow functions can be derived (Fig. 68). In a simplified procedure it is again sufficient to determine only one point of the time flow function and to draw the time flow function parallel to the flow function. Again it is advisable for more precise evaluations to determine two or three time yield loci with their unconfined yield strengths $\sigma_{c,t}$ for the same time.

The procedure of performing time consolidation tests with the help of a ring shear tester is identical. With the new design of a ring shear tester by Schulze [59] it is nearly as inexpensive as with Jenike's shear cell to manufacture and handle many cells. In principle, biaxial testers can also be used for time consolidation, but it is not advisable because only one test can be performed and during time consolidation the tester cannot be used for other investigations.

When describing the uniaxial testers (chapter 4.5) it was explained that the states of stress for consolidation and failure are well defined and are homogeneous throughout the sample if wall friction is decreased to a minimum. The disadvantage with regard to the determination of yield loci and the flow function concerns the state of consolidation. Only estimates of the flow function can be obtained (see procedure I in Fig. 11), which underestimate the unconfined yield strength. When testing coarse plastic granules which are free flowing at room temperature, but which gain strength when stored under pressure at higher temperatures, the Jenike shear tester cannot be used and also the ring shear tester has deficiencies. Thus the uniaxial tester is the best alternative [77,78]. The scatter in the results of time consolidation tests is significantly larger than in tests without time consolidation. The relatively small deviation between the flow function and the dependency $\sigma_c = f(\sigma_{1,c})$ of procedure I in Fig. 11 might therefore not be of very big importance.

The situation is different in the application of the torsional shear tester for time consolidation tests. The disadvantage of the torsional shear tester with regard to the determination of yield loci and the flow function concerns the second step of the shear process, "shear" up to failure. This disadvantage exists also after time consolidation and its influence can hardly be estimated.

Some of the testers mentioned in chapter 4.6, especially the Johanson Indicizer, the caking tester of van der Kraan, the cohesion tester, the tensile strength tester and the monoaxial shear tester, in principle can all be used for time consolidation tests. Adequate time tests will yield an increase in strength, when the bulk solid has a time dependent behaviour. Thus the testers can be used for quality control and comparative measurements also with respect to time consolidation, but one has to keep in mind, that all the deficiencies mentioned in chapter 4.6 still exist. The results from time tests cannot be safer than the ones without time consolidation.

5 Special bulk solid properties

Testers for measuring flow properties were described in chapter 4. Advantages and disadvantages of the testers were discussed, also with respect to special bulk solid properties. In this chapter these special properties will be mentioned first and it will then be shown which testers are best suited for those special bulk solid properties.

5.1 Coarse particles

The standard Jenike shear cell has a diameter of about 95 mm; ring shear cells normally have a width of about 50 mm; the diameter of a torsional shear cell is even smaller. If bulk solids, which contain coarse particles, have to be tested, it is advisable to remove the coarse particles before testing. Two reasons have to be mentioned for doing this. Coarse particles can be squeezed between base and ring in Jenike-type shear testers, when the ring moves relative to the base. Forces caused by this squeezing action influence the overall shearing force by an unknown amount. This squeezing cannot occur in ring shear testers.

The second reason follows from the assumption that the bulk solid behaves as a continuum in the shear cell. The major assumption in continuum mechanics is that the elements forming the continuum – the particles – are very small compared to the volume of the continuum to be handled. In the "Standard Shear Testing Technique" [2] it is stated, that the Jenike-type shear tester is suitable for testing bulk solids having particles with sizes of up to 5% of the shear cell diameter. Coarser particles should be removed. If there are only a few coarse particles, these particles contribute little to the strength of the bulk solid, because they are impeded between the fines across which the bulk solid sample primarily shears. For the standard size Jenike shear-cell particles with sizes $\geq$ 5 mm have to be removed. With regard to the squeezing action mentioned above, 5 mm-particles still seem to be too large. In Jenike's Bulletin 123 [1] it is recommended, to remove particles $\geq$ 2.5 mm.

Neither in the Standard Shear Testing Technique [2] nor in Bulletin 123 [1] the concentration of coarse particles, being smaller than 5 mm or 2.5 mm, is mentioned. Schwedes and Schulze [5] investigated the influence of the ratio of shear cell diameter to particle size on the shear stress at steady state flow. Tests were carried out with monodisperse glass ballotini fractions of 0.5 mm, 1 mm, 2 mm, 3 mm, 5 mm and 10 mm. Three different shear cell diameters were used (66 mm, 93 mm, 132 mm). The results are given in Fig. 69. The shear stress τ at steady state flow under a normal stress of σ = 5.51 kPa versus the ratio D/x of shear cell diameter D to particle size x. The shear stress τ decreases with increasing D/x ratio and levels out for high D/x ratios. This is in accordance with the results obtained with a fine-grained limestone (x_{50} = 4.8 µm) where no influence of the D/x ratio was detected. Hence, a shear cell with a coarse bulk solid leads to realistic results only when using a shear cell with sufficiently large dimensions. Which dimension is the critical one for a specific bulk solid can only be checked by running similar tests.

The tests leading to Fig. 69 were performed with monosized free flowing particles. If those monosized fractions have to be tested regarding their angle of internal friction the conclusions regarding the size of the shear cell mentioned at the end of the last paragraph have to be followed. More often bulk solids having smaller particles and/or wide particle size distributions have to be tested. If the mean particle size is smaller than 1 mm and the particle size distribution is sufficiently wide it is to expect that no size-influence exists when using the standard size shear cell. The largest shear cell of a Jenike-type shear tester, known to the reviewer, was built by Höhne [128]. Comparative tests with a fine-grained limestone between this cell (diameters of 300 and 500 mm) and the standard size showed, that a lot of care has to taken to get similar results. In the large cell it is difficult to get an even distribution of normal stresses and shear stresses in the shear zone (see also Fig. 25 in chapter 4.1).

When removing coarse particles from the bulk solid sample before the shear process it is often mentioned that the result is on the safe side. This is only true if the concentration of coarse particles is large enough to enable

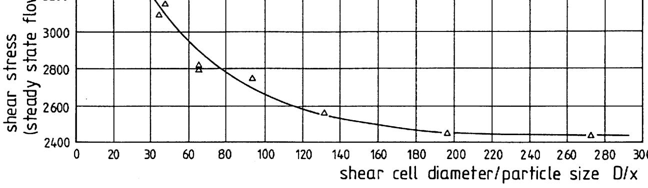

Fig. 69. Shear stress versus ratio of shear cell diameter D to particle size x (glass ballotini, monosized fractions with 0.5 mm <x <10 mm; σ = 5510 Pa)

them to interact. As long as the coarse particles are impeded within the fines as single particles these single coarse particles move bodily while the bulk solid sample shears across the fines. This conception is confirmed by tests of Molerus and Nywlt [129] who tested different mixtures of two narrow, non-overlapping limestone-fractions with mean particle sizes of 3.4 μm and 45 μm. Only with mixtures up to 35% of the coarse fraction an influence of the amount of coarse particles could be detected.

In wall friction tests the influence of coarse particles is significantly smaller since shear takes place between the bulk solid sample and the wall specimen without any relative movement between single particles. Scott and Keys [125] who used a shear cell of 308 mm diameter to measure the wall friction of coal with particle sizes of up to 30 mm stated that for wall friction tests the cell diameter should be at least 10 times larger than the largest particle size (chapter 4.7). It not only depends on the size of the largest particles, but also on the number of large particles being in contact with the wall specimen. Having many coarse particles in contact with the wall specimen it has to be doubted if the shear plane in the tester is still representative of the application. For those cases it is recommended to repeat tests with always new samples, examine the scatter and interpret the results with regard to application. In general it can be expected that the wall friction angle decreases with increasing particle size and that it gets constant for coarse particles.

As was already mentioned in earlier chapters the uniaxial tester can be recommended for testing the strength of plastic granules and pellets (i.e. fertilizer or animal food) with larger particle size, especially if they gain strength when stored under pressure without movement (time consolidation). Also the ring shear tester can be used for this application. The uniaxial tester ensures “reliable” results at low expense. If more precise data are demanded, e.g. for silo design, the ring shear tester has advantages. The use of a Jenike-type shear tester and the torsional shear tester cannot be recommended.

5.2 Elastic behaviour

Elastic behaviour of a bulk solid can be found, if either the particles itself are elastic (plastic or rubber material) or the sample has a very high porosity (=90%) due to very small particle sizes, e.g. Aerosil which is a fine-grained silica with particle sizes of about 10 nm. If a sample of such a bulk solid is sheared in a Jenike-type shear tester, it is hardly possible to obtain steady state flow. Due to the elasticity of the sample the ring of the shear cell nearly moves back into its original position, when the stem for applying the shear force is retracted. In order to get reliable results many preconsolidation tests have to be performed in which the number of twists and the normal stress at preconsolidation have to be determined which ensure steady state flow in “preshear” within the limited strain. Even if steady state flow is obtained during “preshear”, it is difficult to detect a peak shear stress in the subsequent “shear”-process under a smaller normal stress.

Due to its unlimited strain ring shear testers are favourable. No preconsolidation, which requires skill and experience, is necessary and steady state flow can be obtained. If the bulk solid gains strength due to time consolidation, this increase in strength can be detected quantitatively when using the ring shear tester in its new version [18].

The torsional shear tester has advantages compared with the Jenike-type shear tester since steady state flow can be obtained, but there still remains an uncertainty in the correct determination of the unconfined yield strength. When using the uniaxial tester the uncertainty results from the consolidation. Steady state flow cannot be achieved and thus only an estimate of the flow function can be obtained. No comparative tests with an elastic bulk solid in an uniaxial tester and a ring shear tester have been published. Therefore it is not possible to get an idea about the amount of underestimation of unconfined yield strength measured in uniaxial testers. The testers described in chapter 4.6 may be used, but no prediction of the behaviour of elastic bulk solids is possible.

Wall friction tests for elastic bulk solids are easily performed in a Jenike-type shear tester. The shear strain can be enlarged for wall friction tests and the elasticity is of little influence on the shear between the sample and the wall specimen.

5.3 Temperature

In principle all testers mentioned in chapter 4 can be used to measure flow properties at elevated or low temperatures. Either the whole apparatus has to be placed in a heated or cooled chamber or the prepared shear cell containing the bulk solid sample has to be brought to the test temperature in an oven or in a cooler. In the latter case a very quick shear test with “preshear” and “shear” is performed. It is also possible to put the cell into the oven (cooler) again between “preshear” and “shear”. Whether this is necessary depends mainly on the temperature difference to room temperature, the heat capacity of the bulk solid and the speed of performing the shear test. No doubt, a test in a compartment at the desired temperature gives the most exact answer, but it is also more expensive. Often it is sufficient to use the other method.

The need for testing flow properties at temperatures other than room temperature follows from the fact that either the bulk solid is produced or handled at a different temperature. Thus it is of interest to know at which temperature the flow properties are worse, because it is sufficient to perform the tests at that temperature. The above mentioned simpler test gives the right answer and more accurate tests are only necessary when the flow properties are worse at a temperature below or above room temperature. Time consolidation also has to be measured or at least checked because it depends on temperature for some bulk solids. There might be better flowability at elevated temperature without time consolidation but with time consolidation the flowability at the higher temperature could decrease.

The highest temperature realized in a shear tester was achieved by Pilz [110]. He manufactured a Jenike-type shear cell, made from ceramics, stored the shear cell in an oven and performed shear tests at temperatures up to 1000°C. His first approach was to "preshear" the sample to steady state flow outside the oven at room temperature and to "shear" it to failure inside the oven at the elevated temperature. In a second approach he carried out both steps of the shear process, "preshear" and "shear", in the oven at the elevated temperature and only by following this procedure he got reliable results.

5.4 Moisture content

The cohesion of bulk solids results from interparticle adhesion forces. In dry bulk solids van der Waals attractive forces are of main influence. If moisture is added capillary forces are generated, mainly due to capillary pressure of liquid bridges in the contact zone of particles. If shear tests are performed with samples of the same bulk solid and increasing moisture content at the same major principal stress the unconfined yield strengths σ_c always follows the trend shown in Fig. 70. If the dry bulk solid is cohesionless due to coarser particles, the curve starts in the origin, but it has generally a similar shape. There is always one moisture content at which the unconfined yield strength is at a maximum. With a further increase in moisture content the liquid partly acts as a lubricant and the resistance to shear is decreasing again. The moisture content leading to maximum strength depends on the bulk solid, its origin, particle size, size distribution and other parameters and cannot be predicted without shear tests. With hard coal as handled in power stations before grinding and burning this moisture content is in the order of 10%; it is nearly 50% with lignite.

Similar to the influence of different temperatures all testers mentioned in chapter 4 are generally capable of detecting the increase in strength with increasing moisture content. It is also expected that they all can detect the decrease in strength if the moisture content exceeds the value of maximum strength. But it may be doubted that all testers measure maximum strength at an identical moisture content. Only if reliable yield loci are determined identical results have to be expected. This is only possible with some of the testers (see chapter 2.6).

Like dry bulk solids also moist bulk solids can gain strength with time consolidation. As already mentioned with reference to the temperature influence, the relative increase in strength with time and also the absolute values can and will differ with increasing moisture content.

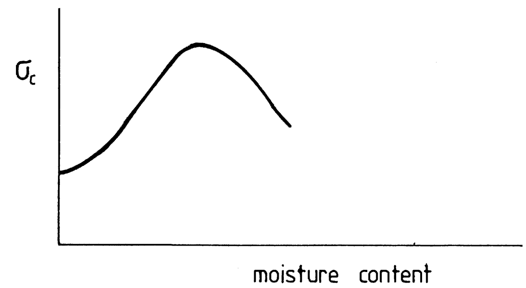

Fig. 70. Unconfined yield strength σ_c versus moisture content

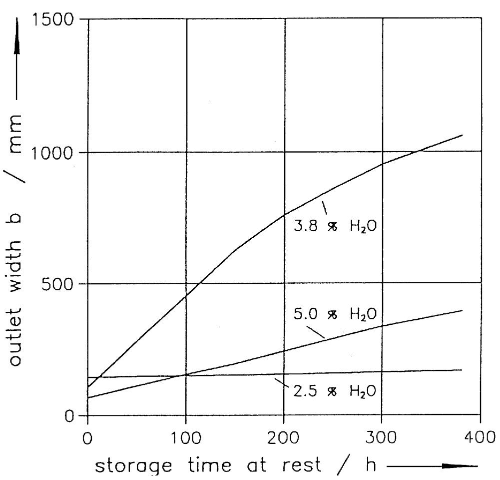

Fig. 71. Minimum outlet width b versus storage time at rest (moist limestone: 0–3mm)

No predictions are possible without running shear tests. As an example the results of shear tests with a moist, crushed limestone (particle size $0 \div 3$ mm, moisture content $2.5 \div 5.0\%$) are shown in Fig. 71. The tests were performed to predict the critical width b of a slotted outlet to prevent doming [130]. The critical width b was calculated from the shear and time shear tests due to the Jenike procedure [1,17]. In Fig. 71 this critical width is plotted versus the storage time at rest for three moisture contents. It can be seen that without time consolidation the sample with a moisture content of 2.5% has the highest strength. The increase in strength with time was most pronounced at a moisture content of 3.8%, whereas at a moisture content of 2.5% hardly any time consolidation could be detected. It would be interesting to find the reasons of this behaviour. This however requires research time and is beyond industrial interest, where fast and reliable answers are required.

The described tests are typically performed at constant moisture content and care has to be taken that the moisture content does not change (no drying nor an additional moistening). In reality the moisture content in stored bulk solids can change, because condensation may occur in cooler areas. If the temperature gradient is reversed, a migration occurs in the opposite direction. Johanson [131, 132] reports of temperature differences of up to 40°C in railcars and trucks due to cold nights and sunny days. Due to those temperature cycles moist bulk solids gain more strength compared to a time consolidation at a constant moisture content. If the bulk solid is soluble in water (salt, sugar, ..) solid bridges might form at the particle contacts due to moisture migration, drying and crystallization. The strength-increase due to these processes can be several times stronger compared to time consolidation at constant moisture content and in addition it is hard to predict. Johanson [132] claims that this behaviour can be predicted with his Indicizers, but not with any other device. Unfortunately he does not explain how the test has to be performed, which results are to expect and how these results are used to calculate critical diameters.

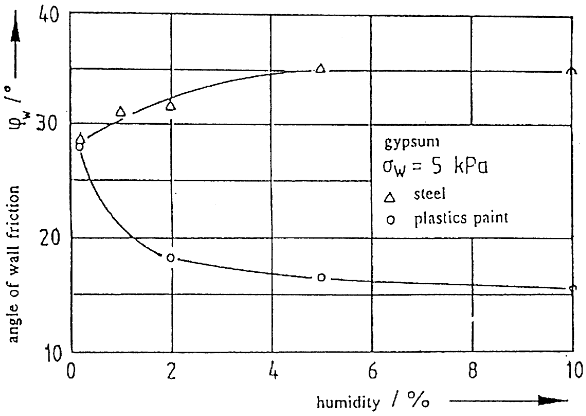

Fig. 72. Angle of wall friction φ_w versus humidity

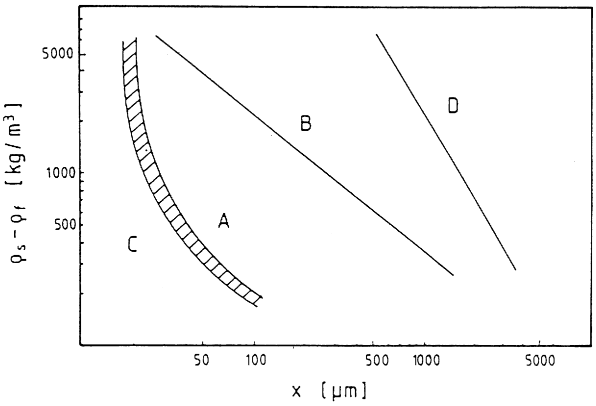

Fig. 73. Fluidization diagram of Geldart

Different moisture contents can also influence the wall friction. Whether an increase or decrease of the wall friction angle occurs with increasing moisture content depends on the bulk solid and more strongly on the wall specimen, especially its affinity to the liquid, hydrophobic or hydrophilic. Figure 72 shows the wall friction angle φ_w of gypsum against stainless steel and a plastic coating as a function of the gypsum's moisture content. No difference can be detected without moisture for the two wall specimens, but with increasing moisture the friction angle is decreasing for the plastic coating, while it increases for stainless steel.

It is often reported that shear tests have to be performed in humidity controlled rooms. That is only partly true. For all bulk solids an equilibrium exists between air humidity and moisture content of the bulk solid. If the equilibrium is disturbed water will evaporate or vapour will condense. This process is time dependent and not very fast as long as there is no relative velocity between the bulk solid and the surrounding air. If no humidity controlled room is available the sensitivity of the bulk solid to humidity change has to be tested first. If a certain moisture content in the bulk solid has to be achieved, the sample should be stored in a small air humidity controlled compartment until equilibrium has been achieved. The following shear test outside this compartment is fast enough and not influenced by the air humidity in the laboratory.

5.5 Flooding

Flooding describes a fluid-like behaviour of fine particles which are fluidized by a counter flowing gas or by entrained gas after extensive shaking. The flooding tendencies of bulk solids are described by Geldart and Williams [133]. They relate the flooding tendency to the fluidization behaviour, expressed in Geldarts well known diagram, where the difference between particle and gas density ($\rho_s - \rho_f$) is plotted versus particle size x (Fig. 73). Bulk solids belonging to group D are coarse and have a low flow resistance and therefore flooding cannot occur. Bulk solids belonging to group B exhibit a higher flow resistance, but this is still low enough to ensure that a fluidized expanded bed settles promptly after stopping the gas flow. Thus there is only a low flooding tendency. Bulk solids belonging to group A are easily fluidized. They exhibit a considerable expansion and need a long time for deaeration and settling after stopping the gas flow. They have a remarkable flooding tendency. Bulk solids belonging to group C are very cohesive. They can hardly be fluidized and tend to form channels through which the gas will mainly flow. But if they are fluidized, they need a very long time for deaeration, compared to group A. Their flooding tendency is lower.

Flooding tendencies cannot be investigated in shear testers, because the samples in shear testers are always in a deaerated and compressed state. The only tester in which flooding might occur and in which flooding experiments could perhaps be performed is the Aero Flow, mentioned in chapter 4.6.9 to investigate the avalanching behaviour. But no reports have been given about such tests.

The problem in handling bulk solids having a flooding tendency results from their high flow resistance. After aeration (caused by counter flowing gas or by self-aeration through intensive shaking) the time for complete deaeration can be very long. As long as the bulk solid is not deaerated, it has fluid-like properties. In the aerated state a gas pressure (surplus pressure against ambient pressure) exists. During deaeration this gas pressure decreases. The way, in which this decrease occurs, can be used as an indicator for flooding intensity.

A simple tester to investigate the deaeration is shown in Fig. 74: A bulk solid sample rests on a gas-permeable

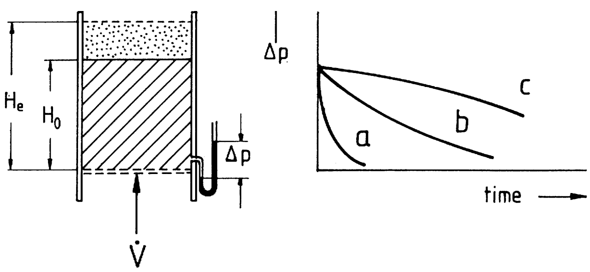

Fig. 74. Test to measure flooding tendencies

distributor plate in a cylinder. The sample of initial height H_O is fluidized by an air flow rate $\dot{V}$ and achieves height H_e in the expanded state. The gas pressure against ambient Δp is measured directly above the distributor plate. After stopping the air flow rate at time t_o the sample starts to settle and the gas pressure starts to decrease. The way how Δp decreases can be taken to classify the flooding behaviour. Sample c has a high flooding tendency, whereas sample a has a very low flooding tendency. Alternatively, the time-dependent decrease in sample height can be taken as an indicator for flooding behaviour, but the tests with measurement of the gas pressure are more reproducible and allow a better classification. At a pressure of $\Delta p \rightarrow 0$ it is expected that the height approaches H_O. But that happens rarely and is another reason to measure Δp instead of H. The test shown in Fig. 74 is used in many laboratories, but it cannot be used to predict the flooding behaviour in an industrial application quantitatively. It only helps to predict which bulk solid will create the biggest problem. No tester or test procedure is known to the author of this report, being able to predict the flooding behaviour quantitatively with respect to application.

5.6 Caking

Caking belongs to time consolidation and describes a very severe increase in strength of a bulk solid being stored under pressure without movement. The aim of investigating caking tendencies is very rarely the design of a silo for flow. Often processes exist in which a bulk solid is compacted under its own weight. Steady state flow does not occur. Such processes are e.g. the storage of sacks or bags on palettes, the storage of large amounts of bulk solids in open storage piles for blending or the storage of bulk solids in silos having a flat bottom instead of a hopper. The compaction of the bulk solid in such applications can best be described by uniaxial compression.

Thus, uniaxial testers (chapter 4.5) and the caking testers of van der Kraan and Bates (chapter 4.6.1) can be used. Their use is recommended compared to the Johanson Indicizer: The states of compaction and failure are well defined and known in the uniaxial testers. In the caking testers of van der Kraan and Bates at least the state of compaction is defined and the state of failure in van der Kraan's tester can be calculated. It is guaranteed that the result of the failure test is not effected by the geometry of the tester, which is the case in Bates tester. In the Johanson Indicizer neither the state of compaction nor the state of failure are well defined and known. Thus only qualitative results can be expected when using the Johansen Indicizer as a caking tester.

The other testers mentioned in chapters 4.6.3, 4.6.5, 4.6.6, 4.6.7 and 4.6.8 can also be used for qualitative caking tests, but only after the testers have been modified for time consolidation. That is easiest for the penetration test, since only additional containers have to be used for the simultaneous compaction of many samples.

5.7 Slip stick

A slip stick behaviour was already shown in Fig. 61 in the plot of wall shear stress τ_w versus strain. Slip stick can occur in internal friction as well as in wall friction. Referring to wall friction it was recommended in chapter 4.7 that the maximum value should be taken for obtaining a wall friction angle φ_w, which has to be used in silo design for mass flow to occur. The situation is not as obvious in internal friction, which is discussed in detail in the PhD-thesis of van der Kraan [72].

Van der Kraan first reviews the papers dealing with slip stick, before explaining what is going on in the bulk solid during slip stick. I refer to van der Kraan's thesis for more details. Figure 75 is taken from his thesis (giving the literature survey van der Kraan has used). Van der Kraan proposes the following explanation for slip stick. In Fig. 76 [72] a typical oscillation of the shear stress during an experiment in Jenike's shear tester is shown. Even though steady state flow is apparently achieved, the shear stress is oscillating although the normal stress is constant. Parallel to the oscillations the state of the sample is changing stepwise from overconsolidated via critical consolidated to underconsolidated (these terms are explained in the introduction to chapter 4). An enlarged section of the shear stress–strain plot is shown in Fig. 77. It is assumed that the shear stress τ_p closely defines steady state flow. Suddenly the measured shear stress drops to $\tau_{p,min}$ (van der Kraan explains this with a sudden release of stored energy). At the same time the sample is compacted, which is confirmed by a measured volume decrease. With respect to the compacted state having a higher bulk density the sample is now overconsolidated because the normal stress has not changed. With increasing strain the shear stress τ is increasing again and has to pass a peak stress $\tau_{p,max}$ at which dilation starts with a decrease of τ towards τ_p at steady state flow, etc.

Looking at Fig. 77 the questions arise which τ-value should be taken as the steady state value and at which instant the stem for shear force application should be retracted. Van der Kraan proposes to take the mean value between $\tau_{p,min}$ and $\tau_{p,max}$ for the determination of a yield locus. This value might underestimate the shear stress at steady state flow, but as long as this shear stress cannot exactly be measured, it is a good compromise yielding a smaller major consolidation stress σ_1 of the yield locus and thus a design on the safe side. The stem for the shear force application shall be retracted – due to van der Kraan – just after the slip has occurred because the compaction of the sample is largest. It might be doubted whether the compaction is really largest at this instant. Simultaneous measurements of shear stress and volume change are necessary to answer this question. After "steady state" has been reached the second step of the shear process ("shear") can be performed in the usual way. The interpretation of that shear stress–strain plot is no problem because the first peak-value is the largest and slip stick starts later at a reduced stress level.

Van der Kraan also performed comparative tests with several bulk solids using Jenike's shear tester, the Delft flexible wall biaxial tester, the Postec uniaxial tester and

Tester	Material	Particle properties			Flowability	Reference
		average particle diameter [μm]	Particle size distr.	Particle shape		
Jenike	Portland cement	coarse and fine	-	-	Cohesive	Schrämli (1967)
	Sugar with calcium stearate	150-425	wide	-	Free flowing	Peleg (1973)
	Wheat flour	65	-	-	Cohesive	Kamath *et al.* (1993a)
Table Model Instron	Different polyester particles	3-17	wide and narrow	irregular	Highly agglomerated to free flowing	Budny (1979)
Triaxial cell	Wheat flour	65	-	-	Cohesive	Kamath *et al.* (1993b)
	Potato starch	5-70	wide	smooth and oval	Cohesive	Gerritsen *et al.* (1980)
	Powdered milk	10-80	wide	-	Free flowing	Gerritsen *et al.* (1980)
	Polystyrene	2000	narrow	-	Free flowing	Kolumbas and Wu (1990)
Rotational shear cell	Coal (Relative humidity > 16.3%)	<800	wide	-	-	Bagster *et al.* (1974)
	Carbonic iron	-	-	-	-	Gebhard (1982)
Uniaxial compaction	Sugar	100-1000	narrow and wide	angular	-	Butler *et al.* (1990)

Fig. 75. Literature survey of slip-stick experiments [72]

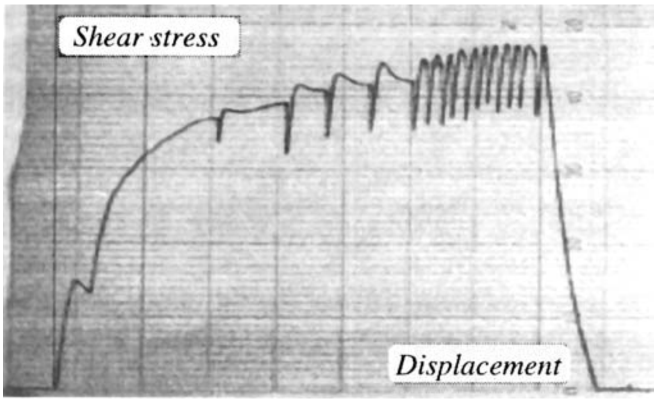

Fig. 76. Oscillation of the shear stress in a test in Jenike's shear tester in order to critically consolidate the sample [72]

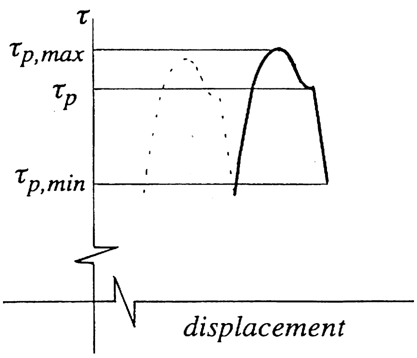

Fig. 77. Shear stress versus displacement in a shear test at "steady state flow" with a sample showing slip-stick behaviour [72]

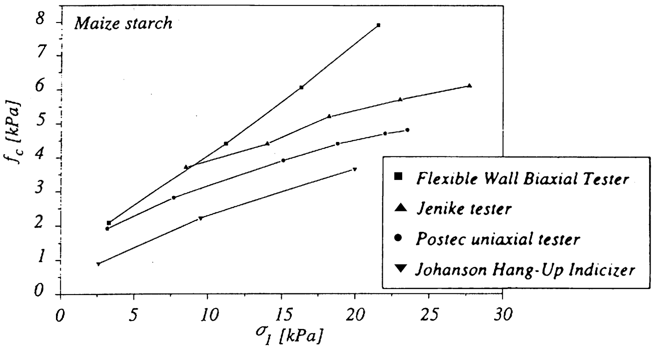

Fig. 78. Flow functions for maize starch (showing slip-stick behaviour) from four different testers [72]

the Johansen Hang-up Indicizer. The flow functions he got for maize starch ($x_{50} = 13.7\,\mu m$) are shown in Fig. 78. The biaxial tester and Jenike's shear tester yield similar results in the low stress region, being of interest for silo design. The results from the uniaxial tester underestimate the strength, being in line with the explanations in chapter 2 and 4.5.

5.8 Attrition and wear

As already pointed out in chapter 4.7 the word "attrition" is used with respect to the bulk solid and the word "wear" with respect to the wall specimen. Attrition means a change in particle size distribution towards smaller values. Attrition occurs, when the applied stress exceeds the strength of single particles. Single particle compression tests up to failure as used in comminution would give the most reliable answer but they are of little use when the attrition behaviour of a bulk solid stored in a silo, in a pile of material or in any other container has to be judged. For those applications it is recommended to perform shear tests. In order to get a significant amount of attrition, a large shear strain is necessary, which best can be obtained in ring shear testers.

It is recommended to perform ring shear tests under different normal stresses to determine the amount of attrition as a function of the normal stress. The applied strain should be large enough to get a measurable amount of attrition, but it has to be the same in all tests under different normal stresses. Those tests can give only qualitative values because the situation in the ring shear tester can hardly be compared to the situation in real applications. But it can be deduced from the results in the ring shear tester, at which state of stress a severe attrition occurs. Transferred to the application it has to be ensured that this state of stress does not occur in the application. In a silo e.g. the maximum stresses due to the Janssen-equation (chapter 3.2) are proportional to the diameter D of the silo and inversely proportional to the wall friction coefficient μ. By choosing or adjusting D and μ accordingly it can be ensured that no attrition takes place in the application. If on the other hand the maximum stresses in an application are known a test in a ring shear tester at these stresses can clearly, but only qualitatively show if a severe attrition has to be expected.

From the testers mentioned in this report only the tester of Haaker [122,123], shown in Fig. 63, and a tester offered by AJAX [124], can be used instead of a ring shear tester. But the tester of Haaker, being designed for wall friction and wear measurements, has to be adjusted to obtain friction between layers of the bulk solid and not between a bulk solid layer and a wall specimen.

6 Comparison of the testers

The testers were described in chapter 4 and partly already in chapter 2. In chapter 5 it was discussed which special properties influence bulk solids handling and which testers are capable of measuring the influence of these properties. As already mentioned in chapter 1 the results of flowability tests are used for different applications, into which an introduction was given in chapter 3. In this chapter the value and the usefulness of the testers for special applications (chapter 6.1) and for quality control and qualitative comparison (chapter 6.2) will be judged. In a concluding chapter (6.3) the results are summarized again in a table following certain criteria.

6.1 Special applications

6.1.1 Design of silos for flow

The first application mentioned in chapter 3 is the design of silos for flow. To get reliable flow the silo has to be designed in such a way that the strength of the stored bulk solid at no time and in no place is sufficient to form stable domes, stable ratholes or other dead zones. The most critical area in a silo is the area directly above the outlet. The bulk solid flowing in the silo from top to bottom is subjected to different stresses. According to these stresses it is consolidated and gets its strength. Above the outlet the flow is converging with decreasing stresses towards the outlet. Due to these decreasing stresses the strength of the bulk solid also decreases towards the outlet. A passive plastic state of stress prevails in this converging flow. At each point within this region steady state flow exists. As already explained in chapter 2.4 only at steady state flow a bulk solid sample looses its memory of the stress history, i.e. that any bulk solid element at a certain location in the convergent geometry is always exposed to the identical state of stress (in steady state flow) and hence has an identical strength independent of the stress history. This is independent of all the possible ways to reach this point.

Closing the outlet and opening it again will decide if the strength of the bulk solid is large enough for doming to occur. The dome is stable if the stress in the dome (parallel to its surface) is smaller than the unconfined yield strength which the bulk solid developed during steady state flow before closing the outlet. To predict the unconfined yield strength shear tests have to be performed. The consolidation in the shear tester must ensure steady state flow, because only steady state flow characterizes the stress history representing the state of stress in converging flow. Having received this stress history ("preshear") samples have to be sheared to failure ("shear") to get yield loci. The Mohr stress circle being tangential to the yield locus and going through the origin yields the unconfined yield strength as the major principal stress of this Mohr stress circle. The minor principal stress is zero because the stress normal to the surface of a dome is zero.

From this explanation it follows that a shear tester, used for the design of silos for flow, has to ensure steady state flow at "preshear" and reliable τ, σ-values in the low stress region at "shear" to get a yield locus, from which the unconfined yield strength can be derived. Steady state flow can be achieved in Jenike-type shear testers, in ring shear testers, in the torsional shear tester and in the true biaxial shear tester, as used in Braunschweig, Delft and Porsgrunn. A reliable value of the unconfined yield strength cannot be derived from tests in the torsional shear tester. The biaxial testers have the additional advantage of being able to measure the unconfined yield strength directly. However, they have the disadvantage of a very high demand for time and equipment. Many bulk solids gain strength when stored under pressure without movement. Time yield loci have to be determined. Again steady state flow in "preshear" is a prerequisite to get reliable values. Many time consolidation tests have to be performed, which can easily be performed with Jenike's shear tester and the ring shear tester in its new version [59]. Thus, only these two testers can be recommended for a reliable design of silos for flow.

Results of the other testers underestimate the strength of the bulk solid (as shown for uniaxial testers and torsional shear tester) or they do not enable a safe quantitative statement, because the states of stress during consolidation and/or failure are neither evenly distributed nor are they known. The Johanson Hang-up Indicizer belongs in this category. Johanson himself has a lot of experience and incorporates all his experience in the evaluation of his test data. But as these calculations cannot be checked, his tester cannot be judged and therefore not recommended for the design of silos for flow.

6.1.2 Other applications

The design of silos for flow is the only known application, where the stress history and its influence on strength and failure is exactly known and can be simulated in shear testers. Shear tests yield the flow function – or time flow functions – describing the (unconfined yield) strength of a bulk solid as a result of consolidation. Consolidation is achieved at steady state flow with the major principal (consolidation) stress σ_1. The minor principal stress follows from the Mohr stress circle being tangential to a yield locus in its endpoint. If a sample is stressed by an identical major principal stress σ_1 but without reaching steady state flow, the minor principal stress will be larger compared to the one at steady state flow. This Mohr stress circle is smaller, the sample is less consolidated and gained less strength. Thus, the highest strength a bulk solid sample can achieve when stressed by a consolidation stress σ_1 results from steady state flow at consolidation. This highest strength is represented by the flow function.

The strength a bulk solid sample gets in a specific application depends on the stress history, i.e. on the way the bulk solid sample is consolidated. If during consolidation steady state flow was not achieved – besides silo flow the most probable situation – the strength will be smaller than indicated by the flow function. The strength of a consolidated sample that was not subjected to steady state flow depends not only on the final stress state of consolidation but also on the stress path, i.e. on the way how the stress was applied. Many stress paths are possible and exist in true applications: uniaxial, biaxial, stepwise, cyclic, mixtures, ... Only if the stress path in an application is known and this stress path is repeated or simulated in a shear tester a shear test can predict the correct strength of the sample.

When discussing caking in chapter 5.6 processes were mentioned in which bulk solids are compacted under their own weight, but without reaching steady state flow. The storage in sacks or bags, the storage in open storage piles for blending and the storage in silos having a flat bottom were mentioned. The compaction in a silo with a flat bottom is similar to the compaction in an uniaxial tester. Thus the uniaxial tester might yield a more realistic value of the unconfined yield strength. The compaction of a bulk solid in a bag is different, because the bag can expand in the horizontal direction. As a result the horizontal minor principal stress decreases and the size of the Mohr stress circle increases which results in larger values of bulk density and strength. For this application the uniaxial tester underestimates the strength. The strength of the bulk solid in the bag is somewhere in between the strength documented by the flow function and the strength following from an uniaxial test. Looking back on Fig. 11 the dependency of procedure I corresponds to an uniaxial test. In open piles for blending an expansion in horizontal direction is also not prohibited. Thus an uniaxial test will also underestimate the strength. Basing a design on the flow function is considered to be on the safe side.

In this chapter up to now only the influence of the stress history was mentioned, but in addition a strong influence of anisotropy exists. Looking again at the design of silos for flow it was demonstrated in chapter 3.1 that the directions of the major principal stresses during steady state flow and during failure or doming nearly coincide. Therefore no influence of anisotropy exists. The anisotropic behaviour after uniaxial compression was also explained (Fig. 11). If the major consolidation stress $\sigma_{1,\mathrm{c}}$ is applied in x-direction and the failure test is also performed in x-direction (Fig. 10), the strength-dependency of procedure I is obtained. However, if the directions of the major principal stresses in consolidation and failure are perpendicular to each other (procedure III) a significant lower strength results. Hence, the strength depends on the direction of stress application.

Coming back to the mentioned storage in a flat bottom silo, in bags and in open piles for blending, the prediction of a strength depends on the direction. For the flat bottom silo the strength in vertical direction is larger than the strength in horizontal direction, following the dependencies of procedure I and procedure III. For the strength of a bulk solid in a bag compacted by bags lying on top, it already was explained that due to a horizontal expansion the strength in vertical direction is larger compared to procedure I but lower than the strength according to the flow function. It is assumed that the strength of the bulk solid in this bag in horizontal direction is even smaller than the prediction from procedure III.

In conclusion, stress history and anisotropic behaviour have a strong influence on the strength of a bulk solid. Only if the stress history and the directions of the major principal stresses during consolidation and failure are known for the application and can be simulated in a shear tester, a reliable prediction of the strength is possible. Possible tendencies can be derived from plots like Fig. 11. The flow function and the time flow functions describe the maximum strength a bulk solid can develop during consolidation. Therefore a design for flow is always on the safe side if it is based on the flow function or time flow functions. The amount of safety can only be checked if the stress history and the directions of stress application at consolidation and failure are known.

A design for flow or the prediction of flowability are not the only problems of interest when handling bulk solids. In chapter 3.2 the design of silos for strength was discussed. It was shown that stresses acting on silo walls can be calculated following the Janssen-equation. The equation contains geometrical terms, the gravity constant g, the bulk solid density ρ_b, the wall friction coefficient μ and the horizontal stress ratio λ (k in English literature). Only the prediction of this stress ratio λ presents a problem. λ as the ratio of the horizontal stress σ_h at the wall to the mean vertical stress σ_v cannot be derived in a satisfactory manner with the help of known properties such as angle of internal friction and angle of wall friction. Thus, the Lambdameter, in which λ can be measured, was designed. This tester is described in chapter 3.2.

If in other applications no satisfactory and proven theoretical description between measured bulk solid properties and application exists, an attempt should be made to measure the required parameters directly in an equivalent tester, as it was done successfully with the Lambdameter.

For the sake of completeness it also should be repeated here – repetition of chapter 3.3 – that shear testers can be used for the calibration of constitutive models. But only those testers can be applied, where the complete state of stress and the complete state of strain can be measured. The biaxial testers described in chapters 2.3 and 4.4 are best qualified, because they allow to perform many different stress – or strain – controlled tests with different strain or stress paths. The states of stress and the states of strain are also known in uniaxial testers, but the homogeneity of the packing is poor compared to the packing in biaxial testers and only two situations can be simulated, consolidation and unconfined yield. In soil mechanics results from tests in a triaxial tester (Fig. 35a) are taken for the calibration of constitutive models. All the other testers mentioned in this report are not appropriate for the calibration of constitutive models.

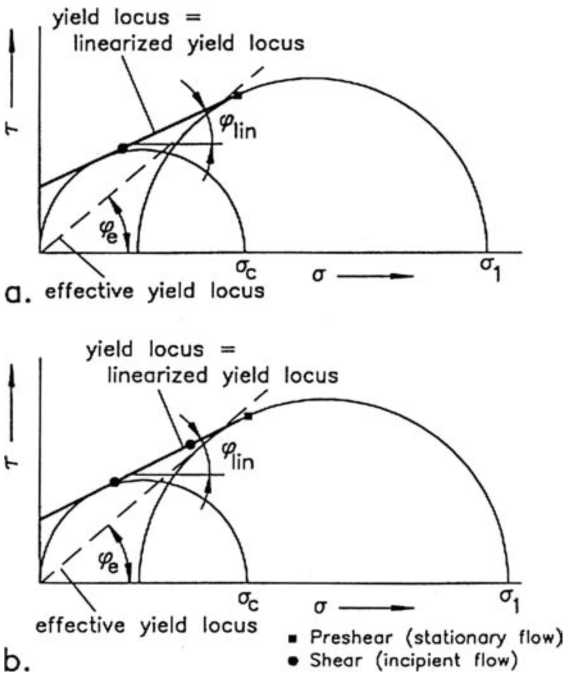

Fig. 79. Yield locus gained with one test (a) or two tests (b)

6.2 Quality control, qualitative comparison

In chapter 3.4 it was already pointed out that very often the testing of bulk solids is not done to design a silo or other solid handling operations. Often it is only of interest, to decide which one out of a number of bulk solids has the best or worst flowability without any need for a quantitative value. Or it is of interest whether the flow properties of a continuously produced bulk solid are changing over time. In this case it is sufficient to use estimates of the flow function, as long as the test procedure does not change from test to test and – with regard to the handling of the bulk solid in further processes – the test is performed at a stress level representative of the application. Testers which can be automated easily and give reproducible results are favourable.

The testers mentioned in chapters 4.1 to 4.5 (Jenike-type shear tester; ring shear tester; torsional shear tester; triaxial, biaxial and uniaxial testers) are often regarded as being too expensive and too time consuming. It is often stated that only well trained personnel can get reliable results. The triaxial and biaxial testers really cannot be recommended here. They have their advantages in research work, where precision is more important than time. For the other four testers (Jenike type, ring, torsional and uniaxial) only the first statement – they are expensive – is correct. They might be more expensive than many of the testers mentioned in chapter 4.6. But if the need is only for quality control or product development, it is also possible to use these testers with a simpler and faster procedure. An estimate of a yield locus can be derived by running only one test (preshear and shear, Fig. 79a) and a repetition test. Therefore, with two tests an estimate of the flow function and thus of the flowability $ff_c = \sigma_1/\sigma_c$ – according to chapter 3.4 – can be found, which is at least as good and reliable as results gained from other testers. If a more precise classification is expected two points of a yield locus can be measured (Fig. 79b); including repetition tests only four points have to be measured.

With the ring shear tester in its new version [18,34, 59] and in principle also with the torsional shear tester [44–46] tests at very low normal stresses are no problem. The Jenike-type shear tester is limited to normal stresses above around 2 kPa to get reliable results (see chapter 4.1). Using the uniaxial tester only cohesive bulk solids can be tested and a minimal normal stress at consolidation is necessary to obtain a sample being stable enough for the failure test. For a very cohesive bulk solid having a flowability of $ff_c = 2$ the necessary minimal consolidation stress is in the order of 10 kPa. It is increasing with better flowability.

If small differences in the flowability of free-flowing bulk solids with a flowability of $ff_c > 10$ have to be detected, tests at very low normal stresses have to be performed [34]. Another advantage of using a ring shear tester or a torsional shear tester, also for quality control, is the fact that for both testers automated versions exist. The test results are now independent of personnel skills. In conclusion, if such testers exist and are used for quality control, they will give the most precise answer about flowability within the analysis time required by any of the other testers mentioned in chapter 4.6.

After this conclusive remark the question arises, whether the testers mentioned in chapter 4.6 can at all be used for quality control and whether they produce results which can be used. Of course they can be used, they will detect differences in flowability and they do not require personnel skill, but it cannot be expected that they are very precise. Without referring to a specific tester it is recommended – before using the tester for quality control or qualitative comparison – to perform comparative tests with representative bulk solids – representative with respect to the special application – either with one of the testers mentioned in chapters 4.1, 4.2 or 4.5 or with regard to the behaviour of the bulk solid in application. Results of comparative tests between a "simpler tester" (chapter 4.6) and a "better tester" will increase the confidence in the use of the simpler tester for routine quality control. Also a more qualitative comparison of the behaviour of the bulk solid in the tester and in application is of help. If a clear correlation between two testers or between the behaviour in the simpler tester and in application was found no reason can be given for not using the results of the simpler testers. But one has to be very careful when transferring positive experiences regarding one bulk solid to other bulk solids. Perhaps additional comparative tests are necessary.

Take again the Johansen Hang-Up Indicizer. It is often used, it is commercially available and results can be produced quickly and without much experience. But the states of stress in this tester during compaction and failure are not homogeneous and unknown. The results are dependent on wall friction and geometrical data. Thus no properties can be achieved which are independent of the tester. For the correct characterization of flow properties it should be the main requirement to get data not affected by the test device. Even for qualitative characterization it has to be assured that the qualitative statement is correct. Comparative tests with different bulk solids and different testers clearly show that the results differ and also that the ranking in flowability is not identical from tester to tester [62, 63].

When using the testers described in chapters 4.6.1 to 4.6.3 and 4.6.5 to 4.6.8 the bulk solids must be cohesive. Only the Hosokawa tester (chapter 4.6.4) and the Aero-Flow for testing the avalanching behaviour (chapter 4.6.9) can test easy and free flowing bulk solids and thus might be capable to detect differences in flowability also for these bulk solids.

It is not the task of the reviewer to make a recommendation, which of the testers of chapter 4.6 should and can be used. It depends on the special bulk solid, on the number of bulk solids to be classified regarding their flowability, on the frequency for the need of test results, on the number of severe problems existing in the handling of bulk solids, on the availability of equipment for measuring properties of particles and bulk solids, on the question, if an equivalent tester is commercially available, if the supplier offers a satisfactory service and background information, etc. If the need for flow testing is low, it is recommended to consult experts, who routinely deal with equivalent problems.

6.3 Summary

In a paper on the "flowability of bulk solids – definition and measuring principles" [3, 4] Schulze has given an overview of various methods used for the characterization of the flowability of bulk solids. He is using the definition of $ff_c = \sigma_1/\sigma_c$, already given in chapter 3.4. Thus σ_1 and σ_c have to be known to estimate the flowability. He also judges the different testers or measuring principles by introducing the following criteria:

1. Consolidation procedure with corresponding measurement of strength
2. Consolidation of the bulk solids sample up to steady state flow
3. Coincidence of the directions of major principle stresses at consolidation and failure
4. Reproducible stressing conditions of the bulk solid sample at consolidation (4a) and failure (4b)
5. Known average stresses and uniform stress distribution in the plane of interest at consolidation (5a) and failure (5b)
6. Possibility for varying the consolidation stresses (with regard to application)
7. Possibility for measuring time consolidation

For quality control and qualitative comparisons criteria 1, 4a/b, 6 and also 7 are important. But for the sake of greater accuracy also the other criteria should be consi-

Testers/Criteria	1	2	3	4	5	6	7
Jenike's shear tester	yes	yes	yes	yes/yes	yes/yes	yes	yes
Ring shear testers	yes	yes	yes	yes/yes	yes/yes	yes	yes
Torsional shear tester	yes	yes	yes	yes/yes	yes/no	yes	yes
Triaxial tester (Fig. 35a)	yes	yes	yes	yes/yes	yes/yes	yes	yes
Biaxial testers	yes	yes	yes	yes/yes	yes/yes	yes	yes
Uniaxial testers	yes	no	yes	yes/yes	yes*/yes	yes	yes
Johanson Hang-up Indicizer	yes	no	no	yes/yes	no/no	yes	yes
Caking tester	yes	no	no	yes/yes	yes/no	yes	yes
J & J Quality Control tester	yes	no	yes	yes/yes	no/no	yes	yes
Penetration test	no	no	no	yes/yes	yes*/no	yes	yes
Hosokawa Tester	no	–	–	–	–	–	no
Powder Bed Tester	yes	no	no	yes/yes	yes*/yes	yes	yes
Cohesion Tester	yes	no	no	yes/yes	yes/no	yes	yes
Tensile Strength Tester	yes	no	no	yes/yes	yes/no	yes	yes
Monoaxial Tester	yes	no	no	yes/yes	yes*/no	yes	yes
Aero Flow	no	–	–	–	–	–	no
Angle of Repose	no	–	–	–	–	–	no

dered. If many time consolidation tests are necessary, it is recommended to use an additional device in which many bulk solid samples can be placed under load without occupying the shear tester. An example for such a device is the Jenike consolidation bench [1].

In the following table all testers mentioned in this report will be judged regarding their potential to determine flowability. It must be accepted that any schematic listing of the many existing testers and test procedures will not be precise in all aspects. An apparently poor valuation does not necessarily exclude the application of the tester from special purposes. This concerns especially the testers of chapter 4.6 regarding their potentials to be used as a means for quality control or qualitative comparisons. If any criterion is not applicable a '-'sign is noted in the table. A* signifies that the judgement is only valid, if wall friction can be ignored (e.g. by using a lubricant or small height to diameter ratios).

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